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N,*N*′-[(2,3,5,6-Tetrafluoro-1,4-phenylene)-dimethylene]bis(pyridine-4-carboxamide)

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Key indicators

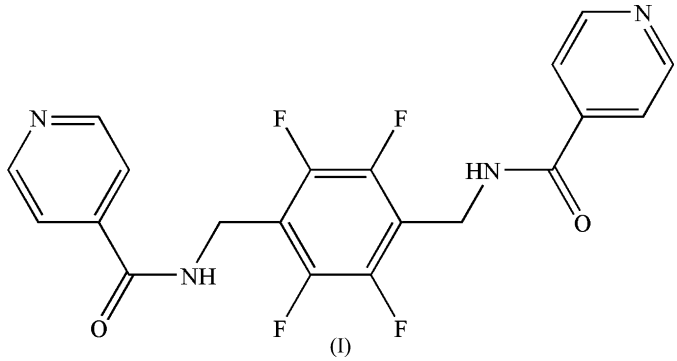
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.126
Data-to-parameter ratio = 16.3

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

In the centrosymmetric molecule of the title compound, $C_{20}H_{14}F_4N_4O_2$, the pyridine ring is twisted with respect to the benzene ring with a dihedral angle of 66.85 (10)°. In the crystal structure, molecules are linked by intermolecular hydrogen-bonding and C—H···π interactions.

Comment

Pyridinecarboxamide derivatives have attracted much attention because of their biological activity. Some derivatives of pyridinecarboxamide possess anticancer (Liu *et al.*, 1996) and anti-allergic activities (Tsuzurahara *et al.*, 1987), and are efficient as IKK inhibitors (Li & Prakash, 2005), monoamine oxidase B inhibitors (Blauenstein *et al.*, 1998) and anticonvulsant drugs (Palmer *et al.*, 1993). As part of a search for new pyridinecarboxamide compounds with better biological activity, the title compound, (I), has been synthesized. Here we report the crystal its structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule of (I) is centrosymmetric. The pyridine ring is twisted with respect to the benzene ring, with a dihedral angle of 66.85 (10)°.

Weak C—H···π interactions are observed in the crystal structure (Fig. 2); C3—H3 = 0.93, H3···Cg^{iv} = 2.97, C3···Cg^{iv} = 3.726 (3) Å and C3—H3···Cg^{iv} = 139°; C5—H5 = 0.93, H5···Cg^{v} = 3.05, C5···Cg^{v} = 3.756 (3) Å and C5—H5···Cg^{v} = 133° [*Cg* is the centroid of the pyridine ring; symmetry codes: (iv) $\frac{5}{2}-x,\frac{1}{2}+y,\frac{3}{2}-z$; (v) $\frac{3}{2}-x,-\frac{1}{2}+y,\frac{3}{2}-z$]. Classical N—H···O and weak C—H···O and C—H···F hydrogen bonding occurs in the crystal structure (Table 1), which helps to stabilize the crystal structure (Fig. 3).

Experimental

To a stirred suspension of isonicotinyl chloride (3.92 g, 22 mmol) and 1,4-bis(aminomethyl)-2,3,5,6-tetrafluorobenzene (2.08 g, 10 mmol) in dry tetrahydrofuran (25 ml) was added dropwise triethylamine (3 ml) at 273 K. The mixture was maintained at room temperature for 48 h

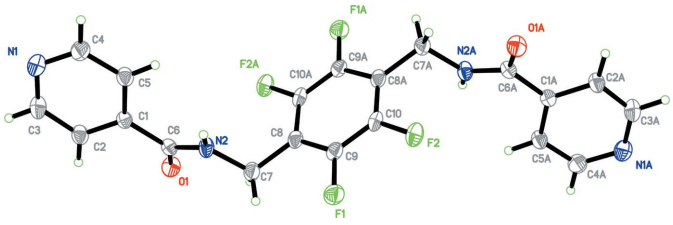


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for H atoms) [symmetry code: (A) $1-x, 1-y, 2-z$].

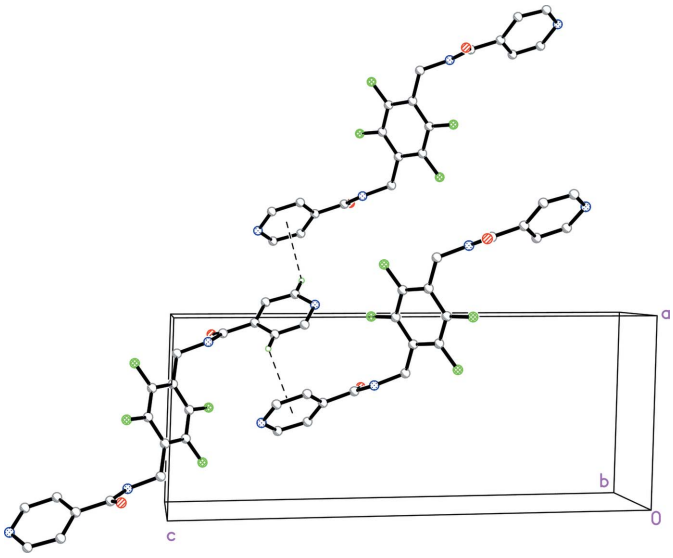


Figure 2
The C—H$\cdots\pi$ interactions (dashed lines) in (I).

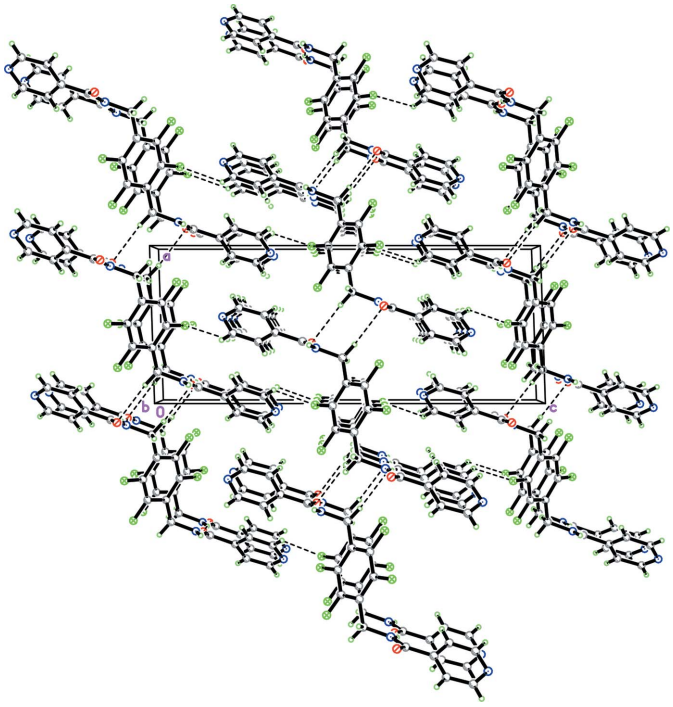


Figure 3
A packing diagram for (I). Dashed lines indicate hydrogen bonds.

with stirring under a nitrogen atmosphere, then evaporated to dryness. The resulting residue was washed with iced water (25 ml), saturated sodium carbonate solution (12 ml), and again with iced water (15 ml). After work-up, the crude product was dried under vacuum and purified by recrystallization from methanol to yield (I) (3.81 g, 91% yield) as a white solid. Single crystals of (I) were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{20}H_{14}F_4N_4O_2$
M_r = 418.35
Monoclinic, $P2_1/n$
a = 8.3488 (17) Å
b = 5.1431 (11) Å
c = 20.665 (4) Å
β = 91.843 (2)°
V = 886.9 (3) Å^3
Z = 2
D_x = 1.567 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 (2) K
Block, colorless
0.30 × 0.20 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7535 measured reflections
2213 independent reflections
1302 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 28.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.126
S = 1.03
2213 reflections
136 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.1461P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$O1^{i}	0.88	2.10	2.927 (2)	155
C4—H4$\cdots$F2^{ii}	0.93	2.53	3.242 (3)	134
C7—H7$B\cdots$O1^{iii}	0.97	2.50	3.393 (3)	153

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

The imino H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were placed in calculated positions, with C—H = 0.97 (methylene) or 0.93 Å (aromatic), and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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