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ISSN 2414-3146

Naphthalene-2,6-diyl bis(4-methylbenzenesulfonate)

Aleksandra Piontek, Dawid Siodłak and Bartosz Zarychta*

Faculty of Chemistry, University of Opole, Oleska 48, 45-052 Opole, Poland. *Correspondence e-mail: bzarychta@uni.opole.pl

Received 12 June 2018
Accepted 18 June 2018

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

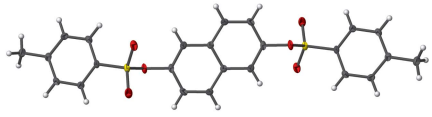
Keywords: tosylates; crystal structure; cross-coupling reactions.

CCDC reference: 1849908

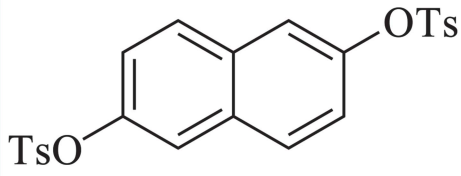
Structural data: full structural data are available from iucrdata.iucr.org

The complete molecule of the title compound, $C_{24}H_{20}O_6S_2$, is generated by a crystallographic inversion centre at the middle of the naphthalene ring system. The dihedral angle between the naphthalene ring system and the pendant benzene ring is 10.23 (6)° and the C—S—O—C torsion angle is −172.05 (10)°. In the crystal, weak C—H···O interactions link the molecules into $(10\bar{1})$ sheets.

3D view



Chemical scheme



Structure description

Aryl tosylates have attracted considerable attention as electrophiles in transition-metal catalyzed cross-coupling reactions (Piontek & Szostak, 2017; Chen *et al.*, 2015; Ackermann *et al.*, 2006). The use of these compounds can have advantages over the corresponding aryl halides in that the phenol group is a useful directing group for the introduction of other functional groups on the aromatic ring and as such can allow access to a wider substrate scope (Bisz & Szostak, 2017*a*,*b*, 2018; Ackermann *et al.*, 2006).

The asymmetric unit of the title compound consists of one independent half-molecule. The complete molecule is generated by an inversion centre at the middle of the C4—C4$(2-x, 1-y, 1-z)$ bond. The molecular structure is shown in Fig. 1.

In the crystal, C—H···O hydrogen bonds (Table 1) connect the molecules into $(10\bar{1})$ sheets (Fig. 2).

Synthesis and crystallization

The title compound was synthesized according to the procedure described by Murai *et al.* (2012). Diethyl ether (0.8 ml) was placed in a reaction vial (8 ml) provided with a rubber septum. The title compound was added to the diethyl ether until a saturated solution was obtained. The resulting solution was then heated and left to stand in a refrigerator (−20°C) and colourless irregular crystals formed.

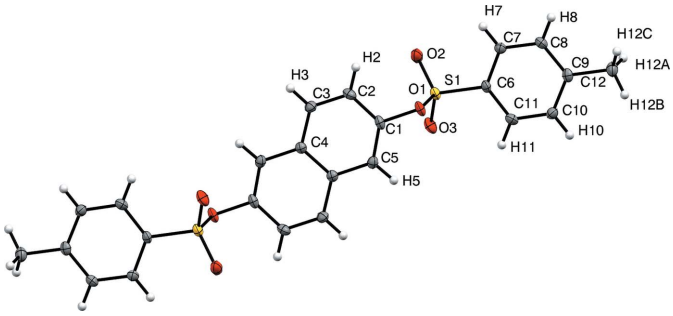


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are generated by the symmetry operation 2 − x, 1 − y, 1 − z.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

We gratefully acknowledge Narodowe Centrum Nauki (grant No. 2014/15/D/ST5/02731).

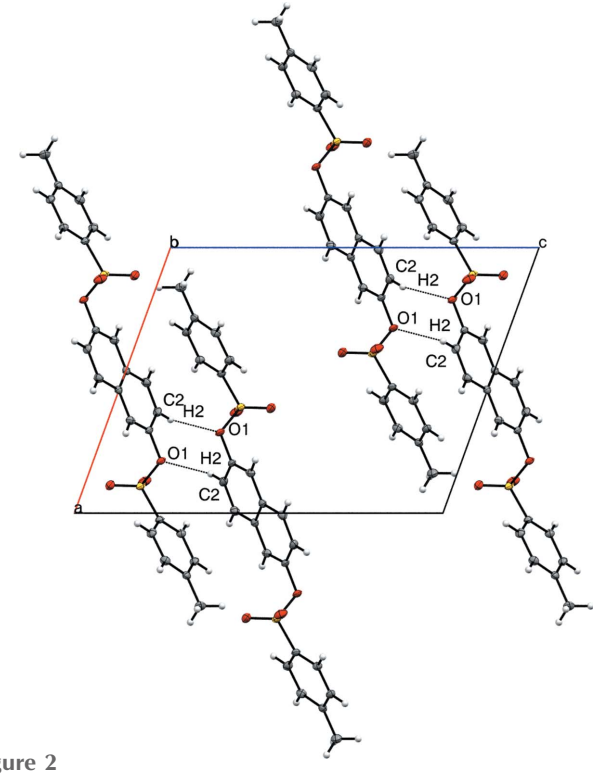


Figure 2
The crystal packing of the title compound, viewed along the *b* axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{i}	0.93	2.46	3.3261 (18)	156

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{20}O_6S_2$
M_r	468.52
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, *b*, *c* (Å)	12.2270 (3), 5.7229 (1), 15.9353 (5)
β (°)	109.869 (3)
V (Å^3)	1048.68 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.30
Crystal size (mm)	0.5 × 0.45 × 0.4
Data collection	
Diffractometer	Oxford Diffraction Xcalibur
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6789, 2046, 1806
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.082, 1.08
No. of reflections	2046
No. of parameters	146
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.34

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2008), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2008).

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full crystallographic data

IUCrData (2018). **3**, x180890 [https://doi.org/10.1107/S2414314618008908]

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Crystal data

$C_{24}H_{20}O_6S_2$
M_r = 468.52
Monoclinic, $P2_1/n$
a = 12.2270 (3) Å
b = 5.7229 (1) Å
c = 15.9353 (5) Å
β = 109.869 (3)°
V = 1048.68 (5) Å^3
Z = 2
$F(000)$ = 488
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6789 reflections
θ = 3.5–26.0°
μ = 0.30 mm^{-1}
T = 100 K
Irregular, colourless
0.5 × 0.45 × 0.4 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 1024 x 1024 with blocks 2 x 2 pixels mm^{-1}
ω scan
6789 measured reflections
2046 independent reflections
1806 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.0°, θ_{min} = 3.5°
h = −15→15
k = −6→7
l = −19→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.082
S = 1.08
2046 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.3531P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

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. All H atoms were found in a difference map but set to idealized positions and treated as riding with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for C—H and with C—H$_3$ = 0.96 Å and U_{iso} = 1.5U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.60178 (3)	0.69746 (7)	0.34689 (2)	0.01589 (13)
O1	0.69632 (9)	0.56049 (19)	0.31607 (7)	0.0179 (3)
O2	0.61916 (9)	0.9428 (2)	0.34060 (7)	0.0237 (3)
O3	0.60474 (9)	0.6029 (2)	0.43036 (7)	0.0226 (3)
C1	0.81150 (12)	0.5696 (3)	0.37842 (9)	0.0152 (3)
C2	0.88107 (13)	0.7606 (3)	0.37395 (10)	0.0180 (3)
H2	0.8523	0.8769	0.3314	0.022*
C3	0.99199 (13)	0.7727 (3)	0.43334 (10)	0.0171 (3)
H3	1.0392	0.8981	0.4308	0.021*
C4	0.96386 (12)	0.4036 (3)	0.50119 (9)	0.0137 (3)
C5	0.84882 (13)	0.3929 (3)	0.43857 (10)	0.0157 (3)
H5	0.8002	0.2680	0.4387	0.019*
C6	0.47502 (12)	0.6087 (3)	0.26206 (10)	0.0145 (3)
C7	0.42327 (13)	0.7571 (3)	0.19026 (10)	0.0156 (3)
H7	0.4582	0.8978	0.1848	0.019*
C8	0.31833 (13)	0.6905 (3)	0.12701 (10)	0.0162 (3)
H8	0.2827	0.7883	0.0788	0.019*
C9	0.26556 (12)	0.4792 (3)	0.13462 (10)	0.0159 (3)
C10	0.32065 (13)	0.3337 (3)	0.20673 (10)	0.0170 (3)
H10	0.2866	0.1916	0.2119	0.020*
C11	0.42491 (13)	0.3965 (3)	0.27083 (10)	0.0173 (3)
H11	0.4608	0.2984	0.3189	0.021*
C12	0.15007 (14)	0.4100 (3)	0.06714 (11)	0.0222 (4)
H12A	0.0889	0.4959	0.0782	0.033*
H12B	0.1378	0.2456	0.0721	0.033*
H12C	0.1500	0.4445	0.0082	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0119 (2)	0.0182 (2)	0.0144 (2)	0.00249 (14)	0.00038 (15)	−0.00300 (14)
O1	0.0111 (5)	0.0247 (6)	0.0138 (5)	0.0040 (4)	−0.0010 (4)	−0.0040 (4)
O2	0.0192 (6)	0.0180 (6)	0.0273 (6)	0.0006 (5)	−0.0008 (5)	−0.0049 (5)
O3	0.0187 (6)	0.0327 (7)	0.0141 (5)	0.0033 (5)	0.0024 (4)	−0.0015 (5)
C1	0.0104 (7)	0.0217 (8)	0.0115 (7)	0.0021 (6)	0.0011 (6)	−0.0036 (6)
C2	0.0187 (8)	0.0200 (8)	0.0148 (7)	0.0046 (6)	0.0051 (6)	0.0052 (6)
C3	0.0164 (7)	0.0168 (8)	0.0190 (8)	−0.0009 (6)	0.0072 (6)	0.0028 (6)
C4	0.0135 (7)	0.0141 (7)	0.0140 (7)	0.0012 (6)	0.0054 (6)	−0.0014 (6)
C5	0.0131 (7)	0.0160 (8)	0.0179 (8)	−0.0006 (6)	0.0051 (6)	−0.0027 (6)
C6	0.0102 (7)	0.0169 (8)	0.0145 (7)	0.0017 (6)	0.0016 (6)	−0.0013 (6)
C7	0.0152 (7)	0.0146 (7)	0.0167 (8)	0.0010 (6)	0.0049 (6)	0.0003 (6)
C8	0.0151 (7)	0.0178 (8)	0.0142 (7)	0.0037 (6)	0.0031 (6)	0.0025 (6)
C9	0.0126 (7)	0.0198 (8)	0.0160 (7)	0.0009 (6)	0.0058 (6)	−0.0031 (6)
C10	0.0171 (8)	0.0142 (8)	0.0205 (8)	−0.0014 (6)	0.0073 (6)	−0.0004 (6)
C11	0.0171 (8)	0.0166 (8)	0.0168 (7)	0.0042 (6)	0.0038 (6)	0.0031 (6)

C12	0.0164 (8)	0.0271 (9)	0.0210 (8)	−0.0036 (7)	0.0036 (6)	−0.0012 (7)

Geometric parameters (Å, º)

S1—O3	1.4249 (12)	C6—C11	1.389 (2)
S1—O2	1.4285 (12)	C6—C7	1.392 (2)
S1—O1	1.6056 (11)	C7—C8	1.389 (2)
S1—C6	1.7503 (15)	C7—H7	0.9300
O1—C1	1.4220 (17)	C8—C9	1.395 (2)
C1—C5	1.361 (2)	C8—H8	0.9300
C1—C2	1.401 (2)	C9—C10	1.393 (2)
C2—C3	1.366 (2)	C9—C12	1.508 (2)
C2—H2	0.9300	C10—C11	1.383 (2)
C3—C4^{i}	1.419 (2)	C10—H10	0.9300
C3—H3	0.9300	C11—H11	0.9300
C4—C3^{i}	1.419 (2)	C12—H12A	0.9600
C4—C4^{i}	1.422 (3)	C12—H12B	0.9600
C4—C5	1.423 (2)	C12—H12C	0.9600
C5—H5	0.9300		
O3—S1—O2	118.79 (7)	C11—C6—S1	118.77 (11)
O3—S1—O1	107.92 (6)	C7—C6—S1	119.57 (12)
O2—S1—O1	108.58 (7)	C8—C7—C6	118.61 (14)
O3—S1—C6	110.22 (7)	C8—C7—H7	120.7
O2—S1—C6	110.17 (7)	C6—C7—H7	120.7
O1—S1—C6	99.36 (6)	C7—C8—C9	121.02 (14)
C1—O1—S1	114.41 (9)	C7—C8—H8	119.5
C5—C1—C2	123.45 (14)	C9—C8—H8	119.5
C5—C1—O1	118.69 (13)	C10—C9—C8	118.78 (13)
C2—C1—O1	117.86 (13)	C10—C9—C12	120.27 (14)
C3—C2—C1	118.76 (14)	C8—C9—C12	120.94 (14)
C3—C2—H2	120.6	C11—C10—C9	121.33 (14)
C1—C2—H2	120.6	C11—C10—H10	119.3
C2—C3—C4^{i}	120.85 (14)	C9—C10—H10	119.3
C2—C3—H3	119.6	C10—C11—C6	118.70 (14)
C4^{i}—C3—H3	119.6	C10—C11—H11	120.6
C3^{i}—C4—C4^{i}	119.12 (16)	C6—C11—H11	120.6
C3^{i}—C4—C5	121.55 (14)	C9—C12—H12A	109.5
C4^{i}—C4—C5	119.33 (17)	C9—C12—H12B	109.5
C1—C5—C4	118.48 (14)	H12A—C12—H12B	109.5
C1—C5—H5	120.8	C9—C12—H12C	109.5
C4—C5—H5	120.8	H12A—C12—H12C	109.5
C11—C6—C7	121.55 (14)	H12B—C12—H12C	109.5
O3—S1—O1—C1	−57.12 (12)	O1—S1—C6—C11	84.27 (13)
O2—S1—O1—C1	72.86 (11)	O3—S1—C6—C7	147.51 (12)
C6—S1—O1—C1	−172.05 (10)	O2—S1—C6—C7	14.51 (14)
S1—O1—C1—C5	93.80 (14)	O1—S1—C6—C7	−99.35 (13)

S1—O1—C1—C2	−86.75 (14)	C11—C6—C7—C8	0.8 (2)
C5—C1—C2—C3	−0.6 (2)	S1—C6—C7—C8	−175.44 (11)
O1—C1—C2—C3	179.94 (13)	C6—C7—C8—C9	−0.2 (2)
C1—C2—C3—C4^{i}	−0.4 (2)	C7—C8—C9—C10	−0.6 (2)
C2—C1—C5—C4	1.0 (2)	C7—C8—C9—C12	178.42 (14)
O1—C1—C5—C4	−179.60 (12)	C8—C9—C10—C11	0.9 (2)
C3^{i}—C4—C5—C1	179.05 (14)	C12—C9—C10—C11	−178.12 (14)
C4^{i}—C4—C5—C1	−0.3 (2)	C9—C10—C11—C6	−0.3 (2)
O3—S1—C6—C11	−28.87 (14)	C7—C6—C11—C10	−0.6 (2)
O2—S1—C6—C11	−161.87 (12)	S1—C6—C11—C10	175.75 (11)

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{ii}	0.93	2.46	3.3261 (18)	156

Symmetry code: (ii) −*x*+3/2, *y*+1/2, −*z*+1/2.