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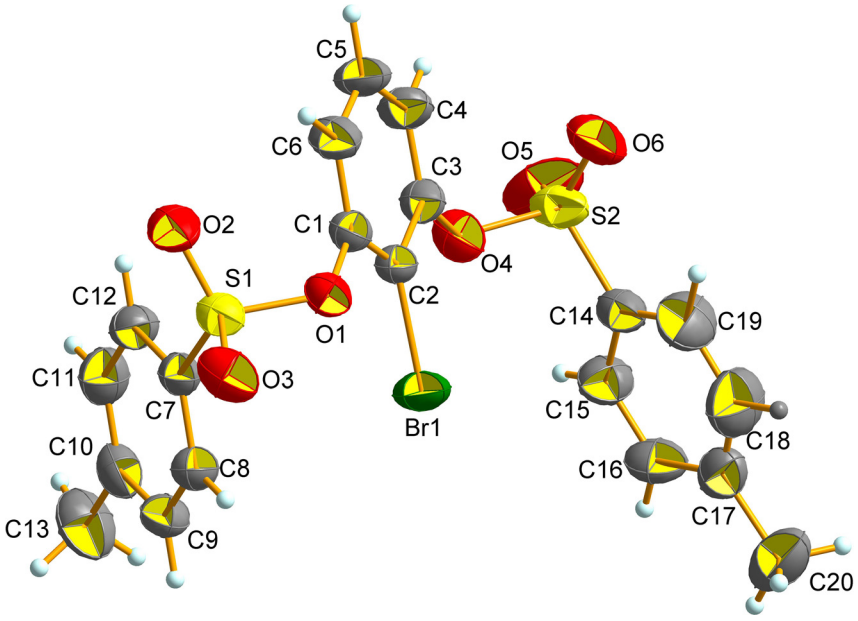
The crystal structure of 2-bromo-1,3-phenylene bis(4-methylbenzenesulfonate), $C_{20}H_{17}BrO_6S_2$



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Abstract

$C_{20}H_{17}BrO_6S_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.173(2) Å, b = 8.256(2) Å, c = 16.514(5) Å, α = 89.947(3)°, β = 83.105(3)°, γ = 72.077(3)°, V = 1051.8(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0334, w$R_{ref}(F^2)$ = 0.0889, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Block
Size:	0.46 × 0.35 × 0.31 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.19 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,043, 3690, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3105
$N(param)_{refined}$:	265
Programs:	Bruker [1], SHELX [2, 3]

1 Source of material

To 2-bromobenzene-1,3-diol (2.35 g, 12.5 mmol) in THF (15 mL) was added 10% K_2CO_3 (6.50 g, 47.1 mmol) as an aqueous solution. After the resulting solution was cooled to 0 °C with an ice-water bath, a solution of Tosylchloride (TsCl) (4.82 g, 25.3 mmol) in THF (35 mL) was slowly added over 15 min at 0 °C. After the addition of TsCI, the ice-water-bath was removed and the reaction mixture was stirred for 2 h. To the mixture was then added EtOAc (100 mL). The two-phase mixture was separated. The organic layer was washed with H_2O and dried ($MgSO_4$). Removal of solvent under reduced pressure gave the

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.58469 (4)	0.41524 (4)	0.30305 (2)	0.06418 (16)
C1	0.8819 (3)	0.5111 (3)	0.32777 (15)	0.0419 (6)
C2	0.7880 (3)	0.4714 (3)	0.27009 (15)	0.0408 (6)
C3	0.8486 (4)	0.4736 (3)	0.18829 (16)	0.0453 (6)
C4	0.9986 (4)	0.5116 (4)	0.16383 (18)	0.0581 (8)
H4	1.036823	0.514470	0.108638	0.070*
C5	1.0921 (4)	0.5457 (4)	0.22257 (19)	0.0630 (8)
H5	1.195066	0.569513	0.206479	0.076*
C6	1.0352 (4)	0.5450 (4)	0.30442 (18)	0.0555 (7)
H6	1.099477	0.567051	0.343449	0.067*
C7	0.5654 (3)	0.7888 (3)	0.43539 (15)	0.0430 (6)
C8	0.4152 (4)	0.7517 (4)	0.46346 (17)	0.0504 (7)
H8	0.417639	0.666776	0.500784	0.060*
C9	0.2625 (4)	0.8410 (4)	0.43579 (19)	0.0561 (7)
H9	0.161559	0.815742	0.454598	0.067*
C10	0.2559 (4)	0.9677 (4)	0.38052 (19)	0.0579 (8)
C11	0.4076 (5)	1.0040 (4)	0.35355 (19)	0.0630 (8)
H11	0.404604	1.089871	0.316708	0.076*
C12	0.5628 (4)	0.9155 (4)	0.38025 (17)	0.0540 (7)
H12	0.663902	0.940519	0.361511	0.065*
C13	0.0861 (5)	1.0662 (5)	0.3514 (3)	0.0883 (12)
H13A	0.106327	1.143018	0.310536	0.132*
H13B	0.007953	1.129968	0.396621	0.132*
H13C	0.036120	0.988358	0.328566	0.132*
C14	0.6797 (4)	0.1736 (4)	0.08991 (16)	0.0505 (7)
C15	0.5093 (4)	0.2265 (4)	0.0773 (2)	0.0615 (8)
H15	0.461932	0.331302	0.054696	0.074*
C16	0.4090 (4)	0.1227 (5)	0.0984 (2)	0.0692 (9)
H16	0.292710	0.159441	0.090391	0.083*
C17	0.4752 (5)	−0.0330 (4)	0.1307 (2)	0.0666 (9)
C18	0.6452 (6)	−0.0803 (5)	0.1438 (3)	0.0945 (13)
H18	0.692034	−0.184010	0.167450	0.113*
C19	0.7492 (5)	0.0199 (5)	0.1234 (3)	0.0839 (11)
H19	0.865007	−0.016134	0.132131	0.101*
C20	0.3675 (7)	−0.1490 (6)	0.1512 (3)	0.1011 (14)
H20A	0.406313	−0.245908	0.113677	0.152*
H20B	0.248119	−0.088736	0.147105	0.152*
H20C	0.379334	−0.186599	0.205809	0.152*
O1	0.8242 (2)	0.5010 (2)	0.41043 (10)	0.0475 (4)
O2	0.8814 (3)	0.7616 (3)	0.45482 (14)	0.0657 (6)
O3	0.7281 (3)	0.6049 (3)	0.54689 (11)	0.0651 (6)
O4	0.7436 (3)	0.4525 (3)	0.13082 (12)	0.0599 (5)
O5	0.7567 (4)	0.3889 (4)	−0.01083 (13)	0.0994 (10)
O6	0.9835 (3)	0.2145 (4)	0.06552 (18)	0.0986 (10)
S1	0.75925 (9)	0.67132 (9)	0.46918 (4)	0.0485 (2)
S2	0.80853 (11)	0.30332 (12)	0.06104 (5)	0.0649 (3)

crude aryl tosylate. The crude product was purified by column chromatography over silica gel with CH_2Cl_2 to afford the title compound (5.50 g, 89% yield). The product was dissolved by CH_2Cl_2 and the crystals of the title compound were obtained by slow evaporation within 1 day.

2 Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the methyl groups were set to 1.5 U_{eq} (C) and the U_{iso} values of all other hydrogen atoms were set to 1.2 U_{eq}(C).

3 Comment

p-Toluenesulfonates have rich biological activities, such as monitoring the merging of lipids, studying membrane fusion during acrosome reaction, development of technology for linking photosensitizers to model monoclonal antibodies and so on [4–7]. Meanwhile, *p*-toluenesulfonates are often used in organic synthesis [8].

Herein, a novel resorcinol *p*-toluenesulfonate compound was synthesized and characterized by single-crystal X-ray diffraction [1–3]. The crystal structure is composed of three structural units containing a central benzene ring connecting these moieties with each other by S–O bonds. The bond lengths of S(1)–O(1), S(1)–O(3), S(1)–O(2), S(2)–O(4), S(2)–O(6) and S(2)–O(6) are 1.6183(19), 1.421(2), 1.418(2), 1.606(2), 1.422(3) and 1.403(3) Å, respectively. The bond angles of O(1)–S(1)–C(7), O(3)–S(1)–C(7) and O(2)–S(1)–O(1) are 103.79(11), 110.82(13) and 108.55(11), indicating the S(1) atom is in the sp^3 unequal hybridization state. Similarly, S(2) atom is also in the sp^3 unequal hybridization [9].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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