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

**Po-Sheng Chen, Chia-Her Lin and Bao-Tsan Ko***

Department of Chemistry, Chung Yuan Christian University, Chung-Li 320, Taiwan
Correspondence e-mail: btko@cycu.edu.tw

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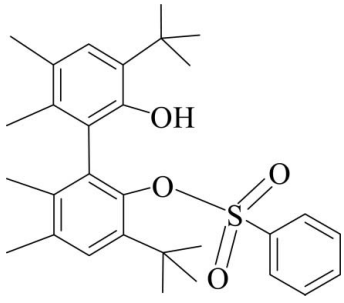
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.056; $wR$ factor = 0.164; data-to-parameter ratio = 21.1.

In the title compound, $C_{30}H_{38}O_4S$, 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 O—H···O hydrogen bond. The dihedral angle between the planes of the two aromatic rings of the biphenyl unit is 70.4 (2)°.

## 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

$C_{30}H_{38}O_4S$ $M_r$ = 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)°
$V$ = 2703.4 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.15 mm$^{-1}$
$T$ = 296 K
0.30 × 0.20 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1$A$···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).

## References

Bruker (2008). *APEX2*, *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chisholm, M. H., Lin, C.-C., Gallucci, J. C. & Ko, B.-T. (2003). *Dalton Trans.* pp. 406–412.
La, D. S., Alexander, J. B., Cefalo, D. R., Craf, D. D., Hoveyda, A. H. & Schrock, R. R. (1998). *J. Am. Chem. Soc.* **120**, 9720–9721.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Solinas, M., Meadows, R. E., Wilson, C., Blake, A. J. & Woodward, S. (2007). *Eur. J. Org. Chem.* pp. 1613–1623.
Wu, J., Chen, Y.-Z., Hung, W.-C. & Lin, C.-C. (2008). *Organometallics*, **27**, 4970–4978.

# supporting information

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

**Po-Sheng Chen, Chia-Her Lin and Bao-Tsan Ko**

## S1. 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 ($BIPHEN^{2-}$) 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-$H_2$. 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).

## S2. Experimental

The title compound (I) was synthesized by the following procedures (Scheme 2): 3,3′-di-tert-butyl-5,5′,6,6′-tetramethyl-biphenyl-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 $CH_2Cl_2$ and the resulting solution cooled to 273 K. Neat triethylamine ($NEt_3$, 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 $MgSO_4$ and filtered through Celite again to remove $MgSO_4$. 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).

### S3. 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 $U_{iso}$(H) = 1.2 and 1.5$U_{eq}$(C) for aryl and methyl groups, respectively; for hydroxy group, O—H = 0.82 Å and $U_{iso}$(H) = 1.2$U_{eq}$(O).

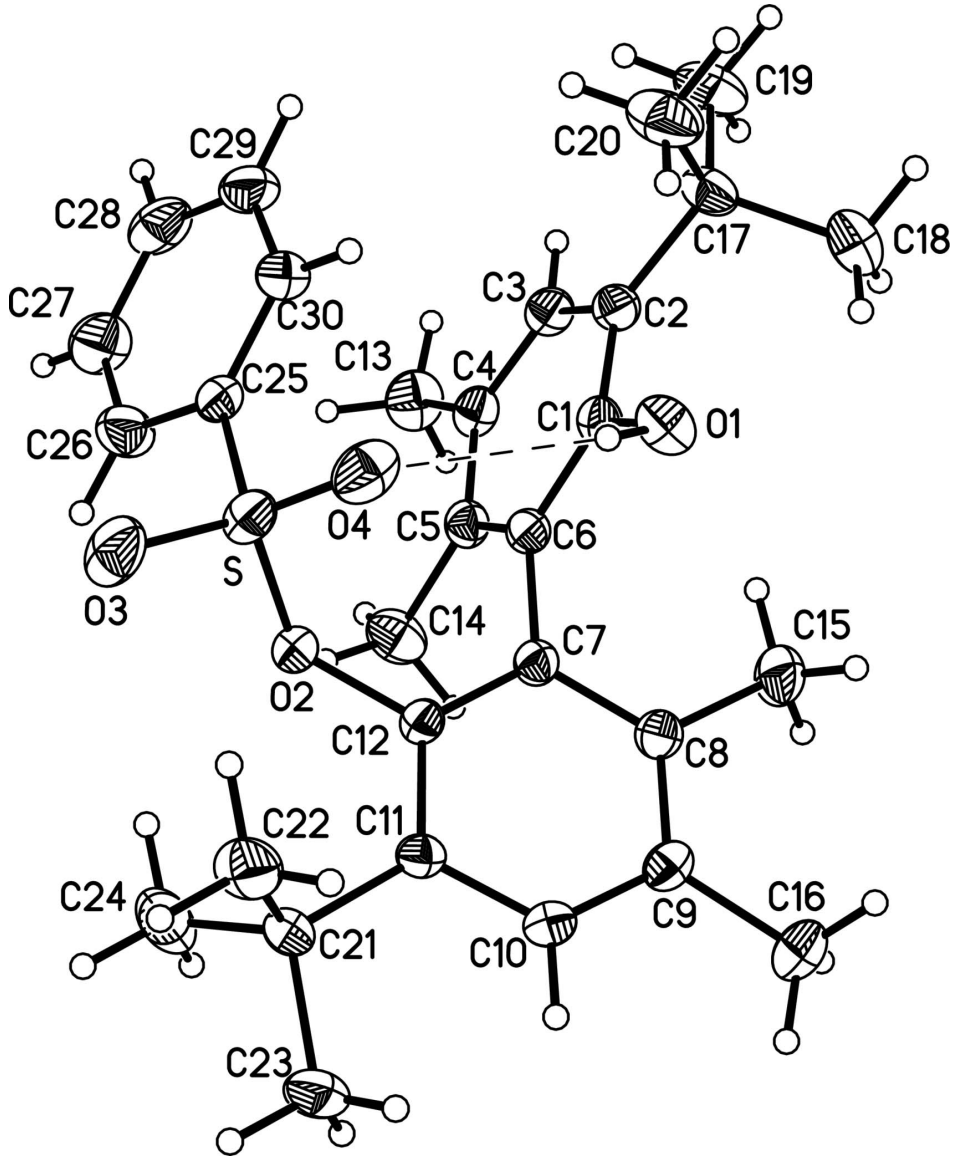


**Figure 1**

A view of the molecular structure of (I) with the atomn umbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

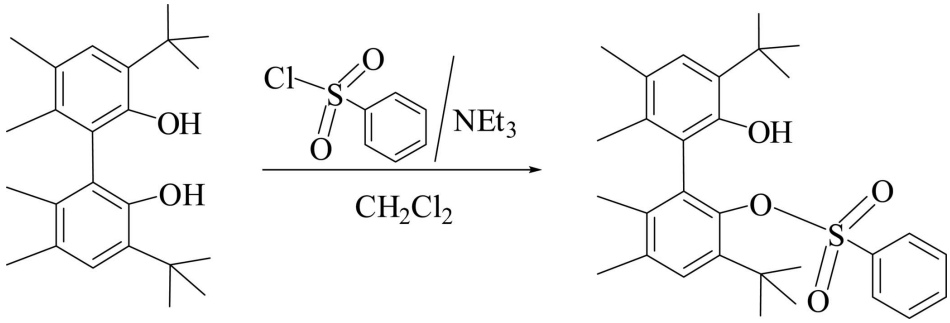


**Figure 2**

The formation of the title compound.

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

*Crystal data*

$C_{30}H_{38}O_4S$
$M_r = 494.66$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.9909$ (7) Å
$b = 13.3610$ (11) Å
$c = 20.2884$ (16) Å
$\beta = 93.428$ (3)°
$V = 2703.4$ (4) Å$^3$
$Z = 4$
$F(000) = 1064$
$D_x = 1.215$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3976 reflections
$\theta = 2.5$–$22.8°$
$\mu = 0.15$ mm$^{-1}$
$T = 296$ K
Columnar, colourless
0.30 × 0.20 × 0.18 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.8°$
$h = -13 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -26 \rightarrow 26$

*Refinement*

Refinement on $F^2$
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
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
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.1593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$

*Special details*

**Experimental**. $^1$H NMR ($CDCl_3$, ppm): $\delta$ 7.20-7.46 (6H, m, ArH), 6.68 (1H, s, ArH), 5.00 (1H, s, OH), 2.32 (1H, s, $CH_3$), 1.77 (1H, s, $CH_3$), 1.74 (1H, s, $CH_3$), 1.59 (9H, s, $C(CH_3)_3$), 1.50 (1H, s, $CH_3$), 1.45 (9H, s, $C(CH_3)_3$).

**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 $F^2$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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) |
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

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

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|---|---|---|---|
| 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) |
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1*A*···O4 | 0.82 | 2.37 | 3.107 (2) | 150 |