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Crystal structure and Hirshfeld surface analysis of dimethyl 4-hydroxy-5,4′-dimethyl-2′-(toluene-4-sulfonylamino)biphenyl-2,3-dicarboxylate

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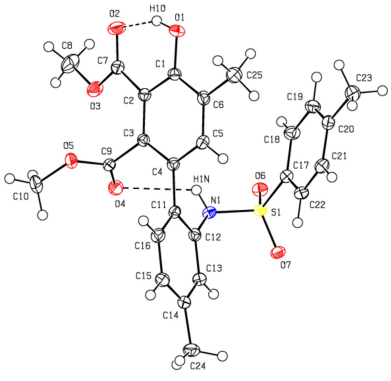
In the title compound, $C_{25}H_{25}NO_7S$, the molecular conformation is stabilized by intramolecular O—H···O and N—H···O hydrogen bonds, which form $S(6)$ and $S(8)$ ring motifs, respectively. The molecules are bent at the S atom with a C—SO_2—NH—C torsion angle of −70.86 (11)°. In the crystal, molecules are linked by C—H···O and N—H···O hydrogen bonds, forming molecular layers parallel to the (100) plane. C—H···π interactions are observed between these layers.

1. Chemical context

Furan contains a system of conjugated *s-cis*-double bonds, closed through an oxygen atom, and as a result, this heterocycle easily participates in Diels–Alder reactions. The [4 + 2] cycloaddition of furan with acetylenedicarboxylic acid esters (as alkynes) was performed for the first time to find a simple route for the preparation of *Cantharidin* (Diels & Alder, 1931). Furan reacts with esters of acetylenedicarboxylic acid when heated to 373 K. The 7-oxabicyclo[2.2.1]heptene scaffold, the product of the reaction between furans and alkynes, has great synthetic potential as a useful tool for the design of a broad diversity of substances with various practical properties. These cycloadducts have been used to construct polycyclic aromatic hydrocarbons (Eda *et al.*, 2015; Criado *et al.*, 2013). The annulated 7-oxabicyclo[2.2.1]heptane moiety also acts as a framework for synthesis of molecular tweezers (Murphy *et al.*, 2016; Warrener *et al.*, 1999), high-molecular-weight materials (Margetić *et al.*, 2010; Vogel *et al.*, 1999) and various supramolecular systems (Abdelhamid *et al.*, 2011; Akbari Afkhami *et al.*, 2017; Khalilov *et al.*, 2021; Safarova *et al.*, 2019). Under acid catalysis, cycloaddition intermediates can be converted into phenols, cyclohexenoles, or substituted aromatic hydrocarbons (Zaytsev *et al.*, 2019; Zubkov *et al.*, 2012*a*,*b*). In this work, we continued our investigations of the cycloaddition of dimethyl acetylenedicarboxylate (DMAD) with substituted furans (Zubkov *et al.*, 2009; Borisova *et al.*, 2018*a*,*b*). In particular, in the course of the thermic [4 + 2] cycloaddition between DMAD and sulfamide **2**, an interesting sequence of reaction steps was observed: a cleavage of the epoxy bridge and a sigmatropic shift of the methyl group



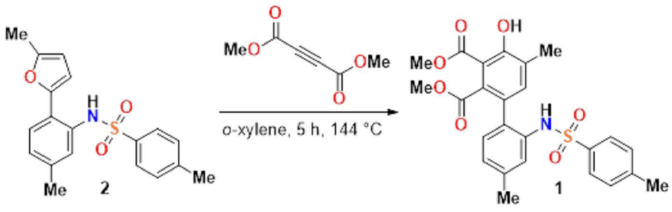
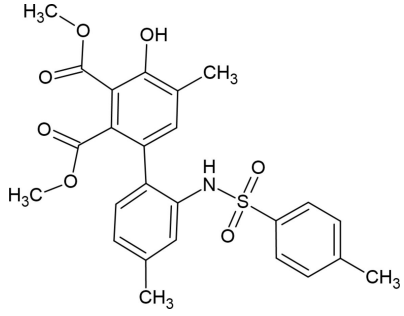


Figure 1
Synthesis of 4-hydroxy-5,4′-dimethyl-2′-(toluene-4-sulfonylamino)-biphenyl-2,3-dicarboxylic acid dimethyl ester (**1**).

(Fig. 1). On the other hand, the biological and catalytic activity as well as coordination ability of the new sulfamide derivative **1** can be dictated by the non-covalent bond-donor or acceptor character of the substituents (Gurbanov *et al.*, 2022*a*,*b*; Kopylovich *et al.*, 2011*a*,*b*; Mahmoudi *et al.*, 2017*a*,*b*; Mahmudov *et al.*, 2013).



2. Structural commentary

In the title compound (Fig. 2), the molecular conformation is stabilized by intramolecular O—H···O and N—H···O hydrogen bonds, which form *S*(6) and *S*(8) ring motifs, respectively. Molecules of the title compound are bent at the S atom with a C17—S1—N1—C12 torsion angle of −70.86 (11)°. The benzene ring (C11–C16) attached to the N atom makes a dihedral angle of 77.99 (6)° with the benzene ring (C1–C6) having the OH group, and these rings make angles of 26.98 (6) and 57.58 (6)°, respectively, with the benzene ring (C17–C22) attached to the S atom. The geometric parameters of the title compound are normal and comparable to those of the related compound listed in the *Database survey* section.

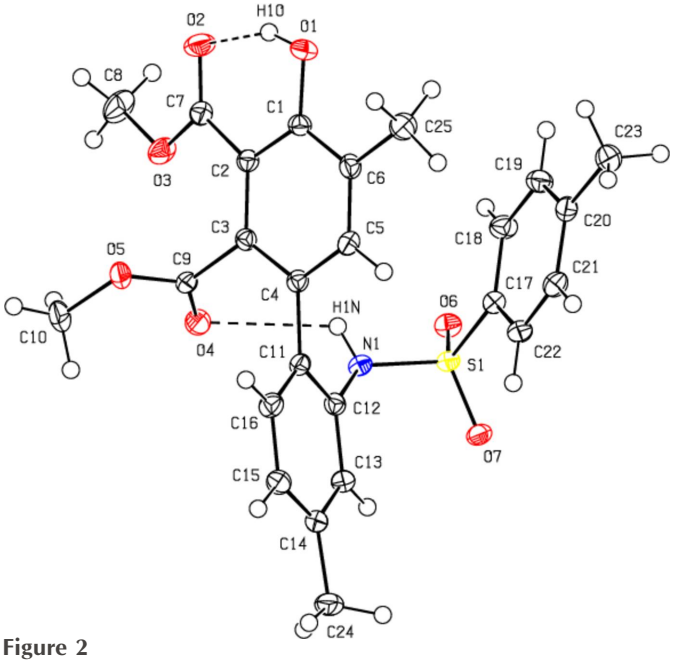


Figure 2
Structure and atomic numbering scheme of the title compound, shown as 50% probability ellipsoids.

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O4	0.836 (18)	2.507 (18)	3.0185 (14)	120.5 (15)
N1—H1*N*···O6^{i}	0.836 (18)	2.280 (18)	3.0643 (14)	156.4 (18)
O1—H1*O*···O2	0.91 (2)	1.72 (2)	2.5596 (14)	152 (2)
C24—H24*A*···O4^{ii}	0.98	2.60	3.3784 (16)	137
C25—H25*C*···O1^{iii}	0.98	2.59	3.2177 (17)	122
C16—H16···*Cg*1^{iv}	0.95	2.61	3.4780 (14)	153

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+2, -y+1, -z+1$.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecules are linked by C—H···O and N—H···O hydrogen bonds, forming molecular layers parallel to the (100) plane (Table 1, Figs. 3, 4 and 5). C—H···π interactions (Table 1) between these layers also add to the crystal cohesion.

To quantify the intermolecular interactions, a Hirshfeld surface analysis was performed and *CrystalExplorer17.5* (Spackman *et al.*, 2021) was used to generate the accompanying two-dimensional fingerprint plots. Fig. 6 shows the Hirshfeld surface mapped over d_{norm}. On the Hirshfeld surface, shorter and longer contacts are indicated by red and blue spots, respectively, and contacts with lengths about equal

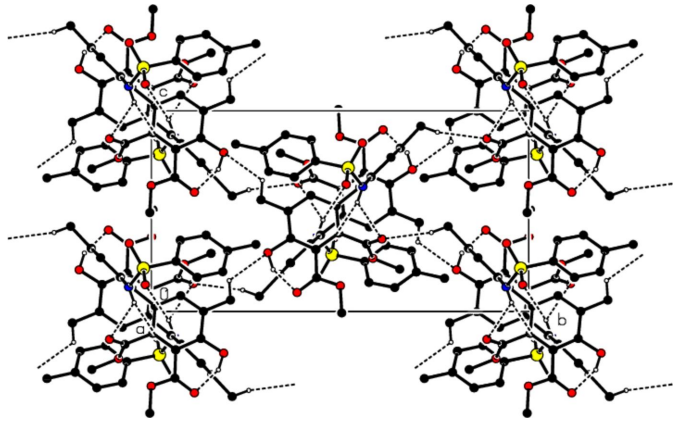


Figure 3
Packing of molecules in the title compound with the N—H···O, O–H···O and C—H···O hydrogen bonds, viewed along the *a* axis.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

Contact	Distance	Symmetry operation
H15···O1	2.67	$2-x, 1-y, 1-z$
H1*O*···H25*C*	2.40	$x, \frac{3}{2}-y, \frac{1}{2}+z$
H10*C*···O2	2.71	$2-x, 1-y, 2-z$
H1*N*···O6	2.28	$1-x, 1-y, 1-z$
O4···H24*A*	2.60	$x, \frac{1}{2}-y, \frac{1}{2}+z$
H22···H22	2.47	$1-x, 1-y, -z$
H8*C*···C15	2.65	$x, y, 1+z$
H13···H19	2.57	$1-x, -\frac{1}{2}+y, \frac{1}{2}-z$

to the sum of the van der Waals radii are indicated by white spots. The C—H···O and N—H···O interactions (Tables 1 and 2) are represented by the two most significant red spots on the d_{norm} surface.

Fig. 7 depicts the two-dimensional fingerprint plots of (d_i, d_e) points from all the contacts contributing to the Hirshfeld surface analysis in normal mode for all atoms. The most important intermolecular interactions are H···H contacts, contributing 52.3% to the overall crystal packing. Other interactions and their respective contributions are O···H/H···O (27.0%), C···H/H···C (15.2%), O···C/C···O (2.5%), O···O (2.0%) and N···H/H···N (1.1%). The Hirshfeld surface analysis confirms the significance of H-atom interactions in the packing formation. The significant frequency of H···H and O···H/H···O interactions implies that van der Waals interactions and hydrogen bonding are important in crystal packing (Hathwar *et al.*, 2015).

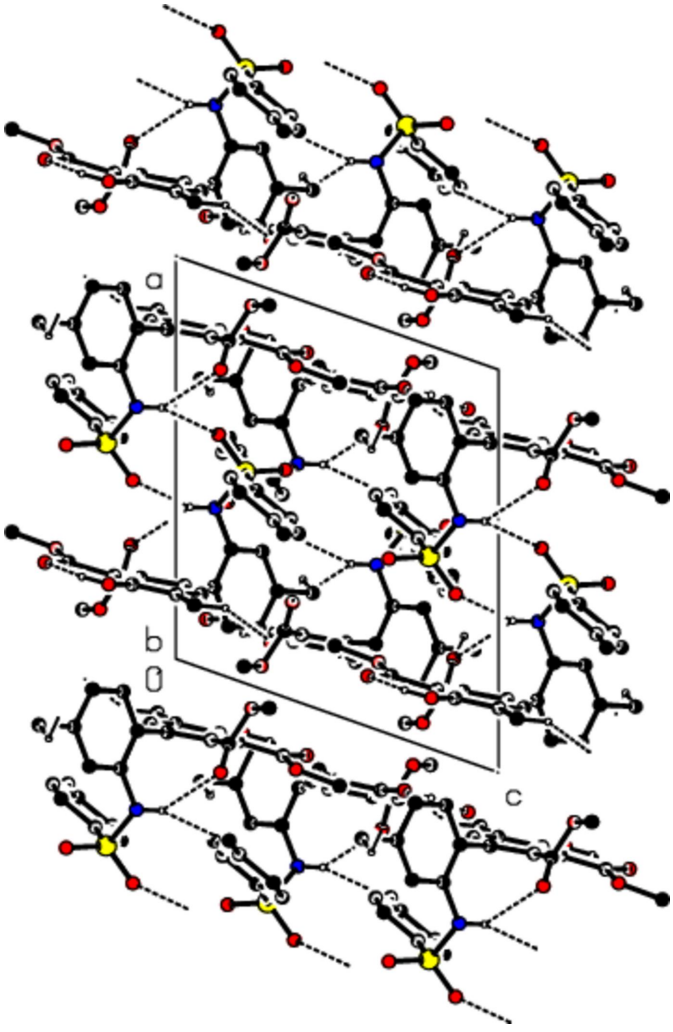


Figure 4
Packing of molecules in the title compound, viewed along the *b* axis.

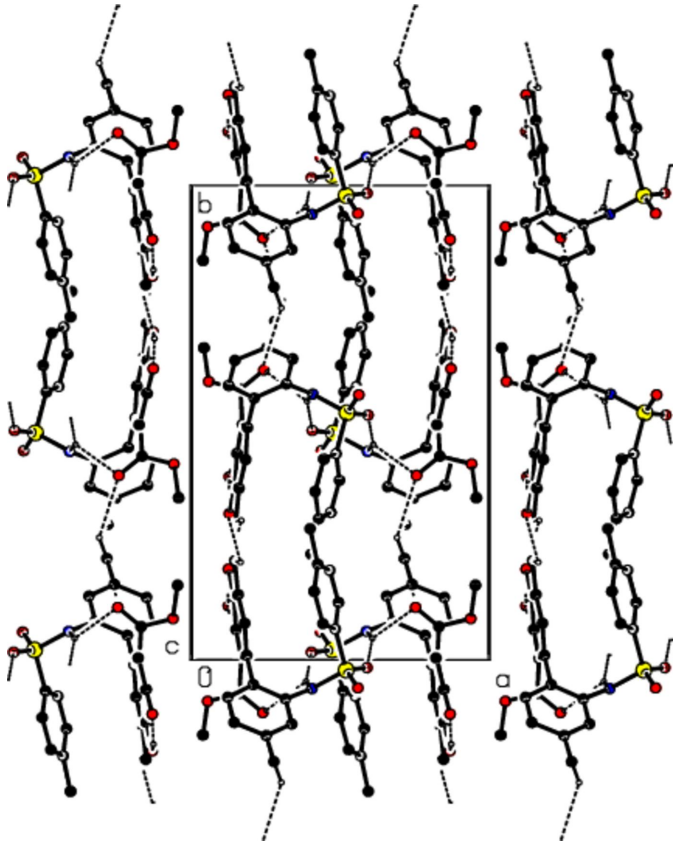


Figure 5
Packing of molecules in the title compound, viewed along the *c* axis.

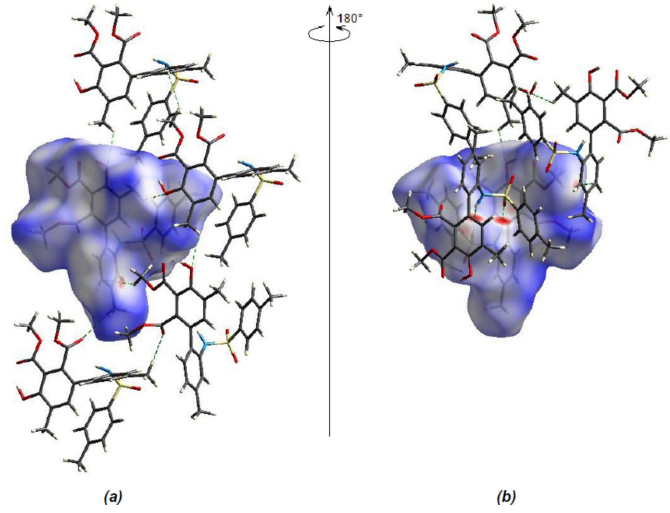


Figure 6
(*a*) Front and (*b*) back views of the three-dimensional Hirshfeld surface for the title compound.

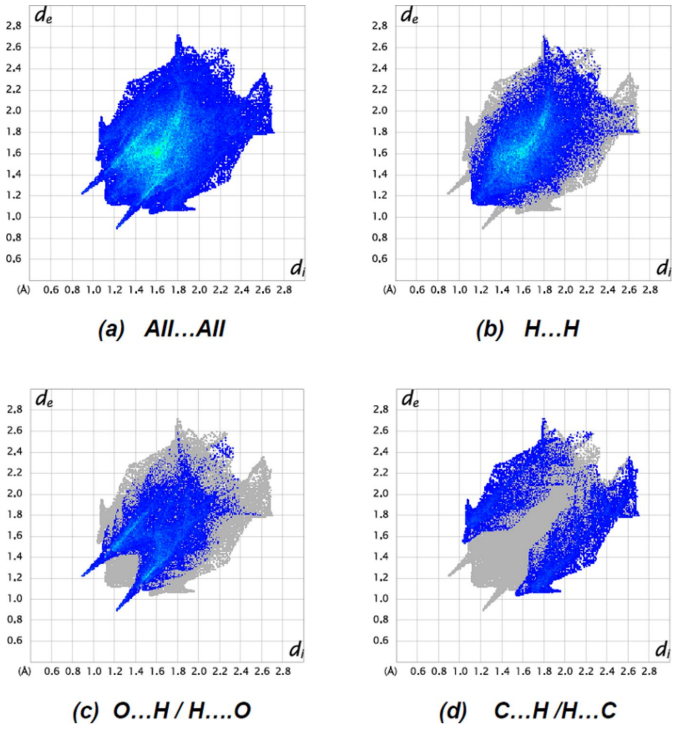


Figure 7
The two-dimensional fingerprint plots for the title compound showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) F···H/H···F, (*d*) O···H/H···O and (*e*) C···H/H···C interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{25}H_{25}NO_7S$
M_r	483.52
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	12.52978 (9), 18.87277 (12), 10.63916 (7)
β (°)	109.4092 (8)
V (Å^3)	2372.88 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.61
Crystal size (mm)	0.22 × 0.20 × 0.15
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.654, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37027, 5051, 4842
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.088, 1.03
No. of reflections	5051
No. of parameters	321
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.44

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT2016/6* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43, last update November 2022; Groom *et al.*, 2016) for the *N*,4-dimethylbenzene-1-sulfonamide unit, resulted in two hits, CSD refcodes EVOJAB (Shakuntala, *et al.*, 2011*a*) and EVOFAX (Shakuntala, *et al.*, 2011*b*).

The molecule of EVOJAB (Shakuntala, *et al.*, 2011*a*) is twisted about the N—S bond with a C—SO_2—NH—C torsion angle of 44.55 (17)°. The two aromatic rings are inclined to each other by 66.2 (1)°. In the crystal, N—H···O hydrogen bonds link the molecules into infinite chains parallel to the *b* axis. Molecules of EVOFAX (Shakuntala, *et al.*, 2011*b*), are bent at the S atom with a C—SO_2—NH—C torsion angle of 57.7 (2)°. The benzene rings are rotated relative to each other by 68.1 (1)°. In the crystal, N—H···O(S) hydrogen bonds pack the molecules into infinite chains parallel to the *b* axis.

5. Synthesis and crystallization

Dimethyl but-2-ynedioate (87.6 µL, 0.7 mmol) was added to a solution of 4-methyl-*N*-(5-methyl-2-(5-methylfuran-2-yl) phenyl)benzenesulfonamide (100 mg, 0.3 mmol) in *o*-xylene (5 mL). The mixture was refluxed for 5 h. After cooling of the reaction to r.t, the solvent was evaporated under reduced pressure and the crude product was purified by column chromatography (eluent: from hexane to ethyl acetate). The title compound was obtained as a colourless powder, yield 68%, 97 mg (0.2 mmol); m.p. > 523 K (with decomp.). A single crystal of the title compound was grown from a mixture of hexane and ethyl acetate. IR (KBr), ν (cm^{-1}): *br*. 3277 (NH, OH), 1745, 1671 (CO_2), 1353 (ν_{as} SO_2), 1238 (C—OH), 1172 (ν_s SO_2). ^{1}H NMR (700.2 MHz, $CDCl_3$) (*J*, Hz): δ 11.31 (*s*, 1H, OH), 7.52 (*s*, 1H, NH), 7.42 (*d*, *J* = 8.2, 2H, H Ar), 7.18 (*d*, *J* = 8.2, 2H, H Ar), 6.96 (*d*, *J* = 7.6, 1H, H Ar), 6.85 (*d*, *J* = 7.6, 1H, H Ar), 6.71 (*s*, 1H, H Ar), 6.08 (*s*, 1H, H Ar), 3.92 (*s*, 3H, OCH_3), 3.51 (*s*, 3H, OCH_3), 2.43 (*s*, 3H, CH_3), 2.39 (*s*, 3H, CH_3), 2.06 (*s*, 3H, CH_3). ^{13}C{^{1}H} NMR (176.1 MHz, $CDCl_3$): δ 169.5, 168.7, 159.9, 143.3, 139.4, 137.3, 136.9, 134.1, 133.0, 130.7, 129.5 (2C), 129.4, 128.5, 127.1 (2C), 126.3, 125.7, 124.7, 107.9, 53.1, 52.3, 21.6, 21.4, 16.0. MS (ESI) *m/z*: $[M + H]^+$ 484. Elemental analysis calculated (%) for $C_{25}H_{25}NO_7S$: C 62.10, H 5.21, N 2.90, S 6.63; found: C 61.87, H 5.48, N 3.09, S 6.37.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were included in the refinement using the riding-model approximation with C—H distances of 0.95–0.98 Å, and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). The H atoms of the NH and OH groups were found in a difference map and refined freely [N1—H1*N* = 0.836 (18) Å and O1—H1*O* = 0.91 (2) Å].

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supporting information

Acta Cryst. (2024). E**80**, 62-66 [https://doi.org/10.1107/S205698902301071X]

Crystal structure and Hirshfeld surface analysis of dimethyl 4-hydroxy-5,4′-dimethyl-2′-(toluene-4-sulfonylamino)biphenyl-2,3-dicarboxylate

Narmina A. Guliyeva, Gleb M. Burkin, Selbi Annadurdyyeva, Victor N. Khrustalev, Zeliha Atioğlu, Mehmet Akkurt and Ajaya Bhattarai

Computing details

Dimethyl 4-hydroxy-5,4′-dimethyl-2′-{[(4-methylphenyl)sulfonyl]amino}biphenyl-\ 2,3-dicarboxylate

Crystal data

$C_{25}H_{25}NO_7S$
$M_r = 483.52$
Monoclinic, $P2_1/c$
$a = 12.52978$ (9) Å
$b = 18.87277$ (12) Å
$c = 10.63916$ (7) Å
$\beta = 109.4092$ (8)°
$V = 2372.88$ (3) Å^3
$Z = 4$
$F(000) = 1016$
$D_x = 1.354$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 26913 reflections
$\theta = 4.4$–$77.8°$
$\mu = 1.61$ mm^{-1}
$T = 100$ K
Prism, colourless
0.22 × 0.20 × 0.15 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min} = 0.654$, $T_{max} = 1.000$
37027 measured reflections
5051 independent reflections
4842 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 77.8°$, $\theta_{min} = 3.7°$
$h = -15\rightarrow15$
$k = -22\rightarrow23$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.088$
$S = 1.03$
5051 reflections
321 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 1.207P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00097 (9)

Special details

Experimental. CrysAlisPro 1.171.41.117a (Rigaku OD, 2021) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.47540 (2)	0.47876 (2)	0.28445 (3)	0.01572 (9)
O1	0.87334 (8)	0.69147 (5)	0.70753 (10)	0.0219 (2)
H1O	0.8780 (19)	0.6776 (12)	0.791 (2)	0.054 (6)*
O2	0.87491 (9)	0.61396 (5)	0.90556 (9)	0.0289 (2)
O3	0.83061 (9)	0.49945 (5)	0.87207 (9)	0.0254 (2)
O4	0.75736 (8)	0.38919 (5)	0.63886 (9)	0.0229 (2)
O5	0.94306 (8)	0.41300 (5)	0.73522 (9)	0.0245 (2)
O6	0.40571 (7)	0.48386 (5)	0.36703 (9)	0.01983 (19)
O7	0.43510 (7)	0.44113 (5)	0.16051 (8)	0.01987 (19)
N1	0.59121 (9)	0.43960 (5)	0.37778 (10)	0.0167 (2)
H1N	0.6110 (15)	0.4544 (9)	0.4562 (18)	0.028 (4)*
C1	0.85341 (10)	0.63093 (6)	0.63507 (12)	0.0172 (2)
C2	0.84786 (10)	0.56433 (6)	0.69114 (12)	0.0165 (2)
C3	0.82958 (10)	0.50356 (6)	0.60943 (12)	0.0155 (2)
C4	0.81082 (10)	0.51001 (6)	0.47363 (12)	0.0159 (2)
C5	0.81682 (10)	0.57750 (7)	0.42116 (12)	0.0181 (2)
H5	0.8049	0.5819	0.3285	0.022*
C6	0.83940 (10)	0.63786 (6)	0.49899 (12)	0.0188 (2)
C7	0.85359 (10)	0.56214 (7)	0.83223 (12)	0.0193 (2)
C8	0.82815 (14)	0.49568 (9)	1.00727 (14)	0.0332 (3)
H8A	0.7721	0.5293	1.0178	0.050*
H8B	0.9030	0.5076	1.0700	0.050*
H8C	0.8077	0.4476	1.0254	0.050*
C9	0.83582 (10)	0.42959 (6)	0.66379 (11)	0.0175 (2)
C10	0.95930 (14)	0.34207 (8)	0.78924 (16)	0.0360 (4)
H10A	0.9373	0.3076	0.7161	0.054*
H10B	0.9125	0.3352	0.8460	0.054*
H10C	1.0391	0.3352	0.8423	0.054*
C11	0.78777 (10)	0.44813 (6)	0.38084 (11)	0.0161 (2)
C12	0.67941 (10)	0.41792 (6)	0.32872 (11)	0.0160 (2)
C13	0.65792 (10)	0.36524 (6)	0.23203 (12)	0.0183 (2)
H13	0.5840	0.3457	0.1970	0.022*
C14	0.74280 (11)	0.34054 (7)	0.18550 (12)	0.0193 (2)
C15	0.85138 (11)	0.36831 (7)	0.24215 (13)	0.0210 (3)
H15	0.9113	0.3506	0.2151	0.025*
C16	0.87306 (10)	0.42142 (7)	0.33738 (12)	0.0201 (3)
H16	0.9475	0.4399	0.3737	0.024*

C17	0.51190 (10)	0.56517 (6)	0.25010 (12)	0.0178 (2)
C18	0.50126 (11)	0.62124 (7)	0.32991 (13)	0.0222 (3)
H18	0.4745	0.6133	0.4024	0.027*
C19	0.53028 (12)	0.68894 (7)	0.30225 (13)	0.0224 (3)
H19	0.5228	0.7273	0.3565	0.027*
C20	0.57021 (10)	0.70204 (7)	0.19664 (12)	0.0198 (2)
C21	0.58170 (11)	0.64480 (7)	0.11927 (12)	0.0211 (3)
H21	0.6100	0.6526	0.0480	0.025*
C22	0.55246 (11)	0.57649 (7)	0.14447 (12)	0.0197 (2)
H22	0.5600	0.5380	0.0904	0.024*
C23	0.59565 (12)	0.77675 (7)	0.16557 (13)	0.0250 (3)
H23A	0.5250	0.8006	0.1147	0.037*
H23B	0.6466	0.7758	0.1128	0.037*
H23C	0.6319	0.8025	0.2489	0.037*
C24	0.71670 (12)	0.28600 (7)	0.07589 (14)	0.0264 (3)
H24A	0.6916	0.2420	0.1064	0.040*
H24B	0.7849	0.2767	0.0528	0.040*
H24C	0.6566	0.3038	−0.0027	0.040*
C25	0.85016 (13)	0.70949 (7)	0.44329 (14)	0.0273 (3)
H25A	0.9281	0.7264	0.4826	0.041*
H25B	0.7985	0.7427	0.4645	0.041*
H25C	0.8308	0.7061	0.3463	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01611 (15)	0.01641 (15)	0.01492 (15)	−0.00118 (10)	0.00555 (11)	−0.00276 (10)
O1	0.0258 (5)	0.0150 (4)	0.0243 (5)	−0.0007 (3)	0.0075 (4)	−0.0048 (3)
O2	0.0414 (6)	0.0258 (5)	0.0193 (4)	−0.0024 (4)	0.0098 (4)	−0.0067 (4)
O3	0.0356 (5)	0.0253 (5)	0.0150 (4)	−0.0039 (4)	0.0081 (4)	0.0016 (4)
O4	0.0268 (5)	0.0180 (4)	0.0221 (4)	−0.0035 (4)	0.0057 (4)	0.0021 (3)
O5	0.0248 (5)	0.0180 (4)	0.0243 (5)	0.0032 (4)	−0.0005 (4)	0.0032 (4)
O6	0.0185 (4)	0.0218 (4)	0.0211 (4)	−0.0015 (3)	0.0091 (4)	−0.0028 (3)
O7	0.0205 (4)	0.0210 (4)	0.0166 (4)	−0.0011 (3)	0.0041 (3)	−0.0050 (3)
N1	0.0180 (5)	0.0184 (5)	0.0138 (5)	−0.0011 (4)	0.0056 (4)	−0.0025 (4)
C1	0.0146 (5)	0.0162 (6)	0.0198 (6)	0.0002 (4)	0.0043 (4)	−0.0032 (4)
C2	0.0148 (5)	0.0173 (6)	0.0166 (5)	0.0004 (4)	0.0043 (4)	−0.0011 (4)
C3	0.0139 (5)	0.0153 (6)	0.0166 (5)	0.0002 (4)	0.0042 (4)	0.0002 (4)
C4	0.0138 (5)	0.0169 (6)	0.0163 (5)	−0.0005 (4)	0.0041 (4)	−0.0002 (4)
C5	0.0176 (5)	0.0202 (6)	0.0148 (5)	−0.0012 (4)	0.0033 (4)	0.0009 (4)
C6	0.0176 (6)	0.0165 (6)	0.0202 (6)	−0.0004 (4)	0.0036 (5)	0.0026 (5)
C7	0.0182 (6)	0.0207 (6)	0.0178 (6)	0.0016 (5)	0.0042 (5)	−0.0006 (5)
C8	0.0423 (8)	0.0419 (9)	0.0158 (6)	−0.0051 (7)	0.0099 (6)	0.0034 (6)
C9	0.0223 (6)	0.0167 (6)	0.0125 (5)	0.0014 (5)	0.0044 (4)	−0.0001 (4)
C10	0.0417 (9)	0.0202 (7)	0.0336 (8)	0.0070 (6)	−0.0043 (6)	0.0068 (6)
C11	0.0179 (6)	0.0163 (6)	0.0130 (5)	−0.0001 (4)	0.0036 (4)	0.0005 (4)
C12	0.0178 (5)	0.0152 (5)	0.0153 (5)	0.0009 (4)	0.0059 (4)	0.0014 (4)
C13	0.0186 (6)	0.0170 (6)	0.0183 (6)	−0.0010 (4)	0.0045 (5)	−0.0016 (4)

C14	0.0223 (6)	0.0170 (6)	0.0169 (5)	0.0028 (5)	0.0043 (5)	−0.0011 (4)
C15	0.0199 (6)	0.0229 (6)	0.0206 (6)	0.0042 (5)	0.0074 (5)	−0.0020 (5)
C16	0.0175 (6)	0.0226 (6)	0.0193 (6)	−0.0008 (5)	0.0048 (5)	−0.0013 (5)
C17	0.0181 (6)	0.0178 (6)	0.0163 (5)	0.0003 (4)	0.0042 (4)	−0.0008 (4)
C18	0.0284 (7)	0.0220 (6)	0.0190 (6)	−0.0007 (5)	0.0114 (5)	−0.0021 (5)
C19	0.0293 (7)	0.0184 (6)	0.0203 (6)	0.0013 (5)	0.0094 (5)	−0.0023 (5)
C20	0.0199 (6)	0.0192 (6)	0.0177 (6)	0.0006 (5)	0.0029 (5)	0.0018 (5)
C21	0.0245 (6)	0.0244 (6)	0.0150 (5)	0.0001 (5)	0.0073 (5)	0.0006 (5)
C22	0.0230 (6)	0.0206 (6)	0.0161 (5)	0.0007 (5)	0.0072 (5)	−0.0027 (5)
C23	0.0301 (7)	0.0206 (6)	0.0233 (6)	−0.0013 (5)	0.0076 (5)	0.0032 (5)
C24	0.0261 (7)	0.0250 (7)	0.0262 (7)	0.0035 (5)	0.0062 (5)	−0.0093 (5)
C25	0.0354 (7)	0.0187 (6)	0.0243 (7)	−0.0053 (5)	0.0054 (6)	0.0031 (5)

Geometric parameters (Å, º)

S1—O6	1.4330 (9)	C11—C16	1.3922 (17)
S1—O7	1.4338 (9)	C11—C12	1.4056 (17)
S1—N1	1.6372 (11)	C12—C13	1.3911 (17)
S1—C17	1.7643 (13)	C13—C14	1.3940 (17)
O1—C1	1.3543 (14)	C13—H13	0.9500
O1—H1O	0.91 (2)	C14—C15	1.3942 (18)
O2—C7	1.2239 (16)	C14—C24	1.5076 (17)
O3—C7	1.3203 (16)	C15—C16	1.3864 (18)
O3—C8	1.4507 (16)	C15—H15	0.9500
O4—C9	1.2020 (16)	C16—H16	0.9500
O5—C9	1.3425 (15)	C17—C18	1.3907 (17)
O5—C10	1.4444 (16)	C17—C22	1.3952 (17)
N1—C12	1.4298 (15)	C18—C19	1.3864 (18)
N1—H1N	0.836 (18)	C18—H18	0.9500
C1—C2	1.4029 (17)	C19—C20	1.3955 (18)
C1—C6	1.4054 (17)	C19—H19	0.9500
C2—C3	1.4112 (16)	C20—C21	1.3943 (18)
C2—C7	1.4794 (17)	C20—C23	1.5064 (17)
C3—C4	1.3899 (16)	C21—C22	1.3904 (18)
C3—C9	1.5031 (16)	C21—H21	0.9500
C4—C5	1.4028 (17)	C22—H22	0.9500
C4—C11	1.4941 (16)	C23—H23A	0.9800
C5—C6	1.3811 (17)	C23—H23B	0.9800
C5—H5	0.9500	C23—H23C	0.9800
C6—C25	1.4999 (17)	C24—H24A	0.9800
C8—H8A	0.9800	C24—H24B	0.9800
C8—H8B	0.9800	C24—H24C	0.9800
C8—H8C	0.9800	C25—H25A	0.9800
C10—H10A	0.9800	C25—H25B	0.9800
C10—H10B	0.9800	C25—H25C	0.9800
C10—H10C	0.9800		
O6—S1—O7	119.80 (5)	C13—C12—C11	120.39 (11)

O6—S1—N1	104.85 (5)	C13—C12—N1	119.43 (11)
O7—S1—N1	107.72 (5)	C11—C12—N1	120.16 (10)
O6—S1—C17	108.52 (6)	C12—C13—C14	121.23 (11)
O7—S1—C17	107.68 (6)	C12—C13—H13	119.4
N1—S1—C17	107.72 (6)	C14—C13—H13	119.4
C1—O1—H1O	104.7 (14)	C13—C14—C15	118.09 (11)
C7—O3—C8	116.17 (11)	C13—C14—C24	120.53 (11)
C9—O5—C10	115.05 (11)	C15—C14—C24	121.37 (11)
C12—N1—S1	123.00 (8)	C16—C15—C14	120.91 (11)
C12—N1—H1N	116.8 (12)	C16—C15—H15	119.5
S1—N1—H1N	111.3 (12)	C14—C15—H15	119.5
O1—C1—C2	122.66 (11)	C15—C16—C11	121.26 (11)
O1—C1—C6	116.37 (11)	C15—C16—H16	119.4
C2—C1—C6	120.97 (11)	C11—C16—H16	119.4
C1—C2—C3	119.15 (11)	C18—C17—C22	120.65 (12)
C1—C2—C7	117.65 (11)	C18—C17—S1	119.52 (10)
C3—C2—C7	123.06 (11)	C22—C17—S1	119.82 (9)
C4—C3—C2	120.39 (11)	C19—C18—C17	119.11 (12)
C4—C3—C9	116.77 (10)	C19—C18—H18	120.4
C2—C3—C9	122.74 (10)	C17—C18—H18	120.4
C3—C4—C5	118.70 (11)	C18—C19—C20	121.58 (12)
C3—C4—C11	123.18 (11)	C18—C19—H19	119.2
C5—C4—C11	118.10 (10)	C20—C19—H19	119.2
C6—C5—C4	122.53 (11)	C21—C20—C19	118.23 (12)
C6—C5—H5	118.7	C21—C20—C23	121.65 (12)
C4—C5—H5	118.7	C19—C20—C23	120.08 (12)
C5—C6—C1	118.13 (11)	C22—C21—C20	121.27 (11)
C5—C6—C25	122.27 (11)	C22—C21—H21	119.4
C1—C6—C25	119.60 (11)	C20—C21—H21	119.4
O2—C7—O3	122.41 (12)	C21—C22—C17	119.15 (11)
O2—C7—C2	123.45 (12)	C21—C22—H22	120.4
O3—C7—C2	114.12 (11)	C17—C22—H22	120.4
O3—C8—H8A	109.5	C20—C23—H23A	109.5
O3—C8—H8B	109.5	C20—C23—H23B	109.5
H8A—C8—H8B	109.5	H23A—C23—H23B	109.5
O3—C8—H8C	109.5	C20—C23—H23C	109.5
H8A—C8—H8C	109.5	H23A—C23—H23C	109.5
H8B—C8—H8C	109.5	H23B—C23—H23C	109.5
O4—C9—O5	124.64 (11)	C14—C24—H24A	109.5
O4—C9—C3	124.75 (11)	C14—C24—H24B	109.5
O5—C9—C3	110.41 (10)	H24A—C24—H24B	109.5
O5—C10—H10A	109.5	C14—C24—H24C	109.5
O5—C10—H10B	109.5	H24A—C24—H24C	109.5
H10A—C10—H10B	109.5	H24B—C24—H24C	109.5
O5—C10—H10C	109.5	C6—C25—H25A	109.5
H10A—C10—H10C	109.5	C6—C25—H25B	109.5
H10B—C10—H10C	109.5	H25A—C25—H25B	109.5
C16—C11—C12	118.01 (11)	C6—C25—H25C	109.5

C16—C11—C4	120.09 (11)	H25A—C25—H25C	109.5
C12—C11—C4	121.80 (11)	H25B—C25—H25C	109.5
O6—S1—N1—C12	173.68 (9)	C3—C4—C11—C16	−102.44 (14)
O7—S1—N1—C12	45.03 (11)	C5—C4—C11—C16	75.74 (15)
C17—S1—N1—C12	−70.86 (11)	C3—C4—C11—C12	81.40 (15)
O1—C1—C2—C3	−178.41 (11)	C5—C4—C11—C12	−100.42 (14)
C6—C1—C2—C3	0.95 (17)	C16—C11—C12—C13	−2.95 (17)
O1—C1—C2—C7	5.80 (17)	C4—C11—C12—C13	173.28 (11)
C6—C1—C2—C7	−174.84 (11)	C16—C11—C12—N1	175.10 (11)
C1—C2—C3—C4	−3.68 (17)	C4—C11—C12—N1	−8.66 (17)
C7—C2—C3—C4	171.87 (11)	S1—N1—C12—C13	−65.05 (14)
C1—C2—C3—C9	172.71 (11)	S1—N1—C12—C11	116.88 (11)
C7—C2—C3—C9	−11.74 (18)	C11—C12—C13—C14	0.72 (18)
C2—C3—C4—C5	3.56 (17)	N1—C12—C13—C14	−177.35 (11)
C9—C3—C4—C5	−173.04 (10)	C12—C13—C14—C15	2.31 (19)
C2—C3—C4—C11	−178.27 (11)	C12—C13—C14—C24	−176.98 (12)
C9—C3—C4—C11	5.13 (17)	C13—C14—C15—C16	−3.09 (19)
C3—C4—C5—C6	−0.73 (18)	C24—C14—C15—C16	176.20 (12)
C11—C4—C5—C6	−178.99 (11)	C14—C15—C16—C11	0.8 (2)
C4—C5—C6—C1	−1.93 (18)	C12—C11—C16—C15	2.20 (18)
C4—C5—C6—C25	177.57 (12)	C4—C11—C16—C15	−174.11 (11)
O1—C1—C6—C5	−178.81 (11)	O6—S1—C17—C18	18.25 (12)
C2—C1—C6—C5	1.80 (18)	O7—S1—C17—C18	149.32 (10)
O1—C1—C6—C25	1.68 (17)	N1—S1—C17—C18	−94.76 (11)
C2—C1—C6—C25	−177.72 (12)	O6—S1—C17—C22	−162.58 (10)
C8—O3—C7—O2	2.03 (19)	O7—S1—C17—C22	−31.51 (12)
C8—O3—C7—C2	−176.26 (11)	N1—S1—C17—C22	84.42 (11)
C1—C2—C7—O2	−8.10 (18)	C22—C17—C18—C19	0.8 (2)
C3—C2—C7—O2	176.29 (12)	S1—C17—C18—C19	179.92 (10)
C1—C2—C7—O3	170.16 (11)	C17—C18—C19—C20	−0.2 (2)
C3—C2—C7—O3	−5.45 (17)	C18—C19—C20—C21	−0.7 (2)
C10—O5—C9—O4	−3.35 (18)	C18—C19—C20—C23	177.09 (12)
C10—O5—C9—C3	−178.35 (11)	C19—C20—C21—C22	1.12 (19)
C4—C3—C9—O4	−68.07 (16)	C23—C20—C21—C22	−176.63 (12)
C2—C3—C9—O4	115.42 (14)	C20—C21—C22—C17	−0.61 (19)
C4—C3—C9—O5	106.91 (12)	C18—C17—C22—C21	−0.35 (19)
C2—C3—C9—O5	−69.60 (14)	S1—C17—C22—C21	−179.51 (9)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O4	0.836 (18)	2.507 (18)	3.0185 (14)	120.5 (15)
N1—H1*N*···O6^{i}	0.836 (18)	2.280 (18)	3.0643 (14)	156.4 (18)
O1—H1*O*···O2	0.91 (2)	1.72 (2)	2.5596 (14)	152 (2)
C13—H13···O7	0.95	2.53	3.0022 (16)	111
C18—H18···O6	0.95	2.58	2.9370 (16)	103

C24—H24*A*···O4^{ii}	0.98	2.60	3.3784 (16)	137
C25—H25*C*···O1^{iii}	0.98	2.59	3.2177 (17)	122
C16—H16···*Cg*1^{iv}	0.95	2.61	3.4780 (14)	153

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*−1/2; (iv) −*x*+2, −*y*+1, −*z*+1.