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2-Methylsulfonyl-4-(trifluoromethyl)-benzoic acid

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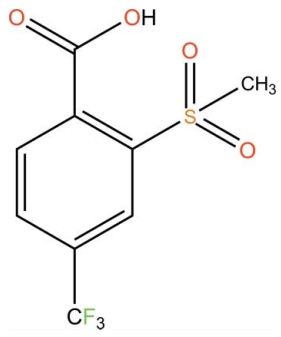
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.123; data-to-parameter ratio = 14.2.

In the title molecule, $C_9H_7F_3O_4S$, the S and the methyl C atoms of the methylsulfonyl group deviate from the benzene ring plane by 0.185 (2) and −1.394 (3) Å, respectively. In the crystal, O—H···O hydrogen bonds link the molecules into chains along [201]. Weak C—H···O interactions further link these chains into layers parallel to the *ac* plane.

Related literature

For details of the synthesis, see: Cain *et al.* (1998).



Experimental

Crystal data

$C_9H_7F_3O_4S$
M_r = 268.21
Monoclinic, $P2_1/c$
a = 5.0804 (10) Å
b = 17.345 (4) Å
c = 11.576 (2) Å
β = 95.41 (3)°
V = 1015.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 293 K
0.39 × 0.32 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.871, T_{max} = 0.926
9132 measured reflections
2250 independent reflections
1879 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.11
2250 reflections
158 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O4^{i}	0.82 (1)	1.92 (1)	2.725 (3)	169 (4)
C9—H9B···O3^{ii}	0.96	2.35	3.208 (3)	148

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: CV5300).

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supplementary materials

Acta Cryst. (2012). E**68**, o1856 [doi:10.1107/S160053681202243X]

2-Methylsulfonyl-4-(trifluoromethyl)benzoic acid

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Comment

The title compound, (I), is a intermediate in the synthesis of sulfonylurea herbicides developed and produced by E. I. du Pont de Nemours and Company. Herein, we report its crystal structure.

In (I) (Fig. 1), the S and the methyl C atoms of the methylsulfonyl group deviate from the benzene ring plane at 0.185 (2) and -1.394 (3) Å, respectively. Intermoleculear O—H···O hydrogen bonds (Table 1) link the molecules into chains along [201] (Fig. 2), and weak C—H···O interactions (Table 1) link further these chains into layers parallel to *ac* plane.

Experimental

The title compound was prepared by the reaction of 2-(methylsulphenyl)-4-trifluoromethylbenzoic acid and hydrogen peroxide in acetic acid at 10 ° (Cain *et al.*, 1998). A colourless crystal suitable for single-crystal X-ray diffraction was obtained by the recrystallization from dichloromethane.

Refinement

C-bound H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 – 0.96 Å , and with U_{iso}(H) = 1.2 – 1.5 U_{eq}(C). O-bound H atoms were located in a differece Fourier map and were refined with restraint as O—H = 0.82 (1) Å, and U_{iso}(H) = 1.5U_{eq}(O).

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

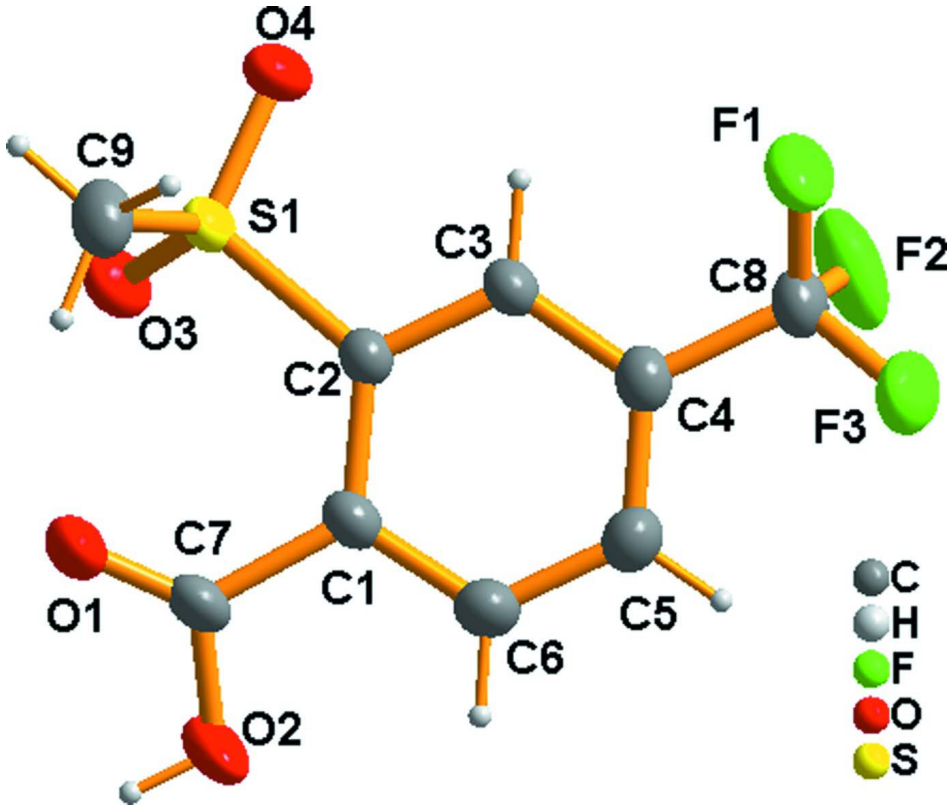

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level for non-H atoms.

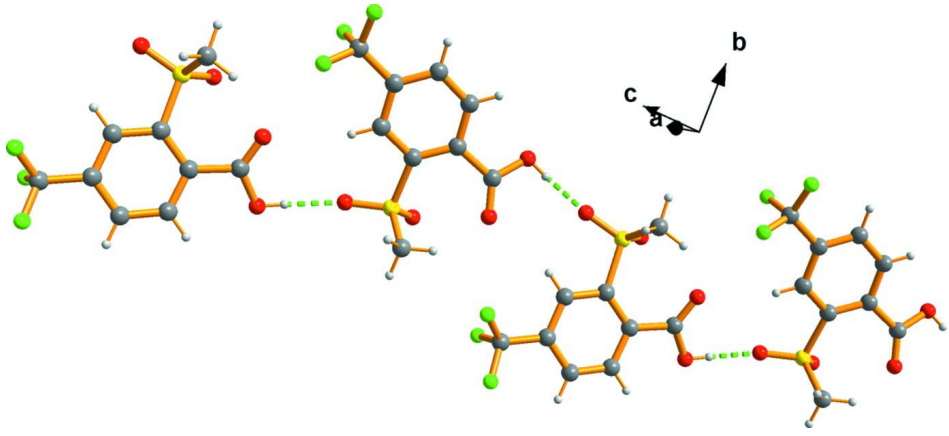

Figure 2

A portion of the crystal packing showing hydrogen-bonded (dashed lines) chains.

2-Methylsulfonyl-4-(trifluoromethyl)benzoic acid

Crystal data

$C_9H_7F_3O_4S$
$M_r = 268.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.0804\ (10)$ Å
$b = 17.345\ (4)$ Å
$c = 11.576\ (2)$ Å
$\beta = 95.41\ (3)°$

V = 1015.6 (4) Å^3
Z = 4
F(000) = 544
D_x = 1.754 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8167 reflections

θ = 3.0–27.5°
μ = 0.36 mm^{-1}
T = 293 K
Block, colorless
0.39 × 0.32 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.871, T_{max} = 0.926

9132 measured reflections
2250 independent reflections
1879 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°, θ_{min} = 3.5°
h = −6→6
k = −22→22
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.11
2250 reflections
158 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.7827P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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. 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 $F^2 > 2$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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.2822 (5)	0.85011 (13)	0.09229 (19)	0.0319 (5)
C2	0.4291 (5)	0.80387 (12)	0.17524 (19)	0.0284 (5)
C3	0.6265 (5)	0.83602 (13)	0.25122 (19)	0.0304 (5)
H3	0.7242	0.8051	0.3051	0.037*
C4	0.6780 (5)	0.91494 (13)	0.2467 (2)	0.0337 (5)
C5	0.5314 (5)	0.96140 (14)	0.1677 (2)	0.0392 (6)
H5	0.5636	1.0141	0.1658	0.047*
C6	0.3360 (5)	0.92880 (14)	0.0911 (2)	0.0396 (6)
H6	0.2386	0.9602	0.0378	0.048*
C7	0.0778 (5)	0.81875 (14)	0.0022 (2)	0.0353 (5)

C8	0.8793 (5)	0.94984 (14)	0.3342 (2)	0.0389 (6)
C9	0.4951 (5)	0.65356 (15)	0.0879 (2)	0.0436 (6)
H9A	0.4715	0.5992	0.0984	0.065*
H9B	0.6803	0.6649	0.0889	0.065*
H9C	0.4062	0.6691	0.0147	0.065*
F1	1.0869 (4)	0.90478 (10)	0.35927 (19)	0.0655 (5)
F2	0.7743 (4)	0.96498 (13)	0.43239 (16)	0.0743 (6)
F3	0.9734 (4)	1.01622 (10)	0.29917 (18)	0.0648 (5)
O1	0.0598 (4)	0.75293 (11)	−0.02770 (17)	0.0511 (5)
O2	−0.0822 (4)	0.87346 (13)	−0.0433 (2)	0.0594 (6)
H2	−0.187 (6)	0.853 (2)	−0.092 (3)	0.089*
O3	0.0826 (3)	0.69334 (11)	0.19817 (16)	0.0409 (4)
O4	0.5177 (4)	0.68310 (10)	0.30716 (16)	0.0420 (4)
S1	0.36264 (11)	0.70384 (3)	0.20026 (5)	0.02965 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0313 (12)	0.0354 (12)	0.0275 (10)	−0.0014 (9)	−0.0052 (9)	−0.0003 (9)
C2	0.0292 (11)	0.0281 (10)	0.0270 (10)	−0.0019 (8)	−0.0027 (9)	−0.0010 (8)
C3	0.0292 (11)	0.0307 (11)	0.0298 (10)	−0.0012 (9)	−0.0056 (9)	−0.0011 (9)
C4	0.0343 (13)	0.0323 (11)	0.0340 (11)	−0.0041 (9)	0.0002 (10)	−0.0039 (9)
C5	0.0465 (15)	0.0295 (11)	0.0402 (13)	−0.0049 (10)	−0.0025 (11)	0.0017 (9)
C6	0.0461 (15)	0.0348 (12)	0.0358 (12)	−0.0008 (10)	−0.0079 (11)	0.0054 (10)
C7	0.0363 (13)	0.0409 (13)	0.0264 (11)	−0.0014 (10)	−0.0080 (10)	0.0021 (9)
C8	0.0381 (14)	0.0344 (12)	0.0426 (13)	−0.0063 (10)	−0.0053 (11)	−0.0041 (10)
C9	0.0411 (14)	0.0387 (13)	0.0500 (15)	0.0002 (11)	−0.0014 (12)	−0.0120 (11)
F1	0.0520 (11)	0.0479 (10)	0.0892 (14)	0.0019 (8)	−0.0329 (10)	−0.0072 (9)
F2	0.0700 (14)	0.1067 (17)	0.0459 (10)	−0.0298 (12)	0.0039 (9)	−0.0328 (10)
F3	0.0673 (12)	0.0446 (9)	0.0773 (12)	−0.0257 (8)	−0.0208 (10)	0.0066 (9)
O1	0.0628 (14)	0.0392 (10)	0.0453 (10)	−0.0044 (9)	−0.0255 (10)	−0.0005 (8)
O2	0.0596 (14)	0.0481 (12)	0.0622 (13)	0.0101 (10)	−0.0377 (11)	−0.0121 (10)
O3	0.0279 (9)	0.0490 (10)	0.0441 (10)	−0.0075 (7)	−0.0047 (8)	0.0036 (8)
O4	0.0404 (10)	0.0411 (10)	0.0410 (10)	−0.0038 (7)	−0.0135 (8)	0.0091 (7)
S1	0.0264 (3)	0.0294 (3)	0.0313 (3)	−0.0036 (2)	−0.0070 (2)	0.0017 (2)

Geometric parameters (Å, º)

C1—C6	1.392 (3)	C7—O1	1.194 (3)
C1—C2	1.410 (3)	C7—O2	1.326 (3)
C1—C7	1.502 (3)	C8—F1	1.322 (3)
C2—C3	1.387 (3)	C8—F3	1.325 (3)
C2—S1	1.796 (2)	C8—F2	1.326 (3)
C3—C4	1.396 (3)	C9—S1	1.753 (3)
C3—H3	0.9300	C9—H9A	0.9600
C4—C5	1.383 (3)	C9—H9B	0.9600
C4—C8	1.498 (3)	C9—H9C	0.9600
C5—C6	1.387 (4)	O2—H2	0.816 (10)
C5—H5	0.9300	O3—S1	1.4325 (18)
C6—H6	0.9300	O4—S1	1.4484 (18)

C6—C1—C2	118.2 (2)	O2—C7—C1	112.0 (2)
C6—C1—C7	118.1 (2)	F1—C8—F3	106.1 (2)
C2—C1—C7	123.6 (2)	F1—C8—F2	107.8 (2)
C3—C2—C1	120.5 (2)	F3—C8—F2	106.0 (2)
C3—C2—S1	114.94 (17)	F1—C8—C4	112.9 (2)
C1—C2—S1	124.32 (17)	F3—C8—C4	112.8 (2)
C2—C3—C4	119.8 (2)	F2—C8—C4	110.8 (2)
C2—C3—H3	120.1	S1—C9—H9A	109.5
C4—C3—H3	120.1	S1—C9—H9B	109.5
C5—C4—C3	120.3 (2)	H9A—C9—H9B	109.5
C5—C4—C8	120.2 (2)	S1—C9—H9C	109.5
C3—C4—C8	119.3 (2)	H9A—C9—H9C	109.5
C4—C5—C6	119.6 (2)	H9B—C9—H9C	109.5
C4—C5—H5	120.2	C7—O2—H2	107 (3)
C6—C5—H5	120.2	O3—S1—O4	116.25 (11)
C5—C6—C1	121.5 (2)	O3—S1—C9	112.01 (13)
C5—C6—H6	119.3	O4—S1—C9	107.14 (13)
C1—C6—H6	119.3	O3—S1—C2	108.76 (11)
O1—C7—O2	122.8 (2)	O4—S1—C2	106.40 (11)
O1—C7—C1	125.2 (2)	C9—S1—C2	105.64 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O4^{i}	0.82 (1)	1.92 (1)	2.725 (3)	169 (4)
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