Crystal structure of *N′*-(5-chloro-2-hydroxybenzylidene)furan-2-carbohydrazide monohydrate, $C_{12}H_9ClN_2O_3 \cdot H_2O$

Hua-Dong Dong*

Key Laboratory of Surface and Interface Science of Henan, School of Material & Chemical Engineering, Zhengzhou University of Light Industry, Zhengzhou 450002, P. R. China

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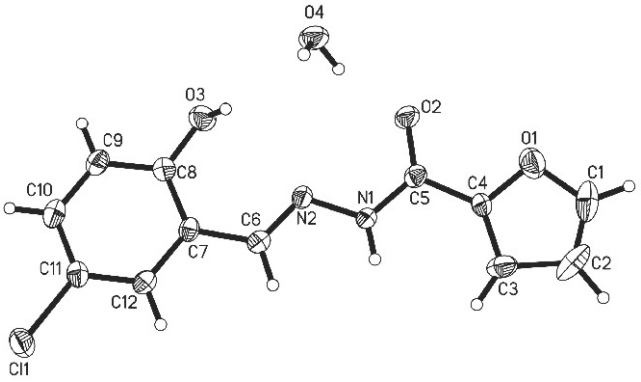

Abstract

$C_{12}H_{11}ClN_2O_4$, orthorhombic, $P2_12_12_1$ (no. 19), a = 4.8534(3) Å, b = 12.446(1) Å, c = 20.734(2) Å, V = 1252.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.055, $wR_{ref}(F^2)$ = 0.134, T = 293 K.

Source of material

5-Chloro-2-hydroxybenzaldehyde (1 mmol, 0.1565 g) and furan-2-carbohydrazide (1 mmol, 0.126 g) were dissolved in methanol (10 ml). The mixture was stirred at refluxing temperature for 2 h, the product was isolated and recrystallized from 95 % methanol. Yellow single crystals were obtained after 2 days.

Experimental details

All H atoms were positioned geometrically and refined as riding with d(C—H) = 0.93 Å (aromatic) and 0.97 Å (methylene) and d(N—H) = 0.86 Å, with U_{iso}(H) = 1.2 U_{eq}(CH_2 or NH) and U_{iso}(H) = 1.5 U_{eq}(C). H atoms of water molecule are located from a difference Fourier map and refined isotropically, with d(O—H) and H···H distances restrained to 0.85(1) Å and 1.37(2) Å. The Flack parameter is –0.11(12).

Discussion

Schiff bases bearing additional donor groups represent an important class of heteropolydentate ligands with metals in coordination chemistry [1].

The title crystal structure consists of Schiff base molecules and lattice water molecules. The Schiff base adopts an *E* geometry with respect the C=N bond. The molecule is not planar, the dihedral angle between the furan ring and benzene ring is 9.4(2)°. The water molecule is linked to the Schiff base molecule through the O–H···O hydrogen bond. In the crystal, inversion dimers are linked by intermolecular O–H···O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.18 × 0.20 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.17 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3816, 2437
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1748
$N(param)_{refined}$:	180
Program:	SHELXTL [2]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	–0.1799	0.0204	0.2489	0.043
H(10A)	4*a*	0.8853	–0.0733	–0.0099	0.057
H(9A)	4*a*	0.6346	–0.2169	0.0281	0.055
H(3A)	4*a*	0.2790	–0.2291	0.1522	0.082
H(12A)	4*a*	0.4854	0.1216	0.1111	0.046
H(6A)	4*a*	0.1332	0.0599	0.1812	0.045
H(2B)	4*a*	–0.8801	0.0875	0.3997	0.090
H(3B)	4*a*	–0.5025	0.0956	0.3130	0.066
H(1B)	4*a*	–0.9820	–0.0957	0.4186	0.089
H(4A)	4*a*	0.31(2)	–0.284(7)	0.273(4)	0.15(3)
H(4B)	4*a*	0.03(2)	–0.283(5)	0.252(3)	0.09(2)

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)	4*a*	0.8943(2)	0.1476(1)	0.01882(6)	0.0528(6)	0.0599(7)	0.0646(7)	–0.0023(6)	0.0128(6)	0.0160(6)
O(2)	4*a*	–0.3157(6)	–0.2157(2)	0.2664(2)	0.045(2)	0.034(2)	0.082(2)	–0.004(1)	0.019(2)	–0.008(2)
N(2)	4*a*	0.0164(6)	–0.0839(2)	0.1954(1)	0.032(2)	0.038(2)	0.036(2)	0.003(2)	0.004(1)	–0.001(2)
C(8)	4*a*	0.4083(7)	–0.1354(3)	0.0935(2)	0.036(2)	0.043(2)	0.036(2)	0.000(2)	–0.002(2)	0.001(2)
N(1)	4*a*	–0.1677(6)	–0.0473(2)	0.2412(2)	0.035(2)	0.030(2)	0.043(2)	0.001(1)	0.008(2)	–0.002(2)
C(10)	4*a*	0.7541(8)	–0.0631(4)	0.0223(2)	0.041(2)	0.062(3)	0.040(2)	0.012(2)	0.005(2)	0.000(2)

* e-mail: donghuadong2011@126.com

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(9)	4*a*	0.6044(8)	–0.1489(4)	0.0452(2)	0.047(2)	0.048(2)	0.043(2)	0.006(2)	0.007(2)	–0.011(2)
O(3)	4*a*	0.2668(7)	–0.2230(2)	0.1129(1)	0.068(2)	0.046(2)	0.051(2)	–0.006(2)	0.005(2)	–0.002(2)
C(5)	4*a*	–0.3270(8)	–0.1162(3)	0.2735(2)	0.029(2)	0.039(2)	0.044(2)	0.004(2)	–0.000(2)	–0.002(2)
C(7)	4*a*	0.3618(7)	–0.0329(3)	0.1191(2)	0.028(2)	0.043(2)	0.032(2)	0.004(2)	–0.001(2)	–0.001(2)
C(4)	4*a*	–0.5223(7)	–0.0672(3)	0.3188(2)	0.031(2)	0.040(2)	0.036(2)	–0.003(2)	0.004(2)	0.002(2)
O(1)	4*a*	–0.6831(7)	–0.1354(3)	0.3539(2)	0.055(2)	0.071(2)	0.067(2)	–0.002(2)	0.013(2)	0.011(2)
C(12)	4*a*	0.5150(7)	0.0530(3)	0.0948(2)	0.037(2)	0.040(2)	0.038(2)	0.007(2)	–0.002(2)	–0.003(2)
C(11)	4*a*	0.7084(8)	0.0383(3)	0.0473(2)	0.034(2)	0.050(2)	0.035(2)	0.002(2)	0.001(2)	0.007(2)
C(6)	4*a*	0.1598(8)	–0.0110(3)	0.1685(2)	0.039(2)	0.037(2)	0.038(2)	0.005(2)	–0.001(2)	–0.002(2)
C(2)	4*a*	–0.796(1)	0.0293(5)	0.3797(3)	0.053(3)	0.086(4)	0.086(4)	0.023(3)	–0.012(3)	–0.052(4)
C(3)	4*a*	–0.5810(9)	0.0346(3)	0.3312(3)	0.047(2)	0.035(2)	0.083(3)	–0.001(2)	0.001(3)	–0.008(2)
C(1)	4*a*	–0.848(1)	–0.0709(6)	0.3901(2)	0.048(3)	0.124(5)	0.049(3)	0.014(4)	0.013(2)	–0.001(3)
O(4)	4*a*	0.1767(8)	–0.3244(2)	0.2535(2)	0.051(2)	0.033(2)	0.099(3)	–0.003(2)	0.015(2)	–0.002(2)

References

1. Borisova, N. E.; Reshetova, M. D.; Ustynyuk, Y. A.: Metal-Free Methods in the Synthesis of Macrocyclic Schiff Bases. Chem. Rev. **107** (2007) 46-79.
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.