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3-(Aminocarbonyl)pyridinium trifluoroacetate

S. Athimoolam* and S. Natarajan

Department of Physics, Madurai Kamaraj University, Madurai 625 021, India

Correspondence e-mail:
xrdsopmku@yahoo.com

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Key indicators

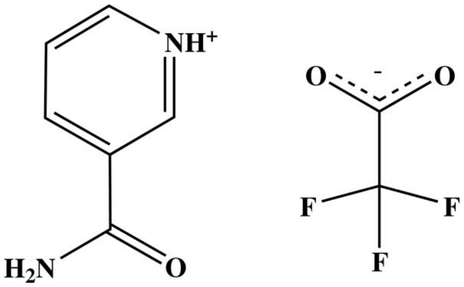
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.066
wR factor = 0.212
Data-to-parameter ratio = 10.0

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

In the title compound, $C_6H_7N_2O^+\cdot C_2O_2F_3^-$, the carboxamide group is twisted by 16.3 (8)° from the pyridine ring plane. The protonated cations are linked to the anions by N—H···O hydrogen bonds to form a distorted lamellar structure.

Comment

Nicotinamide (also known as 3-pyridinecarboxamide), one of the two principal forms of the vitamin B complex, has considerable biological importance. Its major metabolite, NAD^{2+} (nicotinamide adenine dinucleotide), is involved in the production of energy, the synthesis of fatty acids, cholesterol and steroids, signal transduction, and the maintenance of the integrity of the genome (Kuhajda, 2000). It is also used in pharmacological doses as antidiabetogenic (Pociot *et al.* 1993) and antihyperlipidaemic agents (Sampathkumar *et al.*, 2006). Salts of nicotinamide with 3,5-dinitrosalicylate (Koman *et al.*, 2003) and nitrate (Gubin *et al.*, 1988) have been reported previously. The planar nicotinamide molecule has three potential sites for hydrogen-bonding interactions, *viz*. the pyridine N atom, the amine N atom and the carbonyl O atom. These sites make this molecule an important supramolecular organic synthon. Hydrogen-bonded assemblies possess unique chemical and physical properties (Desiraju, 1989). Supramolecular interactions in salts of isonicotinamide have also been described (Aakeroy *et al.*, 2002) and we report the structure of the title compound, (I), here as part of our ongoing investigations of hydrogen bonding in vitamin–inorganic/organic salts.



(I)

The asymmetric unit of (I) contains a nicotinamidium cation and a trifluoroacetate anion (Fig. 1). Deprotonation of the anions and protonation of the cations are confirmed by the C—N and C—O bond distances. Characteristic features of protonated nicotinamide structures are the twisting of the carboximide plane out of the pyridine ring plane (by 2–40°) and a significant widening of the C—N—C angle in the pyri-

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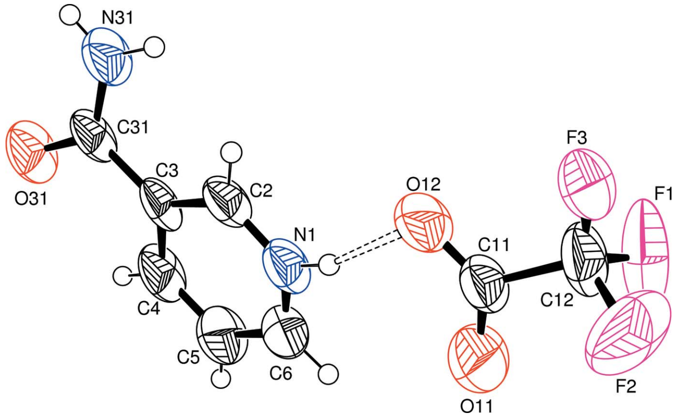

Figure 1
The molecular structure of compound (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. Only the F atoms of the major disorder component are shown. The double dashed line indicates the hydrogen bond.

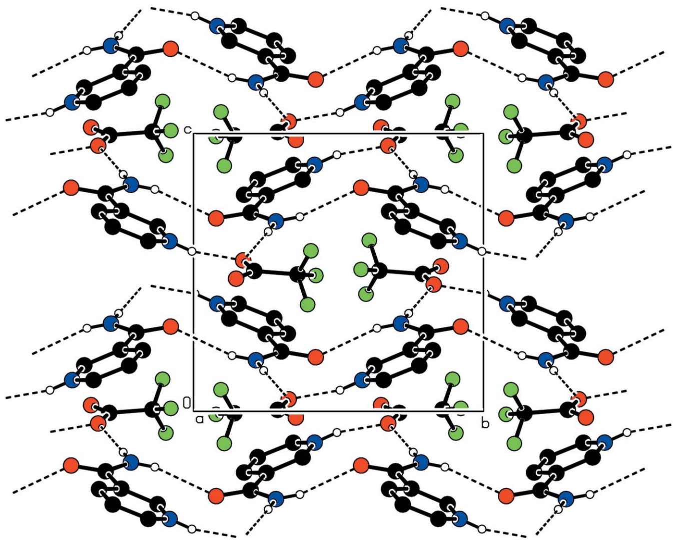

Figure 2
A packing diagram, viewed down the *a* axis. H atoms not involved in the hydrogen bonds and the F atoms of the minor disorder component have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

dine ring (>120°) (from ten nicotinamidium structures in the Cambridge Structural Database, Version 5.27 of 2006; Allen, 2002). Both of these features are observed in (I), the angle between the carboxamide and pyridine ring planes being 16.3 (8)° and the C2—N1—C6 angle being 122.5 (4)°.

The cations of (I) are linked through N—H···O hydrogen bonds, forming a $C(4)$ chain motif (Etter *et al.*, 1990). In general, this type of homonuclear interaction leads to a sheet-like structure (Athimoolam & Natarajan, 2006). However, in (I), the arrangement of the ions can be described as corrugated sheets of cations (Fig. 2) with anions located between the sheets, forming $R_3^2(12)$ ring motifs through N—H···O hydrogen bonds (Fig. 3 and Table 1).

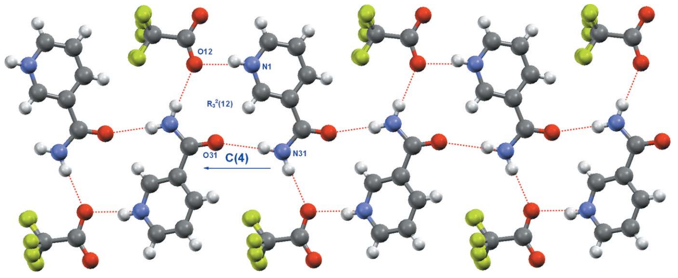

Figure 3
Aggregation of the cations and anions generated by hydrogen bonds (shown as dashed lines), showing graph-set motifs of $C(4)$ chains and $R_3^2(12)$ rings.

Experimental

Compound (I) was crystallized from an aqueous mixture containing nicotinamide and trifluoroacetic acid (1:1) by the technique of slow evaporation.

Crystal data

$C_6H_7N_2O^+ \cdot C_2O_2F_3^-$
$M_r = 236.16$
Monoclinic, $P2_1/c$
$a = 10.045\ (6)$ Å
$b = 10.138\ (8)$ Å
$c = 10.510\ (7)$ Å
$\beta = 112.42\ (4)^\circ$
$V = 989.4\ (12)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 293\ (2)$ K
$0.24 \times 0.21 \times 0.18$ mm

Data collection

Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.928$, $T_{\max} = 0.972$
2054 measured reflections
1727 independent reflections
778 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.212$
$S = 1.01$
1727 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O12	0.86	1.84	2.671 (5)	163
N31—H31A···O12$^{\rm i}$	0.86	2.11	2.969 (5)	172
N31—H31B···O31$^{\rm ii}$	0.86	2.38	3.205 (5)	162

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and N—H = 0.86 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$ (C,N). The F atoms of the trifluoroacetate are disordered over two positions, with site occupancies of 0.55 (1) and 0.45 (1). Despite attempts to model this disorder effectively, the displacement parameters for the F atoms remain very high.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *ORTEP-3* (Farrugia, 1997), *Mercury* (Version 1.4.1; Macrae *et al.*, 2006) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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