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2-(2,2,2-Trifluoroacetylamino)pyridin-3-yl trifluoromethanesulfonate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.087
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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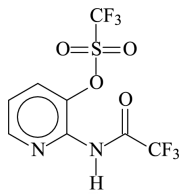
The two independent molecules of $C_8H_4N_2O_4F_6S$ are linked by a pair of $N_{amido}\cdots N_{pyridyl}$ interactions [2.858 (2) and 2.880 (2) Å], giving rise to a hydrogen-bonded dimer. The conformation of the trifluoromethanesulfonate moiety is different in the two molecules.

Comment

The synthesis of aza-7-indoles, which are the starting reagents for the synthesis of pharmacologically active compounds having an indole nucleus (Mérour & Joseph, 2001), requires the title compound, 2-trifluoroacetoamino-3-pyridine trifluoromethanesulfonate (I), in one of the steps. The asymmetric unit of the title compound consists of two independent molecules that are linked by a pair of $N_{amido}\cdots N_{pyridyl}$ hydrogen bonds [2.858 (2) and 2.880 (2) Å] into a dimeric entity (see Fig. 1 and Table 2). In the two molecules, the conformation of the trifluoroacetylamino group is almost the same but the conformation of the trifluoromethanesulfonate group is different (see the torsion angles in Table 1).



(I)

Experimental

The compound was prepared by using a reported procedure (Dai *et al.*, 2001; Dai *et al.*, 2002; Dai *et al.*, 2003). Commercially available 2-amino-3-hydroxypyridine (1.12 g, 10 mmol), trifluoroacetic anhydride (2.36 g, 11 mmol) and pyridine (2.37 g, 30 mmol) were dissolved in THF (60 ml) and the reagents allowed to react for 36 h at room temperature to give trifluoroacetoamino-3-hydroxypyridine, which was purifed by chromatography (ethyl acetate/hexane 1/1). The compound was then reacted with a solution of sodium hydride (0.63 g,15 mmol) dissolved in THF (80 ml) and a solution of *N*-phenylbis(trifluoromethylsulfonylimide) (3.62 g, 10 mmol) dissolved in THF (70 ml). After 24 h, cold water (70 ml) was added to the mixture. The THF–water solution was washed with sodium bicarbonate (40 ml × 3) and sodium chloride solution (40 ml). The crude product was purified by chromatography (ethyl acetate/hexane 1/5) and the pure compound was obtained in a crystalline form in about 70% yield.

Crystal data

$C_8H_4F_6N_2O_4S$
M_r = 338.19
Triclinic, $P\overline{1}$
a = 9.8702 (5) Å
b = 10.0063 (5) Å
c = 12.6434 (6) Å
α = 101.436 (1)°
β = 103.466 (1)°
γ = 95.981 (1)°
V = 1175.4 (1) Å^3
Z = 4
D_x = 1.911 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6114 reflections
θ = 2.4–28.3°
μ = 0.38 mm^{-1}
T = 100 (2) K
Block, colorless
0.40 × 0.35 × 0.30 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
13584 measured reflections
5315 independent reflections
4725 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.087
S = 1.01
5315 reflections
387 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.4147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.406 (1)	S1*a*—O1*a*	1.413 (1)
S1—O2	1.411 (1)	S1*a*—O2*a*	1.415 (1)
S1—O3	1.584 (1)	S1*a*—O3*a*	1.584 (1)
S1—C1	1.837 (2)	S1*a*—C1*a*	1.836 (2)
F1—C1	1.325 (2)	F1*a*—C1*a*	1.315 (2)
F2—C1	1.321 (2)	F2*a*—C1*a*	1.318 (2)
F3—C1	1.322 (2)	F3*a*—C1*a*	1.320 (2)
F4—C8	1.329 (2)	F4*a*—C8*a*	1.336 (2)
F5—C8	1.330 (2)	F5*a*—C8*a*	1.340 (2)
F6—C8	1.328 (2)	F6*a*—C8*a*	1.319 (2)
O3—C3	1.405 (2)	O3*a*—C3*a*	1.413 (2)
O4—C7	1.207 (2)	O4*a*—C7*a*	1.211 (2)
N1—C2	1.332 (2)	N1*a*—C2*a*	1.327 (2)
N1—C6	1.340 (2)	N1*a*—C6*a*	1.340 (2)
N2—C7	1.348 (2)	N2*a*—C7*a*	1.352 (2)
N2—C2	1.410 (2)	N2*a*—C2*a*	1.411 (2)
O1—S1—O2	124.4 (1)	O1*a*—S1*a*—O2*a*	122.4 (1)
O1—S1—O3	105.4 (1)	O1*a*—S1*a*—O3*a*	106.2 (1)
O2—S1—O3	111.4 (1)	O2*a*—S1*a*—O3*a*	111.4 (1)
O1—S1—C1	106.5 (1)	O1*a*—S1*a*—C1*a*	106.6 (1)
O2—S1—C1	106.4 (1)	O2*a*—S1*a*—C1*a*	108.0 (1)
O3—S1—C1	100.0 (1)	O3*a*—S1*a*—C1*a*	99.9 (1)
C3—O3—S1	120.4 (1)	C3*a*—O3*a*—S1*a*	120.5 (1)
C7—N2—C2	121.6 (1)	C7*a*—N2*a*—C2*a*	123.5 (1)
O4—C7—N2	127.0 (1)	O4*a*—C7*a*—N2*a*	127.5 (1)
O4—C7—C8	118.3 (1)	O4*a*—C7*a*—C8*a*	119.6 (1)
N2—C7—C8	114.7 (1)	N2*a*—C7*a*—C8*a*	112.8 (1)
C1—S1—O3—C3	85.7 (1)	C1*a*—S1*a*—O3*a*—C3*a*	−98.9 (1)
C7—N2—C2—C3	−55.8 (2)	C7*a*—N2*a*—C2*a*—C3*a*	−55.5 (2)
S1—O3—C3—C4	67.2 (2)	S1*a*—O3*a*—C3*a*—C4*a*	106.8 (1)
S1—O3—C3—C2	−114.5 (1)	S1*a*—O3*a*—C3*a*—C2*a*	−75.8 (2)
C2—N2—C7—O4	−2.5 (2)	C2*a*—N2*a*—C7*a*—O4*a*	−3.9 (2)
C2—N2—C7—C8	175.6 (1)	C2*a*—N2*a*—C7*a*—C8*a*	172.2 (1)

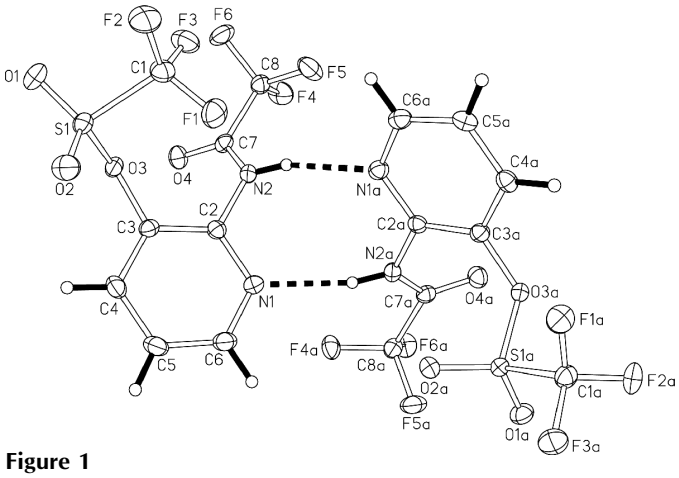


Figure 1
ORTEP (Johnson, 1976) plot of the two independent molecules of $C_8H_4N_2O_4F_6S$, showing 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*n*···N1*a*	0.85 (1)	2.06 (1)	2.880 (2)	164 (2)
N2*a*—H2*na*···N1	0.85 (1)	2.02 (1)	2.858 (2)	167 (2)

The aromatic H atoms were placed at calculated positions [C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C)] and were included in the refinement in the riding-model approximation. The amino H atoms were located in a difference map and refined with a distance restraint [N—H = 0.85 (1) Å].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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2-(2,2,2-Trifluoroacetylamino)pyridin-3-yl trifluoromethanesulfonate. Addendum

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Owing to unfortunate circumstances the paper by Huang, Zhang & Sung [*Acta Cryst.* (2004), E**60**, o708–o710] reports the same structure as the paper by Huang, Liu, Hu & Ng [*Acta Cryst.* (2004), E**60**, o308–o309] and hence the two papers should be read together. The authors of the later paper apologise unreservedly for this problem.