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2,10,12-Trimethyl-12*H*-5,7-dioxa-6-thiadibenzo[*a*,*d*]cyclooctene 6-oxide

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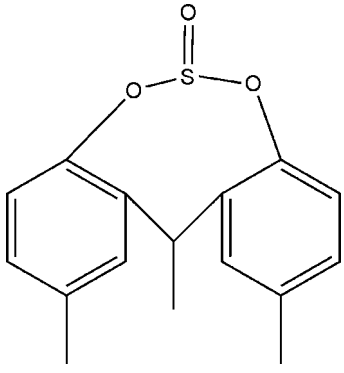
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.054; wR factor = 0.116; data-to-parameter ratio = 8.0.

In the two independent molecules of the title compound, $C_{16}H_{16}O_3S$, the dihedral angles between the benzene rings are 79.06 (2) and 78.46 (2)°. The SO_2C_5 rings are not planar and have boat–chair conformations.

Related literature

For general background, see: Pastor *et al.* (1983); Allen *et al.* (1987); Cremer & Pople (1975).



Experimental

Crystal data

$C_{16}H_{16}O_3S$
M_r = 288.36
Orthorhombic, $Pca2_1$
a = 14.122 (3) Å
b = 8.0880 (16) Å
c = 25.343 (5) Å
V = 2894.6 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.915, T_{max} = 0.958
3213 measured reflections
2890 independent reflections
1444 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.116
S = 1.00
2890 reflections
361 parameters
202 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 2708 Friedel pairs
Flack parameter: −0.06 (17)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2239).

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supplementary materials

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2,10,12-Trimethyl-12*H*-5,7-dioxa-6-thiadibenzo[*a*,*d*]cyclooctene 6-oxide

S.-L. Dong, B. Xu, Z.-Q. Feng and J.-T. Wang

Comment

In a research for novel antioxidants, a series of medium-ring heterocycles derived from sterically hindered phenols has been prepared and investigated (Pastor *et al.*, 1983). We herein report the crystal structure of the title compound, (I).

The asymmetric unit of (I) contains two independent molecules, essentially in the same geometry (Fig. 1). The bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987).

Rings A(C2—C7), B(C9—C14), D(C18—C23) and E(C25—C30) are, of course, planar and the dihedral angles between them are A/B = 79.06 (2)° and D/E=78.46 (2)°. Rings C (S1/O2/O3/C4/C5/C12/C13/C15) and F (S2/O5/O6/C21/C22/C27/C28/C31) are not planar, having total puckering amplitudes, Q_T, of 1.057 (3) and 1.037 (3) Å, respectively, and boat-chair conformations (Cremer & Pople, 1975).

Experimental

2,2'-ethylidenebis(4-methylPhenol)(1.21 g, 5 mmol) and triethylamine (11 mmol, 1.52 ml) and methylene chloride(AR) (100 ml) were added into the four-neck round-bottom flask fitted with a mechanical stirrer, dropping funnel, thermometer, and reflux condenser. The system was put in an ice-water bath and stirred for 30 min. and then thionyl chloride (5 mmol, 0.36 ml) which was dissolved in methylene chloride(AR) (50 ml) was added and stirred. The reaction was kept at 273 K in an ice-water bath for 24 h. The mixture was washed with hydrogen chloride solution (5%, 100 ml) and saturated sodium hydrogen carbonate (7.4%, 100 ml) and distilled water (200 ml). The solvent was distilled under reduced pressure. The residue was exsiccated in a desiccator. The product was purified by repeated crystallization. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of isopropyl alcohol(AR) (10 ml) (yield; 1.04 g, 72%, m.p. 401 K).

Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.98 and 0.96 Å for aromatic, methine and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

Figures

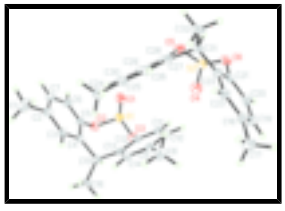


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 15% probability level.

2,10,12-Trimethyl-12*H*-5,7-dioxa-6-thiadibenzo[a,d]cyclooctene 6-oxide

Crystal data

$C_{16}H_{16}O_3S$	D_x = 1.323 Mg m^{-3}
M_r = 288.36	Melting point: 401 K
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 25 reflections
a = 14.122 (3) Å	θ = 10–13º
b = 8.0880 (16) Å	μ = 0.23 mm^{-1}
c = 25.343 (5) Å	*T* = 298 (2) K
V = 2894.6 (10) Å^3	Block, colorless
Z = 8	0.30 × 0.20 × 0.10 mm
F_{000} = 1216	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.021
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.6º
T = 298(2) K	*h* = 0→17
ω/2θ scans	*k* = 0→9
Absorption correction: ψ scan (North *et al.*, 1968)	*l* = −31→0
T_{min} = 0.915, T_{max} = 0.958	3 standard reflections
3213 measured reflections	every 200 reflections
2890 independent reflections	intensity decay: none
1444 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$w = 1/[\sigma^2(F_o^2) + (0.040P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.116	$(\Delta/\sigma)_{max} < 0.001$
S = 1.00	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
2890 reflections	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
361 parameters	Extinction correction: none
202 restraints	Absolute structure: Flack (1983), 208 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.06 (17)
Secondary atom site location: difference Fourier map	

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 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}
S1	0.21883 (16)	0.7088 (3)	0.74303 (10)	0.0973 (7)
O1	0.1300 (4)	0.7391 (6)	0.7223 (2)	0.1098 (19)
O2	0.2261 (3)	0.8052 (6)	0.7991 (2)	0.0862 (16)
O3	0.2965 (3)	0.8038 (6)	0.7086 (2)	0.0833 (15)
C1	0.0108 (6)	1.3898 (10)	0.8340 (3)	0.116 (3)
H1B	−0.0482	1.3606	0.8503	0.175*
H1C	−0.0013	1.4450	0.8011	0.175*
H1D	0.0456	1.4622	0.8570	0.175*
C2	0.0693 (5)	1.2320 (9)	0.8237 (3)	0.0788 (19)
C3	0.1592 (4)	1.2418 (8)	0.8007 (3)	0.0655 (18)
H3A	0.1826	1.3447	0.7906	0.079*
C4	0.2138 (5)	1.1037 (9)	0.7925 (3)	0.0617 (18)
C5	0.1728 (5)	0.9516 (10)	0.8060 (3)	0.0694 (19)
C6	0.0842 (5)	0.9431 (10)	0.8286 (3)	0.083 (2)
H6A	0.0590	0.8416	0.8385	0.099*
C7	0.0340 (5)	1.0837 (10)	0.8362 (3)	0.084 (2)
H7A	−0.0266	1.0770	0.8505	0.101*
C8	0.2456 (6)	1.3871 (9)	0.5856 (3)	0.102 (2)
H8A	0.2534	1.4839	0.6071	0.152*
H8B	0.1839	1.3883	0.5697	0.152*
H8C	0.2931	1.3861	0.5584	0.152*
C9	0.2561 (4)	1.2314 (8)	0.6201 (3)	0.0676 (16)
C10	0.2452 (5)	1.0780 (9)	0.5978 (3)	0.0739 (19)
H10A	0.2296	1.0695	0.5622	0.089*
C11	0.2570 (5)	0.9372 (9)	0.6271 (3)	0.0762 (18)
H11A	0.2511	0.8337	0.6115	0.091*
C12	0.2775 (4)	0.9505 (9)	0.6798 (3)	0.0689 (18)
C13	0.2884 (4)	1.1028 (9)	0.7053 (3)	0.0580 (17)
C14	0.2783 (4)	1.2404 (8)	0.6728 (3)	0.0675 (18)
H14A	0.2871	1.3445	0.6876	0.081*
C15	0.3078 (4)	1.1142 (9)	0.7630 (3)	0.0660 (19)
H15A	0.3439	1.0152	0.7725	0.079*
C16	0.3669 (4)	1.2611 (7)	0.7795 (3)	0.082 (2)

H16A	0.4262	1.2589	0.7610	0.122*
H16B	0.3783	1.2563	0.8168	0.122*
H16C	0.3337	1.3613	0.7711	0.122*
S2	0.04866 (15)	0.7149 (3)	0.51953 (10)	0.0912 (6)
O4	0.1369 (4)	0.7467 (6)	0.5400 (2)	0.1101 (19)
O5	−0.0329 (3)	0.8073 (6)	0.5539 (2)	0.0810 (15)
O6	0.0424 (3)	0.8172 (6)	0.4638 (2)	0.0813 (16)
C17	0.2390 (6)	1.4193 (10)	0.4275 (3)	0.109 (3)
H17A	0.2000	1.5103	0.4385	0.163*
H17B	0.2535	1.4303	0.3907	0.163*
H17C	0.2966	1.4192	0.4476	0.163*
C18	0.1868 (5)	1.2598 (8)	0.4366 (3)	0.0731 (18)
C19	0.2286 (5)	1.1054 (10)	0.4249 (3)	0.080 (2)
H19A	0.2899	1.1010	0.4115	0.096*
C20	0.1795 (6)	0.9635 (11)	0.4330 (3)	0.081 (2)
H20A	0.2053	0.8631	0.4223	0.097*
C21	0.0904 (5)	0.9661 (9)	0.4573 (3)	0.0665 (18)
C22	0.0483 (5)	1.1173 (9)	0.4706 (3)	0.0664 (19)
C23	0.0994 (5)	1.2595 (8)	0.4597 (3)	0.0716 (18)
H23A	0.0725	1.3607	0.4685	0.086*
C24	0.0153 (5)	1.3952 (9)	0.6739 (3)	0.093 (2)
H24A	0.0061	1.4887	0.6511	0.140*
H24B	0.0772	1.4006	0.6894	0.140*
H24C	−0.0317	1.3965	0.7013	0.140*
C25	0.0060 (4)	1.2366 (9)	0.6420 (3)	0.0734 (18)
C26	−0.0158 (4)	1.2464 (7)	0.5889 (3)	0.0636 (17)
H26A	−0.0231	1.3501	0.5736	0.076*
C27	−0.0273 (4)	1.1081 (10)	0.5581 (3)	0.0679 (19)
C28	−0.0154 (4)	0.9546 (9)	0.5820 (3)	0.0642 (17)
C29	0.0086 (5)	0.9411 (10)	0.6343 (3)	0.0750 (19)
H29A	0.0189	0.8378	0.6493	0.090*
C30	0.0170 (4)	1.0803 (10)	0.6639 (3)	0.0755 (19)
H30A	0.0305	1.0707	0.6997	0.091*
C31	−0.0445 (4)	1.1191 (8)	0.4987 (3)	0.0640 (18)
H31A	−0.0772	1.0169	0.4886	0.077*
C32	−0.1105 (4)	1.2619 (8)	0.4832 (3)	0.083 (2)
H32A	−0.1209	1.2601	0.4458	0.124*
H32B	−0.0820	1.3652	0.4930	0.124*
H32C	−0.1700	1.2498	0.5012	0.124*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0971 (16)	0.0757 (12)	0.119 (2)	−0.0127 (12)	0.0042 (14)	−0.0001 (15)
O1	0.127 (5)	0.085 (4)	0.118 (4)	−0.020 (3)	−0.003 (4)	−0.007 (3)
O2	0.108 (4)	0.070 (3)	0.080 (4)	0.003 (3)	0.000 (3)	0.015 (3)
O3	0.072 (3)	0.077 (3)	0.101 (4)	0.010 (3)	0.005 (3)	0.007 (3)
C1	0.108 (6)	0.130 (7)	0.111 (7)	0.025 (6)	0.030 (6)	0.014 (6)

C2	0.074 (4)	0.089 (4)	0.073 (4)	0.009 (4)	−0.011 (3)	−0.001 (4)
C3	0.072 (4)	0.064 (4)	0.060 (4)	−0.007 (3)	0.007 (3)	0.002 (3)
C4	0.057 (4)	0.068 (4)	0.061 (4)	0.001 (3)	−0.017 (3)	0.005 (4)
C5	0.072 (4)	0.075 (4)	0.061 (5)	−0.010 (4)	−0.001 (3)	0.014 (4)
C6	0.080 (4)	0.084 (4)	0.084 (5)	−0.015 (4)	0.004 (4)	−0.002 (4)
C7	0.065 (4)	0.107 (5)	0.081 (5)	−0.005 (4)	0.002 (4)	0.000 (5)
C8	0.098 (5)	0.105 (5)	0.101 (5)	0.014 (5)	0.007 (5)	0.012 (4)
C9	0.054 (3)	0.079 (4)	0.071 (4)	−0.003 (3)	0.002 (3)	0.013 (3)
C10	0.058 (4)	0.088 (4)	0.075 (4)	−0.004 (4)	0.009 (4)	−0.008 (4)
C11	0.061 (4)	0.077 (4)	0.091 (4)	−0.006 (4)	0.012 (4)	−0.014 (4)
C12	0.051 (4)	0.059 (3)	0.096 (5)	0.002 (3)	0.002 (4)	−0.008 (4)
C13	0.044 (3)	0.068 (4)	0.062 (4)	−0.001 (3)	0.005 (3)	0.003 (3)
C14	0.055 (4)	0.068 (4)	0.079 (4)	−0.007 (3)	0.002 (3)	0.001 (3)
C15	0.039 (3)	0.070 (4)	0.090 (5)	0.000 (3)	−0.008 (3)	0.012 (4)
C16	0.066 (4)	0.074 (5)	0.105 (6)	−0.007 (4)	−0.015 (4)	−0.005 (4)
S2	0.0892 (15)	0.0773 (12)	0.1071 (16)	0.0089 (12)	0.0064 (14)	0.0016 (14)
O4	0.116 (5)	0.089 (4)	0.126 (5)	0.022 (3)	−0.005 (4)	−0.009 (3)
O5	0.064 (3)	0.068 (3)	0.110 (4)	−0.007 (3)	0.009 (3)	−0.005 (3)
O6	0.073 (3)	0.072 (3)	0.099 (4)	0.006 (3)	−0.016 (3)	−0.016 (3)
C17	0.105 (6)	0.116 (6)	0.105 (6)	−0.050 (5)	0.003 (5)	0.004 (5)
C18	0.079 (4)	0.083 (4)	0.058 (4)	−0.009 (4)	0.012 (3)	−0.010 (3)
C19	0.074 (4)	0.103 (5)	0.064 (4)	0.009 (4)	0.021 (4)	0.010 (4)
C20	0.085 (4)	0.089 (4)	0.069 (5)	0.023 (4)	0.004 (4)	−0.001 (4)
C21	0.067 (4)	0.069 (4)	0.064 (4)	−0.004 (3)	−0.014 (3)	−0.011 (4)
C22	0.057 (4)	0.069 (4)	0.073 (5)	0.000 (3)	0.000 (3)	−0.011 (4)
C23	0.067 (4)	0.071 (4)	0.077 (4)	−0.004 (3)	0.002 (3)	−0.006 (4)
C24	0.086 (5)	0.111 (6)	0.083 (5)	−0.002 (5)	0.002 (4)	−0.035 (5)
C25	0.054 (4)	0.088 (4)	0.078 (4)	−0.010 (4)	0.005 (3)	0.000 (4)
C26	0.060 (4)	0.049 (3)	0.082 (4)	−0.005 (3)	0.001 (3)	0.004 (3)
C27	0.044 (3)	0.080 (4)	0.080 (5)	0.001 (3)	−0.005 (3)	−0.004 (4)
C28	0.050 (3)	0.063 (4)	0.079 (4)	−0.003 (3)	0.010 (3)	−0.005 (4)
C29	0.060 (4)	0.086 (4)	0.079 (4)	−0.003 (4)	0.004 (4)	0.007 (4)
C30	0.054 (4)	0.099 (5)	0.073 (4)	0.000 (4)	−0.003 (3)	0.006 (4)
C31	0.057 (4)	0.048 (4)	0.087 (5)	0.000 (3)	−0.013 (4)	−0.006 (3)
C32	0.053 (4)	0.087 (5)	0.108 (6)	0.009 (4)	−0.025 (4)	−0.005 (4)

Geometric parameters (Å, °)

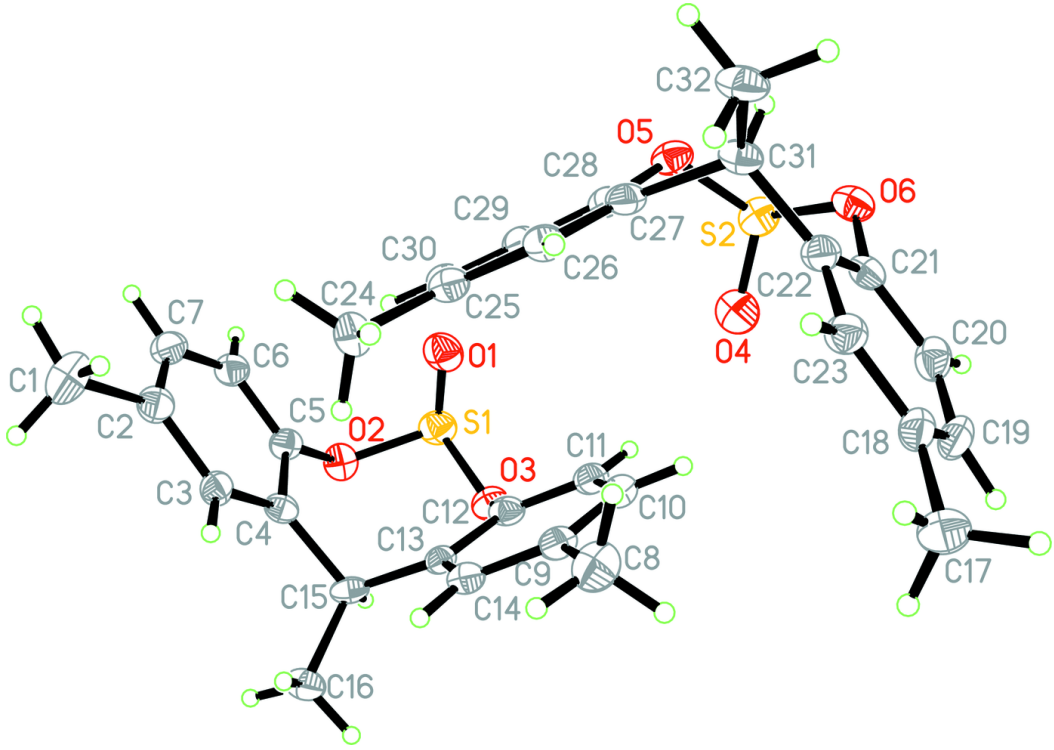
S1—O1	1.381 (5)	S2—O4	1.375 (5)
S1—O3	1.599 (5)	S2—O5	1.626 (5)
S1—O2	1.625 (6)	S2—O6	1.639 (6)
O2—C5	1.414 (8)	O5—C28	1.410 (8)
O3—C12	1.418 (9)	O6—C21	1.391 (8)
C1—C2	1.542 (9)	C17—C18	1.503 (9)
C1—H1B	0.9600	C17—H17A	0.9600
C1—H1C	0.9600	C17—H17B	0.9600
C1—H1D	0.9600	C17—H17C	0.9600
C2—C7	1.337 (9)	C18—C23	1.366 (9)
C2—C3	1.400 (9)	C18—C19	1.413 (9)

C3—C4	1.374 (8)	C19—C20	1.357 (10)
C3—H3A	0.9300	C19—H19A	0.9300
C4—C5	1.403 (10)	C20—C21	1.401 (10)
C4—C15	1.525 (8)	C20—H20A	0.9300
C5—C6	1.377 (10)	C21—C22	1.401 (9)
C6—C7	1.353 (10)	C22—C23	1.385 (9)
C6—H6A	0.9300	C22—C31	1.491 (9)
C7—H7A	0.9300	C23—H23A	0.9300
C8—C9	1.541 (9)	C24—C25	1.522 (9)
C8—H8A	0.9600	C24—H24A	0.9600
C8—H8B	0.9600	C24—H24B	0.9600
C8—H8C	0.9600	C24—H24C	0.9600
C9—C10	1.373 (8)	C25—C26	1.383 (9)
C9—C14	1.374 (9)	C25—C30	1.389 (9)
C10—C11	1.371 (9)	C26—C27	1.374 (9)
C10—H10A	0.9300	C26—H26A	0.9300
C11—C12	1.370 (10)	C27—C28	1.392 (10)
C11—H11A	0.9300	C27—C31	1.528 (10)
C12—C13	1.399 (10)	C28—C29	1.371 (9)
C13—C14	1.392 (9)	C29—C30	1.358 (9)
C13—C15	1.491 (10)	C29—H29A	0.9300
C14—H14A	0.9300	C30—H30A	0.9300
C15—C16	1.511 (8)	C31—C32	1.536 (8)
C15—H15A	0.9800	C31—H31A	0.9800
C16—H16A	0.9600	C32—H32A	0.9600
C16—H16B	0.9600	C32—H32B	0.9600
C16—H16C	0.9600	C32—H32C	0.9600
O1—S1—O3	109.3 (3)	O4—S2—O5	110.7 (3)
O1—S1—O2	107.7 (3)	O4—S2—O6	106.2 (3)
O3—S1—O2	101.8 (3)	O5—S2—O6	101.0 (3)
C5—O2—S1	118.4 (5)	C28—O5—S2	122.3 (4)
C12—O3—S1	123.6 (4)	C21—O6—S2	120.8 (5)
C2—C1—H1B	109.5	C18—C17—H17A	109.5
C2—C1—H1C	109.5	C18—C17—H17B	109.5
H1B—C1—H1C	109.5	H17A—C17—H17B	109.5
C2—C1—H1D	109.5	C18—C17—H17C	109.5
H1B—C1—H1D	109.5	H17A—C17—H17C	109.5
H1C—C1—H1D	109.5	H17B—C17—H17C	109.5
C7—C2—C3	119.2 (7)	C23—C18—C19	117.8 (6)
C7—C2—C1	120.2 (7)	C23—C18—C17	120.6 (7)
C3—C2—C1	120.6 (7)	C19—C18—C17	121.5 (7)
C4—C3—C2	121.8 (6)	C20—C19—C18	120.1 (7)
C4—C3—H3A	119.1	C20—C19—H19A	119.9
C2—C3—H3A	119.1	C18—C19—H19A	119.9
C3—C4—C5	116.4 (7)	C19—C20—C21	120.9 (8)
C3—C4—C15	121.2 (7)	C19—C20—H20A	119.6
C5—C4—C15	121.9 (7)	C21—C20—H20A	119.6
C6—C5—C4	121.4 (7)	O6—C21—C22	121.4 (6)
C6—C5—O2	119.6 (7)	O6—C21—C20	118.4 (7)

C4—C5—O2	118.9 (6)	C22—C21—C20	120.0 (7)
C7—C6—C5	119.5 (8)	C23—C22—C21	117.1 (6)
C7—C6—H6A	120.2	C23—C22—C31	123.0 (7)
C5—C6—H6A	120.2	C21—C22—C31	119.7 (7)
C2—C7—C6	121.6 (7)	C18—C23—C22	123.9 (7)
C2—C7—H7A	119.2	C18—C23—H23A	118.1
C6—C7—H7A	119.2	C22—C23—H23A	118.1
C9—C8—H8A	109.5	C25—C24—H24A	109.5
C9—C8—H8B	109.5	C25—C24—H24B	109.5
H8A—C8—H8B	109.5	H24A—C24—H24B	109.5
C9—C8—H8C	109.5	C25—C24—H24C	109.5
H8A—C8—H8C	109.5	H24A—C24—H24C	109.5
H8B—C8—H8C	109.5	H24B—C24—H24C	109.5
C10—C9—C14	118.3 (6)	C26—C25—C30	117.7 (7)
C10—C9—C8	119.6 (6)	C26—C25—C24	119.2 (7)
C14—C9—C8	122.1 (6)	C30—C25—C24	123.1 (7)
C11—C10—C9	120.9 (7)	C27—C26—C25	122.2 (6)
C11—C10—H10A	119.6	C27—C26—H26A	118.9
C9—C10—H10A	119.6	C25—C26—H26A	118.9
C12—C11—C10	119.3 (8)	C26—C27—C28	117.6 (8)
C12—C11—H11A	120.4	C26—C27—C31	122.1 (7)
C10—C11—H11A	120.4	C28—C27—C31	120.0 (7)
C11—C12—C13	122.8 (8)	C29—C28—C27	121.4 (8)
C11—C12—O3	118.3 (7)	C29—C28—O5	117.7 (7)
C13—C12—O3	118.6 (7)	C27—C28—O5	120.8 (7)
C14—C13—C12	114.8 (7)	C30—C29—C28	119.3 (8)
C14—C13—C15	123.3 (7)	C30—C29—H29A	120.3
C12—C13—C15	121.8 (7)	C28—C29—H29A	120.3
C9—C14—C13	123.8 (6)	C29—C30—C25	121.6 (8)
C9—C14—H14A	118.1	C29—C30—H30A	119.2
C13—C14—H14A	118.1	C25—C30—H30A	119.2
C13—C15—C16	114.9 (6)	C22—C31—C27	109.3 (5)
C13—C15—C4	108.5 (5)	C22—C31—C32	114.8 (6)
C16—C15—C4	112.9 (6)	C27—C31—C32	113.1 (6)
C13—C15—H15A	106.7	C22—C31—H31A	106.4
C16—C15—H15A	106.7	C27—C31—H31A	106.4
C4—C15—H15A	106.7	C32—C31—H31A	106.4
C15—C16—H16A	109.5	C31—C32—H32A	109.5
C15—C16—H16B	109.5	C31—C32—H32B	109.5
H16A—C16—H16B	109.5	H32A—C32—H32B	109.5
C15—C16—H16C	109.5	C31—C32—H32C	109.5
H16A—C16—H16C	109.5	H32A—C32—H32C	109.5
H16B—C16—H16C	109.5	H32B—C32—H32C	109.5
O1—S1—O2—C5	−29.3 (6)	O4—S2—O5—C28	28.1 (7)
O3—S1—O2—C5	85.6 (6)	O6—S2—O5—C28	−84.2 (6)
O1—S1—O3—C12	27.4 (7)	O4—S2—O6—C21	−31.6 (6)
O2—S1—O3—C12	−86.3 (6)	O5—S2—O6—C21	84.0 (5)
C7—C2—C3—C4	−2.7 (11)	C23—C18—C19—C20	4.6 (10)
C1—C2—C3—C4	178.1 (7)	C17—C18—C19—C20	−179.3 (7)

C2—C3—C4—C5	3.1 (10)	C18—C19—C20—C21	−5.6 (11)
C2—C3—C4—C15	174.6 (6)	S2—O6—C21—C22	−89.2 (8)
C3—C4—C5—C6	−2.9 (11)	S2—O6—C21—C20	96.0 (8)
C15—C4—C5—C6	−174.4 (7)	C19—C20—C21—O6	178.9 (7)
C3—C4—C5—O2	−179.2 (7)	C19—C20—C21—C22	4.0 (12)
C15—C4—C5—O2	9.4 (10)	O6—C21—C22—C23	−176.1 (7)
S1—O2—C5—C6	93.5 (8)	C20—C21—C22—C23	−1.4 (11)
S1—O2—C5—C4	−90.2 (7)	O6—C21—C22—C31	8.1 (11)
C4—C5—C6—C7	2.4 (11)	C20—C21—C22—C31	−177.2 (7)
O2—C5—C6—C7	178.6 (6)	C19—C18—C23—C22	−2.1 (11)
C3—C2—C7—C6	2.0 (11)	C17—C18—C23—C22	−178.2 (7)
C1—C2—C7—C6	−178.8 (7)	C21—C22—C23—C18	0.5 (11)
C5—C6—C7—C2	−1.9 (12)	C31—C22—C23—C18	176.2 (7)
C14—C9—C10—C11	−0.6 (10)	C30—C25—C26—C27	0.4 (10)
C8—C9—C10—C11	177.9 (7)	C24—C25—C26—C27	−178.5 (6)
C9—C10—C11—C12	1.7 (10)	C25—C26—C27—C28	−0.5 (10)
C10—C11—C12—C13	−0.9 (10)	C25—C26—C27—C31	−175.5 (6)
C10—C11—C12—O3	−175.3 (6)	C26—C27—C28—C29	−1.1 (10)
S1—O3—C12—C11	−96.8 (7)	C31—C27—C28—C29	174.0 (6)
S1—O3—C12—C13	88.6 (7)	C26—C27—C28—O5	175.2 (6)
C11—C12—C13—C14	−1.0 (9)	C31—C27—C28—O5	−9.6 (10)
O3—C12—C13—C14	173.4 (5)	S2—O5—C28—C29	−94.4 (7)
C11—C12—C13—C15	178.1 (6)	S2—O5—C28—C27	89.1 (7)
O3—C12—C13—C15	−7.6 (9)	C27—C28—C29—C30	2.8 (10)
C10—C9—C14—C13	−1.5 (10)	O5—C28—C29—C30	−173.7 (6)
C8—C9—C14—C13	−180.0 (6)	C28—C29—C30—C25	−2.8 (10)
C12—C13—C14—C9	2.2 (9)	C26—C25—C30—C29	1.3 (10)
C15—C13—C14—C9	−176.8 (6)	C24—C25—C30—C29	−179.8 (7)
C14—C13—C15—C16	−32.3 (8)	C23—C22—C31—C27	−88.7 (9)
C12—C13—C15—C16	148.7 (6)	C21—C22—C31—C27	86.8 (8)
C14—C13—C15—C4	95.1 (7)	C23—C22—C31—C32	39.5 (10)
C12—C13—C15—C4	−83.8 (8)	C21—C22—C31—C32	−145.0 (6)
C3—C4—C15—C13	−85.1 (8)	C26—C27—C31—C22	90.0 (8)
C5—C4—C15—C13	85.9 (8)	C28—C27—C31—C22	−84.9 (8)
C3—C4—C15—C16	43.4 (9)	C26—C27—C31—C32	−39.2 (9)
C5—C4—C15—C16	−145.6 (6)	C28—C27—C31—C32	145.9 (6)

Fig. 1


C32
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