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trans-4-[(Phenylsulfonyloxy)methyl]cyclohexanecarboxylic acid

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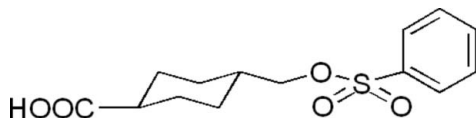
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 Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.055; wR factor = 0.143; data-to-parameter ratio = 14.7.

The title compound, $\text{C}_{14}\text{H}_{18}\text{O}_5\text{S}$, is an important intermediate for the synthesis of poly(amidoamine) dendrimers. The cyclohexane ring adopts a chair conformation with its two substituents in equatorial positions. In the crystal structure, molecules form centrosymmetric dimers via $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

 For related literature, see: Ahmed *et al.* (2001); Grabchev *et al.* (2003); Wang *et al.* (2004).


Experimental

Crystal data

 $\text{C}_{14}\text{H}_{18}\text{O}_5\text{S}$
 $M_r = 298.34$
 Monoclinic, $P2_1/c$
 $a = 17.097$ (5) Å

 $b = 5.960$ (3) Å
 $c = 14.919$ (4) Å
 $\beta = 107.09$ (3)°
 $V = 1453.2$ (10) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹
 $T = 292$ (2) K
 $0.32 \times 0.32 \times 0.13$ mm

Data collection

 Enraf–Nonius CAD-4 diffractometer
 Absorption correction: none
 3702 measured reflections
 2691 independent reflections

 1323 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.008$
 3 standard reflections every 250 reflections
 intensity decay: 0.9%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.142$
 $S = 1.00$
 2691 reflections

 183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4}\cdots\text{O5}^i$	0.82	1.86	2.677 (3)	174

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

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2749).

References

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supporting information

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***trans*-4-[(Phenylsulfonyl)methyl]cyclohexanecarboxylic acid**

Yu-Feng Liang, Qing-Rong Qi and Hu Zheng

S1. Comment

PAMAM (poly(amidoamine)) dendrimers have attracted much interest for their symmetry, high degree of branching and high density of terminal functional groups, which can participate in different reactions. The modification of periphery of PAMAM dendrimer which aimed to change the physical or chemical properties of PAMAM dendrimers, have been reported recently (Grabchev *et al.*,2003; Ahmed *et al.*,2001; Wang *et al.*,2004). To improve the lipophilicity of PAMAM dendrimers and provide a new type of linker with special stereostructure, a series of cyclohexane derivatives were synthesized.

The crystal structure shows that molecules are mainly linked by O—H···O hydrogen bonds and the cyclohexane ring of the title compound exists in the chair conformation.

S2. Experimental

trans-4-(methoxycarbonyl)cyclohexanemethanol (10 mmol), triethylamine (10 mmol) and a small amount of trimethylamine hydrochloride were suspended in dichloromethane (20 mL), benzenesulfonyl chloride (11 mmol) was dropped with vigorous stirring at room temperature, after 1 h the reaction was quenched by addition of water. The organic layer separated was evaporated to give an oil and the oil was hydrolyzed in methanol and aqueous NaOH (11 mmol) solution for 5 h at 323 K. Then the title compound was obtained by acidification with hydrochloride and recrystallized from acetone. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation in cyclohexane and acetone at room temperature.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.93–0.98 Å, O—H = 0.82 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{O})$.

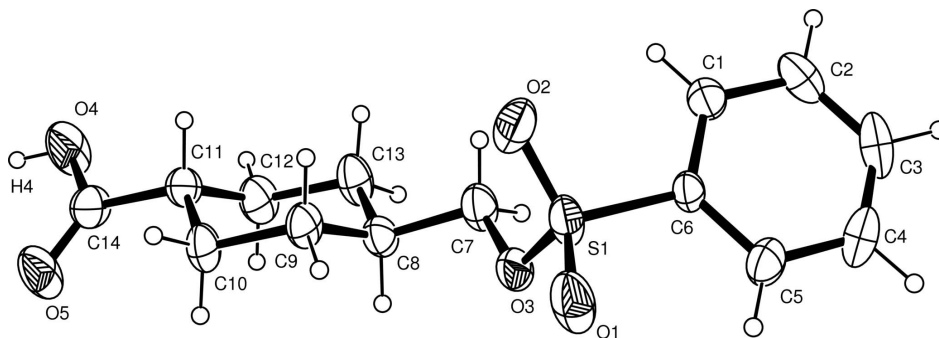


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

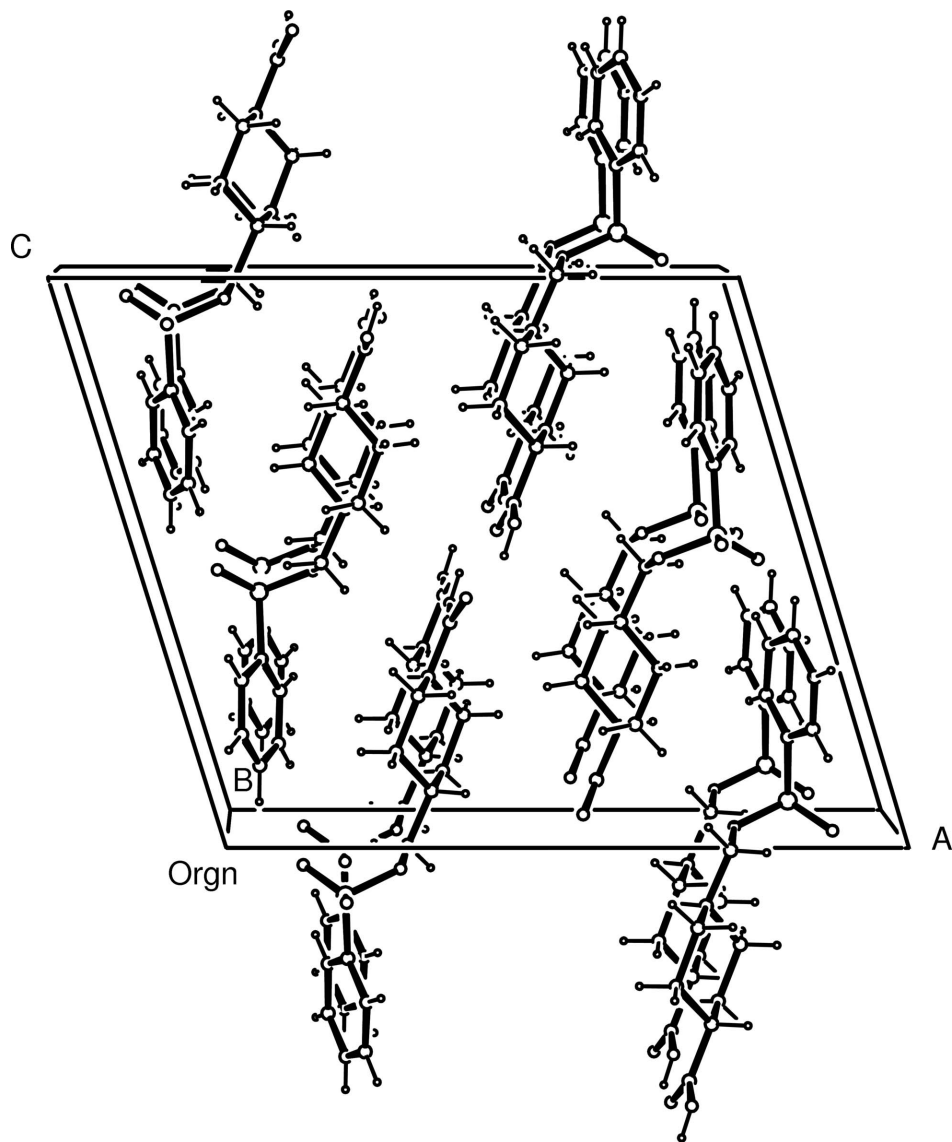


Figure 2

A packing diagram of the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

***trans*-4-[(Phenylsulfonyloxy)methyl]cyclohexanecarboxylic acid**

Crystal data

$C_{14}H_{18}O_5S$

$M_r = 298.34$

Monoclinic, $P2_1/c$

$a = 17.097 (5) \text{ \AA}$

$b = 5.960 (3) \text{ \AA}$

$c = 14.919 (4) \text{ \AA}$

$\beta = 107.09 (3)^\circ$

$V = 1453.2 (10) \text{ \AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.364 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 24 reflections

$\theta = 4.4\text{--}8.7^\circ$

$\mu = 0.24 \text{ mm}^{-1}$

$T = 292 \text{ K}$

Block, colourless

$0.32 \times 0.32 \times 0.13 \text{ mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$ scans

3702 measured reflections

2691 independent reflections

1323 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.008$

$\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 1.3^\circ$

$h = -2 \rightarrow 20$

$k = -7 \rightarrow 0$

$l = -18 \rightarrow 17$

3 standard reflections every 250 reflections

intensity decay: 0.9%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.143$

$S = 1.00$

2691 reflections

183 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

$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2]$

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

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

$\Delta\rho_{\text{max}} = 0.27 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0076 (16)

Special details

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

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.15478 (7)	0.60525 (16)	0.42320 (6)	0.0631 (4)
O1	0.15724 (19)	0.8419 (4)	0.42806 (17)	0.0942 (11)
O2	0.10478 (16)	0.4829 (4)	0.46844 (17)	0.0782 (8)
O3	0.24614 (15)	0.5294 (4)	0.46482 (16)	0.0705 (8)
O4	0.4357 (2)	-0.1520 (5)	0.8999 (2)	0.1010 (11)
H4	0.4645	-0.1623	0.9545	0.151*
O5	0.46270 (19)	0.2062 (5)	0.92678 (18)	0.0973 (11)
C1	0.0928 (2)	0.3172 (6)	0.2785 (2)	0.0558 (10)
H1	0.0855	0.2167	0.3231	0.067*
C2	0.0687 (2)	0.2625 (7)	0.1846 (3)	0.0694 (12)
H2	0.0457	0.1227	0.1654	0.083*
C3	0.0785 (3)	0.4130 (9)	0.1199 (3)	0.0786 (14)
H3	0.0615	0.3752	0.0567	0.094*
C4	0.1127 (3)	0.6184 (9)	0.1467 (3)	0.0807 (13)
H4A	0.1184	0.7202	0.1017	0.097*

C5	0.1386 (2)	0.6739 (6)	0.2393 (2)	0.0636 (11)
H5	0.1632	0.8122	0.2579	0.076*
C6	0.12814 (18)	0.5243 (5)	0.3051 (2)	0.0434 (9)
C7	0.2647 (2)	0.2958 (6)	0.4899 (2)	0.0634 (11)
H7A	0.2877	0.2257	0.4447	0.076*
H7B	0.2147	0.2168	0.4885	0.076*
C8	0.3248 (2)	0.2787 (6)	0.5868 (2)	0.0543 (10)
H8	0.3740	0.3632	0.5870	0.065*
C9	0.2910 (2)	0.3764 (6)	0.6619 (2)	0.0560 (10)
H9A	0.2390	0.3056	0.6579	0.067*
H9B	0.2812	0.5356	0.6504	0.067*
C10	0.3488 (2)	0.3433 (6)	0.7602 (2)	0.0595 (11)
H10A	0.3985	0.4287	0.7669	0.071*
H10B	0.3232	0.3991	0.8058	0.071*
C11	0.3702 (2)	0.0971 (6)	0.7794 (2)	0.0540 (10)
H11	0.3193	0.0165	0.7753	0.065*
C12	0.4053 (2)	-0.0003 (6)	0.7056 (2)	0.0685 (12)
H12A	0.4147	-0.1597	0.7169	0.082*
H12B	0.4577	0.0699	0.7109	0.082*
C13	0.3484 (2)	0.0350 (6)	0.6063 (2)	0.0688 (12)
H13A	0.3754	-0.0171	0.5614	0.083*
H13B	0.2993	-0.0540	0.5981	0.083*
C14	0.4275 (2)	0.0573 (7)	0.8757 (3)	0.0637 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0904 (8)	0.0477 (6)	0.0373 (5)	0.0035 (6)	-0.0028 (5)	-0.0010 (5)
O1	0.152 (3)	0.0478 (16)	0.0584 (17)	0.0040 (17)	-0.0069 (17)	-0.0068 (14)
O2	0.103 (2)	0.0828 (19)	0.0552 (16)	0.0009 (16)	0.0327 (15)	0.0129 (15)
O3	0.0844 (18)	0.0586 (15)	0.0488 (15)	-0.0147 (13)	-0.0111 (13)	0.0161 (12)
O4	0.129 (3)	0.0696 (19)	0.0663 (19)	-0.0009 (17)	-0.0307 (17)	0.0240 (15)
O5	0.125 (3)	0.0689 (18)	0.0601 (18)	-0.0064 (17)	-0.0309 (17)	0.0143 (15)
C1	0.060 (2)	0.053 (2)	0.049 (2)	-0.0035 (18)	0.0074 (18)	0.0019 (18)
C2	0.062 (2)	0.065 (3)	0.065 (3)	0.005 (2)	-0.006 (2)	-0.022 (2)
C3	0.086 (3)	0.104 (4)	0.034 (2)	0.034 (3)	-0.001 (2)	-0.006 (3)
C4	0.105 (3)	0.091 (3)	0.046 (2)	0.020 (3)	0.023 (2)	0.023 (3)
C5	0.077 (3)	0.058 (2)	0.051 (2)	0.004 (2)	0.012 (2)	0.014 (2)
C6	0.049 (2)	0.0432 (19)	0.0327 (18)	0.0033 (16)	0.0031 (15)	0.0025 (15)
C7	0.078 (3)	0.055 (2)	0.046 (2)	0.006 (2)	0.0014 (19)	0.0017 (19)
C8	0.057 (2)	0.053 (2)	0.044 (2)	0.0011 (17)	0.0010 (18)	0.0034 (18)
C9	0.060 (2)	0.054 (2)	0.046 (2)	0.0115 (18)	0.0023 (17)	0.0113 (19)
C10	0.068 (2)	0.064 (2)	0.0378 (19)	0.010 (2)	0.0006 (17)	0.0036 (18)
C11	0.051 (2)	0.057 (2)	0.045 (2)	0.0009 (18)	0.0007 (17)	0.0120 (19)
C12	0.072 (2)	0.063 (2)	0.058 (2)	0.017 (2)	0.000 (2)	0.009 (2)
C13	0.078 (3)	0.067 (3)	0.048 (2)	0.020 (2)	-0.003 (2)	-0.0003 (18)
C14	0.060 (2)	0.066 (3)	0.056 (2)	0.004 (2)	0.0020 (19)	0.020 (2)

Geometric parameters (Å, °)

S1—O1	1.412 (3)	C7—H7A	0.9700
S1—O2	1.434 (3)	C7—H7B	0.9700
S1—O3	1.568 (3)	C8—C13	1.513 (5)
S1—C6	1.754 (3)	C8—C9	1.521 (5)
O3—C7	1.453 (4)	C8—H8	0.9800
O4—C14	1.295 (4)	C9—C10	1.521 (4)
O4—H4	0.8200	C9—H9A	0.9700
O5—C14	1.209 (4)	C9—H9B	0.9700
C1—C2	1.378 (5)	C10—C11	1.519 (4)
C1—C6	1.380 (4)	C10—H10A	0.9700
C1—H1	0.9300	C10—H10B	0.9700
C2—C3	1.364 (6)	C11—C14	1.501 (4)
C2—H2	0.9300	C11—C12	1.516 (5)
C3—C4	1.365 (6)	C11—H11	0.9800
C3—H3	0.9300	C12—C13	1.529 (4)
C4—C5	1.362 (5)	C12—H12A	0.9700
C4—H4A	0.9300	C12—H12B	0.9700
C5—C6	1.376 (4)	C13—H13A	0.9700
C5—H5	0.9300	C13—H13B	0.9700
C7—C8	1.510 (4)		
O1—S1—O2	119.8 (2)	C13—C8—H8	108.2
O1—S1—O3	104.85 (16)	C9—C8—H8	108.2
O2—S1—O3	109.22 (15)	C8—C9—C10	112.4 (3)
O1—S1—C6	108.74 (16)	C8—C9—H9A	109.1
O2—S1—C6	108.57 (16)	C10—C9—H9A	109.1
O3—S1—C6	104.66 (16)	C8—C9—H9B	109.1
C7—O3—S1	119.6 (2)	C10—C9—H9B	109.1
C14—O4—H4	109.5	H9A—C9—H9B	107.9
C2—C1—C6	118.6 (4)	C11—C10—C9	111.1 (3)
C2—C1—H1	120.7	C11—C10—H10A	109.4
C6—C1—H1	120.7	C9—C10—H10A	109.4
C3—C2—C1	120.1 (4)	C11—C10—H10B	109.4
C3—C2—H2	119.9	C9—C10—H10B	109.4
C1—C2—H2	119.9	H10A—C10—H10B	108.0
C2—C3—C4	120.9 (4)	C14—C11—C12	110.4 (3)
C2—C3—H3	119.5	C14—C11—C10	112.7 (3)
C4—C3—H3	119.5	C12—C11—C10	110.9 (3)
C5—C4—C3	119.9 (4)	C14—C11—H11	107.5
C5—C4—H4A	120.1	C12—C11—H11	107.5
C3—C4—H4A	120.1	C10—C11—H11	107.5
C4—C5—C6	119.6 (4)	C11—C12—C13	112.1 (3)
C4—C5—H5	120.2	C11—C12—H12A	109.2
C6—C5—H5	120.2	C13—C12—H12A	109.2
C5—C6—C1	120.8 (3)	C11—C12—H12B	109.2
C5—C6—S1	119.3 (3)	C13—C12—H12B	109.2

C1—C6—S1	119.8 (3)	H12A—C12—H12B	107.9
O3—C7—C8	110.3 (3)	C8—C13—C12	112.1 (3)
O3—C7—H7A	109.6	C8—C13—H13A	109.2
C8—C7—H7A	109.6	C12—C13—H13A	109.2
O3—C7—H7B	109.6	C8—C13—H13B	109.2
C8—C7—H7B	109.6	C12—C13—H13B	109.2
H7A—C7—H7B	108.1	H13A—C13—H13B	107.9
C7—C8—C13	108.5 (3)	O5—C14—O4	122.6 (3)
C7—C8—C9	112.4 (3)	O5—C14—C11	123.5 (3)
C13—C8—C9	111.3 (3)	O4—C14—C11	113.9 (3)
C7—C8—H8	108.2		
O1—S1—O3—C7	166.4 (3)	S1—O3—C7—C8	-132.0 (3)
O2—S1—O3—C7	36.9 (3)	O3—C7—C8—C13	-174.7 (3)
C6—S1—O3—C7	-79.2 (3)	O3—C7—C8—C9	61.8 (4)
C6—C1—C2—C3	-1.2 (5)	C7—C8—C9—C10	175.6 (3)
C1—C2—C3—C4	0.6 (6)	C13—C8—C9—C10	53.7 (4)
C2—C3—C4—C5	0.8 (6)	C8—C9—C10—C11	-55.3 (4)
C3—C4—C5—C6	-1.6 (6)	C9—C10—C11—C14	179.8 (3)
C4—C5—C6—C1	1.0 (5)	C9—C10—C11—C12	55.4 (4)
C4—C5—C6—S1	-175.4 (3)	C14—C11—C12—C13	179.5 (3)
C2—C1—C6—C5	0.4 (5)	C10—C11—C12—C13	-54.7 (4)
C2—C1—C6—S1	176.7 (3)	C7—C8—C13—C12	-176.5 (3)
O1—S1—C6—C5	20.1 (3)	C9—C8—C13—C12	-52.4 (4)
O2—S1—C6—C5	151.9 (3)	C11—C12—C13—C8	53.6 (5)
O3—S1—C6—C5	-91.5 (3)	C12—C11—C14—O5	113.9 (5)
O1—S1—C6—C1	-156.3 (3)	C10—C11—C14—O5	-10.8 (6)
O2—S1—C6—C1	-24.4 (3)	C12—C11—C14—O4	-66.6 (5)
O3—S1—C6—C1	92.1 (3)	C10—C11—C14—O4	168.7 (4)

Hydrogen-bond geometry (Å, °)

<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>
O4—H4 \cdots O5 ⁱ	0.82	1.86	2.677 (3)	174

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