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Pyridinium 4-(trifluoromethyl)benzenesulfonate

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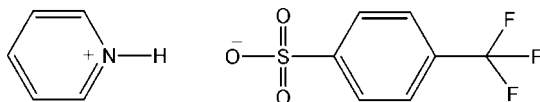
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; disorder in main residue; R factor = 0.088; wR factor = 0.242; data-to-parameter ratio = 12.8.

The title salt, $\text{C}_5\text{H}_6\text{N}^+\cdot\text{C}_7\text{H}_4\text{F}_3\text{O}_3\text{S}^-$, is an ion pair in which the pyridinium cation is linked to the 4-(trifluoromethyl)benzenesulfonate anion by an $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond. The F atoms of the trifluoromethyl group are disordered over two sites in a 0.584 (9):0.416 (9) ratio.

Related literature

For the use of 4-(trifluoromethyl)benzenesulfonate anion as a rust inhibitor, see: Otomo (1993). For comparative bond dimensions for the anion, see: Bats *et al.* (1999); Bernhard *et al.* (1982); Koziol & Podkowińska (1983), and for the cation, see: Djinović & Golič (1992) (1992); Ziemer & Rabis (2000).



Experimental

Crystal data

 $\text{C}_5\text{H}_6\text{N}^+\cdot\text{C}_7\text{H}_4\text{F}_3\text{O}_3\text{S}^-$
 $M_r = 305.27$

Monoclinic, $P2_1/c$
 $a = 5.7905$ (5) Å
 $b = 9.0294$ (7) Å
 $c = 24.988$ (2) Å
 $\beta = 90.8220$ (10)°
 $V = 1306.35$ (18) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 273$ K
 $0.49 \times 0.35 \times 0.25$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.871$, $T_{\max} = 0.931$

6394 measured reflections
 2291 independent reflections
 1880 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.088$
 $wR(F^2) = 0.242$
 $S = 1.07$
 2291 reflections
 179 parameters

15 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.70$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.53$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O3}$	0.86	1.99	2.781 (6)	153

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2593).

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supporting information

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Pyridinium 4-(trifluoromethyl)benzenesulfonate

Gang Chen, Wei Xu, Zhen Yang and Zheng Fan

S1. Comment

4-(Trifluoromethyl)benzenesulfonate was considered as excellent organic rust inhibitors which can effectively prevent corrosion of metals such as steel, copper, manganese and nickel (Otomo *et al.*, 1993). In our laboratory, a new compound containing 4-(trifluoromethyl)benzenesulfonate, (I), has been synthesized, its structure is studied hereafter.

Fig. 1 presents a view of the asymmetric unit: one pyridinium cation and one 4-(trifluoromethyl)benzenesulfonate anion. In the anion the average C—C bond distance in the ring, 1.372 Å, is consistent with the value usually accepted. The internal angle (C4—C5—C6) is decreased to 118.0 (5)°, and endocyclic angle of C3—C2—C7 to 118.3 (5)°. The S—C distance of 1.795 (5)Å is close to the experimental values of 1.77 (Bats *et al.*, 1999), 1.766 (Bernhard *et al.*, 1982) and 1.782 (Kozioł & Podłowińska, 1983). The phenyl ring and the S atom are almost planar, which is displaced from the mean plane of the phenyl ring by 0.010 (6) Å. Three F atom in the trifluoromethyl group is disorder.

In the cation, distances in the pyridinium ring are in the range 1.319 (7)–1.372 (8)Å and angles 118.6 (5)–122.0 (5)°, which are similar to those found in research of Djinović & Golič (1992) and Ziemer *et al.* (2000).

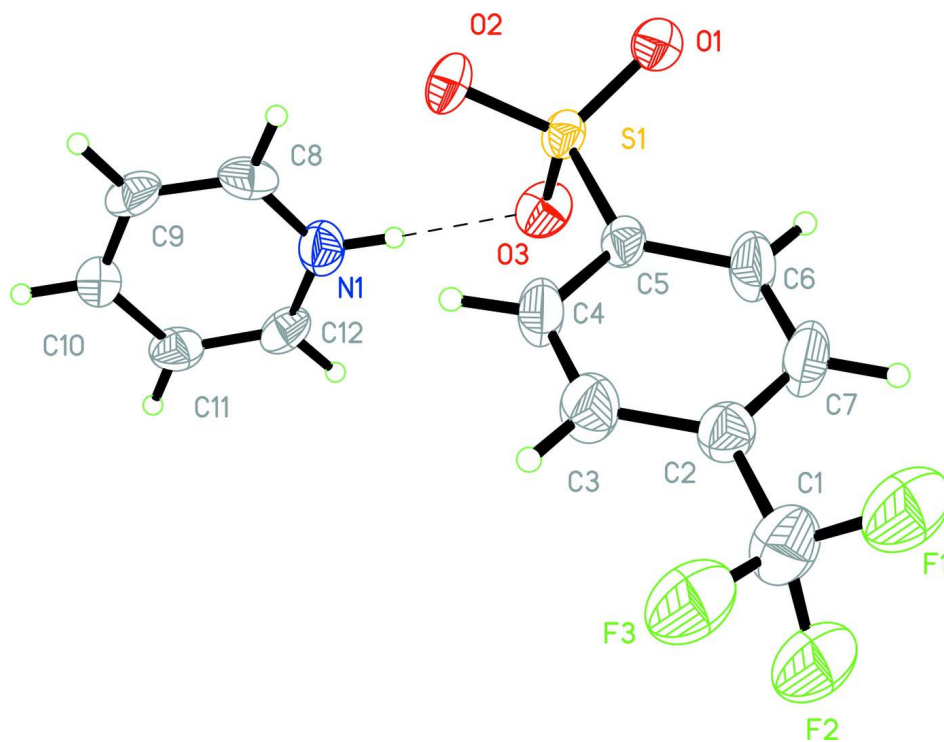
The ion pairs are formed *via* a N1—H1···O3 hydrogen bond (Fig. 2). In addition, weak C—H···O hydrogen bonds stabilize the crystal (Fig. 2 & Table 2).

S2. Experimental

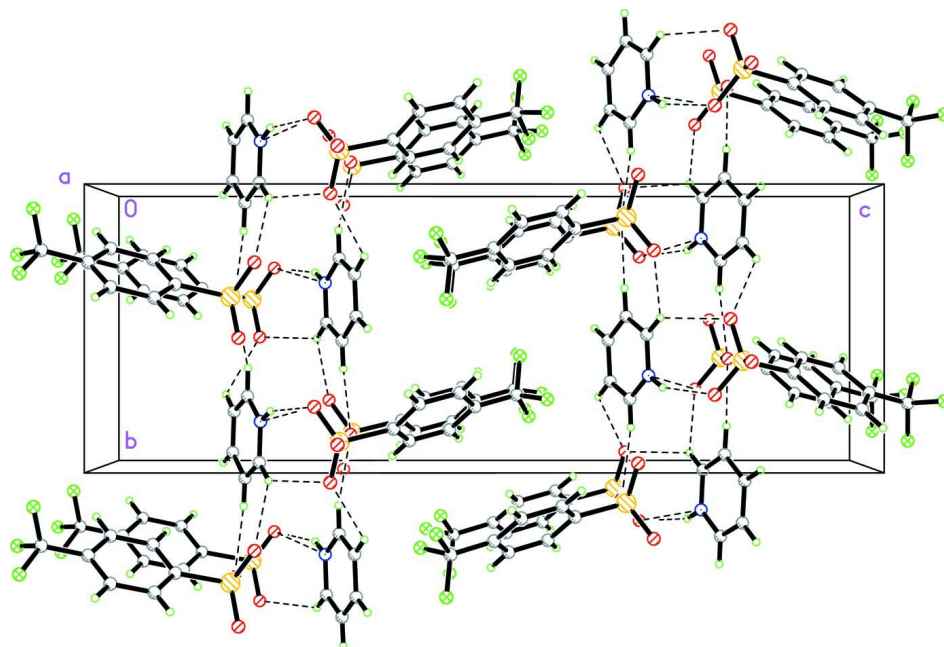
4-(Trifluoromethyl)benzenesulfonic acid and pyridine in a molar ratio of 1:1 were mixed and dissolved in sufficient ethanol by heating to 365 K, when a clear solution resulted. Crystals of (I) were formed by gradual evaporation of excess ethanol over a period of one week at 293 K.

S3. Refinement

All of the H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.86 (N—H), 0.93 Å (aromatic), with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C}, \text{N})$. Three disordered F atoms were split into approximately halves, with occupancies of 0.584 (9) and 0.416 (9). All the C—F distances are restrained to be identical with deviation of 0.01 Å.

**Figure 1**

The cell unit of (I) with atom labels, showing 30% probability displacement ellipsoids. Parts of disorder F atoms was deleted for clarity.

**Figure 2**

A packing diagram viewed down along the *a* axis. Hydrogen bonds are illustrated as thin lines. Parts of disorder F atoms was deleted for clarity.

Pyridinium 4-(trifluoromethyl)benzenesulfonate

Crystal data

 $C_5H_6N^+ \cdot C_7H_4F_3O_3S^-$ $M_r = 305.27$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 5.7905$ (5) Å $b = 9.0294$ (7) Å $c = 24.988$ (2) Å $\beta = 90.822$ (1)° $V = 1306.35$ (18) Å³ $Z = 4$ $F(000) = 624$ $D_x = 1.552$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2733 reflections

 $\theta = 2.4$ – 25.0 ° $\mu = 0.29$ mm⁻¹ $T = 273$ K

Prism, colorless

 $0.49 \times 0.35 \times 0.25$ mm

Data collection

Bruker APEX area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2000)

 $T_{\min} = 0.871$, $T_{\max} = 0.931$

6394 measured reflections

2291 independent reflections

1880 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.031$ $\theta_{\max} = 25.0$ °, $\theta_{\min} = 1.6$ ° $h = -6 \rightarrow 6$ $k = -10 \rightarrow 10$ $l = -22 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.088$ $wR(F^2) = 0.242$ $S = 1.07$

2291 reflections

179 parameters

15 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

 $w = 1/[\sigma^2(F_o^2) + (0.1117P)^2 + 3.3401P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.70$ e Å⁻³ $\Delta\rho_{\min} = -0.53$ 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.

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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	1.1078 (2)	0.88952 (12)	0.32068 (5)	0.0451 (4)	
O1	1.3546 (6)	0.8891 (4)	0.32268 (15)	0.0603 (10)	
O2	1.0094 (6)	1.0326 (4)	0.30758 (14)	0.0595 (10)	
O3	1.0162 (7)	0.7709 (4)	0.28780 (15)	0.0629 (10)	

C1	0.7591 (12)	0.7361 (7)	0.5413 (3)	0.086 (2)	
C2	0.8504 (10)	0.7742 (7)	0.4880 (2)	0.0598 (14)	
C3	0.7200 (11)	0.8601 (8)	0.4550 (3)	0.0756 (18)	
H3	0.5780	0.8954	0.4664	0.091*	
C4	0.7974 (10)	0.8958 (7)	0.4043 (2)	0.0679 (16)	
H4	0.7050	0.9535	0.3818	0.082*	
C5	1.0090 (8)	0.8472 (5)	0.38668 (19)	0.0444 (11)	
C6	1.1354 (10)	0.7576 (8)	0.4195 (2)	0.0730 (18)	
H6	1.2761	0.7203	0.4080	0.088*	
C7	1.0565 (12)	0.7217 (8)	0.4696 (3)	0.081 (2)	
H7	1.1455	0.6602	0.4915	0.098*	
F1'	0.9025 (16)	0.6547 (13)	0.5717 (3)	0.1146 (16)	0.584 (9)
F2'	0.5792 (17)	0.6419 (11)	0.5388 (4)	0.1146 (16)	0.584 (9)
F3'	0.700 (2)	0.8521 (9)	0.5715 (3)	0.1146 (16)	0.584 (9)
F1	0.921 (2)	0.7454 (19)	0.5793 (4)	0.1146 (16)	0.416 (9)
F2	0.672 (3)	0.5995 (12)	0.5458 (5)	0.1146 (16)	0.416 (9)
F3	0.577 (2)	0.8166 (15)	0.5579 (5)	0.1146 (16)	0.416 (9)
N1	0.6745 (8)	0.8321 (6)	0.21165 (18)	0.0650 (13)	
H1	0.7923	0.7923	0.2273	0.078*	
C8	0.6591 (10)	0.9796 (7)	0.2096 (2)	0.0610 (15)	
H8	0.7754	1.0376	0.2248	0.073*	
C9	0.4777 (10)	1.0437 (6)	0.1859 (2)	0.0613 (14)	
H9	0.4666	1.1464	0.1840	0.074*	
C10	0.3107 (10)	0.9570 (7)	0.1649 (2)	0.0628 (14)	
H10	0.1821	1.0001	0.1485	0.075*	
C11	0.3284 (10)	0.8056 (6)	0.1673 (2)	0.0619 (14)	
H11	0.2120	0.7459	0.1531	0.074*	
C12	0.5144 (10)	0.7456 (6)	0.1903 (2)	0.0581 (14)	
H12	0.5314	0.6432	0.1914	0.070*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0535 (8)	0.0315 (6)	0.0502 (7)	0.0055 (5)	-0.0052 (5)	0.0001 (5)
O1	0.057 (2)	0.053 (2)	0.071 (2)	0.0031 (17)	-0.0003 (17)	0.0060 (17)
O2	0.074 (3)	0.039 (2)	0.065 (2)	0.0168 (17)	-0.0059 (18)	0.0063 (16)
O3	0.086 (3)	0.044 (2)	0.058 (2)	-0.0024 (18)	-0.0059 (18)	-0.0113 (16)
C1	0.108 (6)	0.083 (5)	0.068 (4)	0.014 (4)	0.002 (4)	0.004 (4)
C2	0.064 (3)	0.061 (3)	0.055 (3)	0.000 (3)	-0.003 (2)	0.003 (3)
C3	0.067 (4)	0.086 (5)	0.074 (4)	0.025 (3)	0.014 (3)	0.013 (3)
C4	0.061 (3)	0.077 (4)	0.066 (3)	0.023 (3)	-0.006 (3)	0.015 (3)
C5	0.048 (3)	0.030 (2)	0.054 (3)	0.004 (2)	-0.009 (2)	-0.0026 (19)
C6	0.058 (3)	0.093 (5)	0.068 (4)	0.031 (3)	0.001 (3)	0.018 (3)
C7	0.079 (4)	0.096 (5)	0.068 (4)	0.031 (4)	-0.008 (3)	0.029 (4)
F1'	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)
F2'	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)
F3'	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)
F1	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)

F2	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)
F3	0.145 (4)	0.125 (4)	0.074 (2)	-0.006 (3)	0.011 (2)	0.022 (2)
N1	0.061 (3)	0.078 (3)	0.057 (3)	0.011 (3)	-0.002 (2)	0.013 (2)
C8	0.058 (3)	0.063 (4)	0.062 (3)	-0.025 (3)	-0.001 (2)	-0.008 (3)
C9	0.079 (4)	0.032 (3)	0.073 (4)	-0.006 (3)	0.012 (3)	0.000 (2)
C10	0.055 (3)	0.059 (3)	0.074 (4)	0.006 (3)	-0.002 (3)	0.010 (3)
C11	0.066 (4)	0.053 (3)	0.066 (3)	-0.018 (3)	-0.004 (3)	-0.003 (3)
C12	0.079 (4)	0.032 (3)	0.064 (3)	0.001 (2)	0.007 (3)	0.001 (2)

Geometric parameters (Å, °)

S1—O1	1.429 (4)	C5—C6	1.358 (7)
S1—O3	1.446 (4)	C6—C7	1.378 (9)
S1—O2	1.447 (3)	C6—H6	0.9300
S1—C5	1.795 (5)	C7—H7	0.9300
C1—F1	1.330 (8)	N1—C12	1.319 (7)
C1—F2	1.337 (8)	N1—C8	1.336 (8)
C1—F1'	1.338 (7)	N1—H1	0.8600
C1—F3'	1.339 (7)	C8—C9	1.331 (8)
C1—F2'	1.345 (7)	C8—H8	0.9300
C1—F3	1.353 (8)	C9—C10	1.346 (8)
C1—C2	1.479 (9)	C9—H9	0.9300
C2—C3	1.354 (8)	C10—C11	1.372 (8)
C2—C7	1.370 (9)	C10—H10	0.9300
C3—C4	1.388 (8)	C11—C12	1.329 (8)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.379 (7)	C12—H12	0.9300
C4—H4	0.9300		
O1—S1—O3	112.1 (2)	C6—C5—C4	118.0 (5)
O1—S1—O2	113.6 (2)	C6—C5—S1	120.3 (4)
O3—S1—O2	113.1 (2)	C4—C5—S1	121.5 (4)
O1—S1—C5	107.4 (2)	C5—C6—C7	120.4 (5)
O3—S1—C5	104.2 (2)	C5—C6—H6	119.8
O2—S1—C5	105.6 (2)	C7—C6—H6	119.8
F1—C1—F2	105.1 (9)	C2—C7—C6	121.7 (5)
F1'—C1—F3'	105.7 (7)	C2—C7—H7	119.1
F1—C1—F2'	127.7 (9)	C6—C7—H7	119.1
F1'—C1—F2'	98.8 (7)	C12—N1—C8	122.0 (5)
F3'—C1—F2'	108.6 (8)	C12—N1—H1	119.0
F1—C1—F3	107.1 (9)	C8—N1—H1	119.0
F2—C1—F3	100.0 (10)	C9—C8—N1	120.2 (5)
F1—C1—C2	111.7 (8)	C9—C8—H8	119.9
F2—C1—C2	115.5 (8)	N1—C8—H8	119.9
F1'—C1—C2	114.3 (6)	C8—C9—C10	118.6 (5)
F3'—C1—C2	115.0 (6)	C8—C9—H9	120.7
F2'—C1—C2	113.1 (6)	C10—C9—H9	120.7
F3—C1—C2	116.2 (7)	C9—C10—C11	120.6 (6)

C3—C2—C7	118.3 (5)	C9—C10—H10	119.7
C3—C2—C1	118.5 (5)	C11—C10—H10	119.7
C7—C2—C1	123.1 (5)	C12—C11—C10	119.0 (5)
C2—C3—C4	120.3 (5)	C12—C11—H11	120.5
C2—C3—H3	119.9	C10—C11—H11	120.5
C4—C3—H3	119.9	N1—C12—C11	119.6 (5)
C5—C4—C3	121.2 (5)	N1—C12—H12	120.2
C5—C4—H4	119.4	C11—C12—H12	120.2
C3—C4—H4	119.4		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O3	0.86	1.99	2.781 (6)	153
C4—H4 \cdots O1 ⁱ	0.93	2.56	3.255 (7)	132
C8—H8 \cdots O2	0.93	2.47	3.192 (7)	136
C8—H8 \cdots O3 ⁱⁱ	0.93	2.45	3.233 (8)	142
C9—H9 \cdots O1 ⁱⁱ	0.93	2.43	3.274 (6)	151
C11—H11 \cdots O2 ⁱⁱⁱ	0.93	2.52	3.214 (8)	132
C12—H12 \cdots O1 ^{iv}	0.93	2.42	3.324 (7)	166

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, y+1/2, -z+1/2$; (iii) $-x+1, y-1/2, -z+1/2$; (iv) $-x+2, y-1/2, -z+1/2$.