Crystal structure of *N′*-(3,5-dichloro-2-hydroxybenzylidene)-isonicotinohydrazide, $C_{13}H_9Cl_2N_3O_2$

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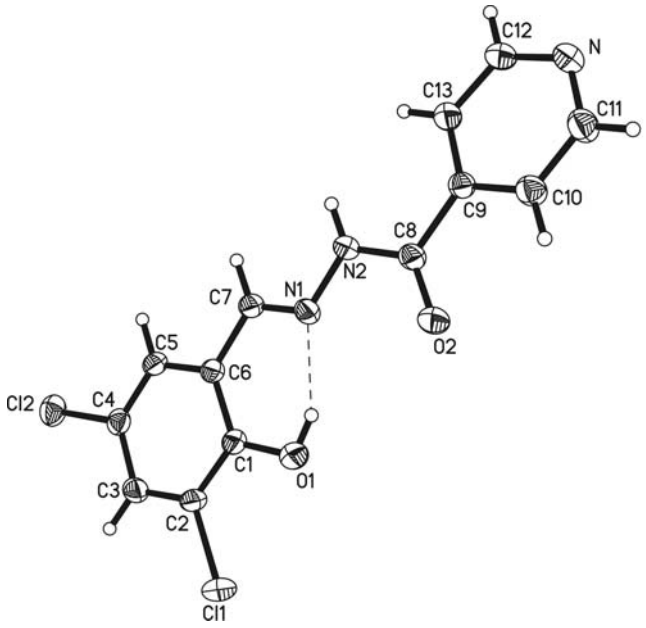

Abstract

$C_{13}H_9Cl_2N_3O_2$, monoclinic, $P12_1/c1$ (no. 14), a = 8.0607(1) Å, b = 10.7551(2) Å, c = 15.6323(2) Å, β = 90.894(1)°, V = 1355.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.094, T = 296 K.

Source of material

Pyridine-4-carboxylic acid hydrazide (1 mmol, 0.137 g) was dissolved in anhydrous ethanol (8 ml), the mixture was stirred for several minitutes at room temperature, 3,5-dichloro-2-hydroxybenzaldehyde (1 mmol, 0.191 g) in ethanol (4 ml) was added dropwise, and the mixture was stirred at refluxing temperature for 2 h. The product was separated and recrystallised in ethanol, brown single crystals of the title compound were obtained after 3 d.

Experimental details

H1 bound to O1 was located from the difference Fourier map and refined freely. The other hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.93 Å, d(N—H) = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(C,N).

Discussion

Hydrazones derived from the reactions of aldehyde and hydrazides show a great deal of interest due to their potential biological properties [1,2]. Untill now, there are only a few examples of hydrazones which have been synthesized and structurallycharacterized [3,4].

The asymmetric unit of the title crystal structure is build up of one molecule, which is not planar. The pyridine ring and the benzene ring form a dihedral angle of 10.26(7)°. The C7/N1/N2/C8 group is slightly twisted with respect to the benzene ring, making a dihedral angle of 2.43(7)°. An intramolecular O–H···N interation is observed involving the hydroxyl moiety and the nitrogen atom. In the crystal, the intermolecular N–H···N hydrogen bonds help to stablize the molecular structure.

Table 1. Data collection and handling.

Crystal:	brown block, size 0.18 × 0.21 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.83 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	55.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10988, 3131
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2580
$N(param)_{refined}$:	186
Programs:	SHELXS-97, SHELXL-97, SHELXTL [5]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	–0.3468	1.0497	–0.1359	0.050
H(7A)	4*e*	–0.1766	0.8885	–0.0935	0.049
H(5A)	4*e*	0.0021	0.7170	–0.0443	0.048
H(3A)	4*e*	0.1049	0.6613	0.2043	0.053
H(10A)	4*e*	–0.6195	1.3748	–0.0539	0.057
H(11A)	4*e*	–0.7192	1.5045	–0.1581	0.065
H(13A)	4*e*	–0.4116	1.1692	–0.2359	0.061
H(12A)	4*e*	–0.5130	1.3081	–0.3349	0.067
H(1A)	4*e*	–0.235(3)	1.017(2)	0.098(2)	0.076(7)

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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}
Cl(2)	4*e*	0.18680(5)	0.54537(4)	0.05031(3)	0.0547(2)	0.0438(2)	0.0681(3)	0.0053(2)	–0.0044(2)	–0.0044(2)
Cl(1)	4*e*	–0.05034(7)	0.86108(5)	0.28548(3)	0.1087(4)	0.0692(3)	0.0314(2)	0.0007(3)	–0.0076(2)	–0.0059(2)
O(1)	4*e*	–0.1972(2)	0.9876(1)	0.14130(8)	0.0766(8)	0.0463(7)	0.0393(6)	0.0102(6)	–0.0001(6)	–0.0046(5)
N(2)	4*e*	–0.3500(2)	1.0741(1)	–0.08360(8)	0.0548(7)	0.0379(7)	0.0321(6)	–0.0018(6)	–0.0051(5)	0.0048(5)
N(1)	4*e*	–0.2719(2)	1.0097(1)	–0.01913(8)	0.0488(7)	0.0367(7)	0.0377(6)	–0.0065(5)	–0.0051(5)	0.0071(5)
C(6)	4*e*	–0.1019(2)	0.8467(1)	0.03159(9)	0.0422(7)	0.0350(7)	0.0336(7)	–0.0086(6)	–0.0031(6)	0.0010(6)
C(8)	4*e*	–0.4330(2)	1.1787(1)	–0.06074(9)	0.0467(7)	0.0385(8)	0.0347(7)	–0.0075(6)	0.0019(6)	0.0060(6)
C(7)	4*e*	–0.1859(2)	0.9146(1)	–0.03703(9)	0.0496(8)	0.0387(8)	0.0337(7)	–0.0087(6)	–0.0041(6)	0.0012(6)
C(1)	4*e*	–0.1135(2)	0.8856(1)	0.11759(9)	0.0488(8)	0.0354(8)	0.0357(7)	–0.0075(6)	–0.0003(6)	–0.0023(6)
C(9)	4*e*	–0.5028(2)	1.2567(1)	–0.13255(9)	0.0416(7)	0.0365(8)	0.0349(7)	–0.0066(6)	–0.0006(6)	0.0033(6)
C(5)	4*e*	–0.0083(2)	0.7419(1)	0.01238(9)	0.0475(8)	0.0387(8)	0.0333(7)	–0.0077(6)	–0.0014(6)	–0.0038(6)
C(3)	4*e*	0.0553(2)	0.7087(2)	0.1612(1)	0.0489(8)	0.0421(9)	0.0419(8)	–0.0074(7)	–0.0118(6)	0.0055(7)
C(4)	4*e*	0.0687(2)	0.6750(1)	0.0762(1)	0.0406(7)	0.0349(8)	0.0463(8)	–0.0052(6)	–0.0044(6)	–0.0016(6)
O(2)	4*e*	–0.4464(2)	1.2108(1)	0.01344(7)	0.0776(8)	0.0528(7)	0.0327(6)	0.0035(6)	0.0047(5)	0.0046(5)
C(10)	4*e*	–0.5962(2)	1.3588(2)	–0.1109(1)	0.0536(9)	0.0494(9)	0.0405(8)	0.0043(7)	0.0066(7)	0.0036(7)
C(2)	4*e*	–0.0335(2)	0.8141(2)	0.18012(9)	0.0549(8)	0.0447(9)	0.0301(7)	–0.0088(7)	–0.0051(6)	–0.0021(6)
N	4*e*	–0.6243(2)	1.4209(1)	–0.25705(9)	0.0629(8)	0.0488(8)	0.0462(8)	0.0028(7)	–0.0072(6)	0.0093(6)
C(11)	4*e*	–0.6548(2)	1.4368(2)	–0.1741(1)	0.0580(9)	0.051(1)	0.053(1)	0.0114(8)	0.0024(8)	0.0055(8)
C(13)	4*e*	–0.4728(2)	1.2376(2)	–0.2182(1)	0.072(1)	0.0425(9)	0.0384(8)	0.0078(8)	–0.0010(7)	–0.0006(7)
C(12)	4*e*	–0.5350(2)	1.3217(2)	–0.2775(1)	0.079(1)	0.053(1)	0.0342(8)	0.0044(9)	–0.0033(8)	0.0032(7)

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