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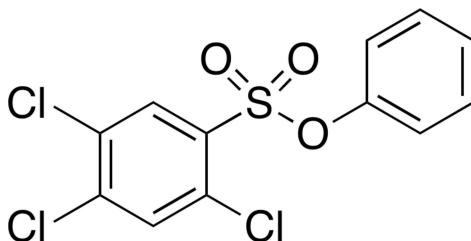
Crystal structure of phenyl 2,4,5-trichlorobenzene-sulfonate

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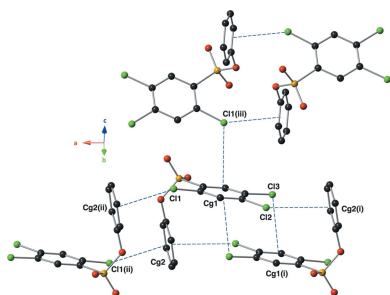
The title compound, C₁₂H₇Cl₃O₃S, was synthesized *via* a nucleophilic substitution reaction between phenol and 2,4,5-trichlorobenzene-sulfonyl chloride. The two aryl rings are oriented *gauche* to one another around the sulfonate S—O bond, with a C—S—O—C torsion angle of $-70.68(16)^\circ$, and the two rings are inclined to one another by $72.40(7)^\circ$. In the crystal, molecules are linked *via* various C—Cl... π interactions, forming ribbons propagating along [100]. Neighboring ribbons are linked by a weak C—Cl... π interaction, forming layers parallel to (010).

1. Chemical context

The use of arene-sulfonates as leaving groups has been explored in synthetic organic chemistry for quite some time (Crossland *et al.*, 1971; Klán *et al.*, 2013; Sardzinski *et al.*, 2015). The stability of sulfonate ester leaving groups and the identification of suitable protecting groups for sulfonates has been reported (Miller, 2010). A competitive C—O and S—O bond fission has been reported in the reaction of amine nucleophiles with arene-sulfonates (Um *et al.*, 2004). The basicity of the amine nucleophile and the electronic nature of the substituent on the sulfonyl moiety are responsible for the difference in regioselectivity. We have synthesized various arene-sulfonate analogues in order to investigate the factors responsible for the competition between C—O and S—O bond fission in the reaction with nitrogen nucleophiles (Atanasova *et al.*, 2015; Cooley *et al.*, 2015).



The sulfonamide moiety has found many useful applications in medicinal chemistry (Navia, 2000). Sulfonamides can be synthesized conveniently from the corresponding sulfonyl chloride and amine nucleophiles. In our recent work, we reported on the synthesis and crystal structure of a chiral sulfonamide (Ngassa *et al.*, 2015). The direct synthesis of sulfonamides from arene-sulfonates has been reported (Caddick *et al.*, 2004). Taking advantage of the regioselectivity of C—O vs S—O bond fission, we have explored the use of arene-sulfonates as electrophilic substrates in the synthesis of



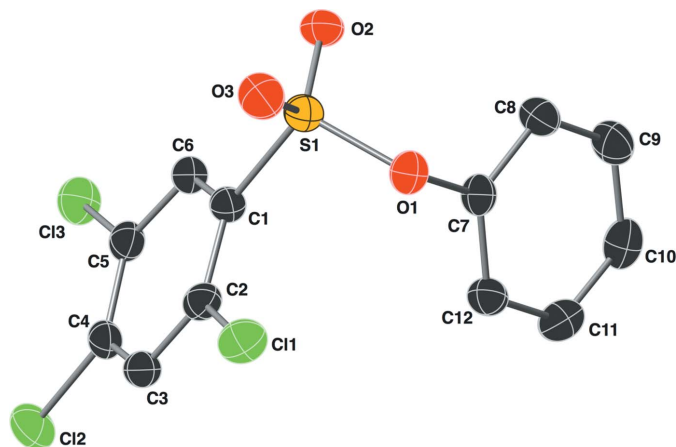


Figure 1
The molecular structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

sulfonamides. We are interested in the role of the substituent on the sulfonyl moiety and the basicity of the amine nucleophile on the nucleophilic substitution. As the title compound is of interest in our ongoing effort to investigate the role of the substituent on the sulfonyl moiety in nucleophilic substitution reactions with nitrogen- and oxygen-nucleophiles, we report herein on the synthesis and crystal structure of this electrophilic arene-sulfonate.

Table 1
Geometric parameters (\AA , $^\circ$) for C—Cl $\cdots\pi$ contacts in the title compound..

Cg 1 and Cg 2 are the centroids of rings C1–C6 and C7–C12, respectively.

C—Cl \cdots Cg	C—Cl	Cl \cdots Cg	C \cdots Cg	C—Cl \cdots Cg
C2—Cl1 \cdots Cg2 ⁱ	1.727 (2)	3.5250 (10)	5.028 (2)	144.23 (7)
C4—Cl2 \cdots Cg2 ⁱⁱ	1.721 (2)	3.7914 (11)	5.160 (2)	135.37 (7)
C5—Cl3 \cdots Cg1 ⁱⁱⁱ	1.725 (2)	3.6298 (10)	4.211 (2)	97.25 (7)
C2—Cl1 \cdots Cg1 ⁱⁱⁱ	1.727 (2)	3.9722 (10)	4.989 (2)	116.56 (7)

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

2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The two aryl rings are oriented gauche to one another around the sulfonate S1—O1 bond, with a C1—S1—O1—C7 torsion angle of $-70.68 (16)^\circ$. The two rings (C1–C6 and C7–C12) are inclined to one another by $72.40 (7)^\circ$.

3. Supramolecular features

In the crystal, molecules are linked by Cl $\cdots\pi$ interactions (Table 1 and Fig. 2). These intermolecular interactions range in Cl \cdots ring centroid distances from 3.525 (1) to 3.972 (1) \AA (Table 1). This distance falls near the accepted average as previously noted (Imai, *et al.*, 2008), and all interactions have a

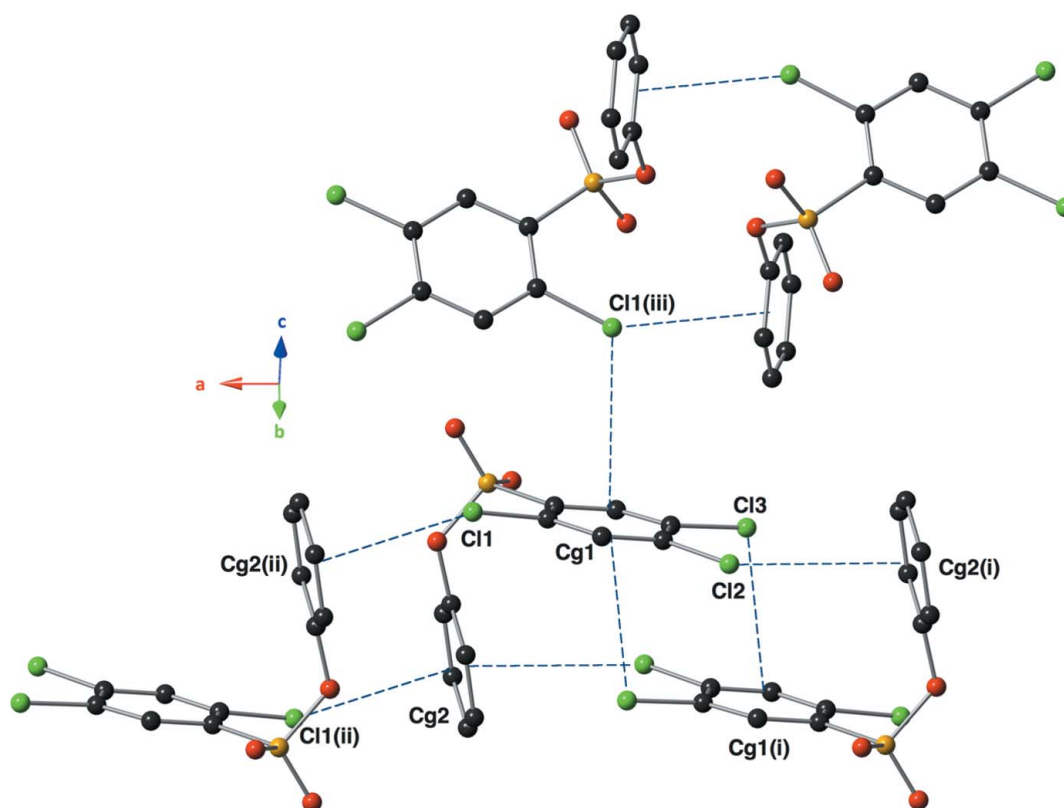


Figure 2
A view of the various C—Cl $\cdots\pi$ interactions (blue dashed lines; see Table 1) present in the crystal lattice of the title compound. H atoms have been omitted for clarity [symmetry codes: (i) $-x + 2, -y + 1, -z + 1$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$].

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₂ H ₇ Cl ₃ O ₃ S
<i>M_r</i>	337.59
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	12.3401 (11), 6.5421 (6), 16.1350 (14)
β (°)	92.1159 (10)
<i>V</i> (Å ³)	1301.7 (2)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.86
Crystal size (mm)	0.24 × 0.18 × 0.10
Data collection	
Diffraction	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2013)
<i>T_{min}</i> , <i>T_{max}</i>	0.689, 0.745
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	10912, 2568, 2172
<i>R_{int}</i>	0.029
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.618
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.031, 0.083, 1.06
No. of reflections	2568
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.26, -0.28

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015), *CrystalMaker* (Palmer, 2007).

'face-on' geometry. The two strong interactions involving atoms Cl1 and Cl2 with the centroid of ring C7–C12 form ribbons propagating along the *a*-axis direction. Within the ribbon there is also a weaker Cl·· π interaction involving atom Cl3 and the centroid of ring C1–C6. Neighbouring ribbons are linked by a second weak Cl1·· π interaction (Table 1 and Fig. 2), forming layers parallel to the *ac* plane. There are no other significant intermolecular interactions present in the crystal.

4. Database survey

The Cambridge Structural Database (CSD, Version 5.37, February 2016; Groom *et al.*, 2016) contains eight structures of phenyl sulfonates where the group bonded directly to the sulfur atom is an aromatic ring. Other substituents on this ring include *p*-tolyl (FIQCIS: Manivannan *et al.*, 2005), nitro (AJIWUL: Vembu *et al.*, 2003; XUKBOV: Vembu & Fronczek, 2009), naphthyl (VOJBOM: Vennila *et al.*, 2008) and amino-naphthyl (LEZWAP: Beyeh *et al.*, 2007). Of particular interest is the structure JEGWEY (Wright *et al.*, 2006) where the substituted aromatic ring bears chlorine atoms in the 2- and 5-positions. The torsion angle around the sulfonate S–O bond is 73.15 (19)°, similar to that seen in the title compound [70.68 (16)°]. In the crystal of this compound, one C–Cl·· π interaction is present [Cl·· π distance: 3.4187 (16) Å] along with C–H··O hydrogen bonds.

Two recent publications describing the crystal structures of benzopyrimidoazepine derivatives have also noted C–Cl·· π interactions present in the lattice (Acosta *et al.*, 2015; Acosta Quintero *et al.*, 2016). In these examples, the C–Cl·· π interactions are complemented by either C–H·· π or π – π interactions between molecules in the solid state.

5. Synthesis and crystallization

Phenol (0.941 g, 10 mmol) was dissolved in 10 ml of chilled dichloromethane. This was followed by the addition of pyridine (1.6 ml, 20 mmol). The resulting solution was cooled in an ice bath under an N₂ atmosphere, followed by the addition of 2,4,5-trichlorobenzenesulfonyl chloride (1.91 g, 10 mmol) portion-wise. The mixture was stirred at 273 K for 30 min and then at room temperature for 12 h. Reaction completion was verified by using TLC analysis. After dilution with 15 ml of CH₂Cl₂, the organic phase was washed with H₂O, brine, and dried over anhydrous Na₂SO₄. After the solvent was evaporated the crude product was obtained as a tan solid. The title compound was recrystallized from CH₂Cl₂/hexanes to afford colourless needle-like crystals (56% yield, m.p. 380–381 K) suitable for X-ray diffraction analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The positions of all hydrogen atoms were calculated geometrically and refined to ride on their parent atoms: C–H = 0.95 Å with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Acknowledgements

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supporting information

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Crystal structure of phenyl 2,4,5-trichlorobenzenesulfonate

Sean Riley, Richard J. Staples, Shannon M. Biros and Felix N. Ngassa

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015); software used to prepare material for publication: *CrystalMaker* (Palmer, 2007).

Phenyl 2,4,5-trichlorobenzenesulfonate

Crystal data

$C_{12}H_7Cl_3O_3S$

$M_r = 337.59$

Monoclinic, $P2_1/n$

$a = 12.3401$ (11) Å

$b = 6.5421$ (6) Å

$c = 16.1350$ (14) Å

$\beta = 92.1159$ (10)°

$V = 1301.7$ (2) Å³

$Z = 4$

$F(000) = 680$

$D_x = 1.723$ Mg m⁻³

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

Cell parameters from 5969 reflections

$\theta = 2.5$ – 26.0 °

$\mu = 0.86$ mm⁻¹

$T = 173$ K

Needle, colourless

$0.24 \times 0.18 \times 0.10$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2013)

$T_{\min} = 0.689$, $T_{\max} = 0.745$

10912 measured reflections

2568 independent reflections

2172 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 26.1$ °, $\theta_{\min} = 2.0$ °

$h = -15 \rightarrow 15$

$k = -8 \rightarrow 8$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.083$

$S = 1.06$

2568 reflections

172 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.7507P]$

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

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

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.28$ e Å⁻³

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl3	0.40330 (4)	0.76082 (9)	0.56242 (3)	0.03498 (15)
Cl2	0.43839 (5)	1.20119 (9)	0.64173 (4)	0.04126 (17)
Cl1	0.86246 (4)	1.05869 (8)	0.68164 (3)	0.03599 (16)
S1	0.83040 (4)	0.61521 (8)	0.58959 (3)	0.02676 (14)
O2	0.78797 (12)	0.4493 (2)	0.54149 (9)	0.0322 (3)
O3	0.88861 (13)	0.5784 (2)	0.66557 (9)	0.0367 (4)
C1	0.72261 (16)	0.7865 (3)	0.60570 (12)	0.0250 (4)
C12	0.86103 (16)	0.9951 (3)	0.43502 (13)	0.0297 (5)
H12	0.8568	1.0940	0.4779	0.036*
O1	0.91219 (11)	0.7437 (2)	0.53709 (8)	0.0283 (3)
C3	0.64884 (17)	1.1015 (3)	0.65532 (12)	0.0287 (5)
H3	0.6587	1.2317	0.6806	0.034*
C6	0.61918 (16)	0.7233 (3)	0.58012 (12)	0.0260 (4)
H6	0.6093	0.5947	0.5535	0.031*
C8	0.89316 (17)	0.6461 (3)	0.39295 (13)	0.0309 (5)
H8	0.9107	0.5088	0.4072	0.037*
C5	0.53069 (16)	0.8467 (3)	0.59320 (12)	0.0267 (4)
C4	0.54605 (17)	1.0382 (3)	0.62926 (12)	0.0285 (5)
C2	0.73697 (17)	0.9755 (3)	0.64467 (12)	0.0273 (4)
C7	0.88520 (16)	0.7940 (3)	0.45289 (12)	0.0256 (4)
C10	0.84980 (18)	0.9035 (4)	0.29115 (13)	0.0352 (5)
H10	0.8371	0.9415	0.2348	0.042*
C11	0.84316 (18)	1.0485 (3)	0.35269 (14)	0.0345 (5)
H11	0.8262	1.1860	0.3385	0.041*
C9	0.87469 (18)	0.7041 (4)	0.31090 (14)	0.0359 (5)
H9	0.8793	0.6053	0.2680	0.043*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl3	0.0269 (3)	0.0401 (3)	0.0379 (3)	0.0003 (2)	0.0017 (2)	0.0008 (2)
Cl2	0.0432 (3)	0.0361 (3)	0.0451 (3)	0.0146 (2)	0.0111 (3)	0.0001 (2)
Cl1	0.0383 (3)	0.0317 (3)	0.0375 (3)	-0.0058 (2)	-0.0056 (2)	-0.0032 (2)
S1	0.0288 (3)	0.0241 (3)	0.0275 (3)	0.0027 (2)	0.0018 (2)	0.0006 (2)
O2	0.0345 (8)	0.0237 (8)	0.0386 (8)	-0.0003 (6)	0.0043 (6)	-0.0037 (6)
O3	0.0419 (9)	0.0386 (9)	0.0295 (8)	0.0100 (7)	-0.0021 (7)	0.0046 (7)
C1	0.0287 (11)	0.0228 (10)	0.0235 (10)	0.0032 (8)	0.0033 (8)	0.0015 (8)
C12	0.0290 (11)	0.0256 (11)	0.0347 (11)	-0.0001 (9)	0.0044 (9)	-0.0039 (9)
O1	0.0244 (7)	0.0334 (8)	0.0271 (7)	-0.0018 (6)	0.0002 (6)	0.0006 (6)

C3	0.0420 (12)	0.0232 (10)	0.0210 (10)	0.0007 (9)	0.0046 (9)	0.0001 (8)
C6	0.0296 (11)	0.0245 (10)	0.0241 (10)	0.0000 (8)	0.0042 (8)	0.0001 (8)
C8	0.0299 (11)	0.0287 (11)	0.0344 (11)	0.0034 (9)	0.0048 (9)	-0.0034 (9)
C5	0.0281 (10)	0.0285 (11)	0.0236 (10)	0.0006 (8)	0.0035 (8)	0.0029 (8)
C4	0.0354 (11)	0.0266 (11)	0.0239 (10)	0.0073 (9)	0.0088 (8)	0.0042 (8)
C2	0.0347 (11)	0.0247 (10)	0.0226 (10)	-0.0043 (9)	0.0011 (8)	0.0006 (8)
C7	0.0212 (10)	0.0305 (11)	0.0251 (10)	-0.0006 (8)	0.0022 (8)	0.0005 (8)
C10	0.0323 (12)	0.0458 (14)	0.0275 (11)	-0.0051 (10)	0.0022 (9)	0.0029 (10)
C11	0.0339 (12)	0.0301 (12)	0.0394 (12)	-0.0003 (9)	-0.0001 (10)	0.0066 (10)
C9	0.0371 (12)	0.0393 (13)	0.0316 (11)	-0.0027 (10)	0.0055 (9)	-0.0108 (10)

Geometric parameters (Å, °)

C13—C5	1.725 (2)	C3—C4	1.385 (3)
C12—C4	1.721 (2)	C3—C2	1.380 (3)
C11—C2	1.727 (2)	C6—H6	0.9500
S1—O2	1.4229 (15)	C6—C5	1.380 (3)
S1—O3	1.4184 (15)	C8—H8	0.9500
S1—C1	1.766 (2)	C8—C7	1.374 (3)
S1—O1	1.5828 (15)	C8—C9	1.388 (3)
C1—C6	1.390 (3)	C5—C4	1.391 (3)
C1—C2	1.395 (3)	C10—H10	0.9500
C12—H12	0.9500	C10—C11	1.378 (3)
C12—C7	1.377 (3)	C10—C9	1.375 (3)
C12—C11	1.383 (3)	C11—H11	0.9500
O1—C7	1.425 (2)	C9—H9	0.9500
C3—H3	0.9500		
O2—S1—C1	107.48 (9)	C6—C5—C13	118.90 (16)
O2—S1—O1	110.01 (8)	C6—C5—C4	119.61 (19)
O3—S1—O2	120.41 (9)	C4—C5—C13	121.47 (16)
O3—S1—C1	109.92 (9)	C3—C4—C12	118.78 (16)
O3—S1—O1	103.89 (9)	C3—C4—C5	120.35 (19)
O1—S1—C1	103.92 (9)	C5—C4—C12	120.87 (17)
C6—C1—S1	117.13 (15)	C1—C2—C11	122.17 (16)
C6—C1—C2	119.83 (19)	C3—C2—C11	117.99 (16)
C2—C1—S1	123.01 (16)	C3—C2—C1	119.84 (19)
C7—C12—H12	121.1	C12—C7—O1	117.49 (18)
C7—C12—C11	117.9 (2)	C8—C7—C12	123.09 (19)
C11—C12—H12	121.1	C8—C7—O1	119.19 (18)
C7—O1—S1	120.10 (12)	C11—C10—H10	119.8
C4—C3—H3	120.0	C9—C10—H10	119.8
C2—C3—H3	120.0	C9—C10—C11	120.3 (2)
C2—C3—C4	120.04 (19)	C12—C11—H11	119.8
C1—C6—H6	119.9	C10—C11—C12	120.4 (2)
C5—C6—C1	120.24 (19)	C10—C11—H11	119.8
C5—C6—H6	119.9	C8—C9—H9	119.7
C7—C8—H8	121.1	C10—C9—C8	120.5 (2)

C7—C8—C9	117.7 (2)	C10—C9—H9	119.7
C9—C8—H8	121.1		
C13—C5—C4—C12	-2.0 (2)	C6—C1—C2—C11	177.38 (15)
C13—C5—C4—C3	178.38 (15)	C6—C1—C2—C3	-2.0 (3)
S1—C1—C6—C5	177.92 (15)	C6—C5—C4—C12	176.71 (15)
S1—C1—C2—C11	-0.5 (3)	C6—C5—C4—C3	-2.9 (3)
S1—C1—C2—C3	-179.86 (15)	C4—C3—C2—C11	-177.78 (15)
S1—O1—C7—C12	109.03 (18)	C4—C3—C2—C1	1.6 (3)
S1—O1—C7—C8	-76.3 (2)	C2—C1—C6—C5	-0.1 (3)
O2—S1—C1—C6	7.64 (18)	C2—C3—C4—C12	-178.78 (15)
O2—S1—C1—C2	-174.44 (16)	C2—C3—C4—C5	0.8 (3)
O2—S1—O1—C7	44.13 (16)	C7—C12—C11—C10	-0.1 (3)
O3—S1—C1—C6	-125.09 (16)	C7—C8—C9—C10	0.3 (3)
O3—S1—C1—C2	52.8 (2)	C11—C12—C7—O1	174.98 (18)
O3—S1—O1—C7	174.30 (14)	C11—C12—C7—C8	0.6 (3)
C1—S1—O1—C7	-70.68 (16)	C11—C10—C9—C8	0.2 (3)
C1—C6—C5—C13	-178.73 (15)	C9—C8—C7—C12	-0.6 (3)
C1—C6—C5—C4	2.5 (3)	C9—C8—C7—O1	-174.96 (18)
O1—S1—C1—C6	124.24 (15)	C9—C10—C11—C12	-0.2 (3)
O1—S1—C1—C2	-57.85 (18)		
