Crystal structure of 2-chloro-5-fluoro-*N*-(4-fluorophenyl)benzamide, $C_{13}H_8ClF_2NO$

Qin Huang*[,I,II,III], Chuang Zhang[I], Xiaoping Rao[II] and Li-Hong Diao[I]

[I] College of Chemistry & Ecological Engineering, Guangxi University for Nationalities, Nanning 53006, P. R. China
[II] Institut of Chemical Forest Products, National Engineering and Technology Research Center of Forest Chemical Industry, Nanjing 210042, P. R. China
[II] Key Laboratory of Development & Application of Forest Chemicals of Guangxi, Nanning 530006, P. R. China

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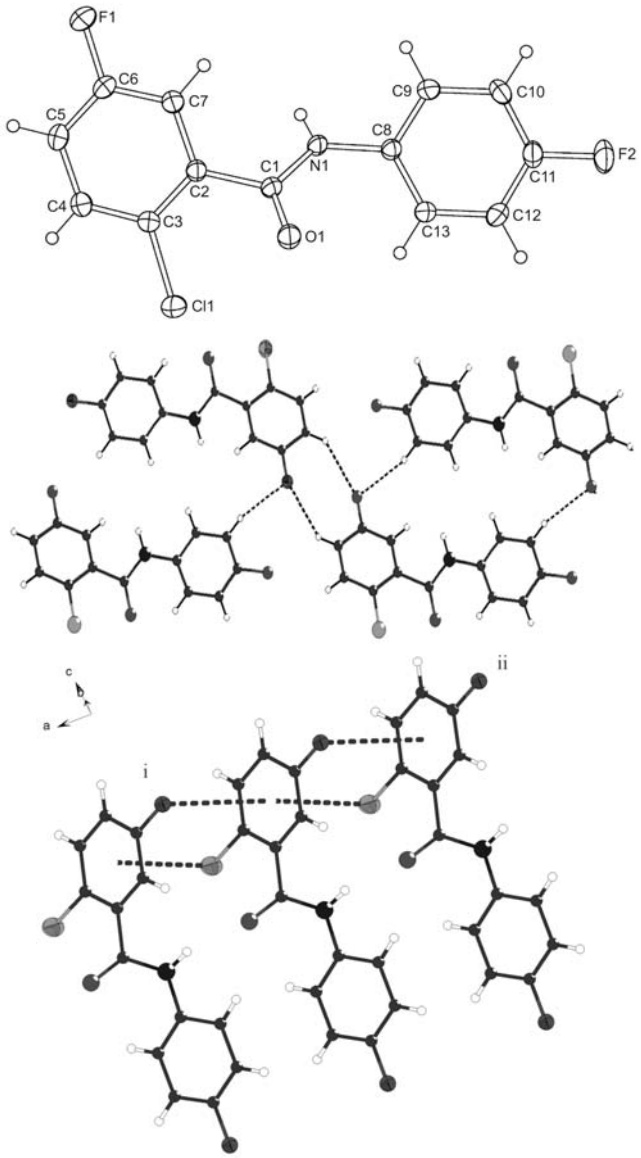

Abstract

$C_{13}H_8ClF_2NO$, triclinic, $P\bar{1}$ (no. 2), a = 5.0624(7) Å, b = 8.6997(8) Å, c = 13.534(1) Å, α = 77.524(1)°, β = 87.022(2)°, γ = 84.251(2)°, V = 578.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.162, T = 298 K.

* Correspondence author (e-mail: huangqinpeking@163.com)

Source of material

A solution of 2-chloro-5-fluoro-*N*-(4-fluorophenyl)benzamide (10 mmol) in 50 ml toluene was added to a solution of aniline (10 mmol) in 10 ml toluene. The reaction mixture was refluxed for 1 h with stirring. Then the resulting white precipitate was obtained by filtration, washed several times with ethanol and dried in vacuo (yield 90 %).

Elemental analysis — found: C, 58.37 %; H, 3.04 %; N, 5.20 %; O, 5.99 %; calculated: C, 58.34 %; H, 3.01 %; N, 5.23 %; O, 5.98 %.

Experimental details

H atoms bonded to C and N were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, d(N—H) = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(C,N).

Discussion

Amide compounds provide potential sites for hydrogen bonds, aromatic ring stacking and weak non-covalent interactions in supermolecular chemistry.

The asymmetric unit of the title compound contains one 2-chloro-5-fluoro-*N*-(4-fluorophenyl)benzamide molecule (figure, top). In the crystal structure, intermolecular N–H···O hydrogen bonds interlink the molecules into chains along [010] (figure, middle). In addition, there are weak C–H···X (X: halogen) and X (lone pair)···π (benzene ring) interactions. The relevant distances are d(C3—Cl1···Cg1^{i}) = 3.729(1) Å, d(C6—F1···Cg1^{ii}) = 3.755(7) Å (symmetry code i: 1+x,y,z; ii: –1+x,y,z); Cg1 is the centroid of the ring defined by the atoms C2-C7 (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.43 × 0.44 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.41 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2969, 1981
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1521
$N(param)_{refined}$:	164
Programs:	SHELXS-97, SHELXL-97 [1]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	1.2905	1.2591	0.6236	0.052
H(4)	2*i*	1.6623	1.1463	1.0052	0.075
H(5)	2*i*	1.3189	1.3368	1.0229	0.076
H(7)	2*i*	1.2534	1.4478	0.7226	0.061
H(9)	2*i*	1.1589	1.3814	0.4489	0.067
H(10)	2*i*	1.1970	1.3520	0.2821	0.083
H(12)	2*i*	1.8623	1.0702	0.3433	0.072
H(13)	2*i*	1.8235	1.0983	0.5103	0.070

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}
Cl(1)	2*i*	1.9256(2)	1.0422(1)	0.84543(9)	0.0707(8)	0.0755(8)	0.0590(7)	0.0269(6)	–0.0081(5)	–0.0156(5)
F(1)	2*i*	1.0541(6)	1.5171(4)	0.8823(2)	0.084(2)	0.122(2)	0.066(2)	0.050(2)	–0.001(1)	–0.039(2)
F(2)	2*i*	1.5567(6)	1.1946(4)	0.2012(2)	0.095(2)	0.130(3)	0.038(1)	–0.004(2)	–0.002(1)	–0.028(2)
N(1)	2*i*	1.4509(5)	1.2560(4)	0.5993(2)	0.028(2)	0.064(2)	0.042(2)	–0.001(1)	0.001(1)	–0.020(1)
O(1)	2*i*	1.8793(5)	1.2633(4)	0.6356(2)	0.028(1)	0.103(2)	0.049(2)	–0.005(1)	0.001(1)	–0.027(2)
C(1)	2*i*	1.6451(7)	1.2632(4)	0.6619(3)	0.033(2)	0.052(2)	0.041(2)	0.000(2)	0.000(2)	–0.015(2)
C(2)	2*i*	1.5500(7)	1.2780(4)	0.7662(3)	0.034(2)	0.052(2)	0.039(2)	–0.005(2)	–0.000(2)	–0.015(2)
C(3)	2*i*	1.6693(7)	1.1873(5)	0.8531(3)	0.042(2)	0.056(2)	0.047(2)	0.003(2)	–0.005(2)	–0.015(2)
C(4)	2*i*	1.5815(9)	1.2084(6)	0.9480(3)	0.068(3)	0.076(3)	0.039(2)	0.012(2)	–0.005(2)	–0.012(2)
C(5)	2*i*	1.3758(9)	1.3204(6)	0.9592(3)	0.069(3)	0.081(3)	0.042(2)	0.007(2)	0.003(2)	–0.024(2)
C(6)	2*i*	1.2588(8)	1.4062(5)	0.8737(3)	0.051(2)	0.076(3)	0.053(2)	0.014(2)	0.004(2)	–0.027(2)
C(7)	2*i*	1.3399(7)	1.3871(5)	0.7788(3)	0.043(2)	0.066(3)	0.044(2)	0.004(2)	–0.003(2)	–0.015(2)
C(8)	2*i*	1.4887(7)	1.2437(4)	0.4957(3)	0.035(2)	0.044(2)	0.038(2)	–0.005(2)	0.001(1)	–0.011(2)
C(9)	2*i*	1.3015(8)	1.3187(5)	0.4280(3)	0.047(2)	0.067(3)	0.046(2)	0.014(2)	0.001(2)	–0.006(2)
C(10)	2*i*	1.3239(9)	1.3014(6)	0.3284(3)	0.061(3)	0.100(4)	0.039(2)	0.012(3)	–0.011(2)	–0.002(2)
C(11)	2*i*	1.5335(9)	1.2097(5)	0.2995(3)	0.059(3)	0.069(3)	0.036(2)	–0.009(2)	0.002(2)	–0.012(2)
C(12)	2*i*	1.7208(9)	1.1331(5)	0.3648(3)	0.056(3)	0.074(3)	0.053(2)	0.012(2)	0.000(2)	–0.030(2)
C(13)	2*i*	1.6970(8)	1.1504(5)	0.4643(3)	0.051(2)	0.079(3)	0.047(2)	0.020(2)	–0.012(2)	–0.026(2)

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References

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