

2-(2,2,2-Trifluoroacetyl)pyridin-3-yl  
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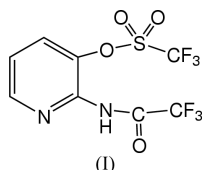
## Key indicators

Single-crystal X-ray study  
 $T = 100\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.032  
 $wR$  factor = 0.081  
Data-to-parameter ratio = 14.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

In the title compound,  $\text{C}_8\text{H}_4\text{N}_2\text{O}_4\text{F}_6\text{S}$ ,  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds [ $\text{N}\cdots\text{N} = 2.857(3)$  and  $2.862(3)\text{ \AA}$ ] form a dimer from the two independent molecules in the asymmetric unit and dictate the conformation of the molecule. The dimers are connected by weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{F}\cdots\text{F}$  interactions to form a two-dimensional supramolecular architecture.

## Comment

Aminopyridines containing polyfluorine groups have attracted the attention of chemists in recent years because of their importance in high optical transparency and thermal stability (Ide & Topacli, 2000). In our designed synthesis for bioactive drugs, trifluoromethanesulfonic and trifluoroacetic acids have been used as protecting groups during further functional synthesis (Park *et al.*, 1998). The title compound, (I), is an aminopyridinyl ester in which the amine and hydroxyl moieties are protected. In the present study, we determined the structure of (I) as part of a continuing study of protectively fluorinated molecules with reference to their optical and thermal properties (Greene & Wuts, 1999).



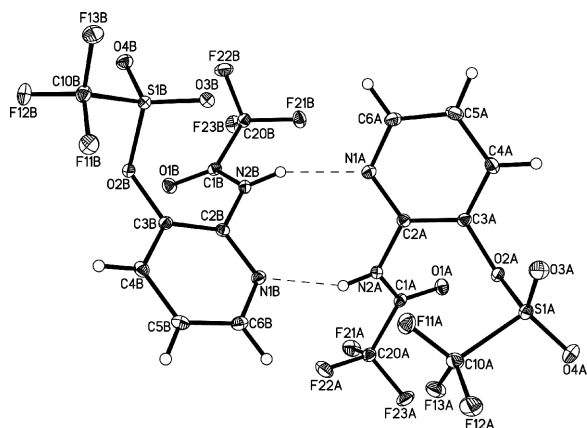
The asymmetric unit of (I) contains two molecules, *A* and *B*. The bond lengths and angles of these molecules are quite similar and are normal. In fact, there is an approximate center of symmetry between molecules *A* and *B*. As illustrated in Fig. 1, the two molecules form a dimer *via* near-linear hydrogen bonds  $\text{N}-\text{H}\cdots\text{N}$  [ $\text{N}\cdots\text{N} = 2.857(3)$  and  $2.862(3)\text{ \AA}$ , and  $\text{N}-\text{H}\cdots\text{N} = 167^\circ$  in both cases]. The C2/C20 acetylamine fragments are almost planar, with the maximum deviation from the least-squares planes not exceeding  $0.020\text{ \AA}$ . These almost planar units make dihedral angles of  $56.1(2)$  and  $56.8(2)^\circ$  with the pyridine ring mean plane in the two molecules. Both  $\text{C}=\text{O}$  and  $\text{S}=\text{O}$  bonds in the acetyl and sulfonic acid fragments have double-bond character. The  $\text{C}-\text{NH}$  bonds (mean  $1.351\text{ \AA}$ ) in the amine fragment are well within the range of the values normally considered standard for single  $\text{C}-\text{N}$  ( $1.47\text{ \AA}$ ) and double  $\text{C}=\text{N}$  ( $1.28\text{ \AA}$ ) bonds, which indicates that the geometry around N2 is normal  $sp^2$ , as expected for  $\pi$ -conjugation of the acetylamine fragment.

The crystal packing in (I) is governed by the  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds which form pseudo-centrosymmetric dimers of molecules *A* and *B*. The dimers are connected by weak

Received 18 February 2004

Accepted 26 March 2004

Online 31 March 2004



**Figure 1**

A view of the hydrogen-bonded dimer of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines

intermolecular C—H...O(C) hydrogen bonds [ $C\cdots O = 3.290(3)$  and  $3.292(3)$  Å, and  $C-H\cdots O = 150$  and  $149^\circ$ ] and  $F\cdots F$  interactions (average  $F\cdots F = 2.825$  Å), forming a two-dimensional supramolecular architecture (Fig. 2).

## Experimental

The title compound was prepared by the elimination reaction of 2-aminophenol (2.5 g, 0.023 mol) with trifluoroacetic acid (3.75 g, 0.025 mol) and trifluoromethanesulfonic acid (2.45 g, 0.025 mol) in the presence of triethylamine (5 ml) as base catalyst. The mixture, in 45 ml tetrahydrofuran, was stirred under reflux until the desired product was obtained (TLC monitoring). The solvent was removed by rotary evaporation and the residue was separated on a silica-gel column with petroleum ether–ethyl acetate (1:2) as eluants. Single crystals suitable for X-ray crystallographic analysis were obtained by slow evaporation of a solution in ethyl acetate.

### Crystal data

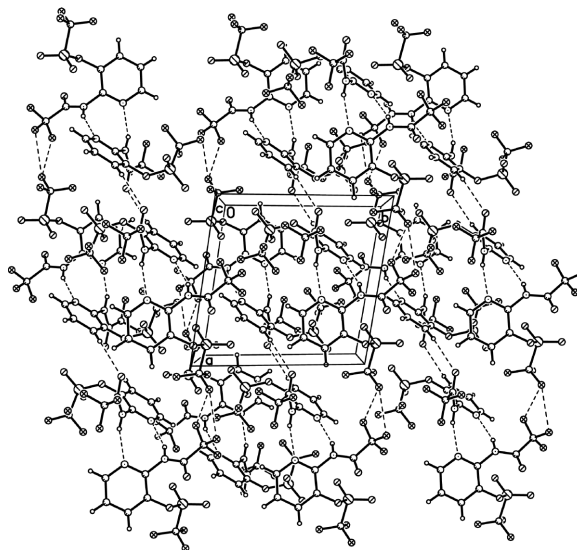
$C_8H_4F_6N_2O_4S$	$Z = 4$
$M_r = 338.19$	$D_x = 1.911$ Mg m $^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.8702(5)$ Å	Cell parameters from 6114 reflections
$b = 10.0063(5)$ Å	$\theta = 2.4\text{--}28.3^\circ$
$c = 12.6434(6)$ Å	$\mu = 0.38$ mm $^{-1}$
$\alpha = 101.436(1)^\circ$	$T = 100(2)$ K
$\beta = 103.466(1)^\circ$	Block, colorless
$\gamma = 95.981(1)^\circ$	$0.40 \times 0.35 \times 0.30$ mm
$V = 1175.37(10)$ Å $^3$	

### Data collection

Bruker SMART CCD area-detector diffractometer	5479 independent reflections
$\varphi$ and $\omega$ scans	4852 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997a)	$R_{int} = 0.018$
$T_{min} = 0.840$ , $T_{max} = 0.893$	$\theta_{max} = 28.3^\circ$
13682 measured reflections	$h = -12 \rightarrow 12$
	$k = -12 \rightarrow 13$
	$l = -16 \rightarrow 16$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.36P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.46$ e Å $^{-3}$
5479 reflections	$\Delta\rho_{min} = -0.30$ e Å $^{-3}$
379 parameters	
H-atom parameters constrained	



**Figure 2**

Crystal packing diagram of (I), with weak intermolecular hydrogen bonds indicated by dashed lines.

**Table 1**

Selected geometric parameters (Å, °).

S1A—O4A	1.4068 (12)	S1B—O3B	1.4139 (11)
S1A—O3A	1.4108 (12)	S1B—O4B	1.4149 (11)
S1A—O2A	1.5842 (10)	S1B—O2B	1.5835 (10)
S1A—C10A	1.8375 (16)	S1B—C10B	1.8385 (15)
O1A—C1A	1.2068 (18)	O1B—C1B	1.2064 (18)
O2A—C3A	1.4057 (17)	O2B—C3B	1.4120 (17)
N1A—C2A	1.3308 (19)	N1B—C2B	1.3254 (18)
N1A—C6A	1.3395 (19)	N1B—C6B	1.341 (2)
N2A—C1A	1.3471 (18)	N2B—C1B	1.3538 (18)
N2A—C2A	1.4109 (18)	N2B—C2B	1.4116 (18)
O4A—S1A—O3A	124.43 (7)	O3B—S1B—O2B	111.37 (6)
O4A—S1A—O2A	105.32 (6)	O4B—S1B—O2B	106.16 (6)
O3A—S1A—O2A	111.47 (6)	O3B—S1B—C10B	108.06 (7)
O4A—S1A—C10A	106.44 (7)	O4B—S1B—C10B	106.50 (7)
O3A—S1A—C10A	106.41 (7)	O2B—S1B—C10B	99.86 (6)
O2A—S1A—C10A	100.03 (6)	C3B—O2B—S1B	120.39 (8)
C3A—O2A—S1A	120.23 (9)	C2B—N1B—C6B	118.46 (13)
C2A—N1A—C6A	118.13 (13)	C1B—N2B—C2B	123.35 (12)
C1A—N2A—C2A	121.49 (12)	O1B—C1B—N2B	127.66 (13)
O1A—C1A—N2A	127.04 (14)	O1B—C1B—C20B	119.68 (13)
O1A—C1A—C20A	118.31 (13)	N2B—C1B—C20B	112.55 (12)
N2A—C1A—C20A	114.62 (12)	N1B—C2B—C3B	121.32 (13)
N1A—C2A—C3A	121.42 (13)	N1B—C2B—N2B	116.56 (12)
N1A—C2A—N2A	116.51 (12)	C3B—C2B—N2B	122.00 (12)
C3A—C2A—N2A	122.00 (13)	C4B—C3B—O2B	119.21 (13)
N1A—C6A—C5A	123.21 (14)	C2B—C3B—O2B	120.02 (12)
O3B—S1B—O4B	122.47 (7)	N1B—C6B—C5B	122.98 (14)

All H atoms were found in a difference electron-density map, but were then placed in calculated positions ( $C-H = 0.95$  Å and  $N-H = 0.88$  Å) and allowed to ride on their parent atoms, with  $U_{iso}(H) = 1.2U_{eq}(\text{parent})$ .

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); program(s) used to solve structure: SHELXTL (Sheldrick, 1997b); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

This work is supported by the Key Scientific Research Foundation of State Education Ministry (grant No. 204067)

and the Natural Science Foundation of China (grant No. 90301005). QFZ is grateful for the assistance of the Alexander von Humboldt Foundation.

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## 2-(2,2,2-Trifluoroacetyl-amino)pyridin-3-yl trifluoromethanesulfonate. Addendum

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Owing to unfortunate circumstances the paper by Huang, Zhang & Sung [*Acta Cryst.* (2004), E60, o708–o710] reports the same structure as the paper by Huang, Liu, Hu & Ng [*Acta Cryst.* (2004), E60, o308–o309] and hence the two papers should be read together. The authors of the later paper apologise unreservedly for this problem.