

2,3,4,5,6-Pentafluorobenzamide

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Key indicators

Single-crystal X-ray study

T = 120 K

Mean $\sigma(C-C)$ = 0.002 Å

R factor = 0.035

wR factor = 0.090

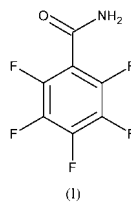
Data-to-parameter ratio = 11.2

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

The dihedral angle between the amide group and the pentafluorophenyl moiety of the title compound, $C_7H_2F_5ON$, is $43.56(5)^\circ$. The molecules are connected *via* $N-H \cdots O=C$ bonds into an infinite, two-dimensional network generated by the cyclic $R_2^2(8)$ and chain $C(4)$ motifs.

Comment

A common pattern of assembly of primary amides is *via* centrosymmetric, cyclic $R_2^2(8)$ hydrogen-bond motif involving the *syn*-oriented NH_s groups, and *via* the $C(4)$ motif with the use of *anti*-oriented NH_a groups. Two situations may arise depending on the symmetry operation relating the amide molecules within the $C(4)$ chain. When amide cyclic dimers are related by *ca* 5.1 Å translation, a one-dimensional assembly of hydrogen-bonded molecules is generated. In turn, the glide plane or 2_1 symmetry operating between the dimers leads to a two-dimensional molecular network (Leiserowitz & Hagler, 1983; Bernstein *et al.*, 1994). The former assembly mode is generally preferred over the latter and is found, for example, in benzamide (Blake & Small, 1972) and in its 2- and 3-fluoro derivatives (Taniguchi *et al.*, 1975; Kato & Sakurai, 1982), while molecules of 4-fluorobenzamide are hydrogen-bonded to form the two-dimensional network (Kubota & Ohba, 1992). On the other hand, 4-chlorobenzamide, which crystallizes in three polymorphic forms, can create both types of assemblies (Taniguchi *et al.*, 1978; Hayashi *et al.*, 1980). Whereas the crystal structure of 2,3,4,5,6-pentafluorobenzoic acid has been known for over 30 years (Benghiat & Leiserowitz, 1972), the structure of its amide analogue, (I), has not been reported so far.



The structure of (I), with the atom-numbering scheme, is shown in Fig. 1. The molecule of (I) is not planar and its amide group is *ca* 15° more twisted relative to the aromatic ring than in the benzamide structure (Blake & Small, 1972). The largest difference in the geometry of the two amide molecules occurs in the value of the endocyclic bond angle at C1, which is $120.1(3)^\circ$ in benzamide and only $116.59(13)^\circ$ in its fluoro analogue. Similar changes have been observed in the geometries of benzoic (Bruno & Randaccio, 1980) and pentafluorobenzoic acids (Benghiat & Leiserowitz, 1972)

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The hydrogen-bonding geometry is given in Table 2. In contrast to benzamide, the molecules of (I) are assembled in the crystal structure into a two-dimensional hydrogen-bonded network parallel to the (001) plane (Fig. 2). The amide NH_a groups take part in hydrogen bonds joining glide-plane-related centrosymmetric R₂²(8) dimers. Each molecule gives rise to five short intermolecular F···F contacts in the range 2.8117 (13)–2.8605 (13) Å. No significant intermolecular π–π stacking interactions are found between the perfluorophenyl rings; however, there are two short F···C contacts of 2.927 (2) and 3.016 (2) Å. They occur between the *meta*-fluoro group F5 and the benzene C3 and carbonyl C7 atoms of molecules related by a unit translation along *b*. As has been shown recently, the C–F···O=C interactions are robust in fluorine-containing organic carbonyl compounds. They are best described as multipolar interactions between the intrinsically polar C–F and C=O groups (Olsen *et al.*, 2003).

Experimental

Commercially available 2,3,4,5,6-pentafluorobenzamide was recrystallized by slow evaporation of an ethanol solution.

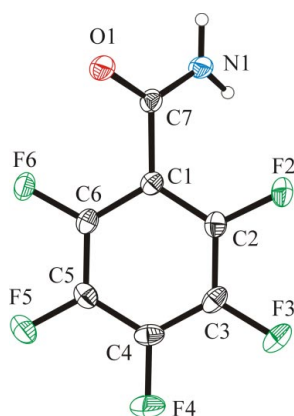


Figure 1
View of the molecule of (I), shown with 50% probability displacement ellipsoids.

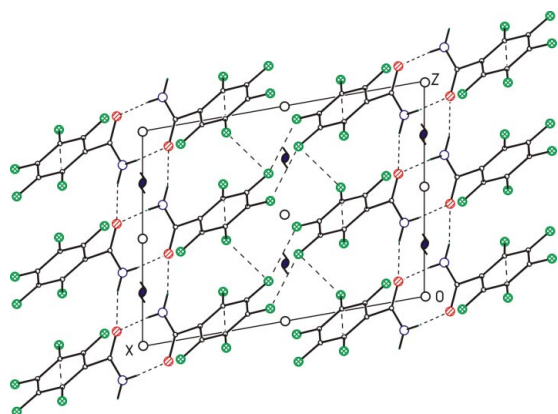


Figure 2
Packing diagram for (I), viewed along the *b* axis. Hydrogen bonds and short F···F contacts are shown by dashed lines.

Crystal data

C₇H₂F₅NO
M_r = 211.10
Monoclinic, P2₁/c
a = 12.7305 (11) Å
b = 6.1929 (6) Å
c = 9.5856 (10) Å
β = 100.073 (8)°
V = 744.07 (12) Å³
Z = 4

D_x = 1.884 Mg m⁻³
Mo Kα radiation
Cell parameters from 2021 reflections
θ = 4–25°
μ = 0.21 mm⁻¹
T = 120 (2) K
Prism, colorless
0.40 × 0.20 × 0.20 mm

Data collection

Kuma KM-4 CCD diffractometer
ω scans
4010 measured reflections
1517 independent reflections
1376 reflections with I > 2σ(I)

R_{int} = 0.016
θ_{max} = 26.4°
h = -15 → 15
k = -7 → 7
l = -11 → 5

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.035
wR(F²) = 0.090
S = 1.13
1517 reflections
135 parameters
All H-atom parameters refined

w = 1/[σ²(F_o²) + (0.048P)² + 0.2307P]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.002
Δρ_{max} = 0.27 e Å⁻³
Δρ_{min} = -0.26 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1–C7	1.2392 (17)	C1–C7	1.513 (2)
N1–C7	1.3217 (19)		
C2–C1–C6	116.59 (13)	O1–C7–C1	119.08 (13)
O1–C7–N1	123.90 (14)	N1–C7–C1	117.02 (12)
C2–C1–C7–N1	44.8 (2)	C6–C1–C7–O1	41.2 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

D–H···A	D–H	H···A	D···A	D–H···A
N1–H1···O1 ⁱ	0.88 (2)	2.05 (2)	2.8902 (17)	158.3 (17)
N1–H2···O1 ⁱⁱ	0.89 (2)	2.03 (2)	2.9137 (17)	175.5 (16)

Symmetry codes: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (ii) $-x, 2 - y, -z$.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2000); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

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