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2-(2-Fluorobenzoylamino)benzoic Acid

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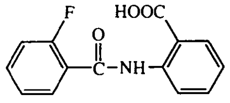
Abstract

The structure of the title compound, $C_{14}H_{10}FNO_3$, was determined at 173 K. The conformation of the molecule is such that both the F atom and the carbonyl O atom of the carboxyl group are located near the amide H atom. Molecules are linked to each other by centrosymmetric hydrogen-bonding interactions through the carboxyl groups, but there are no intermolecular hydrogen bonds involving the amide linkage.

Comment

The title compound, (1), was prepared and its crystal structure determined at low temperature (173 K) as part of a study of isosteric analogs of the nonsteroidal anti-inflammatory and analgesic agent salicylsalicylic acid (salsalate).



(1)

The atom-numbering scheme is shown in Fig. 1. The carboxyl group, the ring to which it is bonded and the amide linkage are nearly coplanar [O(2)—C(14)—C(9)—C(8) = 1.5 (3) and C(7)—N(1)—C(8)—C(9) = 179.9 (2)°]. On the other hand, the fluorinated ring is twisted out of coplanarity with the rest of the molecule [N(1)—C(7)—C(1)—C(2) = 11.4 (3)°]. Nevertheless, this twist is smaller than those found in the two solid-state conformations of 2-fluorobenzamide, where the ring–carbamoyl dihedral angles are 31.7 and 35.4° (Kato & Sakurai, 1982). As a result, both the F atom and the acid carbonyl O atom lie near the amide H atom in (1); F(1)···H(1N) = 2.05 (2) Å and N(1)—H(1N)···F(1) = 133 (2)°, and O(2)···H(1N) = 1.94 (2) Å and N(1)—H(1N)···O(2) = 137 (2)°.

A stereoview of the molecular packing is shown in Fig. 2. The molecules hydrogen bond intermolecularly through their carboxyl groups about crystallographic inversion centers, forming centrosymmetric dimers [O(2)···O(3^i) = 2.637 (2) Å and O(2)···H(1O^i) = 1.70 (2) Å; symmetry code: (i) $-x$, $-1-y$, $1-z$], which are packed in a 'herringbone' arrangement. The amide linkage, with its N—H donor group located in a sterically hindered position, does not participate in any intermolecular hydrogen-bonding interactions.

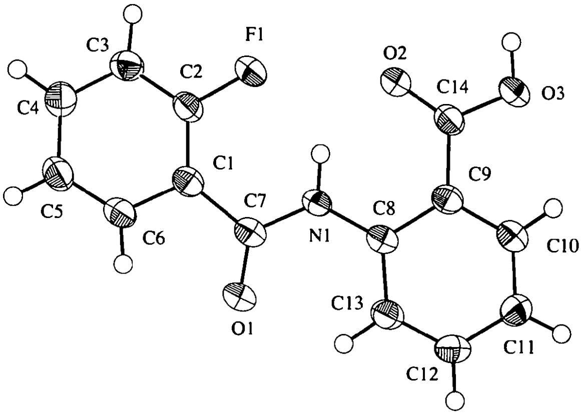


Fig. 1. *ORTEP*II (Johnson, 1976) plot of (1) showing the atom-numbering scheme. H atoms are shown as spheres of fixed size and non-H atoms are shown as 50% probability displacement ellipsoids.

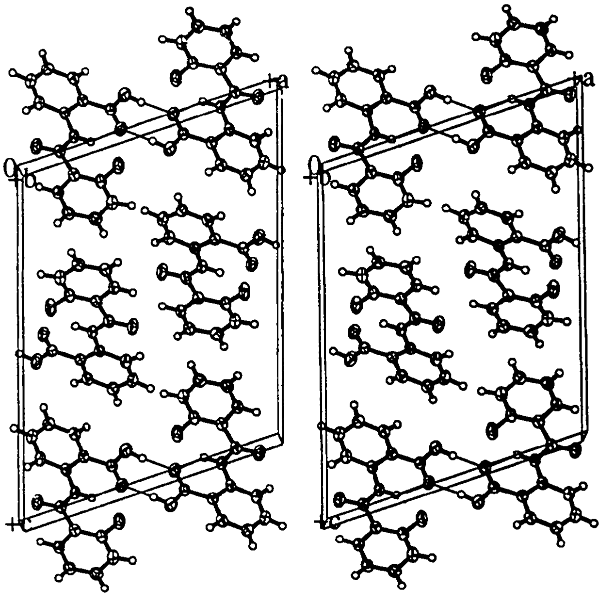


Fig. 2. Stereoview of the molecular packing, with thin solid lines representing hydrogen bonds linking the molecules into dimers.

Experimental

The title compound, (1), was prepared by reaction of anthranilic acid with 2-fluorobenzoyl chloride (Aldrich). Recrystallization was from aqueous methanol.

Crystal data

$C_{14}H_{10}FNO_3$
$M_r = 259.24$
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å

Monoclinic
$P2_1/n$
$a = 13.590(2)$ Å
$b = 5.003(2)$ Å
$c = 17.627(2)$ Å
$\beta = 108.26(1)°$
$V = 1138.1(4)$ Å^3
$Z = 4$
$D_x = 1.513$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 23.9$–$24.9°$
$\mu = 0.96$ mm^{-1}
$T = 173$ K
Plate
$0.50 \times 0.40 \times 0.05$ mm
Light brown

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction:
ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.82$, $T_{\max} = 1.00$
4898 measured reflections
2348 independent reflections
1412 observed reflections
$[I > 3\sigma(I)]$

$R_{\rm int} = 0.035$
$\theta_{\max} = 70.1°$
$h = -16 \rightarrow 0;\ 0 \rightarrow 16$
$k = -6 \rightarrow 0;\ 0 \rightarrow 6$
$l = -20 \rightarrow 21;\ -21 \rightarrow 20$
3 standard reflections monitored every 150 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.034$
$wR = 0.037$
$S = 2.12$
1412 reflections
202 parameters
Only coordinates of H atoms refined

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
F(1)	0.12410 (9)	0.1200 (3)	0.42593 (9)	4.13 (6)
O(1)	0.4403 (1)	0.1349 (4)	0.5584 (1)	4.01 (7)
O(2)	0.1048 (1)	−0.3306 (3)	0.51021 (9)	3.34 (6)
O(3)	0.0840 (1)	−0.6471 (4)	0.5922 (1)	4.12 (7)
N(1)	0.2907 (1)	−0.0963 (4)	0.5427 (1)	2.55 (6)
C(1)	0.2988 (1)	0.2553 (4)	0.4476 (1)	2.58 (8)
C(2)	0.1939 (1)	0.2728 (4)	0.4041 (1)	2.79 (8)
C(3)	0.1545 (2)	0.4399 (5)	0.3402 (1)	3.23 (8)
C(4)	0.2216 (2)	0.6022 (5)	0.3158 (1)	3.24 (8)
C(5)	0.3271 (2)	0.5916 (5)	0.3572 (1)	3.21 (9)
C(6)	0.3635 (2)	0.4212 (5)	0.4202 (1)	2.94 (8)
C(7)	0.3498 (1)	0.0926 (4)	0.5211 (1)	2.67 (7)
C(8)	0.3214 (1)	−0.2676 (4)	0.6091 (1)	2.46 (7)
C(9)	0.2489 (1)	−0.4530 (4)	0.6224 (1)	2.50 (7)
C(10)	0.2806 (2)	−0.6260 (5)	0.6875 (1)	2.91 (8)
C(11)	0.3792 (2)	−0.6227 (5)	0.7398 (1)	3.01 (8)
C(12)	0.4495 (2)	−0.4403 (5)	0.7277 (1)	3.15 (8)
C(13)	0.4213 (2)	−0.2659 (4)	0.6639 (1)	2.92 (8)
C(14)	0.1406 (2)	−0.4691 (4)	0.5698 (1)	2.72 (8)

Table 2. *Selected geometric parameters (Å, °)*

F(1)—C(2)	1.364 (2)	C(3)—C(4)	1.386 (3)
O(1)—C(7)	1.218 (2)	C(4)—C(5)	1.392 (3)
O(2)—C(14)	1.224 (3)	C(5)—C(6)	1.364 (3)
O(3)—C(14)	1.316 (2)	C(8)—C(9)	1.426 (3)
N(1)—C(7)	1.369 (3)	C(8)—C(13)	1.398 (3)
N(1)—C(8)	1.404 (3)	C(9)—C(10)	1.393 (3)
C(1)—C(2)	1.394 (3)	C(9)—C(14)	1.476 (3)
C(1)—C(6)	1.400 (3)	C(10)—C(11)	1.369 (3)
C(1)—C(7)	1.503 (3)	C(11)—C(12)	1.385 (3)
C(2)—C(3)	1.369 (3)	C(12)—C(13)	1.380 (3)
C(7)—N(1)—C(8)	127.3 (2)	N(1)—C(8)—C(9)	119.6 (2)
C(2)—C(1)—C(6)	114.7 (2)	N(1)—C(8)—C(13)	122.9 (2)
C(2)—C(1)—C(7)	128.6 (2)	C(9)—C(8)—C(13)	117.5 (2)
C(6)—C(1)—C(7)	116.7 (2)	C(8)—C(9)—C(10)	119.2 (2)
F(1)—C(2)—C(1)	119.4 (2)	C(8)—C(9)—C(14)	122.5 (2)
F(1)—C(2)—C(3)	116.5 (2)	C(10)—C(9)—C(14)	118.3 (2)
C(1)—C(2)—C(3)	124.0 (2)	C(9)—C(10)—C(11)	122.2 (2)
C(2)—C(3)—C(4)	119.2 (2)	C(10)—C(11)—C(12)	118.7 (2)
C(3)—C(4)—C(5)	119.0 (2)	C(11)—C(12)—C(13)	120.9 (2)
C(4)—C(5)—C(6)	120.1 (2)	C(8)—C(13)—C(12)	121.4 (2)
C(1)—C(6)—C(5)	123.0 (2)	O(2)—C(14)—O(3)	121.9 (2)
O(1)—C(7)—N(1)	123.5 (2)	O(2)—C(14)—C(9)	123.9 (2)
O(1)—C(7)—C(1)	119.1 (2)	O(3)—C(14)—C(9)	114.2 (2)
N(1)—C(7)—C(1)	117.5 (2)		

Non-H atoms were refined anisotropically and H atoms were refined with isotropic displacement parameters fixed at approximately 1.2 times the isotropic equivalent of the attached atom [C—H distances 0.92 (2)–0.99 (2), N—H 0.91 (2) and O—H 0.94 (2) Å].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, torsion angles and intermolecular distances have been deposited with the IUCr (Reference: BK1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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