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2-Trifluoromethyl-1*H*-benzimidazol-3-ium hydrogen sulfate

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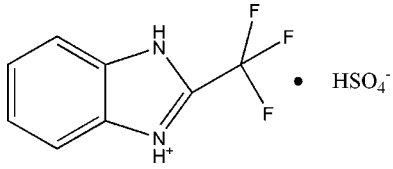
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.052; wR factor = 0.134; data-to-parameter ratio = 11.8.

In the crystal of the title molecular salt, $C_8H_6F_3N_2^+\cdot HSO_4^-$, cation-to-anion N—H···O hydrogen bonds generate [100] chains. Anion-to-anion O—H···O hydrogen bonds generate [001] helices and cross-link the chains into a three-dimensional network.

Related literature

For a related structure and background to molecular salts, see: Liu (2011).



Experimental

Crystal data

$C_8H_6F_3N_2^+\cdot HSO_4^-$
M_r = 284.22
Hexagonal, $P6_5$
a = 9.4119 (13) Å
c = 21.960 (4) Å
V = 1684.7 (5) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.935, T_{max} = 0.935
14287 measured reflections
1977 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.134
S = 1.11
1977 reflections
167 parameters
8 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 957 Friedel pairs
Flack parameter: 0.03 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.86	1.85	2.707 (5)	173
N1—H1A···O4	0.86	1.88	2.740 (7)	174
O1—H1···O2^{ii}	0.86 (2)	1.86 (6)	2.608 (7)	145 (9)

Symmetry codes: (i) $x+1, y, z$; (ii) $y-1, -x+y, z+\frac{1}{6}$.

Data collection: *CrystalClear* (Rigaku, 2005); 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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The author thanks an anonymous reader from the Ordered Matter Science Research Centre, Southeast University, for great help in the revision of this paper.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6491).

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Liu, M.-L. (2011). *Acta Cryst.* E**67**, o2821.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o3473 [https://doi.org/10.1107/S1600536811048811]

2-Trifluoromethyl-1*H*-benzimidazol-3-ium hydrogen sulfate

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S1. Experimental

0.144 g(1 mmol) of 2-Trifluoromethl-1*H*-benzimidazole was firstly dissolved in 30 ml me thanol, to which 0.1 g (1 mmol) of sulfuric acid was then added to afford the solution without any precipitation under stirring at the ambient temperature. Colourless blocks of the title compound were obtained by the slow evaporation of the above solution after 2 days in air.

The dielectric constant of the compound as a function of temperature indicates that the permittivity is basically temperature-independent ($\varepsilon = C/(T–T_0)$), suggesting that this compound is not ferroelectric or there may be no distinct phase transition occurring within the measured temperature within the measured temperature (below the melting point).

S2. Refinement

H atoms were placed in calculated positions (N—H = 0.89 Å; C—H = 0.93Å for Csp^2 atoms and C—H = 0.96Å and 0.97Å for Csp^3 atoms), assigned fixed U_{iso} values [U_{iso} = 1.2Ueq(Csp^2) and 1.5Ueq(Csp^3,N)] and allowed to ride.The H atom bonding with N was found with O—H bond distance of 0.8600Åin the difference electron density map.

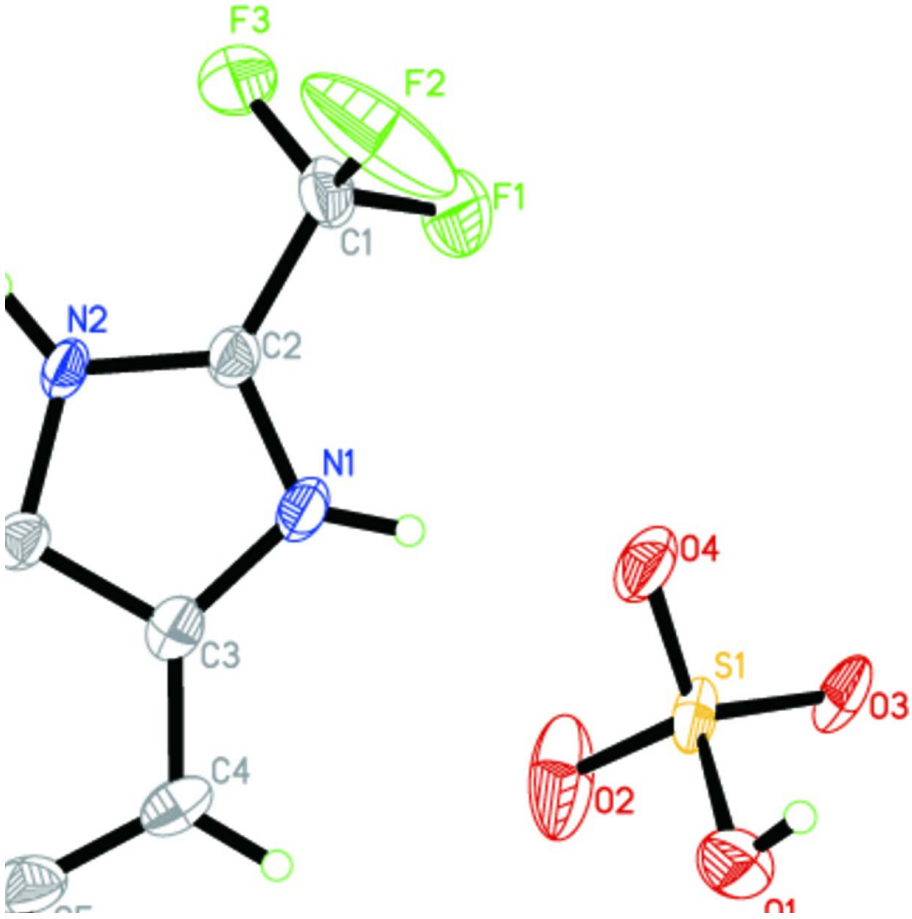

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

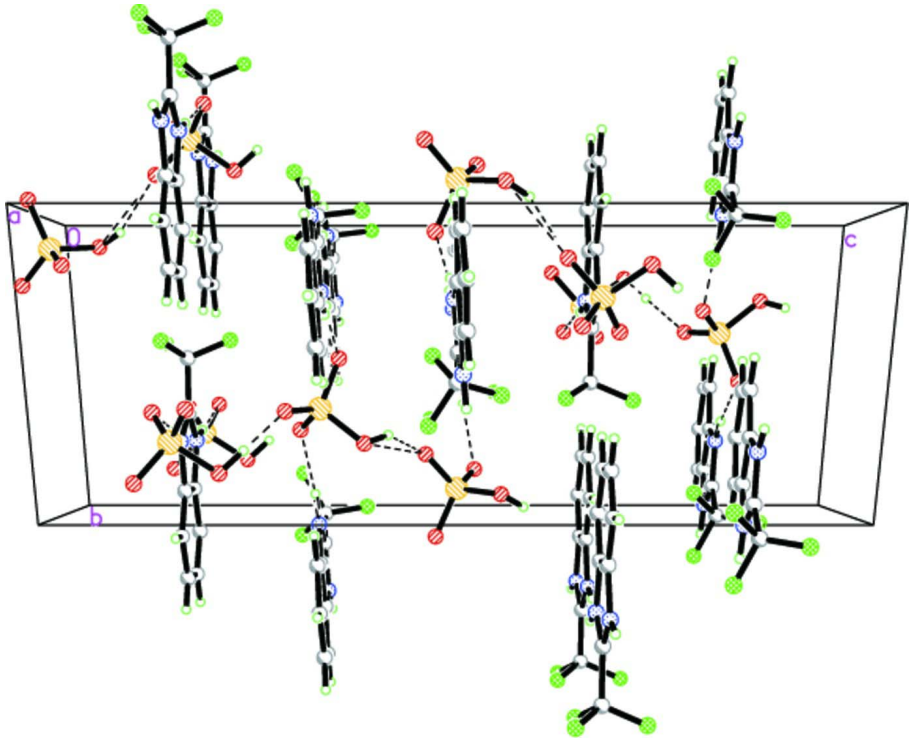

Figure 2

A view of the packing of the title compound, stacking along the *a* axis. Dashed lines indicate hydrogen bonds.

2-Trifluoromethyl-1*H*-benzimidazol-3-ium hydrogen sulfate

Crystal data

$C_8H_6F_3N_2^+\cdot HSO_4^-$
$M_r = 284.22$
Hexagonal, $P6_5$
Hall symbol: P 65
$a = 9.4119\ (13)$ Å
$c = 21.960\ (4)$ Å
$V = 1684.7\ (5)$ Å^3
$Z = 6$
$F(000) = 864$
$D_x = 1.681$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 3.1$–$27.6°$
$\mu = 0.34$ mm^{-1}
$T = 293$ K
Block, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.935$, $T_{max} = 0.935$
14287 measured reflections
1977 independent reflections
1941 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$
$h = -11\rightarrow11$
$k = -11\rightarrow11$
$l = -26\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.134$
$S = 1.11$
1977 reflections
167 parameters
8 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 1.9666P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 957 Friedel pairs
Absolute structure parameter: 0.03 (16)

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.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.09970 (14)	0.72553 (17)	0.16867 (6)	0.0442 (3)
N1	0.4873 (5)	0.7391 (5)	0.1735 (2)	0.0418 (9)
H1A	0.3843	0.7058	0.1770	0.050*
N2	0.7169 (4)	0.7349 (5)	0.16388 (17)	0.0348 (8)
H2A	0.7835	0.6981	0.1596	0.042*
C3	0.6124 (6)	0.9017 (6)	0.1722 (2)	0.0418 (11)
C8	0.7613 (5)	0.9011 (6)	0.1675 (2)	0.0371 (10)
C4	0.6126 (7)	1.0512 (7)	0.1762 (3)	0.0508 (13)
H4	0.5152	1.0535	0.1792	0.061*
O3	−0.0637 (4)	0.6347 (5)	0.14218 (18)	0.0558 (11)
F3	0.5450 (5)	0.4034 (5)	0.1391 (3)	0.0928 (15)
C6	0.9082 (7)	1.1891 (7)	0.1712 (3)	0.0527 (13)
H6	1.0066	1.2883	0.1710	0.063*
O1	0.0866 (5)	0.8248 (5)	0.2230 (2)	0.0627 (11)
C5	0.7592 (8)	1.1907 (7)	0.1755 (3)	0.0589 (15)
H5	0.7623	1.2909	0.1780	0.071*
F1	0.3262 (5)	0.4077 (6)	0.1346 (3)	0.116 (2)
F2	0.4211 (10)	0.3991 (6)	0.2195 (2)	0.157 (3)
O4	0.1589 (5)	0.6207 (5)	0.19150 (19)	0.0571 (11)
C7	0.9120 (6)	1.0455 (7)	0.1670 (3)	0.0456 (11)
H7	1.0103	1.0448	0.1641	0.055*
C1	0.4609 (6)	0.4596 (7)	0.1670 (3)	0.0520 (13)
O2	0.2184 (6)	0.8507 (9)	0.1300 (3)	0.110 (2)

C2	0.5531 (5)	0.6434 (6)	0.1683 (2)	0.0373 (9)
H1	0.041 (10)	0.754 (9)	0.251 (3)	0.10 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0252 (5)	0.0626 (9)	0.0458 (6)	0.0227 (5)	0.0080 (5)	0.0144 (6)
N1	0.0295 (19)	0.051 (2)	0.051 (2)	0.0252 (19)	−0.0012 (18)	−0.0049 (19)
N2	0.0243 (17)	0.044 (2)	0.040 (2)	0.0205 (16)	0.0009 (16)	−0.0062 (18)
C3	0.037 (2)	0.050 (3)	0.043 (3)	0.025 (2)	0.001 (2)	−0.002 (2)
C8	0.037 (2)	0.043 (3)	0.037 (2)	0.024 (2)	−0.006 (2)	−0.005 (2)
C4	0.057 (3)	0.058 (3)	0.059 (3)	0.044 (3)	−0.002 (3)	0.003 (3)
O3	0.0362 (19)	0.087 (3)	0.059 (2)	0.042 (2)	−0.0057 (16)	−0.015 (2)
F3	0.070 (2)	0.058 (2)	0.158 (4)	0.038 (2)	0.018 (3)	−0.012 (3)
C6	0.044 (3)	0.045 (3)	0.062 (3)	0.017 (2)	0.006 (3)	0.009 (3)
O1	0.056 (3)	0.048 (2)	0.079 (3)	0.022 (2)	−0.009 (2)	−0.012 (2)
C5	0.073 (4)	0.052 (3)	0.070 (4)	0.045 (3)	−0.005 (3)	0.005 (3)
F1	0.047 (2)	0.070 (3)	0.213 (6)	0.0161 (19)	−0.039 (3)	−0.044 (3)
F2	0.246 (6)	0.055 (3)	0.072 (3)	0.002 (3)	0.038 (4)	0.009 (2)
O4	0.0403 (19)	0.074 (3)	0.072 (3)	0.039 (2)	0.0007 (17)	0.007 (2)
C7	0.030 (2)	0.055 (3)	0.050 (3)	0.020 (2)	−0.002 (2)	0.003 (2)
C1	0.036 (3)	0.043 (3)	0.067 (3)	0.012 (2)	0.014 (3)	−0.001 (3)
O2	0.057 (3)	0.155 (5)	0.108 (4)	0.045 (3)	0.032 (3)	0.093 (4)
C2	0.030 (2)	0.040 (2)	0.042 (2)	0.0176 (19)	0.0001 (19)	−0.010 (2)

Geometric parameters (Å, º)

S1—O2	1.429 (5)	C4—C5	1.348 (9)
S1—O4	1.444 (4)	C4—H4	0.9300
S1—O3	1.456 (4)	F3—C1	1.304 (6)
S1—O1	1.558 (5)	C6—C7	1.373 (8)
N1—C2	1.329 (6)	C6—C5	1.414 (8)
N1—C3	1.388 (6)	C6—H6	0.9300
N1—H1A	0.8600	O1—H1	0.86 (2)
N2—C2	1.341 (6)	C5—H5	0.9300
N2—C8	1.405 (6)	F1—C1	1.317 (8)
N2—H2A	0.8600	F2—C1	1.258 (8)
C3—C8	1.407 (6)	C7—H7	0.9300
C3—C4	1.409 (7)	C1—C2	1.499 (7)
C8—C7	1.390 (7)		
O2—S1—O4	111.1 (3)	C3—C4—H4	121.2
O2—S1—O3	114.0 (3)	C7—C6—C5	122.0 (5)
O4—S1—O3	113.1 (3)	C7—C6—H6	119.0
O2—S1—O1	103.0 (4)	C5—C6—H6	119.0
O4—S1—O1	108.5 (3)	S1—O1—H1	104 (7)
O3—S1—O1	106.3 (2)	C4—C5—C6	121.9 (5)
C2—N1—C3	108.6 (4)	C4—C5—H5	119.1

C2—N1—H1A	125.7	C6—C5—H5	119.1
C3—N1—H1A	125.7	C6—C7—C8	116.5 (4)
C2—N2—C8	108.5 (3)	C6—C7—H7	121.8
C2—N2—H2A	125.8	C8—C7—H7	121.8
C8—N2—H2A	125.8	F2—C1—F3	110.5 (7)
N1—C3—C8	107.2 (4)	F2—C1—F1	108.3 (6)
N1—C3—C4	132.5 (4)	F3—C1—F1	105.2 (5)
C8—C3—C4	120.3 (5)	F2—C1—C2	112.0 (5)
C7—C8—N2	132.7 (4)	F3—C1—C2	111.0 (4)
C7—C8—C3	121.9 (4)	F1—C1—C2	109.5 (5)
N2—C8—C3	105.4 (4)	N1—C2—N2	110.3 (4)
C5—C4—C3	117.5 (5)	N1—C2—C1	125.9 (4)
C5—C4—H4	121.2	N2—C2—C1	123.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3^{i}	0.86	1.85	2.707 (5)	173
N1—H1*A*···O4	0.86	1.88	2.740 (7)	174
O1—H1···O2^{ii}	0.86 (2)	1.86 (6)	2.608 (7)	145 (9)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *y*−1, −*x*+*y*, *z*+1/6.