Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*R*)- and (*S*)-1-[2-(benzyloxy)-3-methoxyphenyl]-2,2,2-trichloroethyl benzenesulfonate

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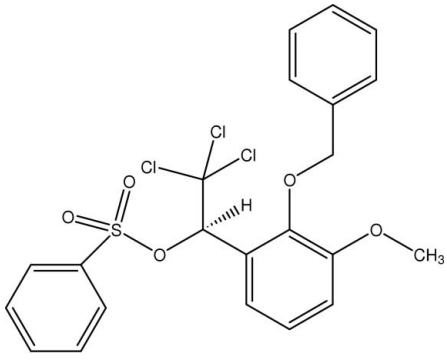
Received 16 May 2007; accepted 4 June 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.040; wR factor = 0.108; data-to-parameter ratio = 48.8.

In the title molecule, $C_{22}H_{19}Cl_3O_5S$, the methoxy group is slightly twisted from the plane of the attached benzene ring by a torsion angle of 18.96 (14)°. In the crystal structure, molecules are linked by intermolecular C—H···O interactions and short Cl···O contacts of 3.0170 (8) Å.

Related literature

For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related literature on values of bond lengths, see: Allen *et al.* (1987). For related structures, see: Al-Douh *et al.* (2006*a*,*b*); Begley *et al.* (1978); Gill *et al.* (1979).



Experimental

Crystal data

$C_{22}H_{19}Cl_3O_5S$
M_r = 501.78
Monoclinic, $P2_1/c$
a = 8.1638 (1) Å
b = 8.8536 (1) Å
c = 30.7221 (5) Å
β = 90.670 (1)°
V = 2220.41 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 100.0 (1) K
0.48 × 0.30 × 0.29 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.779, T_{max} = 0.858
122837 measured reflections
13669 independent reflections
11428 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.08
13669 reflections
280 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7*B*···O2	0.97	2.56	3.040 (1)	110
C14—H14*A*···O1	0.98	2.38	2.820 (1)	107
C14—H14*A*···O3	0.98	2.34	2.838 (1)	111
C21—H21*A*···O3	0.93	2.55	2.919 (1)	104
C3—H3*A*···O2^{i}	0.93	2.49	3.414 (1)	172

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

We thank the Malaysian Government and Universiti Sains Malaysia (USM) for an FRGS grant (203/227/ PKIMIA/ 671046) to conduct this work. MHA thanks the Yemen Government and Hadhramout University of Science and Technology (HUST) for financial scholarship support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2396).

References

Al-Douh, M. H., Hamid, S. A., Osman, H., Ng, S.-L. & Fun, H.-K. (2006*a*). *Acta Cryst.* E**62**, o3954–o3956.
Al-Douh, M. H., Hamid, S. A., Osman, H., Ng, S.-L. & Fun, H.-K. (2006*b*). *Acta Cryst.* E**62**, o4768–o4770.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Begley, M. J., Gill, G. B. & Wallace, B. (1978). *J. Chem. Soc. Perkin Trans. 1*, pp. 93–97.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Gill, G. B., Marrison, K., Parrott, S. J. & Wallace, B. (1979). *Tetrahedron Lett.* **24**, 4867–4870.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

Acta Cryst. (2007). E**63**, o3233 [**doi:10.1107/S1600536807027353**]

(*R*)- and (*S*)-1-[2-(benzyloxy)-3-methoxyphenyl]-2,2,2-trichloroethyl benzenesulfonate

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Comment

In our attempt to synthesize new benzimidazole derivatives (Al-Douh *et al.*, 2006*a*,b), the crystal structure of the title compound, (I), was obtained and determined (Figure 1). Previously, the structure of a 2,2,2-trichloroethyl tosylsulfonate as a functional group has only been determined as an (*S*) derivative in tosylate (Begley *et al.*, 1978) and as enantiomers in sulfonates (Gill *et al.*, 1979). The research on the physical and chemical properties, including the reaction of the title compound to synthesize biologically important compounds is in progress. We present herein its crystal structure.

The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable with those in the related structures (Begley *et al.*, 1978). The dihedral angle between the benzene rings [(C1—C6) and (C8—C13)] is 22.64 (5)° whereas the torsion angle of C8—O1—C7—C6 is −157.96 (7)°. The methoxy group at C9 is slightly twisted from the plane of the attached benzene ring with torsion angle of C22—O2—C9—C10 = −18.96 (14)°.

The intramolecular C7—H7B···O2 interaction generates an S(6) ring motif, while intramolecular C14—H14A···O1, C14—H14A···O3 and C21—H21A···O3 interaction (Table 1 and Figure 1) generate S(5) ring motifs (Bernstein *et al.*, 1995). In the crystal structure, the molecules are linked by short inter Cl2···O4^{ii} contact of 3.0170 (8)Å (symmetry code: (ii) −*x*, −*y*, −*z*) into cyclic centrosymmetric $R^2_2(12)$ dimers. These dimers are interlinked by the C3—H3A···O2^{i} (symmetry code: (i) −*x*, *y* + 1/2, −*z* + 1/2) intermolecular interactions.

Experimental

The title compound (I) was synthesized by adding sulfonyl chloride (535 mg, 3.027 mmol) dropwise to a stirred solution of 2-(2-(benzyloxy)-3-methoxyphenyl)-1*H*-benzimidazole (1000 mg, 3.027 mmol) in dry dichloromethane (50 ml) and dimethylaminopyridine (370 mg, 3.027 mmol). The resulting solution was stirred at room temperature for 2 hr, then at 320 K for 10 hr. Then, it was cooled to room temperature before removing the solvent by rotary evaporator. Crushed ice (25 g) and $CHCl_3$ (50 ml) were added to the crude solution. The solution was shaken using separatory funnel and the organic layer was collected. The resulting solution was washed with 10% NaOH (3 × 25 ml) followed by water (3 × 25 ml), brine (3 × 25 ml) and water (3 × 25 ml) again. The organic layer was dried over $MgSO_4$, filtered and the solvent was removed by rotary evaporator. The crude product was then purified by column chromatography with *n*-hexane:diethyl ether (1:4). The final product was dissolved in petroleum ether, and single crystals suitable for X-ray diffraction were obtained by evaporation the solvent at room temperature.

Refinement

H atoms were placed in calculated positions and constrained to ride on their carrier atoms, with C—H distances in the range 0.93 − 0.98 Å. The U_{iso} values were constrained to be 1.5 U_{eq} of the carrier atom for methyl H atoms and 1.2 U_{eq} for the remaining H atoms.

Figures

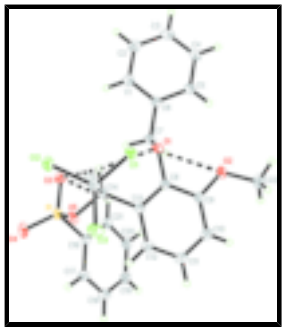

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed lines indicate intramolecular hydrogen bonds.

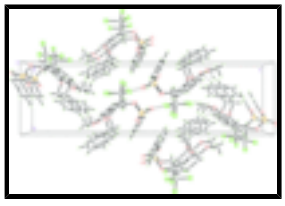

Fig. 2. The crystal packing of (I), viewed down the *a* axis. The intermolecular C—H···O hydrogen bonds and short inter Cl···O contacts are shown as dashed lines.

(*RS*)-1-[2-(Benzyloxy)-3-methoxyphenyl]-2,2,2-trichloroethyl benzenesulfonate

Crystal data

$C_{22}H_{19}Cl_3O_5S$	F_{000} = 1032
M_r = 501.78	D_x = 1.501 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6905 reflections
a = 8.1638 (1) Å	θ = 1.3–40.0º
b = 8.8536 (1) Å	μ = 0.54 mm^{-1}
c = 30.7221 (5) Å	T = 100.0 (1) K
β = 90.670 (1)º	Block, brown
V = 2220.41 (5) Å^3	0.48 × 0.30 × 0.29 mm
Z = 4	

Data collection

Bruker SMART APEX II CCD area-detector diffractometer	13669 independent reflections
Radiation source: fine-focus sealed tube	11428 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.059
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 40.0º
T = 100.0(1) K	θ_{min} = 1.3º
ω scans	h = −14→14
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −16→16
T_{min} = 0.779, T_{max} = 0.858	l = −54→54
122837 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.5813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
13669 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
280 parameters	$\Delta\rho_{min} = -0.55$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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.

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.11494 (3)	−0.07303 (2)	0.165654 (7)	0.01782 (4)
Cl2	0.00495 (3)	−0.10166 (3)	0.076490 (8)	0.02106 (5)
Cl3	0.31730 (3)	−0.22595 (2)	0.103131 (9)	0.02324 (5)
S1	0.29746 (3)	0.21264 (2)	0.024940 (7)	0.01523 (4)
O1	0.22120 (8)	0.28423 (7)	0.17014 (2)	0.01422 (10)
O2	0.46726 (9)	0.36777 (8)	0.22307 (2)	0.01929 (12)
O3	0.17049 (9)	0.30958 (8)	0.04024 (3)	0.02068 (13)
O4	0.28924 (10)	0.14939 (9)	−0.01775 (2)	0.02280 (14)
O5	0.30691 (8)	0.06705 (7)	0.05561 (2)	0.01599 (11)
C1	−0.07517 (13)	0.55311 (13)	0.15528 (4)	0.02359 (18)
H1A	−0.0895	0.5219	0.1266	0.028*
C2	−0.19809 (13)	0.63606 (15)	0.17555 (4)	0.0284 (2)
H2A	−0.2939	0.6600	0.1604	0.034*
C3	−0.17691 (13)	0.68252 (12)	0.21830 (4)	0.02419 (18)
H3A	−0.2575	0.7397	0.2317	0.029*
C4	−0.03532 (12)	0.64364 (11)	0.24112 (3)	0.02053 (15)
H4A	−0.0221	0.6733	0.2700	0.025*

C5	0.08666 (12)	0.56058 (10)	0.22100 (3)	0.01755 (14)
H5A	0.1808	0.5340	0.2365	0.021*
C6	0.06841 (11)	0.51685 (9)	0.17762 (3)	0.01539 (13)
C7	0.20611 (11)	0.43904 (9)	0.15479 (3)	0.01698 (14)
H7A	0.1853	0.4394	0.1236	0.020*
H7B	0.3077	0.4928	0.1604	0.020*
C8	0.37519 (10)	0.22610 (9)	0.16305 (3)	0.01354 (12)
C9	0.50575 (11)	0.26899 (10)	0.19070 (3)	0.01604 (13)
C10	0.66156 (11)	0.21106 (12)	0.18355 (3)	0.02081 (16)
H10A	0.7487	0.2396	0.2015	0.025*
C11	0.68675 (12)	0.11023 (13)	0.14943 (4)	0.02258 (17)
H11A	0.7913	0.0725	0.1446	0.027*
C12	0.55826 (11)	0.06541 (11)	0.12253 (3)	0.01892 (15)
H12A	0.5765	−0.0024	0.1000	0.023*
C13	0.40090 (10)	0.12277 (9)	0.12947 (3)	0.01413 (12)
C14	0.25789 (10)	0.07970 (9)	0.10057 (3)	0.01380 (12)
H14A	0.1740	0.1585	0.1027	0.017*
C15	0.17828 (11)	−0.07436 (9)	0.11099 (3)	0.01580 (13)
C16	0.48763 (10)	0.30045 (10)	0.03280 (3)	0.01484 (12)
C17	0.62592 (12)	0.23099 (11)	0.01579 (3)	0.02071 (16)
H17A	0.6174	0.1392	0.0012	0.025*
C18	0.77654 (12)	0.30144 (14)	0.02105 (4)	0.02500 (19)
H18A	0.8705	0.2563	0.0102	0.030*
C19	0.78741 (12)	0.43929 (13)	0.04253 (3)	0.02332 (18)
H19A	0.8886	0.4867	0.0456	0.028*
C20	0.64852 (13)	0.50693 (11)	0.05949 (3)	0.02105 (16)
H20A	0.6572	0.5987	0.0741	0.025*
C21	0.49626 (11)	0.43739 (10)	0.05464 (3)	0.01730 (14)
H21A	0.4025	0.4818	0.0658	0.021*
C22	0.58060 (14)	0.37768 (13)	0.25894 (4)	0.02435 (18)
H22D	0.5348	0.4396	0.2814	0.037*
H22A	0.6812	0.4217	0.2492	0.037*
H22B	0.6017	0.2784	0.2702	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01973 (9)	0.02031 (9)	0.01344 (8)	−0.00148 (6)	0.00140 (6)	0.00146 (6)
Cl2	0.02243 (9)	0.02403 (10)	0.01665 (9)	−0.00575 (7)	−0.00262 (7)	−0.00215 (7)
Cl3	0.02823 (11)	0.01416 (8)	0.02746 (12)	0.00426 (7)	0.00600 (8)	0.00013 (7)
S1	0.01562 (8)	0.01714 (8)	0.01288 (8)	−0.00106 (6)	−0.00157 (6)	0.00127 (6)
O1	0.0144 (2)	0.0128 (2)	0.0155 (3)	0.00084 (18)	0.00208 (19)	0.00006 (18)
O2	0.0198 (3)	0.0225 (3)	0.0155 (3)	−0.0023 (2)	−0.0018 (2)	−0.0046 (2)
O3	0.0156 (3)	0.0211 (3)	0.0253 (3)	0.0028 (2)	0.0002 (2)	0.0051 (2)
O4	0.0285 (3)	0.0269 (3)	0.0130 (3)	−0.0073 (3)	−0.0038 (2)	0.0000 (2)
O5	0.0213 (3)	0.0150 (2)	0.0118 (2)	0.0006 (2)	0.0021 (2)	−0.00034 (19)
C1	0.0204 (4)	0.0302 (4)	0.0200 (4)	0.0046 (3)	−0.0034 (3)	−0.0051 (3)
C2	0.0185 (4)	0.0368 (5)	0.0298 (5)	0.0079 (4)	−0.0020 (3)	−0.0040 (4)

C3	0.0205 (4)	0.0255 (4)	0.0267 (5)	0.0044 (3)	0.0071 (3)	−0.0024 (4)
C4	0.0240 (4)	0.0204 (4)	0.0173 (4)	0.0002 (3)	0.0061 (3)	−0.0024 (3)
C5	0.0201 (3)	0.0179 (3)	0.0147 (3)	0.0018 (3)	0.0014 (3)	−0.0003 (3)
C6	0.0171 (3)	0.0142 (3)	0.0149 (3)	0.0011 (2)	0.0014 (2)	−0.0009 (2)
C7	0.0208 (3)	0.0141 (3)	0.0162 (3)	0.0024 (2)	0.0039 (3)	0.0014 (2)
C8	0.0134 (3)	0.0141 (3)	0.0131 (3)	0.0005 (2)	0.0010 (2)	0.0001 (2)
C9	0.0160 (3)	0.0177 (3)	0.0144 (3)	−0.0020 (2)	−0.0002 (2)	−0.0002 (3)
C10	0.0149 (3)	0.0277 (4)	0.0198 (4)	−0.0006 (3)	−0.0018 (3)	−0.0005 (3)
C11	0.0149 (3)	0.0304 (4)	0.0224 (4)	0.0045 (3)	0.0002 (3)	−0.0013 (3)
C12	0.0164 (3)	0.0225 (4)	0.0179 (4)	0.0045 (3)	0.0014 (3)	−0.0017 (3)
C13	0.0140 (3)	0.0151 (3)	0.0132 (3)	0.0012 (2)	0.0005 (2)	−0.0001 (2)
C14	0.0157 (3)	0.0137 (3)	0.0119 (3)	0.0009 (2)	0.0006 (2)	−0.0005 (2)
C15	0.0180 (3)	0.0150 (3)	0.0144 (3)	0.0001 (2)	0.0008 (2)	−0.0006 (2)
C16	0.0151 (3)	0.0166 (3)	0.0129 (3)	0.0003 (2)	0.0003 (2)	−0.0002 (2)
C17	0.0181 (3)	0.0229 (4)	0.0211 (4)	0.0028 (3)	0.0017 (3)	−0.0050 (3)
C18	0.0159 (4)	0.0355 (5)	0.0237 (5)	0.0022 (3)	0.0019 (3)	−0.0039 (4)
C19	0.0180 (4)	0.0332 (5)	0.0188 (4)	−0.0057 (3)	−0.0016 (3)	0.0005 (3)
C20	0.0234 (4)	0.0220 (4)	0.0177 (4)	−0.0050 (3)	−0.0010 (3)	−0.0021 (3)
C21	0.0184 (3)	0.0173 (3)	0.0162 (3)	0.0000 (3)	0.0012 (3)	−0.0017 (3)
C22	0.0254 (4)	0.0290 (4)	0.0186 (4)	−0.0060 (3)	−0.0055 (3)	−0.0040 (3)

Geometric parameters (Å, °)

Cl1—C15	1.7631 (9)	C8—C13	1.3964 (12)
Cl2—C15	1.7745 (9)	C8—C9	1.4074 (12)
Cl3—C15	1.7759 (9)	C9—C10	1.3913 (13)
S1—O4	1.4270 (8)	C10—C11	1.3940 (15)
S1—O3	1.4295 (8)	C10—H10A	0.9300
S1—O5	1.5981 (7)	C11—C12	1.3857 (14)
S1—C16	1.7505 (9)	C11—H11A	0.9300
O1—C8	1.3781 (10)	C12—C13	1.4001 (12)
O1—C7	1.4541 (10)	C12—H12A	0.9300
O2—C9	1.3638 (11)	C13—C14	1.5075 (12)
O2—C22	1.4333 (12)	C14—C15	1.5460 (12)
O5—C14	1.4469 (11)	C14—H14A	0.9800
C1—C6	1.3890 (13)	C16—C21	1.3871 (12)
C1—C2	1.3961 (15)	C16—C17	1.3928 (13)
C1—H1A	0.9300	C17—C18	1.3866 (15)
C2—C3	1.3851 (17)	C17—H17A	0.9300
C2—H2A	0.9300	C18—C19	1.3897 (16)
C3—C4	1.3882 (16)	C18—H18A	0.9300
C3—H3A	0.9300	C19—C20	1.3891 (15)
C4—C5	1.3887 (13)	C19—H19A	0.9300
C4—H4A	0.9300	C20—C21	1.3936 (13)
C5—C6	1.3942 (13)	C20—H20A	0.9300
C5—H5A	0.9300	C21—H21A	0.9300
C6—C7	1.4996 (12)	C22—H22D	0.9600
C7—H7A	0.9700	C22—H22A	0.9600
C7—H7B	0.9700	C22—H22B	0.9600

O4—S1—O3	120.76 (5)	C10—C11—H11A	119.5
O4—S1—O5	103.11 (4)	C11—C12—C13	119.66 (9)
O3—S1—O5	108.70 (4)	C11—C12—H12A	120.2
O4—S1—C16	109.46 (4)	C13—C12—H12A	120.2
O3—S1—C16	109.47 (4)	C8—C13—C12	119.76 (8)
O5—S1—C16	103.90 (4)	C8—C13—C14	118.60 (7)
C8—O1—C7	112.01 (7)	C12—C13—C14	121.61 (8)
C9—O2—C22	116.60 (8)	O5—C14—C13	111.16 (7)
C14—O5—S1	119.24 (5)	O5—C14—C15	104.53 (6)
C6—C1—C2	120.53 (10)	C13—C14—C15	115.20 (7)
C6—C1—H1A	119.7	O5—C14—H14A	108.6
C2—C1—H1A	119.7	C13—C14—H14A	108.6
C3—C2—C1	119.79 (10)	C15—C14—H14A	108.6
C3—C2—H2A	120.1	C14—C15—Cl1	108.66 (6)
C1—C2—H2A	120.1	C14—C15—Cl2	109.32 (6)
C2—C3—C4	119.94 (9)	Cl1—C15—Cl2	109.21 (5)
C2—C3—H3A	120.0	C14—C15—Cl3	111.58 (6)
C4—C3—H3A	120.0	Cl1—C15—Cl3	109.24 (5)
C3—C4—C5	120.25 (9)	Cl2—C15—Cl3	108.80 (5)
C3—C4—H4A	119.9	C21—C16—C17	122.05 (8)
C5—C4—H4A	119.9	C21—C16—S1	119.66 (7)
C4—C5—C6	120.21 (9)	C17—C16—S1	118.27 (7)
C4—C5—H5A	119.9	C18—C17—C16	118.63 (9)
C6—C5—H5A	119.9	C18—C17—H17A	120.7
C1—C6—C5	119.23 (8)	C16—C17—H17A	120.7
C1—C6—C7	120.53 (8)	C17—C18—C19	120.14 (9)
C5—C6—C7	120.16 (8)	C17—C18—H18A	119.9
O1—C7—C6	110.03 (7)	C19—C18—H18A	119.9
O1—C7—H7A	109.7	C20—C19—C18	120.58 (9)
C6—C7—H7A	109.7	C20—C19—H19A	119.7
O1—C7—H7B	109.7	C18—C19—H19A	119.7
C6—C7—H7B	109.7	C19—C20—C21	120.03 (9)
H7A—C7—H7B	108.2	C19—C20—H20A	120.0
O1—C8—C13	120.44 (7)	C21—C20—H20A	120.0
O1—C8—C9	119.32 (7)	C16—C21—C20	118.56 (8)
C13—C8—C9	120.22 (8)	C16—C21—H21A	120.7
O2—C9—C10	124.78 (8)	C20—C21—H21A	120.7
O2—C9—C8	115.71 (8)	O2—C22—H22D	109.5
C10—C9—C8	119.51 (8)	O2—C22—H22A	109.5
C9—C10—C11	119.87 (9)	H22D—C22—H22A	109.5
C9—C10—H10A	120.1	O2—C22—H22B	109.5
C11—C10—H10A	120.1	H22D—C22—H22B	109.5
C12—C11—C10	120.97 (9)	H22A—C22—H22B	109.5
C12—C11—H11A	119.5		
O4—S1—O5—C14	−157.73 (6)	C9—C8—C13—C14	179.98 (8)
O3—S1—O5—C14	−28.44 (7)	C11—C12—C13—C8	0.85 (14)
C16—S1—O5—C14	88.06 (7)	C11—C12—C13—C14	178.82 (9)
C6—C1—C2—C3	−0.05 (19)	S1—O5—C14—C13	−89.91 (7)
C1—C2—C3—C4	−1.44 (18)	S1—O5—C14—C15	145.20 (6)

C2—C3—C4—C5	1.17 (16)	C8—C13—C14—O5	140.16 (8)
C3—C4—C5—C6	0.59 (15)	C12—C13—C14—O5	−37.83 (11)
C2—C1—C6—C5	1.79 (16)	C8—C13—C14—C15	−101.19 (9)
C2—C1—C6—C7	−174.91 (10)	C12—C13—C14—C15	80.81 (11)
C4—C5—C6—C1	−2.06 (14)	O5—C14—C15—Cl1	179.41 (5)
C4—C5—C6—C7	174.65 (8)	C13—C14—C15—Cl1	57.13 (8)
C8—O1—C7—C6	−157.96 (7)	O5—C14—C15—Cl2	−61.48 (7)
C1—C6—C7—O1	−109.63 (10)	C13—C14—C15—Cl2	176.24 (6)
C5—C6—C7—O1	73.71 (10)	O5—C14—C15—Cl3	58.91 (7)
C7—O1—C8—C13	−104.57 (9)	C13—C14—C15—Cl3	−63.37 (9)
C7—O1—C8—C9	77.03 (10)	O4—S1—C16—C21	139.64 (8)
C22—O2—C9—C10	−18.96 (14)	O3—S1—C16—C21	5.19 (9)
C22—O2—C9—C8	161.48 (8)	O5—S1—C16—C21	−110.77 (7)
O1—C8—C9—O2	−0.15 (12)	O4—S1—C16—C17	−38.88 (9)
C13—C8—C9—O2	−178.55 (8)	O3—S1—C16—C17	−173.33 (8)
O1—C8—C9—C10	−179.73 (8)	O5—S1—C16—C17	70.71 (8)
C13—C8—C9—C10	1.86 (13)	C21—C16—C17—C18	0.15 (15)
O2—C9—C10—C11	179.85 (9)	S1—C16—C17—C18	178.62 (8)
C8—C9—C10—C11	−0.61 (15)	C16—C17—C18—C19	−0.67 (16)
C9—C10—C11—C12	−0.52 (16)	C17—C18—C19—C20	0.96 (17)
C10—C11—C12—C13	0.41 (16)	C18—C19—C20—C21	−0.70 (16)
O1—C8—C13—C12	179.63 (8)	C17—C16—C21—C20	0.11 (14)
C9—C8—C13—C12	−1.98 (13)	S1—C16—C21—C20	−178.35 (7)
O1—C8—C13—C14	1.60 (12)	C19—C20—C21—C16	0.17 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7B···O2	0.97	2.56	3.040 (1)	110
C14—H14A···O1	0.98	2.38	2.820 (1)	107
C14—H14A···O3	0.98	2.34	2.838 (1)	111
C21—H21A···O3	0.93	2.55	2.919 (1)	104
C3—H3A···O2^{i}	0.93	2.49	3.414 (1)	172

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

Fig. 1

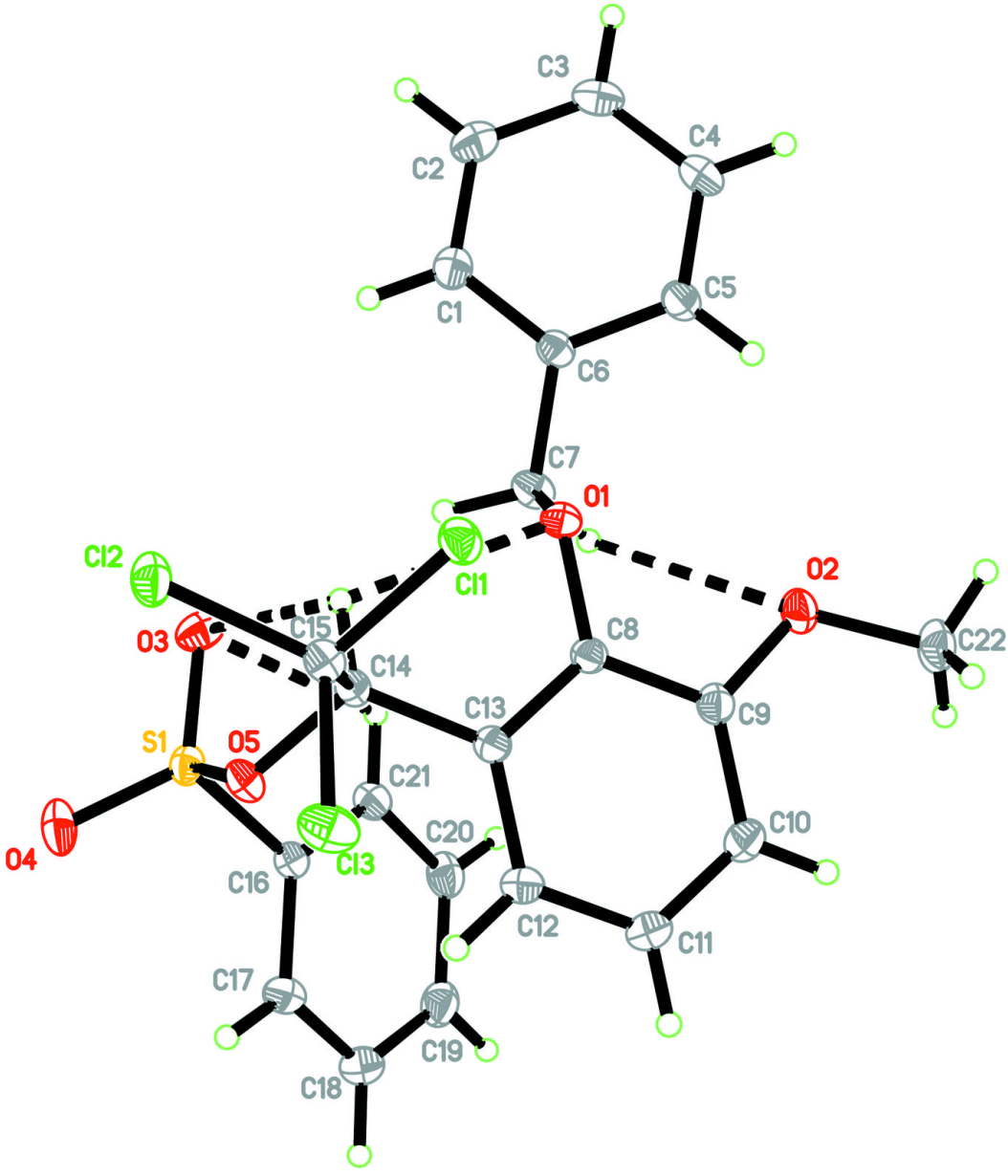
C3
C4
C2
C5
C1
C6
C7
O1
Cl2
Cl1
O2
C22
O3
C15
C14
C8
C13
C9
S1
O5
C21
C10
O4
C20
Cl3
C16
C12
C11
C17
C19
C18

Fig. 2