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Benzene-1,2,3-triyl tris(benzenesulfonate)

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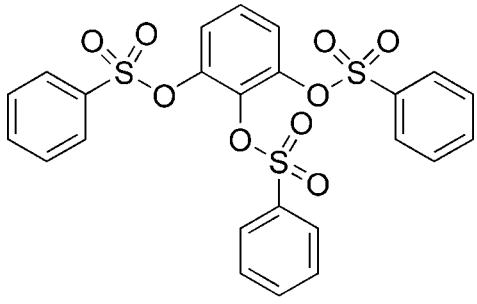
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.044; wR factor = 0.116; data-to-parameter ratio = 7.9.

The title compound, $C_{24}H_{18}O_9S_3$, crystallizes with two molecules per asymmetric unit, with distinctly different conformations, as quantified by the C—O—S—C torsion angles.

Related literature

For background, see: Andersen *et al.* (1982); Linusson *et al.* (2001); Schuda & Price (1987). For reference structural data, see: Allen *et al.* (1995).



Experimental

Crystal data

$C_{24}H_{18}O_9S_3$
M_r = 546.57
Monoclinic, $P2_1$
a = 8.8100 (18) Å
b = 31.318 (6) Å
c = 9.0460 (18) Å
β = 103.42 (3)°
V = 2427.7 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 298 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.932, T_{max} = 0.965
5130 measured reflections
4818 independent reflections
3932 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 1.08
4818 reflections
650 parameters
49 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 156 Friedel pairs
Flack parameter: 0.02 (9)

Table 1
Selected torsion angles (°).

C1—O1—S1—C7	64.6 (4)	C25—O10—S4—C31	−57.5 (5)
C2—O2—S2—C13	127.8 (4)	C30—O11—S5—C37	−128.0 (4)
C3—O3—S3—C19	−76.5 (4)	C29—O12—S6—C43	−66.2 (4)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2522).

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supplementary materials

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Benzene-1,2,3-triyl tris(benzenesulfonate)

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Comment

Benzenesulfonates are important protecting groups (Schuda & Price, 1987) and intermediates (Andersen *et al.*, 1982) in organic synthesis. Benzenesulfonate itself shows some biological acitivities (Linusson *et al.*, 2001). We new report the structure of the title compound, (I), which crystallizes with two molecules in the asymmetric unit. The bond distances and angles in both molecules are normal (Allen *et al.*, 1995)

In the C1 molecule, the three pendant benzene rings and the central benzene ring form a cage-like arrangement (Fig. 1). In the second molecule, the cage is opened because of one benzene ring (C31—C36) turned to the opposite direction. This is reflected in the different C—O—S—C torsion angles for the two molecules (Table 1).

Experimental

Benzenesulfonyl chloride (23 ml, 180 mmol) was added dropwise to a solution of pyrogallol (6.3 g, 50 mmol) in Et_3N (80 ml) and CH_2Cl_2 (100 ml) at 273 K with stirring. After one hour, the residue was filtered, washed with petrol ether (20 ml × 3) and water (20 ml × 3) to yield the pale yellow title compound in a yield of 90%. Colorless blocks of (I) were grown by diffusion of petroleum ether into an AcOEt solution.

Refinement

All H atoms were placed in geometrically idealized positions (C—H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

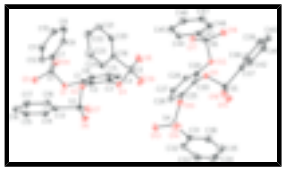


Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level (H atoms omitted for clarity).

Benzene-1,2,3-triyl tris(benzenesulfonate)

Crystal data

$C_{24}H_{18}O_9S_3$	$F_{000} = 1128$
$M_r = 546.57$	$D_x = 1.495$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

Hall symbol: P 2yb	Cell parameters from 3107 reflections
a = 8.8100 (18) Å	θ = 1.1–25.4º
b = 31.318 (6) Å	μ = 0.36 mm^{-1}
c = 9.0460 (18) Å	T = 298 (2) K
β = 103.42 (3)º	Block, colorless
V = 2427.7 (8) $Å^3$	0.20 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	4818 independent reflections
Radiation source: fine-focus sealed tube	3932 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 293(2) K	θ_{max} = 26.0º
ω scans	θ_{min} = 1.3º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = 0→10
T_{min} = 0.932, T_{max} = 0.965	k = 0→38
5130 measured reflections	l = −11→10

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.956P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.115	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.09	$\Delta\rho_{max}$ = 0.26 e $Å^{-3}$
4818 reflections	$\Delta\rho_{min}$ = −0.31 e $Å^{-3}$
650 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
49 restraints	Extinction coefficient: 0.0127 (10)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 156 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.02 (9)

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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}
C1	0.2796 (6)	0.97922 (17)	0.2690 (5)	0.0422 (13)
C2	0.2819 (5)	0.96220 (15)	0.4103 (5)	0.0386 (11)
C3	0.3283 (6)	0.92003 (16)	0.4382 (6)	0.0449 (13)
C4	0.3653 (7)	0.8952 (2)	0.3245 (6)	0.0550 (15)
H4	0.3934	0.8667	0.3426	0.066*
C5	0.3597 (7)	0.91325 (19)	0.1846 (7)	0.0594 (17)
H5	0.3837	0.8966	0.1079	0.071*
C6	0.3194 (7)	0.95559 (19)	0.1555 (6)	0.0526 (15)
H6	0.3192	0.9678	0.0617	0.063*
C7	−0.0573 (6)	1.01924 (16)	0.2242 (6)	0.0416 (12)
C8	−0.1279 (7)	0.98070 (17)	0.1759 (7)	0.0527 (15)
H8	−0.0956	0.9645	0.1026	0.063*
C9	−0.2480 (7)	0.9667 (2)	0.2390 (8)	0.0657 (18)
H9	−0.2992	0.9412	0.2069	0.079*
C10	−0.2912 (7)	0.9909 (2)	0.3499 (8)	0.0621 (17)
H10	−0.3720	0.9815	0.3919	0.075*
C11	−0.2178 (7)	1.02861 (19)	0.3995 (7)	0.0557 (16)
H11	−0.2478	1.0443	0.4752	0.067*
C12	−0.0996 (7)	1.04307 (18)	0.3366 (6)	0.0502 (14)
H12	−0.0487	1.0686	0.3694	0.060*
C13	0.2727 (6)	1.06295 (17)	0.6259 (5)	0.0408 (12)
C14	0.3127 (7)	1.09213 (17)	0.5274 (7)	0.0550 (15)
H14	0.3865	1.0853	0.4724	0.066*
C15	0.2417 (8)	1.1315 (2)	0.5118 (8)	0.072 (2)
H15	0.2659	1.1513	0.4445	0.086*
C16	0.1348 (8)	1.1414 (2)	0.5956 (8)	0.075 (2)
H16	0.0869	1.1681	0.5840	0.090*
C17	0.0975 (8)	1.1125 (2)	0.6964 (8)	0.073 (2)
H17	0.0262	1.1198	0.7535	0.087*
C18	0.1669 (7)	1.07252 (19)	0.7122 (7)	0.0568 (16)
H18	0.1427	1.0526	0.7795	0.068*
C19	0.0667 (6)	0.89228 (17)	0.6361 (6)	0.0474 (13)
C20	−0.0660 (6)	0.87825 (19)	0.5367 (7)	0.0556 (15)
H20	−0.0606	0.8578	0.4635	0.067*
C21	−0.2081 (7)	0.8951 (2)	0.5475 (8)	0.0647 (17)
H21	−0.2992	0.8859	0.4808	0.078*
C22	−0.2156 (7)	0.9250 (2)	0.6556 (7)	0.0684 (18)
H22	−0.3121	0.9352	0.6648	0.082*
C23	−0.0816 (7)	0.9401 (2)	0.7509 (7)	0.072 (2)
H23	−0.0872	0.9616	0.8203	0.086*
C24	0.0621 (7)	0.9233 (2)	0.7433 (7)	0.0605 (17)
H24	0.1534	0.9327	0.8091	0.073*

C25	0.5994 (6)	0.78725 (16)	0.8570 (5)	0.0423 (13)
C26	0.6434 (7)	0.80936 (18)	0.7417 (6)	0.0497 (14)
H26	0.6764	0.8376	0.7563	0.060*
C27	0.6383 (7)	0.78949 (18)	0.6056 (6)	0.0535 (15)
H27	0.6686	0.8046	0.5287	0.064*
C28	0.5895 (7)	0.74777 (17)	0.5794 (6)	0.0472 (13)
H28	0.5875	0.7345	0.4870	0.057*
C29	0.5436 (6)	0.72644 (16)	0.6953 (5)	0.0390 (12)
C30	0.5464 (6)	0.74536 (15)	0.8328 (5)	0.0364 (11)
C31	0.8991 (6)	0.81516 (17)	1.1089 (6)	0.0512 (14)
C32	1.0032 (8)	0.8323 (2)	1.0314 (8)	0.0697 (19)
H32	0.9755	0.8556	0.9670	0.084*
C33	1.1494 (8)	0.8140 (3)	1.0523 (10)	0.086 (3)
H33	1.2217	0.8253	1.0029	0.104*
C34	1.1887 (9)	0.7792 (3)	1.1452 (9)	0.088 (3)
H34	1.2871	0.7670	1.1578	0.106*
C35	1.0832 (8)	0.7622 (3)	1.2198 (8)	0.080 (2)
H35	1.1104	0.7385	1.2824	0.096*
C36	0.9368 (7)	0.78024 (19)	1.2019 (7)	0.0601 (16)
H36	0.8650	0.7689	1.2521	0.072*
C37	0.5128 (6)	0.64779 (17)	1.0596 (5)	0.0427 (12)
C38	0.4055 (7)	0.6402 (2)	1.1456 (7)	0.0577 (16)
H38	0.3805	0.6613	1.2084	0.069*
C39	0.3362 (9)	0.6005 (2)	1.1361 (9)	0.080 (2)
H39	0.2634	0.5947	1.1932	0.096*
C40	0.3736 (9)	0.5696 (2)	1.0433 (9)	0.082 (2)
H40	0.3257	0.5430	1.0377	0.098*
C41	0.4813 (9)	0.5776 (2)	0.9586 (8)	0.075 (2)
H41	0.5063	0.5565	0.8960	0.090*
C42	0.5522 (7)	0.61703 (18)	0.9665 (7)	0.0571 (16)
H42	0.6255	0.6227	0.9098	0.068*
C43	0.1977 (6)	0.69222 (15)	0.6702 (5)	0.0394 (12)
C44	0.1502 (6)	0.73387 (16)	0.6415 (6)	0.0488 (14)
H44	0.1898	0.7505	0.5741	0.059*
C45	0.0424 (7)	0.7504 (2)	0.7151 (7)	0.0598 (17)
H45	0.0082	0.7784	0.6965	0.072*
C46	−0.0147 (7)	0.7257 (2)	0.8159 (7)	0.0609 (17)
H46	−0.0878	0.7370	0.8646	0.073*
C47	0.0361 (7)	0.6843 (2)	0.8446 (7)	0.0588 (17)
H47	−0.0024	0.6679	0.9132	0.071*
C48	0.1439 (7)	0.66703 (19)	0.7725 (6)	0.0507 (14)
H48	0.1793	0.6392	0.7924	0.061*
O1	0.2479 (4)	1.02316 (13)	0.2505 (4)	0.0481 (10)
O2	0.2327 (4)	0.98597 (12)	0.5206 (4)	0.0436 (9)
O3	0.3503 (4)	0.90587 (14)	0.5870 (5)	0.0548 (10)
O4	0.0723 (5)	1.01793 (18)	−0.0037 (5)	0.0674 (12)
O5	0.0995 (6)	1.08356 (15)	0.1488 (6)	0.0724 (13)
O6	0.5018 (4)	1.01278 (16)	0.5969 (5)	0.0618 (12)
O7	0.3547 (6)	0.99497 (14)	0.7888 (4)	0.0620 (12)

O8	0.2226 (6)	0.83636 (16)	0.5216 (7)	0.0864 (16)
O9	0.3271 (6)	0.8596 (2)	0.7861 (6)	0.0960 (19)
O10	0.5939 (5)	0.80350 (13)	0.9990 (4)	0.0519 (10)
O11	0.4876 (4)	0.72388 (13)	0.9426 (4)	0.0423 (9)
O12	0.4998 (4)	0.68312 (12)	0.6795 (4)	0.0432 (9)
O13	0.7046 (6)	0.87594 (15)	0.9994 (6)	0.0819 (15)
O14	0.6723 (6)	0.84022 (18)	1.2304 (5)	0.0802 (15)
O15	0.7514 (4)	0.69465 (15)	1.0327 (5)	0.0612 (12)
O16	0.5972 (6)	0.71699 (15)	1.2124 (4)	0.0651 (12)
O17	0.3137 (5)	0.69192 (15)	0.4325 (4)	0.0594 (11)
O18	0.3319 (5)	0.62518 (13)	0.5817 (5)	0.0642 (12)
S1	0.08707 (18)	1.03852 (5)	0.13813 (17)	0.0502 (4)
S2	0.35990 (15)	1.01224 (5)	0.64660 (15)	0.0432 (3)
S3	0.2467 (2)	0.86794 (5)	0.6354 (2)	0.0633 (4)
S4	0.71387 (19)	0.83832 (5)	1.08912 (19)	0.0580 (4)
S5	0.60581 (15)	0.69739 (5)	1.07434 (14)	0.0426 (3)
S6	0.33127 (17)	0.67046 (5)	0.57364 (16)	0.0454 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.029 (3)	0.048 (3)	0.050 (3)	−0.006 (2)	0.012 (2)	−0.004 (3)
C2	0.027 (2)	0.042 (3)	0.048 (3)	−0.006 (2)	0.013 (2)	−0.008 (2)
C3	0.032 (3)	0.047 (3)	0.054 (3)	−0.005 (2)	0.008 (2)	−0.005 (3)
C4	0.044 (3)	0.052 (4)	0.068 (4)	0.003 (3)	0.011 (3)	−0.010 (3)
C5	0.049 (3)	0.066 (4)	0.066 (4)	0.003 (3)	0.017 (3)	−0.025 (3)
C6	0.042 (3)	0.073 (4)	0.045 (3)	−0.001 (3)	0.014 (2)	−0.006 (3)
C7	0.036 (3)	0.045 (3)	0.044 (3)	−0.001 (2)	0.011 (2)	0.007 (2)
C8	0.057 (4)	0.045 (3)	0.061 (4)	−0.003 (3)	0.025 (3)	−0.010 (3)
C9	0.060 (4)	0.055 (4)	0.091 (5)	−0.010 (3)	0.034 (4)	−0.006 (4)
C10	0.047 (4)	0.064 (4)	0.085 (5)	−0.003 (3)	0.033 (3)	0.007 (4)
C11	0.053 (4)	0.058 (4)	0.061 (4)	0.012 (3)	0.023 (3)	0.004 (3)
C12	0.052 (3)	0.043 (3)	0.055 (3)	0.000 (3)	0.012 (3)	−0.003 (3)
C13	0.036 (3)	0.045 (3)	0.040 (3)	−0.006 (2)	0.008 (2)	−0.006 (2)
C14	0.056 (4)	0.048 (4)	0.062 (4)	−0.013 (3)	0.018 (3)	−0.001 (3)
C15	0.073 (5)	0.054 (4)	0.079 (5)	−0.016 (4)	0.000 (4)	0.008 (4)
C16	0.067 (5)	0.056 (4)	0.086 (5)	0.006 (4)	−0.017 (4)	−0.011 (4)
C17	0.061 (4)	0.084 (6)	0.073 (5)	0.021 (4)	0.016 (4)	−0.019 (4)
C18	0.062 (4)	0.062 (4)	0.051 (3)	0.009 (3)	0.022 (3)	−0.001 (3)
C19	0.041 (3)	0.048 (3)	0.050 (3)	−0.003 (3)	0.006 (2)	0.008 (3)
C20	0.063 (4)	0.045 (3)	0.054 (3)	−0.004 (3)	0.004 (3)	−0.003 (3)
C21	0.049 (4)	0.059 (4)	0.079 (4)	−0.004 (3)	0.000 (3)	−0.001 (3)
C22	0.052 (4)	0.076 (5)	0.079 (5)	0.003 (4)	0.019 (3)	0.009 (4)
C23	0.090 (5)	0.074 (5)	0.060 (4)	0.002 (4)	0.033 (4)	−0.013 (4)
C24	0.056 (4)	0.072 (4)	0.051 (3)	−0.011 (3)	0.006 (3)	−0.006 (3)
C25	0.035 (3)	0.053 (4)	0.037 (3)	0.007 (2)	0.004 (2)	−0.006 (2)
C26	0.054 (3)	0.035 (3)	0.059 (4)	−0.002 (3)	0.011 (3)	0.003 (3)
C27	0.054 (3)	0.064 (4)	0.046 (3)	0.000 (3)	0.018 (3)	0.016 (3)

C28	0.050 (3)	0.053 (4)	0.039 (3)	−0.001 (3)	0.013 (2)	−0.002 (2)
C29	0.038 (3)	0.039 (3)	0.041 (3)	0.003 (2)	0.011 (2)	−0.002 (2)
C30	0.031 (3)	0.042 (3)	0.036 (3)	0.010 (2)	0.007 (2)	0.007 (2)
C31	0.049 (3)	0.050 (4)	0.051 (3)	0.000 (3)	0.005 (3)	−0.012 (3)
C32	0.077 (5)	0.060 (4)	0.075 (5)	−0.018 (4)	0.024 (4)	−0.007 (4)
C33	0.063 (5)	0.100 (7)	0.103 (6)	−0.022 (5)	0.035 (4)	−0.032 (5)
C34	0.053 (4)	0.108 (7)	0.095 (6)	0.003 (5)	0.002 (4)	−0.032 (5)
C35	0.082 (5)	0.078 (5)	0.067 (4)	0.021 (4)	−0.007 (4)	−0.002 (4)
C36	0.060 (4)	0.065 (4)	0.053 (3)	0.001 (3)	0.009 (3)	0.004 (3)
C37	0.043 (3)	0.047 (3)	0.037 (3)	0.003 (3)	0.007 (2)	0.004 (2)
C38	0.054 (4)	0.067 (4)	0.057 (4)	−0.007 (3)	0.022 (3)	0.006 (3)
C39	0.063 (4)	0.088 (6)	0.086 (5)	−0.020 (4)	0.011 (4)	0.018 (5)
C40	0.077 (5)	0.047 (4)	0.098 (6)	−0.015 (4)	−0.025 (4)	0.018 (4)
C41	0.090 (6)	0.045 (4)	0.074 (5)	0.010 (4)	−0.014 (4)	−0.011 (3)
C42	0.060 (4)	0.059 (4)	0.050 (3)	0.016 (3)	0.008 (3)	−0.002 (3)
C43	0.037 (3)	0.042 (3)	0.039 (3)	−0.002 (2)	0.007 (2)	−0.008 (2)
C44	0.048 (3)	0.046 (3)	0.055 (3)	0.002 (3)	0.019 (3)	−0.001 (3)
C45	0.054 (4)	0.055 (4)	0.074 (4)	0.010 (3)	0.022 (3)	−0.005 (3)
C46	0.045 (3)	0.072 (5)	0.073 (4)	−0.001 (3)	0.027 (3)	−0.016 (4)
C47	0.051 (4)	0.069 (4)	0.061 (4)	−0.016 (3)	0.024 (3)	0.000 (3)
C48	0.055 (4)	0.049 (3)	0.049 (3)	−0.005 (3)	0.014 (3)	0.001 (3)
O1	0.040 (2)	0.056 (3)	0.049 (2)	−0.0062 (18)	0.0113 (17)	−0.0005 (19)
O2	0.0356 (19)	0.054 (2)	0.0437 (19)	−0.0046 (17)	0.0146 (16)	−0.0118 (17)
O3	0.040 (2)	0.060 (3)	0.062 (3)	0.0002 (19)	0.0061 (18)	0.006 (2)
O4	0.066 (3)	0.097 (4)	0.043 (2)	−0.011 (3)	0.020 (2)	0.010 (2)
O5	0.073 (3)	0.053 (3)	0.095 (3)	−0.006 (2)	0.027 (3)	0.025 (2)
O6	0.033 (2)	0.076 (3)	0.078 (3)	−0.002 (2)	0.0172 (19)	−0.024 (2)
O7	0.083 (3)	0.057 (3)	0.042 (2)	0.000 (2)	0.007 (2)	0.002 (2)
O8	0.088 (4)	0.051 (3)	0.133 (5)	−0.003 (3)	0.050 (3)	−0.017 (3)
O9	0.069 (3)	0.117 (5)	0.100 (4)	0.027 (3)	0.016 (3)	0.061 (4)
O10	0.056 (2)	0.052 (2)	0.047 (2)	0.0027 (19)	0.0097 (18)	−0.0092 (19)
O11	0.038 (2)	0.049 (2)	0.043 (2)	0.0048 (17)	0.0151 (16)	0.0050 (17)
O12	0.041 (2)	0.043 (2)	0.046 (2)	0.0035 (17)	0.0112 (16)	−0.0060 (16)
O13	0.098 (4)	0.045 (3)	0.091 (3)	0.012 (3)	−0.003 (3)	−0.009 (2)
O14	0.076 (3)	0.096 (4)	0.066 (3)	0.011 (3)	0.013 (2)	−0.041 (3)
O15	0.039 (2)	0.079 (3)	0.068 (3)	0.006 (2)	0.0169 (19)	0.021 (2)
O16	0.092 (3)	0.059 (3)	0.042 (2)	−0.013 (2)	0.011 (2)	−0.0043 (19)
O17	0.064 (3)	0.071 (3)	0.044 (2)	0.005 (2)	0.0125 (19)	−0.007 (2)
O18	0.069 (3)	0.041 (2)	0.085 (3)	−0.003 (2)	0.022 (2)	−0.020 (2)
S1	0.0505 (8)	0.0540 (9)	0.0484 (8)	−0.0048 (7)	0.0160 (7)	0.0123 (7)
S2	0.0375 (7)	0.0471 (8)	0.0446 (7)	−0.0002 (6)	0.0091 (6)	−0.0057 (6)
S3	0.0542 (9)	0.0539 (10)	0.0823 (11)	0.0079 (7)	0.0171 (8)	0.0175 (9)
S4	0.0629 (10)	0.0482 (9)	0.0586 (9)	0.0092 (8)	0.0051 (8)	−0.0169 (7)
S5	0.0408 (7)	0.0524 (9)	0.0346 (6)	−0.0024 (6)	0.0087 (5)	0.0025 (6)
S6	0.0471 (8)	0.0456 (8)	0.0444 (7)	0.0016 (6)	0.0125 (6)	−0.0107 (6)

Geometric parameters (Å, °)

C1—C6	1.375 (6)	C29—C30	1.373 (5)

C1—C2	1.381 (5)	C29—O12	1.409 (6)
C1—O1	1.406 (7)	C30—O11	1.395 (6)
C2—C3	1.388 (6)	C31—C36	1.373 (6)
C2—O2	1.392 (6)	C31—C32	1.385 (6)
C3—C4	1.387 (6)	C31—S4	1.757 (6)
C3—O3	1.388 (6)	C32—C33	1.383 (7)
C4—C5	1.377 (6)	C32—H32	0.9300
C4—H4	0.9300	C33—C34	1.370 (7)
C5—C6	1.382 (6)	C33—H33	0.9300
C5—H5	0.9300	C34—C35	1.376 (7)
C6—H6	0.9300	C34—H34	0.9300
C7—C12	1.381 (6)	C35—C36	1.383 (6)
C7—C8	1.382 (6)	C35—H35	0.9300
C7—S1	1.745 (5)	C36—H36	0.9300
C8—C9	1.385 (6)	C37—C42	1.376 (6)
C8—H8	0.9300	C37—C38	1.377 (6)
C9—C10	1.379 (6)	C37—S5	1.747 (6)
C9—H9	0.9300	C38—C39	1.378 (6)
C10—C11	1.370 (6)	C38—H38	0.9300
C10—H10	0.9300	C39—C40	1.372 (7)
C11—C12	1.374 (6)	C39—H39	0.9300
C11—H11	0.9300	C40—C41	1.375 (7)
C12—H12	0.9300	C40—H40	0.9300
C13—C14	1.378 (6)	C41—C42	1.378 (6)
C13—C18	1.381 (6)	C41—H41	0.9300
C13—S2	1.755 (5)	C42—H42	0.9300
C14—C15	1.374 (6)	C43—C44	1.376 (6)
C14—H14	0.9300	C43—C48	1.381 (6)
C15—C16	1.375 (7)	C43—S6	1.757 (5)
C15—H15	0.9300	C44—C45	1.381 (6)
C16—C17	1.377 (7)	C44—H44	0.9300
C16—H16	0.9300	C45—C46	1.377 (6)
C17—C18	1.387 (6)	C45—H45	0.9300
C17—H17	0.9300	C46—C47	1.376 (6)
C18—H18	0.9300	C46—H46	0.9300
C19—C20	1.371 (6)	C47—C48	1.381 (6)
C19—C24	1.380 (6)	C47—H47	0.9300
C19—S3	1.761 (6)	C48—H48	0.9300
C20—C21	1.382 (6)	O1—S1	1.614 (4)
C20—H20	0.9300	O2—S2	1.624 (4)
C21—C22	1.368 (6)	O3—S3	1.619 (4)
C21—H21	0.9300	O4—S1	1.415 (5)
C22—C23	1.374 (6)	O5—S1	1.416 (5)
C22—H22	0.9300	O6—S2	1.423 (4)
C23—C24	1.388 (6)	O7—S2	1.406 (4)
C23—H23	0.9300	O8—S3	1.407 (6)
C24—H24	0.9300	O9—S3	1.408 (5)
C25—C26	1.380 (6)	O10—S4	1.603 (4)
C25—O10	1.392 (6)	O11—S5	1.618 (4)

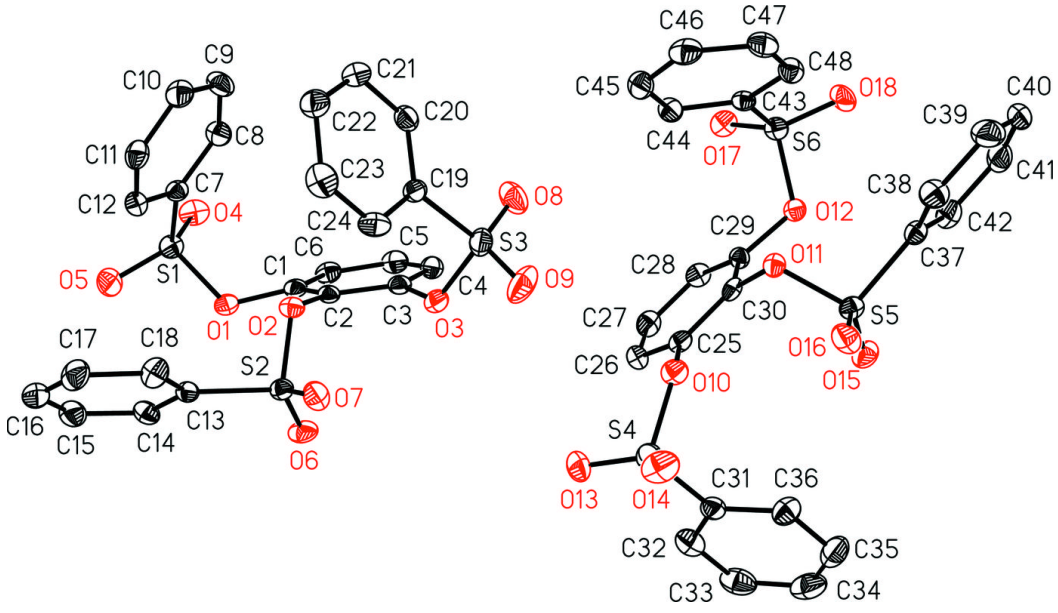
C25—C30	1.393 (6)	O12—S6	1.618 (4)
C26—C27	1.371 (6)	O13—S4	1.422 (5)
C26—H26	0.9300	O14—S4	1.410 (5)
C27—C28	1.379 (6)	O15—S5	1.421 (4)
C27—H27	0.9300	O16—S5	1.409 (4)
C28—C29	1.380 (6)	O17—S6	1.419 (4)
C28—H28	0.9300	O18—S6	1.420 (4)
C6—C1—C2	122.1 (5)	C33—C32—C31	118.3 (7)
C6—C1—O1	121.0 (5)	C33—C32—H32	120.8
C2—C1—O1	116.6 (4)	C31—C32—H32	120.8
C1—C2—C3	118.4 (5)	C34—C33—C32	120.6 (7)
C1—C2—O2	121.1 (4)	C34—C33—H33	119.7
C3—C2—O2	120.4 (4)	C32—C33—H33	119.7
C4—C3—O3	122.4 (5)	C33—C34—C35	120.3 (7)
C4—C3—C2	120.6 (5)	C33—C34—H34	119.8
O3—C3—C2	116.7 (4)	C35—C34—H34	119.8
C5—C4—C3	119.2 (6)	C34—C35—C36	120.2 (7)
C5—C4—H4	120.4	C34—C35—H35	119.9
C3—C4—H4	120.4	C36—C35—H35	119.9
C4—C5—C6	121.4 (5)	C31—C36—C35	118.9 (6)
C4—C5—H5	119.3	C31—C36—H36	120.5
C6—C5—H5	119.3	C35—C36—H36	120.5
C1—C6—C5	118.2 (5)	C42—C37—C38	121.9 (6)
C1—C6—H6	120.9	C42—C37—S5	119.3 (4)
C5—C6—H6	120.9	C38—C37—S5	118.8 (4)
C12—C7—C8	121.8 (5)	C37—C38—C39	118.2 (6)
C12—C7—S1	119.4 (4)	C37—C38—H38	120.9
C8—C7—S1	118.9 (4)	C39—C38—H38	120.9
C7—C8—C9	118.4 (5)	C40—C39—C38	120.6 (7)
C7—C8—H8	120.8	C40—C39—H39	119.7
C9—C8—H8	120.8	C38—C39—H39	119.7
C10—C9—C8	119.5 (6)	C39—C40—C41	120.5 (7)
C10—C9—H9	120.2	C39—C40—H40	119.8
C8—C9—H9	120.2	C41—C40—H40	119.8
C11—C10—C9	121.5 (6)	C40—C41—C42	119.9 (6)
C11—C10—H10	119.3	C40—C41—H41	120.1
C9—C10—H10	119.3	C42—C41—H41	120.1
C10—C11—C12	119.6 (6)	C37—C42—C41	118.9 (6)
C10—C11—H11	120.2	C37—C42—H42	120.6
C12—C11—H11	120.2	C41—C42—H42	120.6
C11—C12—C7	119.1 (5)	C44—C43—C48	122.1 (5)
C11—C12—H12	120.4	C44—C43—S6	118.9 (4)
C7—C12—H12	120.4	C48—C43—S6	119.0 (4)
C14—C13—C18	121.9 (5)	C43—C44—C45	118.6 (5)
C14—C13—S2	119.9 (4)	C43—C44—H44	120.7
C18—C13—S2	118.2 (4)	C45—C44—H44	120.7
C15—C14—C13	118.9 (6)	C46—C45—C44	120.4 (6)
C15—C14—H14	120.6	C46—C45—H45	119.8
C13—C14—H14	120.6	C44—C45—H45	119.8

C14—C15—C16	120.0 (6)	C47—C46—C45	120.1 (6)
C14—C15—H15	120.0	C47—C46—H46	120.0
C16—C15—H15	120.0	C45—C46—H46	120.0
C15—C16—C17	121.0 (7)	C46—C47—C48	120.7 (6)
C15—C16—H16	119.5	C46—C47—H47	119.7
C17—C16—H16	119.5	C48—C47—H47	119.7
C16—C17—C18	119.7 (6)	C47—C48—C43	118.2 (5)
C16—C17—H17	120.2	C47—C48—H48	120.9
C18—C17—H17	120.2	C43—C48—H48	120.9
C13—C18—C17	118.5 (6)	C1—O1—S1	119.2 (3)
C13—C18—H18	120.8	C2—O2—S2	119.7 (3)
C17—C18—H18	120.8	C3—O3—S3	122.4 (3)
C20—C19—C24	121.8 (5)	C25—O10—S4	123.9 (4)
C20—C19—S3	119.4 (4)	C30—O11—S5	119.4 (3)
C24—C19—S3	118.7 (4)	C29—O12—S6	119.2 (3)
C19—C20—C21	118.7 (5)	O4—S1—O5	120.3 (3)
C19—C20—H20	120.7	O4—S1—O1	108.5 (3)
C21—C20—H20	120.7	O5—S1—O1	102.2 (3)
C22—C21—C20	120.4 (6)	O4—S1—C7	108.9 (3)
C22—C21—H21	119.8	O5—S1—C7	111.5 (3)
C20—C21—H21	119.8	O1—S1—C7	103.9 (2)
C21—C22—C23	120.5 (6)	O7—S2—O6	120.7 (3)
C21—C22—H22	119.7	O7—S2—O2	106.4 (2)
C23—C22—H22	119.7	O6—S2—O2	107.3 (2)
C22—C23—C24	119.9 (6)	O7—S2—C13	109.8 (3)
C22—C23—H23	120.0	O6—S2—C13	110.5 (3)
C24—C23—H23	120.0	O2—S2—C13	99.8 (2)
C19—C24—C23	118.6 (6)	O8—S3—O9	122.3 (4)
C19—C24—H24	120.7	O8—S3—O3	108.5 (3)
C23—C24—H24	120.7	O9—S3—O3	102.1 (3)
C26—C25—O10	126.1 (5)	O8—S3—C19	108.9 (3)
C26—C25—C30	119.8 (5)	O9—S3—C19	109.1 (3)
O10—C25—C30	114.0 (4)	O3—S3—C19	104.4 (2)
C27—C26—C25	119.6 (5)	O14—S4—O13	119.3 (3)
C27—C26—H26	120.2	O14—S4—O10	102.1 (3)
C25—C26—H26	120.2	O13—S4—O10	109.2 (3)
C26—C27—C28	121.9 (5)	O14—S4—C31	110.9 (3)
C26—C27—H27	119.0	O13—S4—C31	109.2 (3)
C28—C27—H27	119.0	O10—S4—C31	105.0 (2)
C27—C28—C29	117.5 (5)	O16—S5—O15	119.9 (3)
C27—C28—H28	121.2	O16—S5—O11	105.8 (3)
C29—C28—H28	121.2	O15—S5—O11	107.6 (2)
C30—C29—C28	122.2 (5)	O16—S5—C37	109.5 (2)
C30—C29—O12	117.0 (4)	O15—S5—C37	111.2 (3)
C28—C29—O12	120.7 (4)	O11—S5—C37	100.9 (2)
C29—C30—C25	118.9 (5)	O17—S6—O18	121.2 (3)
C29—C30—O11	120.4 (4)	O17—S6—O12	108.0 (2)
C25—C30—O11	120.6 (4)	O18—S6—O12	102.8 (3)
C36—C31—C32	121.7 (6)	O17—S6—C43	108.5 (2)

C36—C31—S4	118.5 (4)	O18—S6—C43	110.9 (3)
C32—C31—S4	119.8 (5)	O12—S6—C43	103.9 (2)
C6—C1—C2—C3	1.0 (7)	C44—C45—C46—C47	0.4 (10)
O1—C1—C2—C3	−173.5 (4)	C45—C46—C47—C48	−0.4 (10)
C6—C1—C2—O2	−176.4 (5)	C46—C47—C48—C43	−0.6 (9)
O1—C1—C2—O2	9.2 (7)	C44—C43—C48—C47	1.6 (8)
C1—C2—C3—C4	−2.6 (7)	S6—C43—C48—C47	−178.5 (5)
O2—C2—C3—C4	174.8 (5)	C6—C1—O1—S1	72.7 (6)
C1—C2—C3—O3	171.2 (4)	C2—C1—O1—S1	−112.8 (4)
O2—C2—C3—O3	−11.5 (7)	C1—C2—O2—S2	−94.4 (5)
O3—C3—C4—C5	−171.5 (5)	C3—C2—O2—S2	88.3 (5)
C2—C3—C4—C5	1.9 (8)	C4—C3—O3—S3	−69.1 (6)
C3—C4—C5—C6	0.5 (9)	C2—C3—O3—S3	117.3 (5)
C2—C1—C6—C5	1.3 (8)	C26—C25—O10—S4	−34.4 (7)
O1—C1—C6—C5	175.5 (5)	C30—C25—O10—S4	149.2 (4)
C4—C5—C6—C1	−2.1 (9)	C29—C30—O11—S5	95.4 (5)
C12—C7—C8—C9	2.4 (9)	C25—C30—O11—S5	−88.7 (5)
S1—C7—C8—C9	−176.6 (5)	C30—C29—O12—S6	108.0 (5)
C7—C8—C9—C10	−1.5 (10)	C28—C29—O12—S6	−75.3 (6)
C8—C9—C10—C11	−0.1 (11)	C1—O1—S1—O4	−51.2 (4)
C9—C10—C11—C12	0.8 (11)	C1—O1—S1—O5	−179.3 (4)
C10—C11—C12—C7	0.1 (9)	C1—O1—S1—C7	64.6 (4)
C8—C7—C12—C11	−1.7 (9)	C12—C7—S1—O4	−159.5 (5)
S1—C7—C12—C11	177.3 (5)	C8—C7—S1—O4	19.6 (6)
C18—C13—C14—C15	−1.8 (9)	C12—C7—S1—O5	−24.3 (6)
S2—C13—C14—C15	179.3 (5)	C8—C7—S1—O5	154.7 (5)
C13—C14—C15—C16	1.1 (10)	C12—C7—S1—O1	85.0 (5)
C14—C15—C16—C17	0.3 (11)	C8—C7—S1—O1	−96.0 (5)
C15—C16—C17—C18	−1.0 (11)	C2—O2—S2—O7	−118.0 (4)
C14—C13—C18—C17	1.1 (9)	C2—O2—S2—O6	12.6 (4)
S2—C13—C18—C17	−180.0 (5)	C2—O2—S2—C13	127.8 (4)
C16—C17—C18—C13	0.3 (10)	C14—C13—S2—O7	156.0 (5)
C24—C19—C20—C21	−1.6 (9)	C18—C13—S2—O7	−22.9 (5)
S3—C19—C20—C21	174.4 (5)	C14—C13—S2—O6	20.4 (5)
C19—C20—C21—C22	−0.2 (10)	C18—C13—S2—O6	−158.5 (4)
C20—C21—C22—C23	2.7 (11)	C14—C13—S2—O2	−92.4 (5)
C21—C22—C23—C24	−3.6 (11)	C18—C13—S2—O2	88.7 (5)
C20—C19—C24—C23	0.8 (10)	C3—O3—S3—O8	39.5 (5)
S3—C19—C24—C23	−175.3 (5)	C3—O3—S3—O9	169.9 (4)
C22—C23—C24—C19	1.8 (10)	C3—O3—S3—C19	−76.5 (4)
O10—C25—C26—C27	−177.9 (5)	C20—C19—S3—O8	1.4 (6)
C30—C25—C26—C27	−1.7 (8)	C24—C19—S3—O8	177.6 (5)
C25—C26—C27—C28	0.3 (9)	C20—C19—S3—O9	−134.4 (5)
C26—C27—C28—C29	0.7 (9)	C24—C19—S3—O9	41.8 (6)
C27—C28—C29—C30	−0.3 (8)	C20—C19—S3—O3	117.1 (5)
C27—C28—C29—O12	−176.8 (5)	C24—C19—S3—O3	−66.7 (5)
C28—C29—C30—C25	−1.0 (8)	C25—O10—S4—O14	−173.2 (4)
O12—C29—C30—C25	175.6 (4)	C25—O10—S4—O13	59.5 (5)
C28—C29—C30—O11	174.9 (5)	C25—O10—S4—C31	−57.5 (5)

O12—C29—C30—O11	−8.5 (7)	C36—C31—S4—O14	41.4 (6)
C26—C25—C30—C29	2.0 (8)	C32—C31—S4—O14	−138.4 (5)
O10—C25—C30—C29	178.7 (4)	C36—C31—S4—O13	174.9 (5)
C26—C25—C30—O11	−173.9 (5)	C32—C31—S4—O13	−5.0 (6)
O10—C25—C30—O11	2.7 (7)	C36—C31—S4—O10	−68.1 (5)
C36—C31—C32—C33	−1.4 (10)	C32—C31—S4—O10	112.0 (5)
S4—C31—C32—C33	178.5 (5)	C30—O11—S5—O16	117.9 (4)
C31—C32—C33—C34	1.2 (11)	C30—O11—S5—O15	−11.3 (4)
C32—C33—C34—C35	−0.4 (12)	C30—O11—S5—C37	−128.0 (4)
C33—C34—C35—C36	−0.2 (12)	C42—C37—S5—O16	−157.0 (5)
C32—C31—C36—C35	0.8 (9)	C38—C37—S5—O16	20.9 (5)
S4—C31—C36—C35	−179.1 (5)	C42—C37—S5—O15	−22.1 (5)
C34—C35—C36—C31	0.0 (11)	C38—C37—S5—O15	155.8 (4)
C42—C37—C38—C39	−0.3 (9)	C42—C37—S5—O11	91.7 (5)
S5—C37—C38—C39	−178.2 (5)	C38—C37—S5—O11	−90.3 (5)
C37—C38—C39—C40	0.0 (11)	C29—O12—S6—O17	48.9 (4)
C38—C39—C40—C41	0.2 (12)	C29—O12—S6—O18	178.1 (3)
C39—C40—C41—C42	−0.1 (11)	C29—O12—S6—C43	−66.2 (4)
C38—C37—C42—C41	0.4 (9)	C44—C43—S6—O17	−27.4 (5)
S5—C37—C42—C41	178.3 (5)	C48—C43—S6—O17	152.7 (4)
C40—C41—C42—C37	−0.2 (10)	C44—C43—S6—O18	−162.8 (4)
C48—C43—C44—C45	−1.6 (9)	C48—C43—S6—O18	17.3 (5)
S6—C43—C44—C45	178.5 (5)	C44—C43—S6—O12	87.4 (5)
C43—C44—C45—C46	0.6 (10)	C48—C43—S6—O12	−92.5 (5)

Fig. 1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