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2,4-Dichlorophenyl 4-bromobenzene-sulfonate

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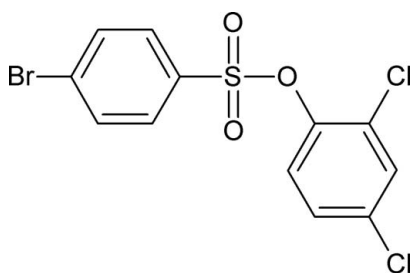
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 Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.074; data-to-parameter ratio = 23.7.

In the title molecule, $\text{C}_{12}\text{H}_7\text{BrCl}_2\text{O}_3\text{S}$, the dihedral angle between the two benzene rings is $55.18(5)^\circ$. The notable intermolecular contacts include $\text{C}-\text{H}\cdots\text{O}$ and $\pi-\pi$ interactions [centroid-centroid distances = $4.037(1)$ and $3.349(1)$ Å].

Related literature

For a detailed account of the molecular and supramolecular architectures of aromatic sulfonates, see Vembu *et al.* (2007). For a general background to aromatic sulfonates, see: Yachi *et al.* (1989); Spungin *et al.* (1992); Tharakan *et al.* (1992); Alford *et al.* (1991); Jiang *et al.* (1990); Narayanan & Krakow (1983). For the criteria to describe $\text{C}-\text{H}\cdots\text{O}$ interactions, see: Desiraju & Steiner, (1999) and for the classification of aromatic stacking interactions, see: Spek (2009).



Experimental

Crystal data

$\text{C}_{12}\text{H}_7\text{BrCl}_2\text{O}_3\text{S}$
 $M_r = 382.05$
 Triclinic, $P\bar{1}$
 $a = 7.2955(10)$ Å
 $b = 8.3955(11)$ Å
 $c = 11.1251(15)$ Å

$\alpha = 95.737(8)^\circ$
 $\beta = 98.645(7)^\circ$
 $\gamma = 96.231(8)^\circ$
 $V = 664.98(15)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 3.65$ mm⁻¹
 $T = 90$ K

$0.17 \times 0.10 \times 0.07$ mm

Data collection

Nonius KappaCCD diffractometer with Oxford Cryostream
 Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)
 $T_{\min} = 0.576$, $T_{\max} = 0.784$

18308 measured reflections
 4745 independent reflections
 4091 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.05$
 4745 reflections

200 parameters
 All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.76$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{O}3^i$	0.95 (3)	2.58 (3)	3.482 (2)	160 (2)
$\text{C}11-\text{H}11\cdots\text{O}1^{\text{ii}}$	0.91 (2)	2.52 (2)	3.390 (2)	160 (2)

Symmetry codes: (i) $x + 1, y, z$; (ii) $x, y - 1, z$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2286).

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supporting information

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2,4-Dichlorophenyl 4-bromobenzenesulfonate

Nagarajan Vembu and Frank R. Fronczek

S1. Comment

Aromatic sulfonates are used in monitoring the merging of lipids (Yachi *et al.*, 1989) and in many other fields (Spungin *et al.*, 1992; Tharakan *et al.*, 1992; Alford *et al.*, 1991; Jiang *et al.*, 1990; Narayanan & Krakow, 1983). An X-ray study of the title compound was undertaken in order to determine its crystal and molecular structure owing to the biological importance of its analogues. The molecular structure is shown in Fig. 1.

The C4–S–O3–C7 torsion angle of 65.86 (14)° corresponds to +synclinal conformation; as expected the dihedral angle between the mean planes of the 2,4-dichlorophenyl and bromobenzene rings of 55.18 (5)° shows that the two rings are not coplanar. This is similar to the situation reported by us for other aromatic sulfonates (Vembu *et al.* 2007 and references cited therein).

The crystal structure exhibits weak intermolecular C—H⋯O interactions (Desiraju & Steiner, 1999) (Table 1). There are two face to face $\pi\cdots\pi$ aromatic stacking interactions. The coordinates of Cg1⋯Cg1 (-x, 1 - y, 1 - z) at 4.037 Å are $\alpha = 0.00$, $\beta = 24.13$, $\gamma = 24.13$, the two perpendicular distances involving the aromatic rings being 3.684 Å. The coordinates of Cg2⋯Cg2 (-1 - x, -y, 2 - z) at 3.751 Å are $\alpha = 0.03$, $\beta = 26.79$, $\gamma = 26.79$, the two perpendicular distances involving the aromatic rings being 3.349 Å. Cg1 is the centroid of the aromatic ring formed by the atoms C1, C2, C3, C4, C5 & C6, Cg2 is the centroid of the aromatic ring formed by the atoms C7, C8, C9, C10, C11 & C12. α is the dihedral angle between the planes of the two aromatic rings, β is the angle of the aromatic ring formed by the atoms C1—C6 through the aromatic ring formed by the atoms C7—C12, γ is the angle of the vector through the centroids of the planes of the two aromatic rings and normal to the plane of the aromatic ring formed by C7—C12 (Spek, 2009).

S2. Experimental

4-Bromobenzenesulfonyl chloride (10 mmol), dissolved in acetone (10 ml), was added dropwise to 2,4-dichlorophenol (10 mmol) in aqueous NaOH (8 ml, 5%) with constant stirring. The precipitate (6.5 mmol, yield 65%) was filtered and recrystallized from aqueous ethanol.

S3. Refinement

All H-atoms were located in difference maps and their positions and isotropic displacement parameters freely refined.

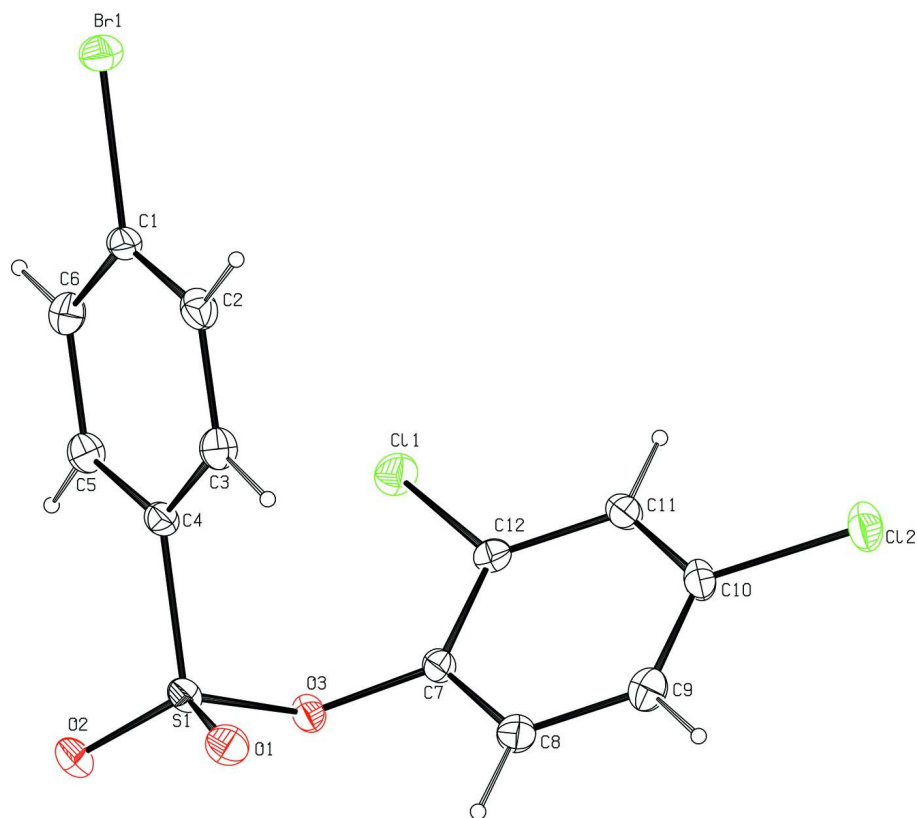


Figure 1

The asymmetric unit with the atoms labelled and displacement ellipsoids depicted at the 50% probability level for all non-H atoms. H-atoms are drawn as spheres of arbitrary radius.

2,4-Dichlorophenyl 4-bromobenzenesulfonate

Crystal data

$C_{12}H_7BrCl_2O_3S$
 $M_r = 382.05$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 7.2955$ (10) Å
 $b = 8.3955$ (11) Å
 $c = 11.1251$ (15) Å
 $\alpha = 95.737$ (8)°
 $\beta = 98.645$ (7)°
 $\gamma = 96.231$ (8)°
 $V = 664.98$ (15) Å³

$Z = 2$
 $F(000) = 376$
 $D_x = 1.908$ Mg m⁻³
 Melting point: 398 K
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 4109 reflections
 $\theta = 2.5$ – 33.6 °
 $\mu = 3.65$ mm⁻¹
 $T = 90$ K
 Prism, colorless
 $0.17 \times 0.10 \times 0.07$ mm

Data collection

Nonius KappaCCD
 diffractometer with Oxford Cryostream
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans with κ offsets

Absorption correction: multi-scan
 (HKL SCALEPACK; Otwinowski & Minor
 1997)
 $T_{\min} = 0.576$, $T_{\max} = 0.784$
 18308 measured reflections
 4745 independent reflections
 4091 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 33.5^\circ$, $\theta_{\text{min}} = 2.9^\circ$
 $h = -10 \rightarrow 11$

$k = -12 \rightarrow 12$
 $l = -16 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.05$
 4745 reflections
 200 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: difference Fourier map
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 0.6284P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.42 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.76 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All su's (except the su in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell su's are taken into account individually in the estimation of su's in distances, angles and torsion angles; correlations between su's in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell su's is used for estimating su's 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.64701 (3)	0.17437 (2)	0.428948 (18)	0.01931 (6)
Cl1	0.02233 (6)	0.03498 (5)	0.69619 (4)	0.01838 (9)
Cl2	0.36290 (6)	-0.08990 (6)	1.13426 (5)	0.02035 (10)
S1	0.14495 (6)	0.51012 (5)	0.77797 (4)	0.01350 (8)
O1	0.26445 (19)	0.59769 (16)	0.88237 (13)	0.0180 (3)
O2	0.00437 (19)	0.58520 (17)	0.70897 (13)	0.0193 (3)
O3	0.02261 (17)	0.36107 (15)	0.82191 (12)	0.0136 (2)
C1	0.4977 (2)	0.2748 (2)	0.53130 (17)	0.0139 (3)
C2	0.5760 (2)	0.3301 (2)	0.65200 (18)	0.0161 (3)
C3	0.4664 (3)	0.4019 (2)	0.72856 (17)	0.0154 (3)
C4	0.2818 (2)	0.4176 (2)	0.68176 (16)	0.0129 (3)
C5	0.2045 (2)	0.3631 (2)	0.56058 (17)	0.0150 (3)
C6	0.3137 (3)	0.2911 (2)	0.48443 (17)	0.0159 (3)
C7	0.1140 (2)	0.2578 (2)	0.89617 (16)	0.0125 (3)
C8	0.1862 (3)	0.3101 (2)	1.01796 (17)	0.0157 (3)
C9	0.2658 (3)	0.2032 (2)	1.09162 (17)	0.0169 (3)
C10	0.2681 (2)	0.0454 (2)	1.04188 (17)	0.0148 (3)
C11	0.1949 (2)	-0.0090 (2)	0.92034 (17)	0.0145 (3)
C12	0.1179 (2)	0.0995 (2)	0.84746 (16)	0.0129 (3)
H2	0.702 (4)	0.319 (3)	0.682 (2)	0.023 (6)*
H3	0.511 (4)	0.442 (3)	0.811 (2)	0.021 (6)*
H5	0.075 (4)	0.373 (3)	0.533 (2)	0.027 (7)*

H6	0.266 (3)	0.253 (3)	0.405 (2)	0.020 (6)*
H8	0.177 (3)	0.419 (3)	1.050 (2)	0.019 (6)*
H9	0.316 (4)	0.241 (3)	1.174 (3)	0.025 (7)*
H11	0.201 (3)	-0.113 (3)	0.889 (2)	0.014 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02086 (9)	0.01838 (10)	0.02144 (10)	0.00506 (7)	0.01007 (7)	0.00289 (7)
C11	0.0235 (2)	0.0159 (2)	0.01452 (19)	0.00065 (16)	0.00259 (16)	-0.00138 (15)
C12	0.01585 (19)	0.0236 (2)	0.0246 (2)	0.00547 (16)	0.00383 (16)	0.01331 (18)
S1	0.01517 (18)	0.01071 (18)	0.01559 (19)	0.00379 (14)	0.00343 (15)	0.00253 (14)
O1	0.0223 (6)	0.0124 (6)	0.0186 (6)	0.0010 (5)	0.0036 (5)	-0.0010 (5)
O2	0.0210 (6)	0.0179 (7)	0.0223 (7)	0.0101 (5)	0.0054 (5)	0.0071 (5)
O3	0.0125 (5)	0.0132 (6)	0.0159 (6)	0.0027 (4)	0.0023 (4)	0.0044 (5)
C1	0.0160 (7)	0.0112 (7)	0.0168 (8)	0.0032 (6)	0.0072 (6)	0.0034 (6)
C2	0.0134 (7)	0.0167 (8)	0.0192 (8)	0.0030 (6)	0.0024 (6)	0.0054 (7)
C3	0.0146 (7)	0.0158 (8)	0.0150 (8)	0.0010 (6)	0.0002 (6)	0.0024 (6)
C4	0.0139 (7)	0.0104 (7)	0.0151 (8)	0.0026 (6)	0.0035 (6)	0.0021 (6)
C5	0.0136 (7)	0.0158 (8)	0.0151 (8)	0.0022 (6)	0.0002 (6)	0.0024 (6)
C6	0.0167 (8)	0.0167 (8)	0.0138 (8)	0.0010 (6)	0.0013 (6)	0.0029 (6)
C7	0.0123 (7)	0.0120 (7)	0.0146 (7)	0.0025 (6)	0.0043 (6)	0.0035 (6)
C8	0.0185 (8)	0.0141 (8)	0.0145 (8)	0.0021 (6)	0.0031 (6)	0.0008 (6)
C9	0.0166 (8)	0.0188 (9)	0.0148 (8)	0.0004 (7)	0.0017 (6)	0.0028 (7)
C10	0.0110 (7)	0.0163 (8)	0.0191 (8)	0.0032 (6)	0.0038 (6)	0.0088 (6)
C11	0.0136 (7)	0.0124 (8)	0.0192 (8)	0.0033 (6)	0.0053 (6)	0.0040 (6)
C12	0.0123 (7)	0.0137 (8)	0.0128 (7)	0.0010 (6)	0.0035 (6)	0.0005 (6)

Geometric parameters (Å, °)

Br1—C1	1.8909 (17)	C4—C5	1.392 (2)
C11—C12	1.7306 (18)	C5—C6	1.387 (3)
C12—C10	1.7361 (18)	C5—H5	0.96 (3)
S1—O1	1.4267 (15)	C6—H6	0.91 (3)
S1—O2	1.4279 (14)	C7—C8	1.386 (2)
S1—O3	1.6163 (13)	C7—C12	1.390 (2)
S1—C4	1.7520 (18)	C8—C9	1.390 (3)
O3—C7	1.407 (2)	C8—H8	0.96 (3)
C1—C2	1.390 (3)	C9—C10	1.387 (3)
C1—C6	1.391 (3)	C9—H9	0.94 (3)
C2—C3	1.391 (3)	C10—C11	1.389 (3)
C2—H2	0.95 (3)	C11—C12	1.387 (2)
C3—C4	1.394 (2)	C11—H11	0.91 (2)
C3—H3	0.94 (3)		
O1—S1—O2	120.61 (9)	C5—C6—C1	119.11 (17)
O1—S1—O3	108.71 (8)	C5—C6—H6	120.8 (16)
O2—S1—O3	102.04 (8)	C1—C6—H6	120.0 (16)

O1—S1—C4	109.04 (8)	C8—C7—C12	120.82 (16)
O2—S1—C4	110.81 (9)	C8—C7—O3	120.41 (16)
O3—S1—C4	104.20 (8)	C12—C7—O3	118.60 (16)
C7—O3—S1	119.14 (11)	C7—C8—C9	119.49 (17)
C2—C1—C6	121.93 (16)	C7—C8—H8	119.2 (15)
C2—C1—Br1	118.56 (13)	C9—C8—H8	121.3 (15)
C6—C1—Br1	119.51 (14)	C10—C9—C8	119.06 (17)
C1—C2—C3	119.00 (16)	C10—C9—H9	122.2 (16)
C1—C2—H2	120.7 (16)	C8—C9—H9	118.7 (17)
C3—C2—H2	120.3 (16)	C9—C10—C11	122.05 (17)
C2—C3—C4	119.06 (17)	C9—C10—C12	119.28 (15)
C2—C3—H3	123.1 (16)	C11—C10—C12	118.66 (14)
C4—C3—H3	117.8 (16)	C12—C11—C10	118.29 (17)
C5—C4—C3	121.71 (16)	C12—C11—H11	121.4 (15)
C5—C4—S1	119.34 (13)	C10—C11—H11	120.2 (15)
C3—C4—S1	118.94 (14)	C11—C12—C7	120.28 (16)
C6—C5—C4	119.18 (16)	C11—C12—C11	119.48 (14)
C6—C5—H5	121.9 (17)	C7—C12—C11	120.22 (14)
C4—C5—H5	118.9 (16)		
O1—S1—O3—C7	-50.31 (14)	C2—C1—C6—C5	-0.6 (3)
O2—S1—O3—C7	-178.76 (13)	Br1—C1—C6—C5	179.19 (14)
C4—S1—O3—C7	65.86 (14)	S1—O3—C7—C8	72.63 (19)
C6—C1—C2—C3	0.8 (3)	S1—O3—C7—C12	-112.06 (16)
Br1—C1—C2—C3	-179.02 (14)	C12—C7—C8—C9	0.9 (3)
C1—C2—C3—C4	-0.5 (3)	O3—C7—C8—C9	176.11 (16)
C2—C3—C4—C5	0.0 (3)	C7—C8—C9—C10	-1.1 (3)
C2—C3—C4—S1	-179.30 (14)	C8—C9—C10—C11	0.6 (3)
O1—S1—C4—C5	-162.54 (14)	C8—C9—C10—C12	-178.65 (14)
O2—S1—C4—C5	-27.52 (17)	C9—C10—C11—C12	0.2 (3)
O3—S1—C4—C5	81.53 (15)	C12—C10—C11—C12	179.41 (13)
O1—S1—C4—C3	16.82 (17)	C10—C11—C12—C7	-0.4 (3)
O2—S1—C4—C3	151.84 (15)	C10—C11—C12—C11	-179.04 (13)
O3—S1—C4—C3	-99.11 (15)	C8—C7—C12—C11	-0.1 (3)
C3—C4—C5—C6	0.1 (3)	O3—C7—C12—C11	-175.43 (15)
S1—C4—C5—C6	179.47 (14)	C8—C7—C12—C11	178.50 (14)
C4—C5—C6—C1	0.2 (3)	O3—C7—C12—C11	3.2 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots O1	0.94 (3)	2.51 (3)	2.919 (2)	106.2 (18)
C2—H2 \cdots O3 ⁱ	0.95 (3)	2.58 (3)	3.482 (2)	160 (2)
C11—H11 \cdots O1 ⁱⁱ	0.91 (2)	2.52 (2)	3.390 (2)	160 (2)

Symmetry codes: (i) $x+1, y, z$; (ii) $x, y-1, z$.