

3,3'-Di-tert-butyl-2'-hydroxy-5,5',6,6'-tetramethylbiphenyl-2-yl benzene-sulfonate

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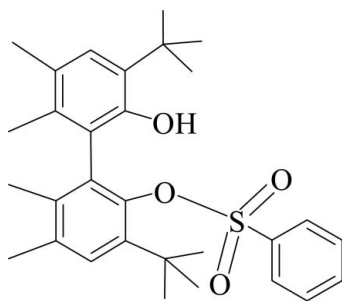
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
R factor = 0.056; wR factor = 0.164; data-to-parameter ratio = 21.1.

In the title compound, $\text{C}_{30}\text{H}_{38}\text{O}_4\text{S}$, the hydroxyl group bonded to one phenyl ring and an O atom of the benzenesulfonate group attached to the other phenyl ring of the biphenyl backbone of the structure are involved in an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond. The dihedral angle between the planes of the two aromatic rings of the biphenyl unit is $70.4(2)^\circ$.

Related literature

For the use of the binolate ligand 5,5',6,6'-tetramethyl-3,3'-di-tert-butyl-1,1'-bi-2,2'-phenolate in ring-closing metathesis reactions, see: La *et al.* (1998); For binolate-metal complexes, see: Chisholm *et al.* (2003); Wu *et al.* (2008). For related structures: see: Solinas *et al.* (2007).



Experimental

Crystal data

$\text{C}_{30}\text{H}_{38}\text{O}_4\text{S}$

$M_r = 494.66$

Monoclinic, $P2_1/c$
 $a = 9.9909(7)$ Å
 $b = 13.3610(11)$ Å
 $c = 20.2884(16)$ Å
 $\beta = 93.428(3)^\circ$
 $V = 2703.4(4)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.15$ mm⁻¹
 $T = 296$ K
 $0.30 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{\min} = 0.964$, $T_{\max} = 0.973$

24566 measured reflections
6653 independent reflections
3695 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.164$
 $S = 1.02$
6653 reflections

316 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O4}$	0.82	2.37	3.107 (2)	150

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT-Plus (Bruker, 2008); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: PV2156).

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supplementary materials

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3,3'-Di-tert-butyl-2'-hydroxy-5,5',6,6'-tetramethylbiphenyl-2-yl benzenesulfonate

P.-S. Chen, C.-H. Lin and B.-T. Ko

Comment

During the last decade, bulky bis(phenolate) compounds have been attracting considerable attention, mainly due to their importance in the development of coordination chemistry related to catalytic applications. These bulky ligands are designed to provide a steric barrier around active metal center for minimizing the side reaction. Bulky binolate ligand, 5,5',6,6'-tetramethyl-3,3'-di-tert-butyl-1,1'-bi-2,2'-phenolate (BIPHEN²⁻) has proved to be important in ring-closing metathesis reactions in both its racemic and resolved forms (La *et al.*, 1998). Recently, a series of binolate metal complexes where the metal atoms are Li, Zn and Al have been synthesized, structurally characterized and studied for the catalytic activity of lactide polymerization (Chisholm *et al.*, 2003). Most recently, Wu and his co-workers, (Wu *et al.*, 2008) have also reported the magnesium complexes supported by mono-benzenesulfonylate phenol ligand and these complexes have been demonstrated as efficient initiators to catalyze ring-opening polymerization of lactides. Our group is interested in the synthesis and preparation of monovalent phenol derived from BIPHEN-H₂. Here, we report the synthesis and crystal structure of the title compound, (I), a potential ligand for the preparation of magnesium and zinc complexes.

The structure of (I) is composed of a biphenyl moiety containing a benzenesulfonate and a hydroxyl group (Fig. 1). The dihedral angle between the planes of the two aromatic rings of the biphenyl unit is 70.4 (2)°. There is an intramolecular O—H...O hydrogen bond between the phenol and benzenesulfonate groups (Table 1). The distance of O4...H is substantially shorter than the van der Waals distance of 2.77 Å for the O and H distance. However, the distances O4...H and O4...O1 are around 0.3-0.4 Å longer than that found in the other mono-sulfonylated biaryl derivative with an intermolecular hydrogen-bonded motif (1.97 Å & 2.795 (2) Å; Solinas *et al.*, 2007).

Experimental

The title compound (I) was synthesized by the following procedures (Scheme 2): 3,3'-di-tert-butyl-5,5',6,6'-tetramethylbiphenyl-2,2'-diol (1.24 g, 3.50 mmol), benzenesulfonyl chloride (0.45 mL, 3.50 mmol) and 4-(dimethylamino)pyridine (DMAP, 0.069 g, 0.57 mmol, catalyst) were dissolved in 50 mL of freshly distilled CH₂Cl₂ and the resulting solution cooled to 273 K. Neat triethylamine (NEt₃, 0.6 mL, 3.85 mmol) was added dropwise to the solution, which was then stirred at ambient temperature for 48 h. The solution was filtered, and the filtrate was washed with 50 mL of water three times. The dichloromethane layer was collected and dried over anhydrous MgSO₄ and filtered through Celite again to remove MgSO₄. The resulting filtrate was then dried under vacuum to obtain the white solids (yield: 78 %). The resulting solid was crystallized from a toluene solution to yield colourless crystals of (I).

Refinement

The H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 and 0.96 Å allowing $U_{\text{iso}}(\text{H}) = 1.2$ and $1.5U_{\text{eq}}(\text{C})$ for aryl and methyl groups, respectively; for hydroxy group, O—H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

Figures

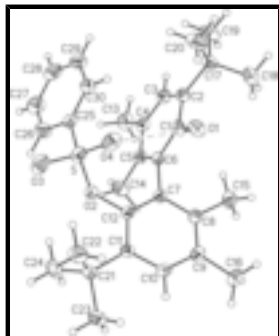


Fig. 1. A view of the molecular structure of (I) with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.



Fig. 2. The formation of the title compound.

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Crystal data

$C_{30}H_{38}O_4S$

$M_r = 494.66$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.9909 (7) \text{ \AA}$

$b = 13.3610 (11) \text{ \AA}$

$c = 20.2884 (16) \text{ \AA}$

$\beta = 93.428 (3)^\circ$

$V = 2703.4 (4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 1064$

$D_x = 1.215 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3976 reflections

$\theta = 2.5\text{--}22.8^\circ$

$\mu = 0.15 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Columnar, colourless

$0.30 \times 0.20 \times 0.18 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296 \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2008)

$T_{\min} = 0.964$, $T_{\max} = 0.973$

24566 measured reflections

6653 independent reflections

3695 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\max} = 28.4^\circ$

$\theta_{\min} = 1.8^\circ$

$h = -13 \rightarrow 8$

$k = -17 \rightarrow 17$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.056$$

$$wR(F^2) = 0.164$$

$$S = 1.02$$

6653 reflections

316 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.1593P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

Experimental. ¹H NMR (CDCl₃, ppm): δ 7.20-7.46 (6H, m, ArH), 6.68 (1H, s, ArH), 5.00 (1H, s, OH), 2.32 (1H, s, CH₃), 1.77 (1H, s, CH₃), 1.74 (1H, s, CH₃), 1.59 (9H, s, C(CH₃)₃), 1.50 (1H, s, CH₃), 1.45 (9H, s, C(CH₃)₃).

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.

Refinement. Refinement of F² against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F², conventional R-factors R are based on F, with F set to zero for negative F². The threshold expression of F² > 2sigma(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² 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 (Å²)

	x	y	z	U _{iso} */U _{eq}
S	0.36621 (6)	0.38183 (5)	0.17811 (3)	0.04538 (19)
O1	0.65811 (15)	0.19585 (13)	0.25711 (8)	0.0520 (5)
H1A	0.5798	0.2118	0.2612	0.078*
O2	0.39067 (14)	0.32215 (11)	0.11199 (7)	0.0418 (4)
O3	0.23658 (16)	0.42523 (15)	0.16868 (11)	0.0713 (6)
O4	0.39560 (19)	0.31853 (13)	0.23306 (8)	0.0614 (5)
C1	0.7141 (2)	0.25910 (17)	0.21289 (11)	0.0372 (5)
C2	0.8289 (2)	0.31260 (18)	0.23429 (11)	0.0403 (5)
C3	0.8784 (2)	0.37703 (18)	0.18794 (12)	0.0438 (6)
H3B	0.9535	0.4151	0.2008	0.053*
C4	0.8245 (2)	0.38863 (17)	0.12427 (12)	0.0425 (6)
C5	0.7154 (2)	0.32891 (18)	0.10304 (11)	0.0401 (5)
C6	0.6595 (2)	0.26372 (17)	0.14839 (11)	0.0357 (5)
C7	0.5539 (2)	0.18961 (16)	0.12617 (10)	0.0354 (5)
C8	0.5890 (2)	0.08878 (18)	0.12009 (11)	0.0397 (5)
C9	0.4930 (2)	0.01987 (17)	0.09643 (11)	0.0403 (5)
C10	0.3647 (2)	0.05417 (17)	0.07881 (11)	0.0401 (5)
H10A	0.3020	0.0076	0.0625	0.048*
C11	0.3231 (2)	0.15299 (17)	0.08380 (10)	0.0360 (5)

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C12	0.4217 (2)	0.21839 (16)	0.10941 (10)	0.0343 (5)
C13	0.8876 (3)	0.4624 (2)	0.07860 (14)	0.0648 (8)
H13A	0.9607	0.4961	0.1021	0.097*
H13B	0.8217	0.5107	0.0632	0.097*
H13C	0.9203	0.4272	0.0416	0.097*
C14	0.6595 (3)	0.3336 (2)	0.03255 (12)	0.0587 (7)
H14A	0.7095	0.3814	0.0086	0.088*
H14B	0.5671	0.3536	0.0316	0.088*
H14C	0.6663	0.2689	0.0125	0.088*
C15	0.7310 (2)	0.0537 (2)	0.13777 (14)	0.0599 (7)
H15A	0.7846	0.1096	0.1533	0.090*
H15B	0.7684	0.0256	0.0994	0.090*
H15C	0.7299	0.0039	0.1718	0.090*
C16	0.5247 (2)	-0.08952 (19)	0.08847 (13)	0.0549 (7)
H16A	0.4457	-0.1242	0.0716	0.082*
H16B	0.5541	-0.1172	0.1305	0.082*
H16C	0.5943	-0.0969	0.0582	0.082*
C17	0.8981 (2)	0.2976 (2)	0.30302 (12)	0.0506 (6)
C18	0.9429 (3)	0.1879 (2)	0.31150 (15)	0.0734 (9)
H18A	0.8662	0.1448	0.3057	0.110*
H18B	0.9845	0.1786	0.3549	0.110*
H18C	1.0059	0.1720	0.2791	0.110*
C19	1.0244 (3)	0.3626 (3)	0.31303 (15)	0.0776 (10)
H19A	1.0002	0.4319	0.3083	0.116*
H19B	1.0866	0.3452	0.2806	0.116*
H19C	1.0652	0.3514	0.3564	0.116*
C20	0.8038 (3)	0.3252 (3)	0.35657 (13)	0.0757 (9)
H20A	0.7767	0.3938	0.3513	0.114*
H20B	0.8492	0.3164	0.3992	0.114*
H20C	0.7261	0.2829	0.3529	0.114*
C21	0.1775 (2)	0.18286 (18)	0.06284 (12)	0.0428 (6)
C22	0.1001 (2)	0.2033 (2)	0.12392 (14)	0.0609 (8)
H22A	0.1426	0.2567	0.1489	0.091*
H22B	0.0993	0.1440	0.1506	0.091*
H22C	0.0097	0.2220	0.1107	0.091*
C23	0.1028 (2)	0.0976 (2)	0.02594 (15)	0.0625 (8)
H23A	0.0127	0.1183	0.0137	0.094*
H23B	0.1009	0.0399	0.0540	0.094*
H23C	0.1480	0.0812	-0.0131	0.094*
C24	0.1724 (3)	0.2737 (2)	0.01644 (15)	0.0714 (9)
H24A	0.2181	0.3290	0.0379	0.107*
H24B	0.0806	0.2917	0.0057	0.107*
H24C	0.2152	0.2572	-0.0233	0.107*
C25	0.4840 (2)	0.47824 (17)	0.17560 (11)	0.0402 (5)
C26	0.4767 (3)	0.5430 (2)	0.12214 (13)	0.0592 (7)
H26A	0.4130	0.5331	0.0874	0.071*
C27	0.5643 (3)	0.6219 (2)	0.12097 (16)	0.0681 (8)
H27A	0.5614	0.6648	0.0848	0.082*
C28	0.6552 (3)	0.6378 (2)	0.17246 (16)	0.0652 (8)

H28A	0.7140	0.6917	0.1715	0.078*
C29	0.6605 (3)	0.5750 (2)	0.22544 (15)	0.0627 (8)
H29A	0.7223	0.5869	0.2607	0.075*
C30	0.5747 (2)	0.4935 (2)	0.22742 (12)	0.0492 (6)
H30A	0.5791	0.4502	0.2634	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0427 (3)	0.0365 (4)	0.0576 (4)	-0.0017 (3)	0.0083 (3)	-0.0061 (3)
O1	0.0436 (9)	0.0635 (12)	0.0487 (10)	-0.0086 (8)	0.0011 (7)	0.0110 (9)
O2	0.0448 (8)	0.0324 (9)	0.0471 (9)	0.0014 (7)	-0.0058 (7)	-0.0024 (7)
O3	0.0406 (10)	0.0567 (12)	0.1176 (17)	0.0051 (9)	0.0124 (10)	-0.0200 (12)
O4	0.0897 (14)	0.0449 (11)	0.0511 (11)	-0.0069 (10)	0.0174 (9)	0.0024 (9)
C1	0.0339 (11)	0.0369 (13)	0.0409 (13)	0.0006 (9)	0.0034 (9)	0.0030 (10)
C2	0.0316 (11)	0.0422 (14)	0.0468 (14)	0.0012 (10)	0.0004 (10)	-0.0057 (11)
C3	0.0319 (11)	0.0409 (14)	0.0584 (16)	-0.0040 (10)	0.0019 (10)	-0.0043 (12)
C4	0.0364 (11)	0.0364 (13)	0.0553 (15)	-0.0015 (10)	0.0076 (10)	0.0019 (11)
C5	0.0363 (11)	0.0396 (14)	0.0443 (13)	0.0030 (10)	0.0020 (10)	0.0005 (11)
C6	0.0306 (10)	0.0349 (13)	0.0414 (13)	0.0003 (9)	0.0008 (9)	-0.0021 (10)
C7	0.0331 (10)	0.0340 (12)	0.0389 (12)	0.0004 (9)	0.0013 (9)	-0.0002 (10)
C8	0.0376 (11)	0.0406 (14)	0.0409 (13)	0.0027 (10)	0.0034 (9)	-0.0022 (11)
C9	0.0467 (13)	0.0344 (13)	0.0405 (13)	0.0005 (10)	0.0086 (10)	-0.0035 (10)
C10	0.0416 (12)	0.0361 (13)	0.0424 (13)	-0.0059 (10)	0.0012 (10)	-0.0046 (11)
C11	0.0378 (11)	0.0376 (13)	0.0322 (11)	-0.0017 (10)	-0.0008 (9)	-0.0033 (10)
C12	0.0380 (11)	0.0292 (12)	0.0354 (12)	0.0001 (9)	0.0011 (9)	-0.0002 (9)
C13	0.0617 (17)	0.0595 (19)	0.0741 (19)	-0.0114 (14)	0.0112 (14)	0.0113 (15)
C14	0.0577 (15)	0.074 (2)	0.0443 (15)	-0.0078 (14)	0.0011 (12)	0.0079 (14)
C15	0.0458 (14)	0.0536 (17)	0.0795 (19)	0.0144 (12)	-0.0017 (13)	-0.0046 (15)
C16	0.0606 (16)	0.0386 (15)	0.0667 (17)	0.0049 (12)	0.0130 (13)	-0.0053 (13)
C17	0.0393 (12)	0.0666 (18)	0.0446 (14)	-0.0009 (12)	-0.0078 (10)	-0.0047 (13)
C18	0.0639 (17)	0.081 (2)	0.073 (2)	0.0148 (16)	-0.0195 (15)	0.0068 (17)
C19	0.0595 (17)	0.100 (3)	0.070 (2)	-0.0212 (17)	-0.0197 (15)	-0.0072 (18)
C20	0.0661 (18)	0.114 (3)	0.0466 (16)	0.0055 (18)	-0.0005 (14)	-0.0178 (17)
C21	0.0334 (11)	0.0445 (14)	0.0493 (14)	-0.0013 (10)	-0.0077 (10)	-0.0032 (11)
C22	0.0354 (12)	0.074 (2)	0.0736 (19)	-0.0008 (13)	0.0059 (12)	-0.0162 (16)
C23	0.0447 (14)	0.0646 (19)	0.0760 (19)	-0.0079 (13)	-0.0155 (13)	-0.0168 (15)
C24	0.0560 (16)	0.073 (2)	0.082 (2)	-0.0045 (15)	-0.0219 (15)	0.0193 (17)
C25	0.0418 (12)	0.0325 (13)	0.0460 (13)	0.0005 (10)	-0.0003 (10)	-0.0050 (11)
C26	0.0673 (17)	0.0497 (17)	0.0584 (17)	-0.0112 (14)	-0.0145 (13)	0.0060 (14)
C27	0.081 (2)	0.0491 (18)	0.074 (2)	-0.0146 (15)	0.0029 (16)	0.0102 (15)
C28	0.0588 (17)	0.0476 (18)	0.090 (2)	-0.0145 (13)	0.0143 (16)	-0.0160 (17)
C29	0.0523 (15)	0.0607 (19)	0.073 (2)	-0.0030 (14)	-0.0115 (13)	-0.0236 (17)
C30	0.0505 (14)	0.0503 (16)	0.0462 (14)	0.0018 (12)	-0.0036 (11)	-0.0071 (12)

Geometric parameters (\AA , $^\circ$)

S—O4	1.4161 (18)	C16—H16B	0.9600
S—O3	1.4215 (18)	C16—H16C	0.9600

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S—O2	1.5920 (16)	C17—C20	1.526 (4)
S—C25	1.748 (2)	C17—C19	1.535 (4)
O1—C1	1.376 (3)	C17—C18	1.538 (4)
O1—H1A	0.8200	C18—H18A	0.9600
O2—C12	1.422 (3)	C18—H18B	0.9600
C1—C6	1.389 (3)	C18—H18C	0.9600
C1—C2	1.398 (3)	C19—H19A	0.9600
C2—C3	1.388 (3)	C19—H19B	0.9600
C2—C17	1.532 (3)	C19—H19C	0.9600
C3—C4	1.378 (3)	C20—H20A	0.9600
C3—H3B	0.9300	C20—H20B	0.9600
C4—C5	1.397 (3)	C20—H20C	0.9600
C4—C13	1.516 (3)	C21—C22	1.524 (3)
C5—C6	1.407 (3)	C21—C23	1.532 (3)
C5—C14	1.505 (3)	C21—C24	1.535 (4)
C6—C7	1.497 (3)	C22—H22A	0.9600
C7—C12	1.398 (3)	C22—H22B	0.9600
C7—C8	1.399 (3)	C22—H22C	0.9600
C8—C9	1.394 (3)	C23—H23A	0.9600
C8—C15	1.517 (3)	C23—H23B	0.9600
C9—C10	1.388 (3)	C23—H23C	0.9600
C9—C16	1.506 (3)	C24—H24A	0.9600
C10—C11	1.390 (3)	C24—H24B	0.9600
C10—H10A	0.9300	C24—H24C	0.9600
C11—C12	1.394 (3)	C25—C30	1.362 (3)
C11—C21	1.544 (3)	C25—C26	1.386 (3)
C13—H13A	0.9600	C26—C27	1.371 (4)
C13—H13B	0.9600	C26—H26A	0.9300
C13—H13C	0.9600	C27—C28	1.360 (4)
C14—H14A	0.9600	C27—H27A	0.9300
C14—H14B	0.9600	C28—C29	1.362 (4)
C14—H14C	0.9600	C28—H28A	0.9300
C15—H15A	0.9600	C29—C30	1.389 (4)
C15—H15B	0.9600	C29—H29A	0.9300
C15—H15C	0.9600	C30—H30A	0.9300
C16—H16A	0.9600		
O4—S—O3	119.60 (12)	H16B—C16—H16C	109.5
O4—S—O2	109.22 (10)	C20—C17—C2	110.6 (2)
O3—S—O2	105.99 (10)	C20—C17—C19	107.8 (2)
O4—S—C25	110.79 (11)	C2—C17—C19	111.7 (2)
O3—S—C25	107.77 (11)	C20—C17—C18	109.8 (2)
O2—S—C25	101.95 (10)	C2—C17—C18	109.8 (2)
C1—O1—H1A	109.5	C19—C17—C18	107.1 (2)
C12—O2—S	124.34 (13)	C17—C18—H18A	109.5
O1—C1—C6	119.32 (19)	C17—C18—H18B	109.5
O1—C1—C2	118.01 (19)	H18A—C18—H18B	109.5
C6—C1—C2	122.6 (2)	C17—C18—H18C	109.5
C3—C2—C1	115.2 (2)	H18A—C18—H18C	109.5
C3—C2—C17	122.5 (2)	H18B—C18—H18C	109.5

C1—C2—C17	122.2 (2)	C17—C19—H19A	109.5
C4—C3—C2	124.7 (2)	C17—C19—H19B	109.5
C4—C3—H3B	117.7	H19A—C19—H19B	109.5
C2—C3—H3B	117.7	C17—C19—H19C	109.5
C3—C4—C5	118.6 (2)	H19A—C19—H19C	109.5
C3—C4—C13	119.5 (2)	H19B—C19—H19C	109.5
C5—C4—C13	121.9 (2)	C17—C20—H20A	109.5
C4—C5—C6	119.0 (2)	C17—C20—H20B	109.5
C4—C5—C14	120.5 (2)	H20A—C20—H20B	109.5
C6—C5—C14	120.5 (2)	C17—C20—H20C	109.5
C1—C6—C5	119.66 (19)	H20A—C20—H20C	109.5
C1—C6—C7	118.98 (19)	H20B—C20—H20C	109.5
C5—C6—C7	120.87 (19)	C22—C21—C23	105.9 (2)
C12—C7—C8	118.76 (19)	C22—C21—C24	110.9 (2)
C12—C7—C6	122.01 (19)	C23—C21—C24	106.9 (2)
C8—C7—C6	119.20 (18)	C22—C21—C11	109.74 (18)
C9—C8—C7	119.73 (19)	C23—C21—C11	111.5 (2)
C9—C8—C15	119.5 (2)	C24—C21—C11	111.68 (19)
C7—C8—C15	120.8 (2)	C21—C22—H22A	109.5
C10—C9—C8	118.4 (2)	C21—C22—H22B	109.5
C10—C9—C16	119.3 (2)	H22A—C22—H22B	109.5
C8—C9—C16	122.3 (2)	C21—C22—H22C	109.5
C9—C10—C11	124.8 (2)	H22A—C22—H22C	109.5
C9—C10—H10A	117.6	H22B—C22—H22C	109.5
C11—C10—H10A	117.6	C21—C23—H23A	109.5
C10—C11—C12	114.52 (19)	C21—C23—H23B	109.5
C10—C11—C21	120.42 (19)	H23A—C23—H23B	109.5
C12—C11—C21	125.1 (2)	C21—C23—H23C	109.5
C11—C12—C7	123.7 (2)	H23A—C23—H23C	109.5
C11—C12—O2	118.27 (18)	H23B—C23—H23C	109.5
C7—C12—O2	117.67 (18)	C21—C24—H24A	109.5
C4—C13—H13A	109.5	C21—C24—H24B	109.5
C4—C13—H13B	109.5	H24A—C24—H24B	109.5
H13A—C13—H13B	109.5	C21—C24—H24C	109.5
C4—C13—H13C	109.5	H24A—C24—H24C	109.5
H13A—C13—H13C	109.5	H24B—C24—H24C	109.5
H13B—C13—H13C	109.5	C30—C25—C26	120.8 (2)
C5—C14—H14A	109.5	C30—C25—S	120.40 (19)
C5—C14—H14B	109.5	C26—C25—S	118.62 (18)
H14A—C14—H14B	109.5	C27—C26—C25	119.3 (2)
C5—C14—H14C	109.5	C27—C26—H26A	120.3
H14A—C14—H14C	109.5	C25—C26—H26A	120.3
H14B—C14—H14C	109.5	C28—C27—C26	120.3 (3)
C8—C15—H15A	109.5	C28—C27—H27A	119.9
C8—C15—H15B	109.5	C26—C27—H27A	119.9
H15A—C15—H15B	109.5	C27—C28—C29	120.2 (3)
C8—C15—H15C	109.5	C27—C28—H28A	119.9
H15A—C15—H15C	109.5	C29—C28—H28A	119.9
H15B—C15—H15C	109.5	C28—C29—C30	120.7 (3)

supplementary materials

C9—C16—H16A	109.5	C28—C29—H29A	119.6
C9—C16—H16B	109.5	C30—C29—H29A	119.6
H16A—C16—H16B	109.5	C25—C30—C29	118.6 (3)
C9—C16—H16C	109.5	C25—C30—H30A	120.7
H16A—C16—H16C	109.5	C29—C30—H30A	120.7

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1A...O4	0.82	2.37	3.107 (2)	150

Fig. 1

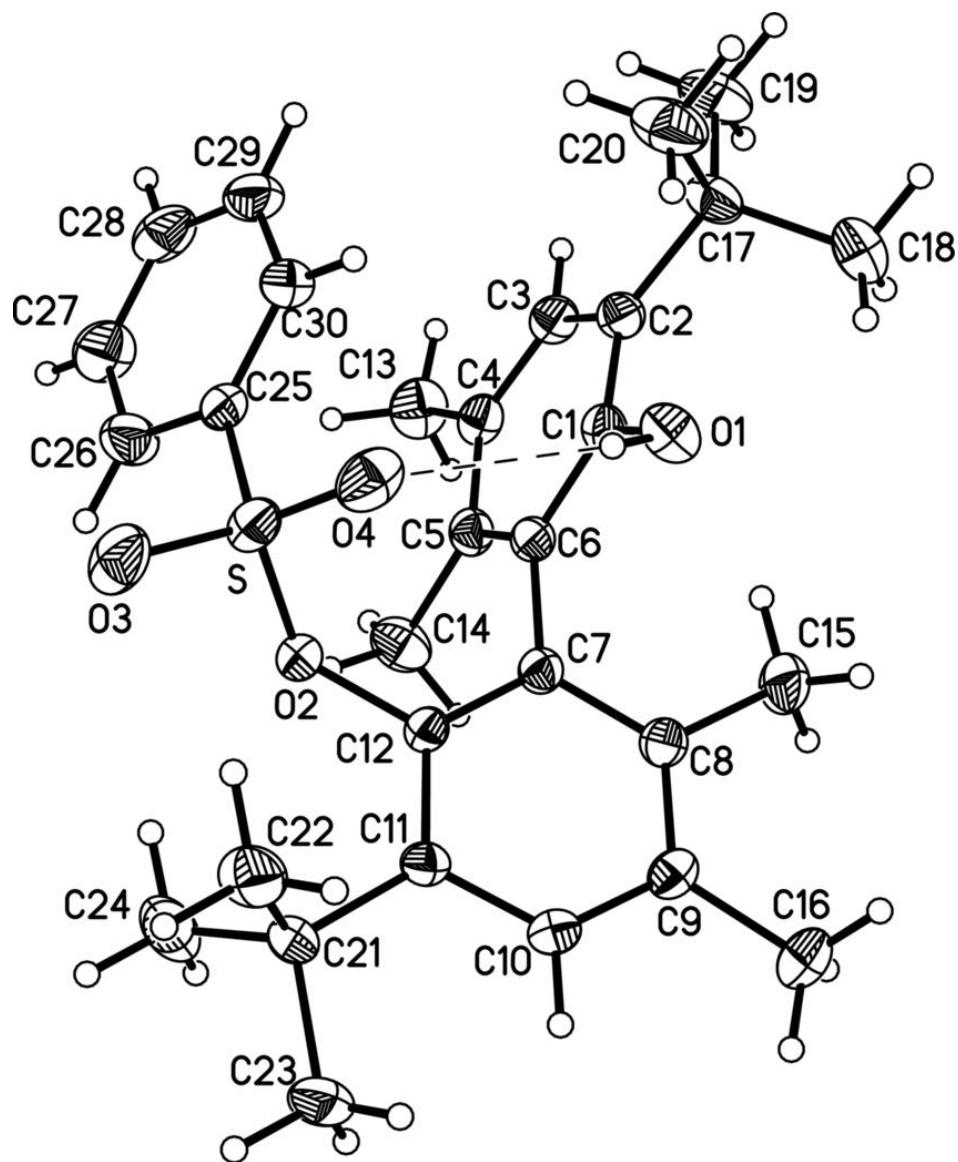


Fig. 2

