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2-[4-Chloro-3-(4-ethoxybenzyl)phenyl]-1,3-dithiane

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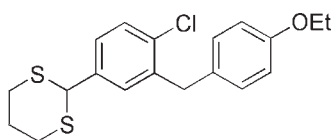
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.095; data-to-parameter ratio = 15.6.

In the title compound, $\text{C}_{19}\text{H}_{21}\text{ClO}_2$, the dithiane ring adopts a chair conformation. The dihedral angle between the benzene rings is $87.88(4)^\circ$. In the crystal, inversion dimmers linked by pairs of $\text{C}-\text{H}\cdots\text{O}$ interactions occur.

Related literature

For a related structure, see: Fun *et al.* (2009). For diaryl-methane motifs, see: Xu *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{19}\text{H}_{21}\text{ClO}_2$ $M_r = 364.93$ Monoclinic, $P2_1/c$ $a = 15.8214(3)$ Å $b = 12.2444(2)$ Å $c = 9.4191(2)$ Å $\beta = 100.715(1)^\circ$ $V = 1792.89(6)$ Å³

$Z = 4$
Cu $K\alpha$ radiation
 $\mu = 4.06$ mm⁻¹

$T = 100$ K
 $0.19 \times 0.13 \times 0.03$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)
 $T_{\min} = 0.513$, $T_{\max} = 0.888$

13885 measured reflections
3257 independent reflections
3040 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.095$
 $S = 1.06$
3257 reflections

209 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}19-\text{H}19\text{A}\cdots\text{O}1^i$	0.97	2.40	3.367 (2)	173

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

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT-Plus (Bruker, 2006); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZZ206).

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supporting information

Acta Cryst. (2010). E66, o1386 [https://doi.org/10.1107/S1600536810017393]

2-[4-Chloro-3-(4-ethoxybenzyl)phenyl]-1,3-dithiane**Brian Samas, Cathy Prévile, Benjamin A Thuma and Vincent Mascitti****S1. Comment**

Diarylmethane motifs are found in some C-aryl glucoside anti-diabetic agents prototypes. Gaining understanding of these motifs is of interest to the pharmaceutical industry (Xu *et al.*, 2009). The asymmetric unit contains one molecule of the title compound. There are no classic hydrogen bonds in the structure. A weak C-H \cdots O interaction pairs the molecules in a head-to-tail fashion. Crystal packing is controlled by van der Waals forces and π - π stacking interactions between both rings.

The separate π - π interactions, rings C3-C8 and C10-C15, are almost normal to each other at 87.88 (4) $^\circ$ and stabilize the structure. The rings are configured in a parallel-displaced configuration, the distance from closest contacts (C5-C15) is 3.599 (2) Å and measured from centroid-to-centroid these rings are separated by 4.8391 (1) Å.

The dithiane ring is in the expected chair conformation with a slight twisting in reference to the benzene ring, to avoid steric repulsion. The saturated ring exhibits a slight outward distortion from the C—S, due to the longer C—S bond. From the dithiane ring the benzene ring is in the expected equatorial position. These features are all common to dithiane crystal structures (Fun *et al.*, 2009). Viewed down the c axis, the dithiane rings inter-leave (Figure 2).

S2. Experimental

A synthesis of compound **1** is described on Fig. 3. Friedel Craft acylation of ethoxy benzene with 2-chloro-5-methyl benzoyl chloride in dichloromethane in presence of aluminum trichloride produced intermediate **2** in 30% yield. Radical bromination of **2** under classical conditions (AIBN, NBS, CCl₄) gave **3** (52% yield) which was subsequently treated with triethylsilane and boron trifluoride diethyl etherate to provide **4** in 81% yield. Nucleophilic displacement with sodium acetate followed by deacetylation (NaOMe/MeOH) led to intermediate **5** in 73% yield over 2 steps. Parikh-Doering oxidation produced the desired aldehyde intermediate **6** in 85% yield. Alternatively, Kornblum oxidation of **4** using 2,4,6-collidine in presence of dimethylsulfoxide gave **6** in 51% yield. Treatment of **6** with 1,3-propanedithiol in the presence of boron trifluoride diethyl etherate cleanly produced dithiane **1** in 74% yield. **1** was dissolved in ethyl acetate and allowed to evaporate over twelve hours to form X-ray diffraction quality crystals. Further details of the synthesis are included in the Experimental Special Details section.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aryl, 0.97 Å for methylene, and 0.96 Å for methyl H atoms. $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aryl and methylene H atoms, and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

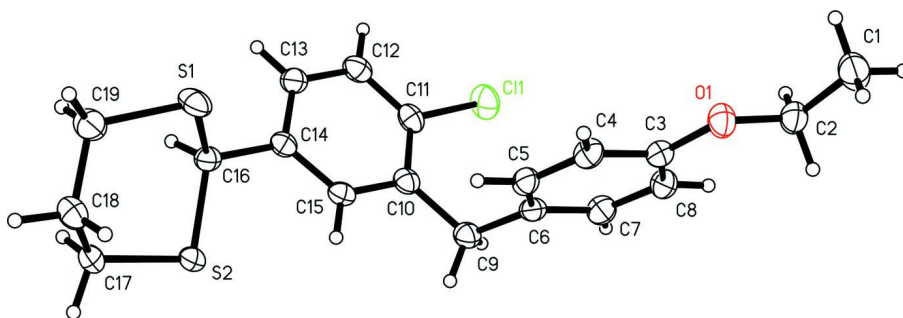


Figure 1

View of the constituents of (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

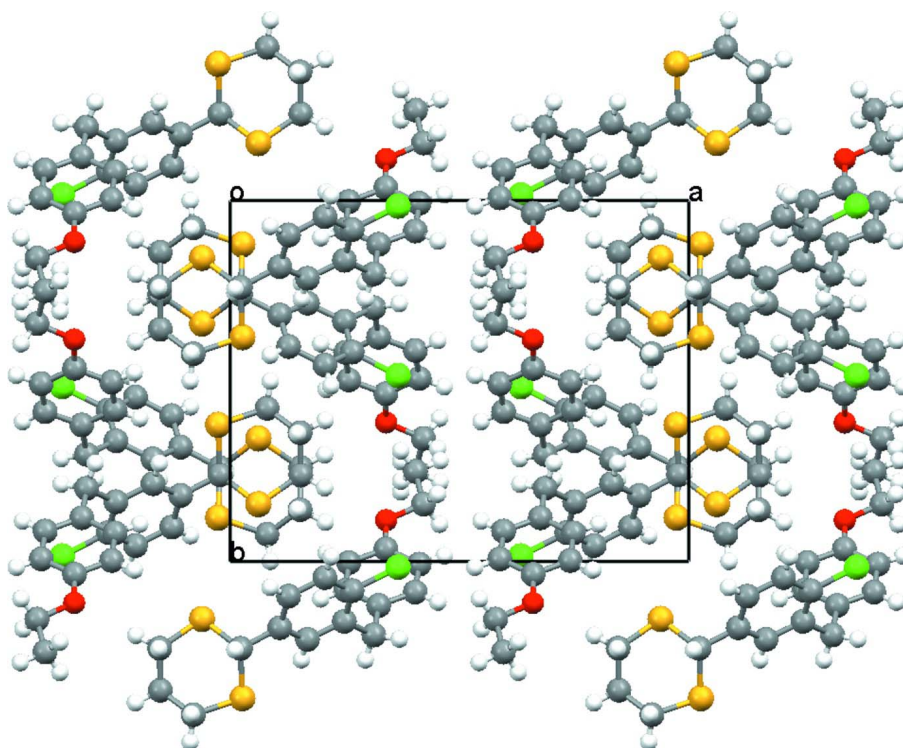


Figure 2

A partial packing diagram viewed along the c direction.

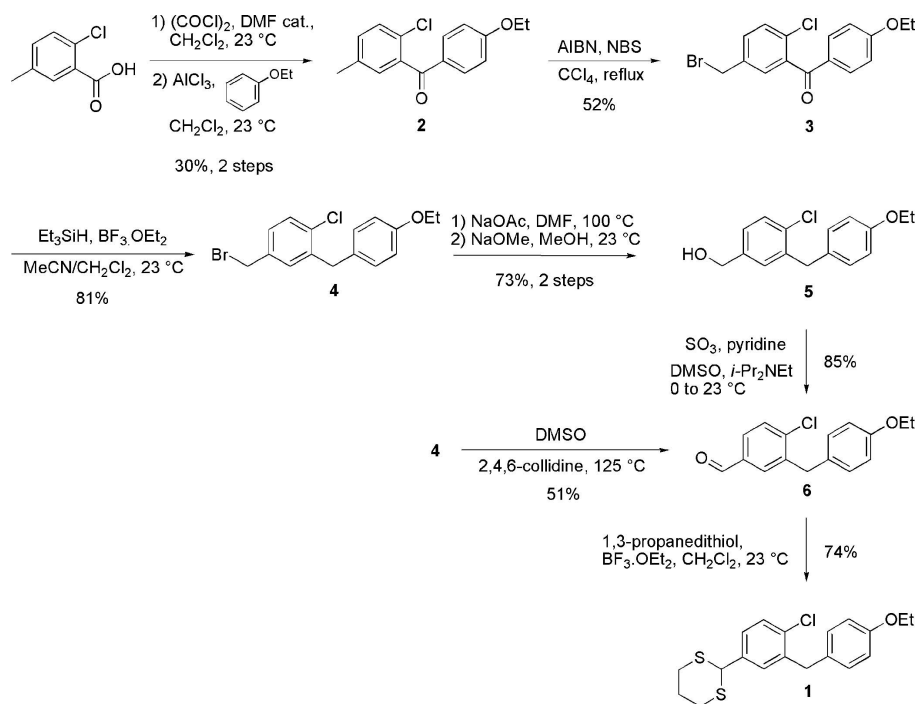


Figure 3
Reaction scheme.

2-[4-Chloro-3-(4-ethoxybenzyl)phenyl]-1,3-dithiane

Crystal data

C₁₉H₂₁ClOS₂
M_r = 364.93
 Monoclinic, *P*2₁/*c*
 Hall symbol: -P 2ybc
a = 15.8214 (3) Å
b = 12.2444 (2) Å
c = 9.4191 (2) Å
 β = 100.715 (1)°
V = 1792.89 (6) Å³
Z = 4

Data collection

Bruker APEXII CCD
 diffractometer
 Radiation source: rotating anode
 Montel Multilayer optics monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 2002)
T_{min} = 0.513, *T_{max}* = 0.888

F(000) = 768
D_x = 1.352 Mg m⁻³
 Cu *K* α radiation, λ = 1.54178 Å
 Cell parameters from 9696 reflections
 θ = 2.8–68.7°
 μ = 4.06 mm⁻¹
T = 100 K
 Block, colourless
 0.19 × 0.13 × 0.03 mm

13885 measured reflections
 3257 independent reflections
 3040 reflections with *I* > 2 σ (*I*)
R_{int} = 0.025
 θ_{\max} = 69.3°, θ_{\min} = 2.8°
h = -19→19
k = -13→14
l = -7→11

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.095$ $S = 1.06$

3257 reflections

209 parameters

0 restraints

0 constraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.711P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. (2-Chloro-5-methyl-phenyl)-(4-ethoxy-phenyl)-methanone (**2**):

To a solution of 2-chloro-5-methyl-benzoic acid (1.0 g, 5.86 mmol) in dichloromethane (12 ml) was added at room temperature (~23°C) oxalyl chloride (0.54 ml, 6.2 mmol) followed by the dropwise addition of *N,N*-dimethylformamide (0.1 ml). The resulting solution was allowed to stir overnight at room temperature before being concentrated under reduced pressure to produce crude 2-chloro-5-methylbenzoyl chloride which was used in the next step without further purification. The crude 2-chloro-5-methylbenzoyl chloride (1.15 g, 5.86 mmol) and ethoxybenzene (0.75 g, 6.14 mmol) were dissolved in 10 ml of dichloromethane and the resulting solution was cooled to 0°C. Aluminum trichloride (0.85 g, 6.38 mmol) was added and the reaction mixture was allowed to stir at 0°C for 4 hours before being allowed to warm to room temperature overnight (~16 hours). The reaction was quenched by pouring the solution over ice. The mixture was diluted with water and extracted three times with dichloromethane. The combined organic layers were successively washed with aqueous 1 N hydrochloric acid solution, aqueous 1 N sodium hydroxide solution, brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The crude material was chromatographed using an ISCO automated chromatography unit (40 g silica gel column) and eluting with a gradient of 0-40% ethyl acetate in heptane yielding 650 mg (30% yield) of an oil that solidified upon standing. MS (LCMS) 274.9 (M+H⁺; positive mode). HRMS calculated for C₁₆H₁₆ClO₂ (M+H⁺) 275.0833 found 275.0832. ¹H NMR (400 MHz, CHLOROFORM-d) δ ppm 1.43 (t, J=7.0 Hz, 3 H), 2.34 (s, 3 H), 4.09 (q, J=7.0 Hz, 2 H), 6.90 (d, J=8.8 Hz, 2 H), 7.14 (s, 1 H), 7.17 - 7.22 (m, 1 H), 7.30 (d, J=8.2 Hz, 1 H), 7.76 (d, J=8.8 Hz, 2 H); ¹³C NMR (100 MHz, CHLOROFORM-d) δ ppm 194.3, 163.7, 139.0, 136.9, 132.7 (2 C), 132.5, 131.7, 129.8, 129.5, 128.4, 114.4 (2 C), 64.0, 21.0, 14.8.

(5-Bromomethyl-2-chloro-phenyl)-(4-ethoxy-phenyl)-methanone (**3**):

To a solution of (2-chloro-5-methyl-phenyl)-(4-ethoxy-phenyl)-methanone (**2**; 650 mg, 2.37 mmol) and *N*-bromosuccinimide (465 mg, 2.61 mmol) in carbon tetrachloride (8 ml) was added 2,2'-azobisisobutyronitrile (AIBN, 8 mg, 0.047 mmol) and the reaction mixture was heated to reflux for 26 hours under nitrogen. The reaction was cooled to room temperature, quenched with water (50 ml) and the mixture diluted with dichloromethane (25 ml). The layers were separated and the aqueous layer was extracted two additional times with dichloromethane (25 ml). The combined organic layers were washed with brine, dried over magnesium sulfate, filtered through a pad of celite, and concentrated under reduced pressure. The crude material was chromatographed with an ISCO automated chromatography unit (40 g silica gel column) eluting with a gradient of 0-20% ethyl acetate in heptane to produce 434 mg (52% yield) of the desired product as a white solid. HRMS calculated for C₁₆H₁₅BrClO₂ (M+H⁺) 352.9938 found 352.9939. ¹H NMR (400 MHz, CHLOROFORM-d) δ ppm 1.43 (t, J=7.0 Hz, 3 H), 4.10 (q, J=6.9 Hz, 2 H), 4.45 (s, 2 H), 6.91 (d, J=8.8 Hz, 2 H), 7.36 (s, 1 H), 7.37 - 7.40 (m, 2 H), 7.76 (d, J=9.0 Hz, 2 H). ¹³C NMR (100 MHz, CHLOROFORM-d) δ ppm 193.3, 163.3, 139.6, 136.9 132.7 (2 C), 131.5, 131.3, 130.6, 129.5, 129.1, 114.6 (2 C), 64.1, 31.8, 14.8.

4-Bromomethyl-1-chloro-2-(4-ethoxy-benzyl)-benzene (**4**):

To a solution of (5-bromomethyl-2-chloro-phenyl)-(4-ethoxy-phenyl)-methanone (**3**; 400 mg, 1.13 mmol) dissolved in dichloromethane (2 ml) and acetonitrile (4 ml) cooled to 0°C was added triethylsilane (0.7 mL, 4 mmol). Boron trifluoride diethyl ether complex (0.34 ml, 2.71 mmol) was added dropwise to the stirring solution and the reaction mixture was allowed to warm to room temperature overnight. The reaction was quenched by addition of aqueous 1 N sodium hydroxide (30 ml) and the resulting biphasic mixture was extracted three times with ethyl acetate (30 ml). The combined organic layers were successively washed with aqueous 1 N sodium hydroxide solution (50 ml), brine (50 ml), dried over sodium sulfate, filtered and concentrated under reduced pressure. The crude material was chromatographed using an ISCO automated chromatography unit (12 g silica gel column) eluting with a gradient of 0-50% ethyl acetate in heptane to produce 310 mg (81% yield) of the desired product as a clear oil. HRMS calculated for C₁₆H₁₇BrClO (M+H⁺) 339.0145 found 339.0137. ¹H NMR (400 MHz, CHLOROFORM-d) δ ppm 1.39 (t, J=7.0 Hz, 3 H), 4.00 (q, J=8.4 Hz, 2 H), 4.01 (s, 2 H), 4.38 (s, 2 H), 6.82 (d, J=8.6 Hz, 2 H), 7.08 (d, J=8.6 Hz, 2 H), 7.12 (d, J=2.0 Hz, 1 H), 7.17 (dd, J=8.1, 2.0 Hz, 1 H), 7.32 (d, J=8.2 Hz, 1 H). ¹³C NMR (100 MHz, CHLOROFORM-d) δ ppm 157.7, 139.8, 136.7, 134.4, 131.5, 131.1, 130.2, 130.1 (2 C), 128.4, 114.7 (2 C), 63.6, 38.4, 32.8, 15.1.

[4-Chloro-3-(4-ethoxy-benzyl)-phenyl]-methanol (**5**):

A solution of 4-bromomethyl-1-chloro-2-(4-ethoxy-benzyl)-benzene (**4**; 300 mg, 0.883 mmol) and sodium acetate (217 mg, 2.65 mmol) in *N,N*-dimethylformamide (2 ml) was heated at 100°C for 18 hours. After cooling to room temperature, the reaction was quenched with water (30 ml) and the resulting mixture was extracted with ethyl acetate (30 ml). The aqueous layer was extracted with ethyl acetate (30 ml) two additional times. The combined organic layers were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The crude material was chromatographed using an ISCO automated chromatography unit (12 g silica gel column) eluting with a gradient of 0-50% ethyl acetate in heptane yielding 237 mg (84% yield) of acetic acid 4-chloro-3-(4-ethoxy-benzyl)-benzyl ester as a clear oil. HRMS calculated for C₁₈H₁₉ClNaO₃ (M+Na⁺) 341.0914 found 341.0925. ¹H NMR (400 MHz, CHLOROFORM-d) δ ppm 1.39 (t, J=6.9 Hz, 3 H), 2.06 (s, 3 H), 3.99 (q, 2 H), 4.02 (s, 2 H), 4.99 (s, 2 H), 6.81 (d, J=8.6 Hz, 2 H), 7.08 (d, J=8.8 Hz, 2 H), 7.10 (br. s., 1 H), 7.13 (dd, J=8.2, 2.0 Hz, 1 H), 7.34 (d, J=8.0 Hz, 1 H). ¹³C NMR (100 MHz, CHLOROFORM-d) δ ppm 170.9, 157.7, 139.6, 134.9, 134.2, 131.3, 130.9 (2 C), 130.1, 129.8, 127.5, 114.7 (2 C), 67.7, 63.6, 38.5, 21.2, 15.1.

To a solution of acetic acid 4-chloro-3-(4-ethoxy-benzyl)-benzyl ester (221 mg, 0.693 mmol) dissolved in methanol (8 p-6 ml) was added 25% sodium methoxide in methanol (1 ml) until pH = 12 was reached and the reaction mixture was allowed to stir at room temperature under nitrogen for 18 hours. The mixture was neutralized with the addition of Dowex Monosphere 650 C (H) cation exchange resin (the resin was washed with methanol 3 times before using) until the pH of the solution was ~7. The reaction mixture was filtered and the filtrate was concentrated under reduced pressure. The

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. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	−0.06149 (3)	0.34104 (3)	0.16942 (5)	0.03161 (14)
S2	0.02647 (3)	0.12144 (3)	0.18334 (5)	0.02839 (13)
Cl1	0.36610 (3)	0.48468 (4)	0.26092 (5)	0.03858 (15)
O1	0.34010 (7)	0.61472 (10)	0.88988 (13)	0.0309 (3)
C10	0.24904 (10)	0.33442 (13)	0.31254 (17)	0.0245 (3)
C17	−0.08039 (10)	0.07566 (13)	0.10028 (18)	0.0273 (3)
H17A	−0.0852	0.0772	−0.0039	0.033*
H17B	−0.0875	0.0005	0.1284	0.033*
C4	0.25848 (10)	0.49708 (14)	0.72116 (18)	0.0271 (4)
H4	0.2093	0.5232	0.7506	0.033*
C18	−0.15249 (11)	0.14349 (14)	0.14036 (19)	0.0303 (4)
H18A	−0.1464	0.1448	0.2447	0.036*
H18B	−0.2070	0.1090	0.1013	0.036*
C12	0.20648 (11)	0.45554 (14)	0.10697 (18)	0.0286 (4)
H12	0.2196	0.5135	0.0510	0.034*
C3	0.33940 (10)	0.53507 (13)	0.78734 (17)	0.0255 (3)
C7	0.40363 (10)	0.41546 (14)	0.63581 (19)	0.0297 (4)
H7	0.4529	0.3877	0.6083	0.036*
C16	0.02258 (10)	0.25970 (12)	0.11252 (17)	0.0233 (3)
H16	0.0101	0.2553	0.0068	0.028*
C11	0.26600 (10)	0.41954 (14)	0.22447 (18)	0.0267 (3)
C14	0.10798 (10)	0.31682 (13)	0.15714 (17)	0.0228 (3)
C15	0.16857 (10)	0.28457 (13)	0.27638 (17)	0.0236 (3)
H15	0.1550	0.2279	0.3339	0.028*
C2	0.42134 (11)	0.65916 (14)	0.95732 (18)	0.0307 (4)
H2A	0.4520	0.6867	0.8847	0.037*
H2B	0.4563	0.6032	1.0128	0.037*
C5	0.25158 (10)	0.42072 (13)	0.61192 (17)	0.0256 (3)
H5	0.1973	0.3967	0.5677	0.031*
C6	0.32365 (10)	0.37868 (13)	0.56612 (18)	0.0248 (3)
C9	0.31334 (10)	0.29688 (13)	0.44322 (19)	0.0279 (4)
H9A	0.3687	0.2854	0.4154	0.034*
H9B	0.2946	0.2276	0.4764	0.034*
C13	0.12736 (11)	0.40436 (13)	0.07374 (17)	0.0261 (3)
H13	0.0869	0.4284	−0.0045	0.031*

C19	-0.15389 (11)	0.26023 (14)	0.0851 (2)	0.0354 (4)
H19A	-0.2059	0.2956	0.1020	0.043*
H19B	-0.1559	0.2587	-0.0185	0.043*
C8	0.41212 (10)	0.49263 (14)	0.74548 (18)	0.0286 (4)
H8	0.4665	0.5158	0.7907	0.034*
C1	0.40403 (13)	0.75029 (16)	1.0549 (2)	0.0393 (4)
H1A	0.3717	0.8067	0.9983	0.059*
H1B	0.4576	0.7797	1.1048	0.059*
H1C	0.3717	0.7227	1.1239	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0281 (2)	0.0190 (2)	0.0491 (3)	0.00118 (15)	0.01076 (19)	-0.00376 (17)
S2	0.0267 (2)	0.0175 (2)	0.0379 (2)	-0.00016 (14)	-0.00212 (17)	0.00027 (15)
C11	0.0302 (2)	0.0440 (3)	0.0424 (3)	-0.01370 (18)	0.00921 (19)	0.00073 (19)
O1	0.0252 (6)	0.0339 (7)	0.0321 (6)	0.0000 (5)	0.0014 (5)	-0.0065 (5)
C10	0.0250 (8)	0.0209 (8)	0.0282 (8)	0.0021 (6)	0.0064 (6)	-0.0043 (6)
C17	0.0271 (8)	0.0212 (8)	0.0319 (8)	-0.0036 (6)	0.0014 (7)	-0.0030 (7)
C4	0.0200 (8)	0.0317 (9)	0.0297 (8)	0.0017 (6)	0.0049 (6)	0.0024 (7)
C18	0.0287 (8)	0.0277 (8)	0.0353 (9)	-0.0054 (7)	0.0082 (7)	-0.0024 (7)
C12	0.0379 (9)	0.0231 (8)	0.0266 (8)	-0.0037 (7)	0.0107 (7)	0.0002 (7)
C3	0.0270 (8)	0.0241 (8)	0.0244 (7)	0.0006 (6)	0.0023 (6)	0.0028 (6)
C7	0.0209 (8)	0.0313 (9)	0.0362 (9)	0.0046 (7)	0.0034 (7)	-0.0004 (7)
C16	0.0263 (8)	0.0206 (7)	0.0231 (7)	0.0018 (6)	0.0045 (6)	0.0001 (6)
C11	0.0248 (8)	0.0254 (8)	0.0314 (8)	-0.0045 (6)	0.0094 (6)	-0.0057 (7)
C14	0.0259 (8)	0.0190 (7)	0.0242 (7)	0.0016 (6)	0.0067 (6)	-0.0039 (6)
C15	0.0284 (8)	0.0175 (7)	0.0256 (8)	0.0008 (6)	0.0073 (6)	-0.0011 (6)
C2	0.0287 (8)	0.0326 (9)	0.0287 (8)	-0.0036 (7)	0.0000 (7)	-0.0005 (7)
C5	0.0206 (7)	0.0275 (8)	0.0274 (8)	-0.0032 (6)	0.0013 (6)	0.0044 (7)
C6	0.0240 (8)	0.0211 (8)	0.0283 (8)	0.0011 (6)	0.0020 (6)	0.0045 (6)
C9	0.0246 (8)	0.0227 (8)	0.0356 (9)	0.0017 (6)	0.0031 (7)	-0.0005 (7)
C13	0.0326 (8)	0.0229 (8)	0.0225 (7)	0.0011 (7)	0.0045 (6)	0.0000 (6)
C19	0.0244 (8)	0.0271 (9)	0.0548 (11)	0.0011 (7)	0.0072 (8)	-0.0013 (8)
C8	0.0198 (8)	0.0305 (9)	0.0336 (9)	-0.0005 (6)	-0.0002 (7)	-0.0002 (7)
C1	0.0415 (10)	0.0375 (10)	0.0376 (10)	-0.0058 (8)	0.0037 (8)	-0.0065 (8)

Geometric parameters (Å, °)

S1—C16	1.8202 (16)	C7—C6	1.388 (2)
S1—C19	1.8209 (18)	C7—C8	1.388 (2)
S2—C17	1.8146 (16)	C7—H7	0.9300
S2—C16	1.8165 (16)	C16—C14	1.510 (2)
C11—C11	1.7494 (16)	C16—H16	0.9800
O1—C3	1.371 (2)	C14—C15	1.391 (2)
O1—C2	1.431 (2)	C14—C13	1.396 (2)
C10—C11	1.389 (2)	C15—H15	0.9300
C10—C15	1.396 (2)	C2—C1	1.503 (3)

C10—C9	1.515 (2)	C2—H2A	0.9700
C17—C18	1.515 (2)	C2—H2B	0.9700
C17—H17A	0.9700	C5—C6	1.391 (2)
C17—H17B	0.9700	C5—H5	0.9300
C4—C5	1.379 (2)	C6—C9	1.516 (2)
C4—C3	1.395 (2)	C9—H9A	0.9700
C4—H4	0.9300	C9—H9B	0.9700
C18—C19	1.520 (2)	C13—H13	0.9300
C18—H18A	0.9700	C19—H19A	0.9700
C18—H18B	0.9700	C19—H19B	0.9700
C12—C13	1.383 (2)	C8—H8	0.9300
C12—C11	1.385 (2)	C1—H1A	0.9600
C12—H12	0.9300	C1—H1B	0.9600
C3—C8	1.385 (2)	C1—H1C	0.9600
C16—S1—C19	98.37 (8)	C13—C14—C16	118.54 (14)
C17—S2—C16	99.46 (7)	C14—C15—C10	122.22 (15)
C3—O1—C2	118.16 (13)	C14—C15—H15	118.9
C11—C10—C15	116.81 (15)	C10—C15—H15	118.9
C11—C10—C9	122.53 (14)	O1—C2—C1	107.64 (14)
C15—C10—C9	120.65 (14)	O1—C2—H2A	110.2
C18—C17—S2	114.06 (11)	C1—C2—H2A	110.2
C18—C17—H17A	108.7	O1—C2—H2B	110.2
S2—C17—H17A	108.7	C1—C2—H2B	110.2
C18—C17—H17B	108.7	H2A—C2—H2B	108.5
S2—C17—H17B	108.7	C4—C5—C6	121.76 (14)
H17A—C17—H17B	107.6	C4—C5—H5	119.1
C5—C4—C3	119.87 (15)	C6—C5—H5	119.1
C5—C4—H4	120.1	C7—C6—C5	117.46 (15)
C3—C4—H4	120.1	C7—C6—C9	122.32 (15)
C17—C18—C19	113.19 (14)	C5—C6—C9	120.21 (14)
C17—C18—H18A	108.9	C10—C9—C6	112.37 (13)
C19—C18—H18A	108.9	C10—C9—H9A	109.1
C17—C18—H18B	108.9	C6—C9—H9A	109.1
C19—C18—H18B	108.9	C10—C9—H9B	109.1
H18A—C18—H18B	107.8	C6—C9—H9B	109.1
C13—C12—C11	119.27 (15)	H9A—C9—H9B	107.9
C13—C12—H12	120.4	C12—C13—C14	120.33 (15)
C11—C12—H12	120.4	C12—C13—H13	119.8
O1—C3—C8	124.69 (14)	C14—C13—H13	119.8
O1—C3—C4	116.00 (14)	C18—C19—S1	113.71 (13)
C8—C3—C4	119.31 (15)	C18—C19—H19A	108.8
C6—C7—C8	121.77 (15)	S1—C19—H19A	108.8
C6—C7—H7	119.1	C18—C19—H19B	108.8
C8—C7—H7	119.1	S1—C19—H19B	108.8
C14—C16—S2	111.22 (10)	H19A—C19—H19B	107.7
C14—C16—S1	109.25 (10)	C3—C8—C7	119.78 (15)
S2—C16—S1	112.24 (8)	C3—C8—H8	120.1

C14—C16—H16	108.0	C7—C8—H8	120.1
S2—C16—H16	108.0	C2—C1—H1A	109.5
S1—C16—H16	108.0	C2—C1—H1B	109.5
C12—C11—C10	122.54 (15)	H1A—C1—H1B	109.5
C12—C11—C11	117.90 (13)	C2—C1—H1C	109.5
C10—C11—C11	119.56 (13)	H1A—C1—H1C	109.5
C15—C14—C13	118.78 (15)	H1B—C1—H1C	109.5
C15—C14—C16	122.67 (14)		
C16—S2—C17—C18	-59.13 (14)	C16—C14—C15—C10	176.73 (14)
S2—C17—C18—C19	65.33 (18)	C11—C10—C15—C14	0.8 (2)
C2—O1—C3—C8	1.2 (2)	C9—C10—C15—C14	-179.94 (14)
C2—O1—C3—C4	-177.84 (14)	C3—O1—C2—C1	175.34 (14)
C5—C4—C3—O1	176.93 (14)	C3—C4—C5—C6	0.8 (2)
C5—C4—C3—C8	-2.2 (2)	C8—C7—C6—C5	-1.1 (2)
C17—S2—C16—C14	-176.03 (11)	C8—C7—C6—C9	178.18 (15)
C17—S2—C16—S1	61.23 (10)	C4—C5—C6—C7	0.8 (2)
C19—S1—C16—C14	174.28 (11)	C4—C5—C6—C9	-178.52 (15)
C19—S1—C16—S2	-61.87 (10)	C11—C10—C9—C6	71.3 (2)
C13—C12—C11—C10	-1.0 (3)	C15—C10—C9—C6	-107.93 (17)
C13—C12—C11—C11	178.80 (12)	C7—C6—C9—C10	-129.56 (17)
C15—C10—C11—C12	0.8 (2)	C5—C6—C9—C10	49.7 (2)
C9—C10—C11—C12	-178.46 (15)	C11—C12—C13—C14	-0.5 (2)
C15—C10—C11—C11	-178.94 (12)	C15—C14—C13—C12	2.0 (2)
C9—C10—C11—C11	1.8 (2)	C16—C14—C13—C12	-176.95 (15)
S2—C16—C14—C15	-22.01 (19)	C17—C18—C19—S1	-66.73 (18)
S1—C16—C14—C15	102.44 (15)	C16—S1—C19—C18	61.15 (15)
S2—C16—C14—C13	156.87 (12)	O1—C3—C8—C7	-177.15 (15)
S1—C16—C14—C13	-78.68 (16)	C4—C3—C8—C7	1.8 (3)
C13—C14—C15—C10	-2.1 (2)	C6—C7—C8—C3	-0.2 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C19—H19 <i>A</i> ...O1 ⁱ	0.97	2.40	3.367 (2)	173

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