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Biphenyl-4-yl 4-methylbenzenesulfonate

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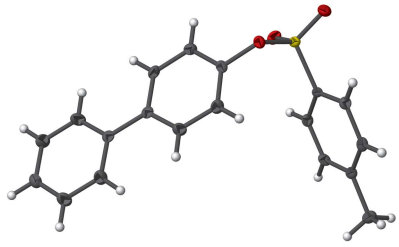
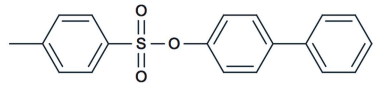
Structural data: full structural data are available from iucrdata.iucr.org

Molecules of the title compound, $C_{19}H_{16}O_3S$, are composed of a biphenyl moiety substituted with a toluene-4-sulfonate group. The dihedral angle between the two coplanar biphenyl rings and the toluene ring is 52.72 (6)°.

3D view



Chemical scheme



Structure description

Aryl tosylates are important substrates in organic synthesis (Hugo *et al.*, 2014; Xu & Zhang, 2011). Recently, the first-row metal catalysts became promising alternatives when used in cross-coupling reactions (Torborg & Beller, 2009). Aryl tosylates seem to be suitable substrates for this type of reaction (So *et al.*, 2008; Zim *et al.* 2001).

In the asymmetric unit of the title compound, there is one independent molecule. The crystal is twinned by inversion. The molecular structure is shown in Fig. 1. In the molecular structure the bond lengths and angles are within normal ranges (Allen *et al.*, 2002). The dihedral angle between the two coplanar biphenyl rings and the toluene ring is 52.72 (6)°. The crystal packing is shown in Fig. 2.

Synthesis and crystallization

Biphenyl-4-yl 4-methylbenzenesulfonate was synthesized according to a procedure described by Murai and co-workers (Murai *et al.*, 2012). The crystallization was performed in a diethyl ether solution. Diethyl ether (0.6 ml) was placed in storage reaction vials (8 ml) with silicone septa. The title compound was added in small portions until a saturated solution was obtained. The solution was warmed, and then, it was left to stand in a refrigerator (−20°C).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

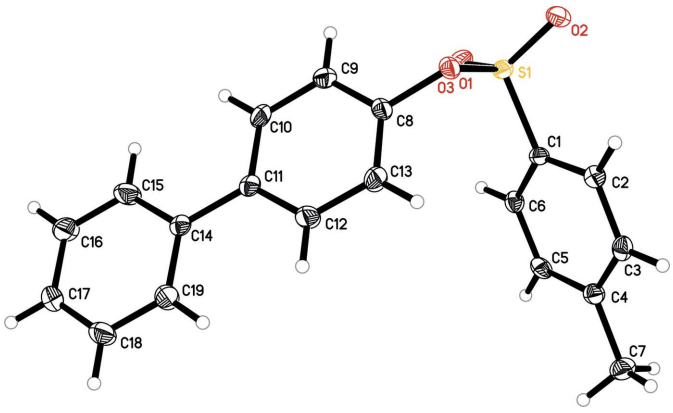


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

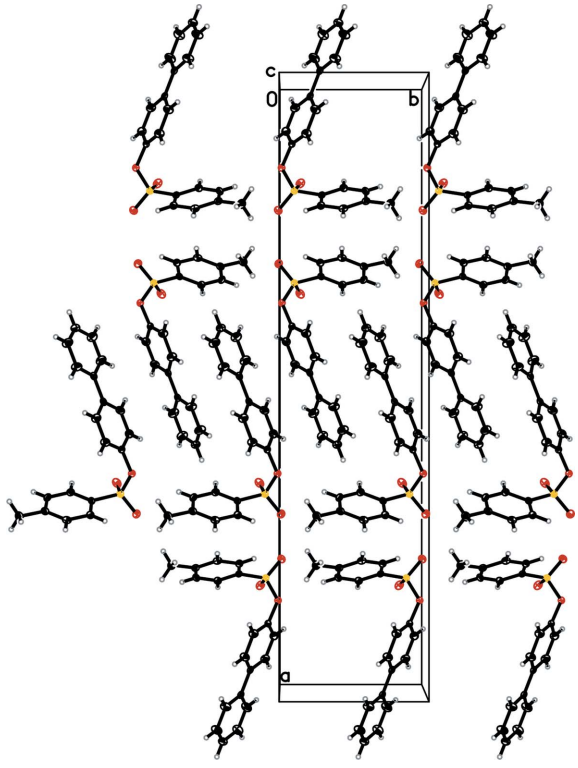


Figure 2
The crystal packing of the title compound, viewed along the *c* axis.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{16}O_3S$
M_r	324.38
Crystal system, space group	Orthorhombic, $Pca2_1$
Temperature (K)	100
a, b, c (Å)	33.2932 (10), 7.9284 (2), 5.7903 (2)
V (Å^3)	1528.42 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.23
Crystal size (mm)	0.3 × 0.25 × 0.12
Data collection	
Diffractometer	Oxford Diffraction Xcalibur
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9829, 2222, 1978
R_{int}	0.027
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.057, 0.98
No. of reflections	2222
No. of parameters	210
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.27
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.32 (11)

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2008), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *SHELXTL* (Sheldrick, 2008).

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Hugo, A. G., Jimena, M. M., Gladys, M. C., Carlos, E. T. & Carlos, R. P. (2014). *Bioorg. Med. Chem. Lett.* **24**, 760–764.
Murai, N., Miyano, M., Yonaga, M. & Tanaka, K. (2012). *Org. Lett.* **14**, 2818–2821.
Oxford Diffraction (2008). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
So, C. M., Lau, C. P., Chan, A. S. C. & Kwong, F. Y. (2008). *J. Org. Chem.* **73**, 7731–7734.
Torborg, C. & Beller, M. (2009). *Adv. Synth. Catal.* **351**, 3027–3043.
Xu, H. & Zhang, J.-L. (2011). *Bioorg. Med. Chem. Lett.* **21**, 5177–5180.
Zim, D., Lando, V. R., Dupont, J. & Monteiro, A. L. (2001). *Org. Lett.* **3**, 3049–3051.

full crystallographic data

IUCrData (2017). **2**, x170982 [https://doi.org/10.1107/S2414314617009828]

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Crystal data

$C_{19}H_{16}O_3S$
M_r = 324.38
Orthorhombic, $Pca2_1$
a = 33.2932 (10) Å
b = 7.9284 (2) Å
c = 5.7903 (2) Å
V = 1528.42 (8) Å^3
Z = 4
F(000) = 680
D_x = 1.410 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9829 reflections
θ = 3.2–26.0°
μ = 0.23 mm^{-1}
T = 100 K
Irregular, colourless
0.3 × 0.25 × 0.12 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
9829 measured reflections
2222 independent reflections
1978 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°, θ_{min} = 3.2°
h = −41→40
k = −9→9
l = −4→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.057
S = 0.98
2222 reflections
210 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.32 (11)

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. Refined as a 2-component inversion twin. All H atoms were found in a difference map but set to idealized positions and treated as riding with $C_{aromatic}$—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) and with C_{methyl}—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.32398 (2)	0.10318 (6)	−0.15317 (14)	0.01524 (13)
O1	0.33997 (5)	0.14325 (19)	−0.3745 (3)	0.0215 (4)
O2	0.29105 (4)	−0.00997 (18)	−0.1300 (3)	0.0226 (4)
O3	0.35856 (4)	0.01401 (17)	−0.0021 (3)	0.0166 (3)
C1	0.31403 (6)	0.2901 (3)	−0.0019 (4)	0.0138 (4)
C2	0.29338 (6)	0.2828 (3)	0.2065 (4)	0.0156 (5)
H2	0.2843	0.1803	0.2644	0.019*
C3	0.28667 (6)	0.4316 (2)	0.3261 (4)	0.0177 (4)
H3	0.2730	0.4282	0.4661	0.021*
C4	0.30000 (6)	0.5864 (3)	0.2409 (4)	0.0169 (5)
C5	0.31986 (6)	0.5899 (3)	0.0296 (4)	0.0176 (5)
H5	0.3283	0.6925	−0.0309	0.021*
C6	0.32716 (6)	0.4425 (3)	−0.0919 (4)	0.0164 (5)
H6	0.3407	0.4457	−0.2322	0.020*
C7	0.29299 (6)	0.7459 (3)	0.3755 (5)	0.0238 (5)
H7A	0.3169	0.8135	0.3732	0.036*
H7B	0.2863	0.7182	0.5323	0.036*
H7C	0.2713	0.8079	0.3070	0.036*
C8	0.39783 (6)	0.0843 (2)	−0.0111 (4)	0.0159 (5)
C9	0.42271 (6)	0.0434 (3)	−0.1928 (4)	0.0208 (5)
H9	0.4137	−0.0252	−0.3122	0.025*
C10	0.46155 (6)	0.1068 (3)	−0.1938 (4)	0.0209 (5)
H10	0.4785	0.0803	−0.3163	0.025*
C11	0.47596 (6)	0.2096 (3)	−0.0158 (4)	0.0152 (4)
C12	0.44964 (6)	0.2455 (3)	0.1645 (4)	0.0213 (5)
H12	0.4585	0.3126	0.2860	0.026*
C13	0.41057 (7)	0.1839 (3)	0.1682 (4)	0.0205 (5)
H13	0.3934	0.2097	0.2899	0.025*
C14	0.51792 (6)	0.2769 (2)	−0.0208 (4)	0.0149 (4)
C15	0.54399 (7)	0.2397 (3)	−0.2010 (4)	0.0271 (6)
H15	0.5351	0.1713	−0.3210	0.032*
C16	0.58282 (7)	0.3019 (3)	−0.2067 (4)	0.0276 (6)
H16	0.5996	0.2747	−0.3296	0.033*
C17	0.59683 (7)	0.4040 (3)	−0.0314 (4)	0.0223 (5)
H17	0.6229	0.4464	−0.0355	0.027*
C18	0.57163 (7)	0.4421 (3)	0.1493 (5)	0.0276 (6)
H18	0.5807	0.5107	0.2686	0.033*
C19	0.53276 (7)	0.3792 (3)	0.1556 (5)	0.0248 (5)
H19	0.5163	0.4058	0.2799	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0146 (2)	0.0136 (2)	0.0175 (2)	0.0014 (2)	−0.0020 (2)	−0.0023 (3)
O1	0.0248 (8)	0.0230 (8)	0.0168 (8)	0.0061 (7)	−0.0012 (7)	−0.0020 (7)

O2	0.0182 (8)	0.0168 (7)	0.0329 (9)	−0.0020 (6)	−0.0032 (8)	−0.0050 (8)
O3	0.0140 (7)	0.0130 (7)	0.0229 (8)	−0.0004 (6)	−0.0009 (7)	0.0024 (7)
C1	0.0125 (10)	0.0122 (10)	0.0167 (11)	0.0017 (8)	−0.0039 (9)	−0.0024 (9)
C2	0.0154 (11)	0.0155 (11)	0.0158 (12)	0.0008 (8)	−0.0010 (9)	0.0017 (9)
C3	0.0141 (9)	0.0223 (11)	0.0167 (11)	0.0017 (8)	−0.0002 (10)	0.0004 (11)
C4	0.0145 (11)	0.0172 (11)	0.0190 (11)	0.0018 (9)	−0.0041 (9)	−0.0021 (10)
C5	0.0164 (10)	0.0139 (10)	0.0223 (13)	−0.0038 (9)	−0.0016 (10)	0.0036 (10)
C6	0.0150 (10)	0.0170 (11)	0.0173 (13)	−0.0012 (8)	0.0021 (9)	0.0019 (8)
C7	0.0240 (11)	0.0199 (11)	0.0274 (13)	0.0034 (8)	−0.0014 (11)	−0.0060 (11)
C8	0.0136 (10)	0.0116 (10)	0.0224 (12)	−0.0006 (8)	−0.0013 (9)	0.0034 (10)
C9	0.0197 (11)	0.0207 (11)	0.0221 (15)	0.0010 (9)	−0.0021 (10)	−0.0077 (10)
C10	0.0178 (10)	0.0245 (11)	0.0204 (15)	0.0040 (9)	0.0034 (10)	−0.0078 (10)
C11	0.0167 (10)	0.0113 (10)	0.0176 (11)	0.0037 (8)	−0.0015 (9)	0.0023 (9)
C12	0.0206 (11)	0.0266 (12)	0.0167 (12)	−0.0032 (10)	0.0004 (10)	−0.0070 (10)
C13	0.0192 (11)	0.0259 (12)	0.0162 (12)	0.0003 (10)	0.0039 (10)	−0.0038 (11)
C14	0.0169 (10)	0.0118 (10)	0.0161 (11)	0.0032 (8)	−0.0022 (9)	0.0020 (9)
C15	0.0256 (12)	0.0318 (13)	0.0239 (15)	−0.0073 (10)	0.0033 (11)	−0.0126 (10)
C16	0.0243 (12)	0.0334 (13)	0.0251 (15)	−0.0056 (10)	0.0100 (10)	−0.0119 (12)
C17	0.0165 (11)	0.0244 (12)	0.0261 (13)	−0.0021 (9)	−0.0003 (10)	−0.0007 (11)
C18	0.0230 (12)	0.0353 (14)	0.0244 (13)	−0.0066 (11)	0.0007 (11)	−0.0118 (12)
C19	0.0193 (12)	0.0349 (14)	0.0201 (12)	−0.0025 (10)	0.0031 (11)	−0.0094 (12)

Geometric parameters (Å, º)

S1—O2	1.4228 (14)	C9—C10	1.388 (3)
S1—O1	1.4238 (18)	C9—H9	0.9300
S1—O3	1.6095 (16)	C10—C11	1.399 (3)
S1—C1	1.753 (2)	C10—H10	0.9300
O3—C8	1.422 (2)	C11—C12	1.392 (3)
C1—C6	1.386 (3)	C11—C14	1.496 (3)
C1—C2	1.390 (3)	C12—C13	1.389 (3)
C2—C3	1.386 (3)	C12—H12	0.9300
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.395 (3)	C14—C15	1.388 (3)
C3—H3	0.9300	C14—C19	1.395 (3)
C4—C5	1.391 (3)	C15—C16	1.384 (3)
C4—C7	1.504 (3)	C15—H15	0.9300
C5—C6	1.386 (3)	C16—C17	1.379 (3)
C5—H5	0.9300	C16—H16	0.9300
C6—H6	0.9300	C17—C18	1.375 (3)
C7—H7A	0.9600	C17—H17	0.9300
C7—H7B	0.9600	C18—C19	1.387 (3)
C7—H7C	0.9600	C18—H18	0.9300
C8—C13	1.372 (3)	C19—H19	0.9300
C8—C9	1.378 (3)		
O2—S1—O1	120.90 (12)	C8—C9—C10	118.6 (2)
O2—S1—O3	102.89 (9)	C8—C9—H9	120.7

O1—S1—O3	108.64 (9)	C10—C9—H9	120.7
O2—S1—C1	109.91 (10)	C9—C10—C11	121.8 (2)
O1—S1—C1	109.37 (10)	C9—C10—H10	119.1
O3—S1—C1	103.59 (10)	C11—C10—H10	119.1
C8—O3—S1	117.76 (13)	C12—C11—C10	117.11 (19)
C6—C1—C2	121.25 (19)	C12—C11—C14	121.99 (19)
C6—C1—S1	119.31 (17)	C10—C11—C14	120.90 (19)
C2—C1—S1	119.44 (17)	C13—C12—C11	122.0 (2)
C3—C2—C1	118.5 (2)	C13—C12—H12	119.0
C3—C2—H2	120.7	C11—C12—H12	119.0
C1—C2—H2	120.7	C8—C13—C12	118.7 (2)
C2—C3—C4	121.4 (2)	C8—C13—H13	120.7
C2—C3—H3	119.3	C12—C13—H13	120.7
C4—C3—H3	119.3	C15—C14—C19	116.86 (19)
C5—C4—C3	118.7 (2)	C15—C14—C11	121.54 (19)
C5—C4—C7	120.9 (2)	C19—C14—C11	121.6 (2)
C3—C4—C7	120.5 (2)	C16—C15—C14	121.8 (2)
C6—C5—C4	120.9 (2)	C16—C15—H15	119.1
C6—C5—H5	119.6	C14—C15—H15	119.1
C4—C5—H5	119.6	C17—C16—C15	120.5 (2)
C5—C6—C1	119.3 (2)	C17—C16—H16	119.8
C5—C6—H6	120.4	C15—C16—H16	119.8
C1—C6—H6	120.4	C18—C17—C16	118.9 (2)
C4—C7—H7A	109.5	C18—C17—H17	120.6
C4—C7—H7B	109.5	C16—C17—H17	120.6
H7A—C7—H7B	109.5	C17—C18—C19	120.7 (2)
C4—C7—H7C	109.5	C17—C18—H18	119.7
H7A—C7—H7C	109.5	C19—C18—H18	119.7
H7B—C7—H7C	109.5	C18—C19—C14	121.4 (2)
C13—C8—C9	121.8 (2)	C18—C19—H19	119.3
C13—C8—O3	118.8 (2)	C14—C19—H19	119.3
C9—C8—O3	119.2 (2)		

O2—S1—O3—C8	172.32 (15)	O3—C8—C9—C10	−177.12 (19)
O1—S1—O3—C8	43.04 (17)	C8—C9—C10—C11	0.3 (3)
C1—S1—O3—C8	−73.17 (17)	C9—C10—C11—C12	0.3 (3)
O2—S1—C1—C6	−143.74 (17)	C9—C10—C11—C14	−179.9 (2)
O1—S1—C1—C6	−8.8 (2)	C10—C11—C12—C13	−0.6 (3)
O3—S1—C1—C6	106.90 (17)	C14—C11—C12—C13	179.5 (2)
O2—S1—C1—C2	36.7 (2)	C9—C8—C13—C12	0.3 (3)
O1—S1—C1—C2	171.61 (16)	O3—C8—C13—C12	176.82 (19)
O3—S1—C1—C2	−72.69 (17)	C11—C12—C13—C8	0.3 (3)
C6—C1—C2—C3	−1.1 (3)	C12—C11—C14—C15	179.7 (2)
S1—C1—C2—C3	178.48 (15)	C10—C11—C14—C15	−0.1 (3)
C1—C2—C3—C4	0.3 (3)	C12—C11—C14—C19	0.0 (3)
C2—C3—C4—C5	1.0 (3)	C10—C11—C14—C19	−179.9 (2)
C2—C3—C4—C7	−179.0 (2)	C19—C14—C15—C16	−0.4 (3)
C3—C4—C5—C6	−1.5 (3)	C11—C14—C15—C16	179.9 (2)

C7—C4—C5—C6	178.5 (2)	C14—C15—C16—C17	−0.1 (4)
C4—C5—C6—C1	0.7 (3)	C15—C16—C17—C18	0.3 (4)
C2—C1—C6—C5	0.6 (3)	C16—C17—C18—C19	−0.1 (4)
S1—C1—C6—C5	−178.97 (16)	C17—C18—C19—C14	−0.4 (4)
S1—O3—C8—C13	101.5 (2)	C15—C14—C19—C18	0.7 (3)
S1—O3—C8—C9	−81.9 (2)	C11—C14—C19—C18	−179.6 (2)
C13—C8—C9—C10	−0.6 (3)		