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3,6-Dibromonaphthalene-2,7-diyl bis(trifluoromethanesulfonate)

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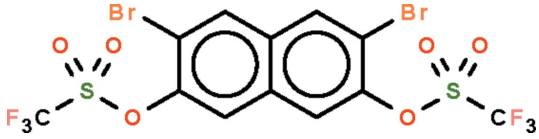
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.094; data-to-parameter ratio = 15.5.

The naphthalene fused ring of the title compound, $C_{12}H_4Br_2F_6O_6S_2$, is slightly buckled (r.m.s. deviation = 0.036 Å) along the common C—C bond and the benzene rings are twisted by 3.2 (3)°. The two trifluoromethylsulfonyl groups lie on opposite sides of the fused-ring system. The crystal structure features short intermolecular F···F contacts [2.715 (4) and 2.832 (4) Å].

Related literature

For the synthesis and background chemistry, see: Shinamura *et al.* (2011).



Experimental

Crystal data

$C_{12}H_4Br_2F_6O_6S_2$
M_r = 582.09
Monoclinic, $P2_1/n$
a = 5.2413 (11) Å
b = 26.450 (6) Å
c = 12.429 (3) Å
β = 90.169 (3)°
V = 1723.1 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.04 mm^{-1}
T = 173 K
0.32 × 0.30 × 0.20 mm

Data collection

Rigaku Saturn724+ CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
T_{min} = 0.573, T_{max} = 1.000
17398 measured reflections
3933 independent reflections
3662 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.094
S = 1.16
3933 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2112).

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supporting information

Acta Cryst. (2011). E**67**, o2698 [https://doi.org/10.1107/S1600536811037755]

3,6-Dibromonaphthalene-2,7-diyl bis(trifluoromethanesulfonate)

Xiang-Xiang Wu, Yan Wan and Seik Weng Ng

S1. Comment

The title compound (Scheme I), which is synthesized from commercially available 3,6-dibromonaphthalene-2,7-diol, has trifluoromethysulfonyloxy (abbreviated OTf) substituents that can be replaced by an acetylenic –C≡C–*R* radical. The product is then converted to a naphthodithiophene by treatment with sodium sulfide. Such naphthodithiophenes can be used as an organic field-effect transistors (Shinamura *et al.*, 2011). We intend to examine these compounds for such applications; the title compound is a precursor. The naphthalene fused-ring is slightly buckled along the common carbon-carbon bond as the benzene rings are offset by 3.2 (3) °. The two trifluoromethylsulfonyl groups lie on opposite sides of the fused-ring (Fig. 1). The crystal structure features short intermolecular F···F contacts (2.715 (4) Å between F3 and inversion-related F4, and 2.832 (4) Å between F3 and inversion symmetry-related F5) that give rise to the formation of a ribbon motif.

S2. Experimental

3,6-Dibromonaphthalene-2,7-diol (3.15 g,10 mmol) was dissolved in the mixture of dichloromethane (100 ml) and triethylamine (3.04 g,30 mmol). The solution was cooled to 273 K. Trifluoromethanesulfonic acid anhydride (3.21 g, 22 mmol) dissolved in dichloromethane (20 ml) was added dropwise. The mxiture was kept cold for 12 h. The solvent was evaporated and the residue purified by column chromatography on silica gel (petroleum ether:ethyl acetate =10:1) to provide the desired product as a white solid (3.78 g, yield 65%). The procedure was that reported by Shinamura *et al.* (2011).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 Å, U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation. Omitted from the refinement because of bad disagreement between observed and calculated structure factors were reflections (0 1 1), (0 2 0) and (0 2 1).

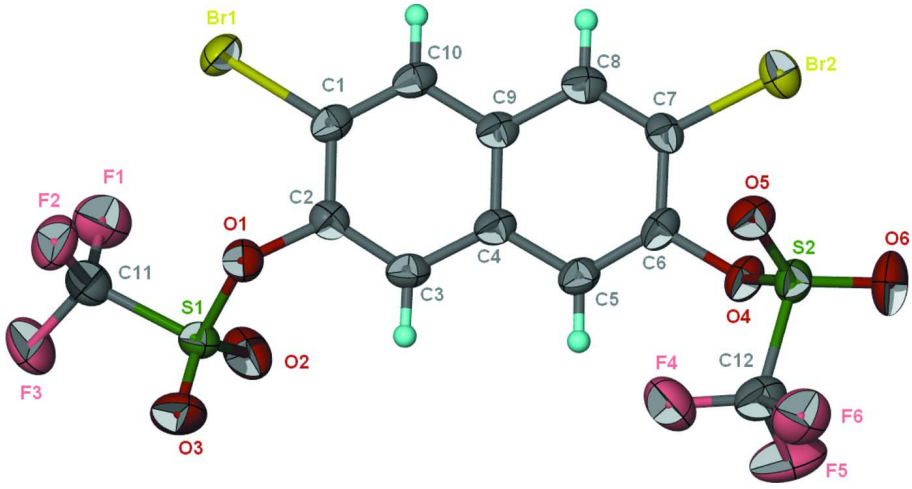

Figure 1

Displacement ellipsoid plot (Barbour, 2001) of $C_{12}H_4Br_2F_6O_6S_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3,6-Dibromonaphthalene-2,7-diyl bis(trifluoromethanesulfonate)

Crystal data

$C_{12}H_4Br_2F_6O_6S_2$
$M_r = 582.09$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.2413$ (11) Å
$b = 26.450$ (6) Å
$c = 12.429$ (3) Å
$\beta = 90.169$ (3)°
$V = 1723.1$ (7) Å^3
$Z = 4$
$F(000) = 1120$
$D_x = 2.244$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5210 reflections
$\theta = 0.8$–$27.5°$
$\mu = 5.04$ mm^{-1}
$T = 173$ K
Prism, colourless
0.32 × 0.30 × 0.20 mm

Data collection

Rigaku Saturn724+ CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans at fixed $\chi = 45°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min} = 0.573$, $T_{max} = 1.000$
17398 measured reflections
3933 independent reflections
3662 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
$h = -6 \rightarrow 6$
$k = -34 \rightarrow 34$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.094$
$S = 1.16$
3933 reflections
253 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 2.4738P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.38667 (8)	0.159089 (15)	0.15955 (3)	0.03395 (12)
Br2	0.69892 (7)	0.232466 (15)	0.59710 (3)	0.03228 (12)
S1	−0.51801 (18)	0.01955 (4)	0.30367 (8)	0.0297 (2)
S2	0.36288 (18)	0.17543 (4)	0.81831 (7)	0.0286 (2)
F1	−0.4753 (6)	0.03529 (11)	0.0989 (2)	0.0519 (7)
F2	−0.8431 (5)	0.05488 (10)	0.1624 (2)	0.0486 (7)
F3	−0.7393 (6)	−0.02313 (10)	0.1425 (2)	0.0576 (8)
F4	−0.0166 (5)	0.11242 (10)	0.8258 (2)	0.0501 (7)
F5	0.3216 (6)	0.08682 (11)	0.9073 (2)	0.0634 (8)
F6	0.0909 (5)	0.14491 (11)	0.9781 (2)	0.0506 (7)
O1	−0.5105 (5)	0.07808 (10)	0.3257 (2)	0.0295 (6)
O2	−0.2705 (6)	−0.00101 (11)	0.2966 (3)	0.0435 (7)
O3	−0.7065 (6)	−0.00149 (13)	0.3702 (3)	0.0523 (9)
O4	0.4608 (5)	0.14368 (10)	0.71859 (19)	0.0296 (6)
O5	0.1917 (5)	0.21376 (10)	0.7854 (2)	0.0339 (6)
O6	0.5748 (6)	0.18540 (13)	0.8852 (2)	0.0453 (8)
C1	−0.2103 (7)	0.14519 (14)	0.2887 (3)	0.0246 (7)
C2	−0.2856 (7)	0.10442 (14)	0.3545 (3)	0.0243 (7)
C3	−0.1645 (7)	0.09387 (13)	0.4487 (3)	0.0260 (7)
H3	−0.2168	0.0658	0.4910	0.031*
C4	0.0403 (7)	0.12493 (13)	0.4835 (3)	0.0247 (7)
C5	0.1600 (7)	0.11762 (14)	0.5845 (3)	0.0264 (8)
H5	0.1130	0.0900	0.6292	0.032*
C6	0.3434 (7)	0.15073 (14)	0.6165 (3)	0.0260 (8)
C7	0.4289 (7)	0.19063 (13)	0.5502 (3)	0.0247 (7)
C8	0.3173 (7)	0.19787 (14)	0.4520 (3)	0.0263 (7)
H8	0.3742	0.2245	0.4068	0.032*
C9	0.1171 (7)	0.16576 (13)	0.4172 (3)	0.0239 (7)
C10	−0.0091 (7)	0.17479 (14)	0.3192 (3)	0.0249 (7)
H10	0.0457	0.2016	0.2740	0.030*
C11	−0.6552 (9)	0.02223 (16)	0.1672 (3)	0.0374 (9)
C12	0.1767 (9)	0.12624 (16)	0.8865 (3)	0.0376 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0434 (2)	0.0326 (2)	0.0258 (2)	0.00171 (17)	−0.00934 (16)	0.00457 (15)
Br2	0.0305 (2)	0.0321 (2)	0.0343 (2)	−0.00321 (15)	0.00105 (15)	−0.00809 (15)
S1	0.0307 (5)	0.0236 (5)	0.0348 (5)	−0.0030 (4)	−0.0049 (4)	0.0053 (4)

S2	0.0322 (5)	0.0296 (5)	0.0239 (4)	0.0006 (4)	−0.0044 (4)	−0.0032 (4)
F1	0.0710 (19)	0.0505 (17)	0.0342 (14)	−0.0086 (14)	0.0094 (13)	−0.0054 (12)
F2	0.0517 (16)	0.0439 (15)	0.0501 (15)	0.0067 (12)	−0.0195 (12)	0.0014 (12)
F3	0.072 (2)	0.0351 (15)	0.0652 (18)	−0.0132 (14)	−0.0185 (15)	−0.0114 (13)
F4	0.0533 (16)	0.0500 (17)	0.0472 (15)	−0.0194 (13)	0.0033 (12)	−0.0053 (12)
F5	0.083 (2)	0.0451 (17)	0.0623 (18)	0.0213 (15)	0.0097 (16)	0.0221 (14)
F6	0.0611 (17)	0.0595 (18)	0.0313 (13)	−0.0012 (14)	0.0112 (12)	−0.0017 (12)
O1	0.0299 (13)	0.0263 (14)	0.0321 (14)	0.0015 (11)	−0.0038 (11)	−0.0013 (11)
O2	0.0354 (16)	0.0268 (15)	0.068 (2)	0.0047 (12)	−0.0101 (14)	−0.0043 (14)
O3	0.0495 (19)	0.056 (2)	0.0515 (19)	−0.0178 (16)	−0.0005 (15)	0.0233 (16)
O4	0.0347 (14)	0.0324 (15)	0.0218 (12)	0.0063 (11)	−0.0046 (10)	−0.0033 (10)
O5	0.0431 (16)	0.0275 (14)	0.0312 (14)	0.0057 (12)	−0.0002 (12)	−0.0021 (11)
O6	0.0403 (16)	0.059 (2)	0.0362 (16)	0.0000 (15)	−0.0135 (13)	−0.0151 (14)
C1	0.0309 (19)	0.0239 (18)	0.0191 (16)	0.0057 (15)	0.0018 (14)	−0.0001 (13)
C2	0.0241 (17)	0.0225 (18)	0.0263 (18)	0.0016 (14)	0.0020 (14)	−0.0025 (14)
C3	0.036 (2)	0.0194 (17)	0.0228 (17)	0.0014 (15)	0.0035 (14)	0.0012 (13)
C4	0.0329 (19)	0.0198 (17)	0.0213 (17)	0.0040 (15)	0.0016 (14)	−0.0008 (13)
C5	0.036 (2)	0.0210 (18)	0.0219 (17)	0.0040 (15)	−0.0018 (14)	0.0002 (14)
C6	0.0307 (19)	0.030 (2)	0.0176 (16)	0.0070 (15)	−0.0020 (14)	−0.0015 (14)
C7	0.0259 (17)	0.0194 (17)	0.0289 (18)	0.0007 (14)	−0.0002 (14)	−0.0055 (14)
C8	0.0316 (19)	0.0209 (18)	0.0265 (18)	0.0026 (15)	0.0050 (14)	−0.0013 (14)
C9	0.0311 (19)	0.0171 (17)	0.0237 (17)	0.0053 (14)	0.0026 (14)	−0.0026 (13)
C10	0.0321 (19)	0.0227 (18)	0.0200 (16)	0.0053 (15)	0.0042 (14)	0.0018 (13)
C11	0.044 (2)	0.028 (2)	0.040 (2)	−0.0033 (18)	−0.0039 (19)	−0.0027 (17)
C12	0.048 (2)	0.034 (2)	0.031 (2)	0.0055 (19)	0.0030 (18)	0.0018 (17)

Geometric parameters (Å, º)

Br1—C1	1.886 (3)	O4—C6	1.421 (4)
Br2—C7	1.887 (3)	C1—C10	1.367 (5)
S1—O3	1.405 (3)	C1—C2	1.410 (5)
S1—O2	1.409 (3)	C2—C3	1.359 (5)
S1—O1	1.573 (3)	C3—C4	1.418 (5)
S1—C11	1.841 (4)	C3—H3	0.9500
S2—O6	1.410 (3)	C4—C5	1.415 (5)
S2—O5	1.414 (3)	C4—C9	1.417 (5)
S2—O4	1.584 (3)	C5—C6	1.359 (5)
S2—C12	1.835 (5)	C5—H5	0.9500
F1—C11	1.317 (5)	C6—C7	1.412 (5)
F2—C11	1.311 (5)	C7—C8	1.366 (5)
F3—C11	1.314 (5)	C8—C9	1.417 (5)
F4—C12	1.313 (5)	C8—H8	0.9500
F5—C12	1.315 (5)	C9—C10	1.405 (5)
F6—C12	1.321 (5)	C10—H10	0.9500
O1—C2	1.414 (4)		
O3—S1—O2	122.2 (2)	C4—C5—H5	120.6
O3—S1—O1	107.7 (2)	C5—C6—C7	122.4 (3)

O2—S1—O1	111.60 (16)	C5—C6—O4	118.7 (3)
O3—S1—C11	106.5 (2)	C7—C6—O4	118.8 (3)
O2—S1—C11	108.3 (2)	C8—C7—C6	119.3 (3)
O1—S1—C11	97.59 (16)	C8—C7—Br2	120.8 (3)
O6—S2—O5	122.32 (19)	C6—C7—Br2	119.8 (3)
O6—S2—O4	107.69 (17)	C7—C8—C9	120.2 (3)
O5—S2—O4	111.13 (15)	C7—C8—H8	119.9
O6—S2—C12	106.2 (2)	C9—C8—H8	119.9
O5—S2—C12	107.73 (19)	C10—C9—C8	120.6 (3)
O4—S2—C12	99.18 (17)	C10—C9—C4	120.0 (3)
C2—O1—S1	123.3 (2)	C8—C9—C4	119.4 (3)
C6—O4—S2	119.3 (2)	C1—C10—C9	120.3 (3)
C10—C1—C2	119.6 (3)	C1—C10—H10	119.8
C10—C1—Br1	120.1 (3)	C9—C10—H10	119.8
C2—C1—Br1	120.4 (3)	F3—C11—F2	109.8 (4)
C3—C2—C1	121.7 (3)	F3—C11—F1	109.2 (4)
C3—C2—O1	120.2 (3)	F2—C11—F1	109.7 (4)
C1—C2—O1	117.7 (3)	F3—C11—S1	108.1 (3)
C2—C3—C4	119.6 (3)	F2—C11—S1	111.0 (3)
C2—C3—H3	120.2	F1—C11—S1	109.0 (3)
C4—C3—H3	120.2	F4—C12—F5	109.7 (4)
C5—C4—C9	119.6 (3)	F4—C12—F6	109.5 (4)
C5—C4—C3	121.6 (3)	F5—C12—F6	108.9 (3)
C9—C4—C3	118.7 (3)	F4—C12—S2	110.0 (3)
C6—C5—C4	118.9 (3)	F5—C12—S2	110.2 (3)
C6—C5—H5	120.6	F6—C12—S2	108.4 (3)

O3—S1—O1—C2	125.8 (3)	C7—C8—C9—C10	176.1 (3)
O2—S1—O1—C2	−10.9 (3)	C7—C8—C9—C4	−2.4 (5)
C11—S1—O1—C2	−124.1 (3)	C5—C4—C9—C10	−176.9 (3)
O6—S2—O4—C6	−146.3 (3)	C3—C4—C9—C10	0.0 (5)
O5—S2—O4—C6	−9.9 (3)	C5—C4—C9—C8	1.7 (5)
C12—S2—O4—C6	103.3 (3)	C3—C4—C9—C8	178.5 (3)
C10—C1—C2—C3	0.6 (6)	C2—C1—C10—C9	−2.1 (5)
Br1—C1—C2—C3	−179.1 (3)	Br1—C1—C10—C9	177.7 (3)
C10—C1—C2—O1	173.5 (3)	C8—C9—C10—C1	−176.8 (3)
Br1—C1—C2—O1	−6.2 (4)	C4—C9—C10—C1	1.8 (5)
S1—O1—C2—C3	−63.9 (4)	O3—S1—C11—F3	−53.1 (4)
S1—O1—C2—C1	123.0 (3)	O2—S1—C11—F3	80.0 (3)
C1—C2—C3—C4	1.1 (5)	O1—S1—C11—F3	−164.2 (3)
O1—C2—C3—C4	−171.6 (3)	O3—S1—C11—F2	67.4 (4)
C2—C3—C4—C5	175.4 (3)	O2—S1—C11—F2	−159.5 (3)
C2—C3—C4—C9	−1.4 (5)	O1—S1—C11—F2	−43.7 (3)
C9—C4—C5—C6	1.2 (5)	O3—S1—C11—F1	−171.6 (3)
C3—C4—C5—C6	−175.5 (3)	O2—S1—C11—F1	−38.5 (3)
C4—C5—C6—C7	−3.5 (5)	O1—S1—C11—F1	77.3 (3)
C4—C5—C6—O4	179.2 (3)	O6—S2—C12—F4	−176.0 (3)
S2—O4—C6—C5	−97.9 (4)	O5—S2—C12—F4	51.3 (3)

S2—O4—C6—C7	84.7 (4)	O4—S2—C12—F4	−64.5 (3)
C5—C6—C7—C8	2.8 (6)	O6—S2—C12—F5	−55.0 (3)
O4—C6—C7—C8	−179.9 (3)	O5—S2—C12—F5	172.4 (3)
C5—C6—C7—Br2	−176.1 (3)	O4—S2—C12—F5	56.6 (3)
O4—C6—C7—Br2	1.2 (4)	O6—S2—C12—F6	64.2 (3)
C6—C7—C8—C9	0.3 (5)	O5—S2—C12—F6	−68.5 (3)
Br2—C7—C8—C9	179.2 (3)	O4—S2—C12—F6	175.8 (3)