Crystal structure of 4-fluoro-*N*-(2-hydroxy-4-nitrophenyl)benzamide, $C_{13}H_9FN_2O_4$

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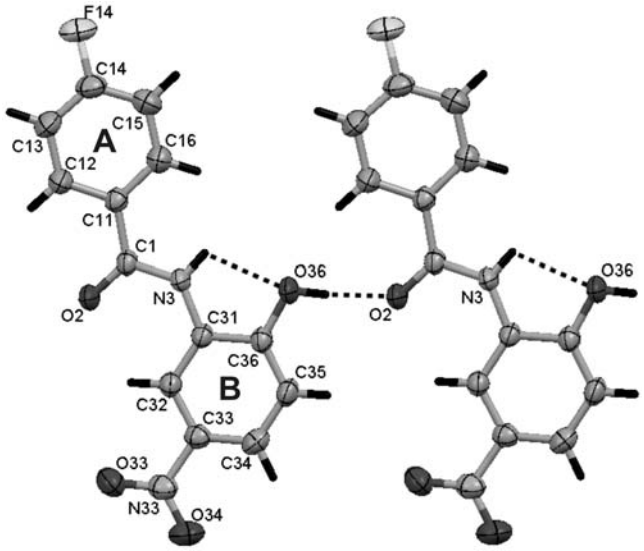

Abstract

$C_{13}H_9FN_2O_4$, monoclinic, $P12_1/c1$ (no. 14), a = 7.3477(7) Å, b = 7.7697(9) Å, c = 21.295(3) Å, β = 99.25(1)°, V = 1199.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.087, T = 298 K.

Source of material

The title structure synthesis was described and published previously [1]. The crystals for X-ray studies were obtained from ethanol solution by slow solvent evaporation.

Discussion

The crystal structure studies of the title compound were conducted with the aim to obtain 3D structure which subsequently can be used as a starting point for molecular modeling of other (2-hydroxyphenyl)benzamides. The dihedral angle between two aromatic rings, describing whole molecule deviation from planarity, is the most important geometrical parameter in the studied structure. The CSD [2] search for (2-hydroxyphenyl)benzamide leading as the basic structure moiety gave three similar structures. Among them one is almost flat (angle close to 0°) [3], while remaining two molecules assume butterfly shape with the higher dihedral angle values exciding 8° and 14°, respectively [3,4]. In the title crystal structure, the dihedral angle between the aromatic rings is 14.1(2)°. The angles between the plane of the amide group and both aromatic rings are not equal. The 4-fluoro-substituted phenyl (ring A) is inclined at 20.9(2)°, while 2-hydroxy-4-nitrophenyl (ring B) - only at 6.8(2)°. This important flattening of the B ring and amide group is caused by stable five-membered ring constructed by intramolecular hydrogen bond of d(N3–H3···O36) = 2.573(1) Å. The crystal structure consists of infinite ribbons of 4-fluoro-*N*-(2-hydroxy-4-nitrophenyl)-benzamide molecules connected by intermolecular hydrogen bonds between the hydroxyl and carbonyl groups of adjacent molecules. The interaction O36–H36···O2 occurring over a distance of 2.591(1) Å and possessing angle of 174.9°, can be classified as a strong hydrogen bond.

Table 1. Data collection and handling.

Crystal:	colourless prisma, size 0.15 × 0.35 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.25 cm^{-1}
Diffractometer, scan mode:	Kuma KM-4 CCD, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13469, 2118
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1588
$N(param)_{refined}$:	182
Programs:	SHELXTL [5], Mercury [6], PLATON [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.2196	0.6100	0.4076	0.045
H(12)	4*e*	–0.3537	0.5925	0.3497	0.058
H(13)	4*e*	–0.4599	0.4413	0.2578	0.069
H(15)	4*e*	0.0452	0.2545	0.2564	0.062
H(16)	4*e*	0.1519	0.4038	0.3489	0.052
H(32)	4*e*	0.0327	0.8239	0.5279	0.044
H(34)	4*e*	0.5290	0.9534	0.6242	0.052
H(35)	4*e*	0.6704	0.8127	0.5488	0.052
H(36)	4*e*	0.6257	0.6552	0.4520	0.070

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	–0.0296(2)	0.6125(2)	0.41899(6)	0.0256(6)	0.0443(8)	0.0342(7)	–0.0006(5)	0.0056(5)	0.0021(6)
O(2)	4*e*	–0.1396(1)	0.6627(1)	0.45321(4)	0.0253(5)	0.0737(7)	0.0458(6)	–0.0041(4)	0.0098(4)	–0.0174(5)
N(3)	4*e*	0.1516(1)	0.6467(1)	0.43413(5)	0.0243(6)	0.0551(7)	0.0342(6)	–0.0015(4)	0.0078(5)	–0.0098(5)
C(11)	4*e*	–0.0895(2)	0.5145(2)	0.35956(6)	0.0308(7)	0.0411(7)	0.0327(7)	–0.0022(5)	0.0055(5)	0.0015(6)
C(12)	4*e*	–0.2727(2)	0.5242(2)	0.33133(7)	0.0341(7)	0.0698(9)	0.0411(8)	0.0022(6)	0.0037(6)	–0.0135(7)
C(13)	4*e*	–0.3368(2)	0.4343(2)	0.27652(7)	0.0394(8)	0.086(1)	0.0443(9)	–0.0002(7)	–0.0038(7)	–0.0148(8)
C(14)	4*e*	–0.2163(2)	0.3353(2)	0.25046(7)	0.0571(9)	0.0610(9)	0.0338(8)	–0.0062(7)	0.0033(7)	–0.0107(7)
F(14)	4*e*	–0.2809(1)	0.2470(1)	0.19644(4)	0.0786(7)	0.1041(8)	0.0511(6)	–0.0027(5)	–0.0020(5)	–0.0371(5)
C(15)	4*e*	–0.0344(2)	0.3222(2)	0.27576(7)	0.0529(9)	0.0548(9)	0.0485(9)	0.0031(7)	0.0134(7)	–0.0122(8)
C(16)	4*e*	0.0284(2)	0.4120(2)	0.33075(7)	0.0356(7)	0.0501(8)	0.0442(8)	0.0000(6)	0.0051(6)	–0.0046(7)
C(31)	4*e*	0.2443(2)	0.7342(2)	0.48749(6)	0.0273(6)	0.0405(7)	0.0314(7)	–0.0014(5)	0.0033(5)	0.0015(6)
C(32)	4*e*	0.1606(2)	0.8200(2)	0.53186(6)	0.0295(7)	0.0449(8)	0.0365(7)	0.0020(5)	0.0046(6)	0.0000(6)
C(33)	4*e*	0.2696(2)	0.8997(2)	0.58219(6)	0.0413(8)	0.0380(7)	0.0333(7)	0.0019(5)	0.0055(6)	0.0002(6)
N(33)	4*e*	0.1770(2)	0.9881(1)	0.62859(6)	0.0558(8)	0.0490(7)	0.0373(7)	0.0055(6)	0.0053(6)	–0.0030(6)
O(33)	4*e*	0.0083(2)	0.9935(1)	0.61987(5)	0.0523(7)	0.0749(7)	0.0531(7)	0.0139(5)	0.0142(5)	–0.0079(6)
O(34)	4*e*	0.2721(2)	1.0550(2)	0.67453(6)	0.0745(8)	0.0897(8)	0.0520(7)	0.0027(6)	–0.0007(6)	–0.0316(6)
C(34)	4*e*	0.4588(2)	0.8986(2)	0.58972(7)	0.0410(8)	0.0480(8)	0.0380(8)	–0.0052(6)	–0.0035(6)	–0.0021(6)
C(35)	4*e*	0.5425(2)	0.8143(2)	0.54477(6)	0.0270(7)	0.0543(9)	0.0467(8)	–0.0038(6)	0.0004(6)	–0.0005(7)
C(36)	4*e*	0.4375(2)	0.7327(2)	0.49411(6)	0.0281(7)	0.0421(7)	0.0368(7)	0.0000(5)	0.0075(6)	0.0020(6)
O(36)	4*e*	0.5059(1)	0.6493(1)	0.44779(4)	0.0239(5)	0.0709(6)	0.0473(6)	–0.0014(4)	0.0092(4)	–0.0121(5)

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