

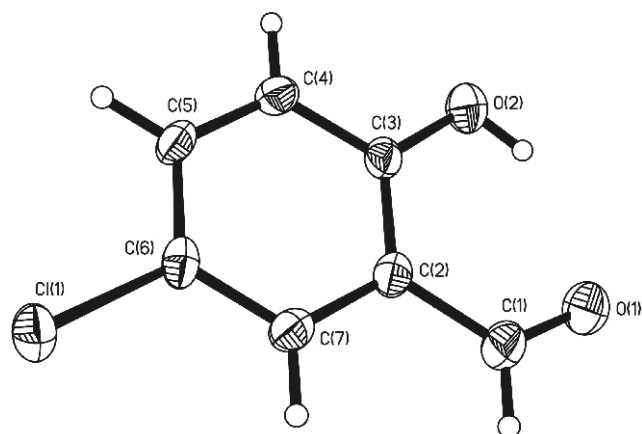
Crystal structure of 5-chloro-2-hydroxybenzaldehyde, $C_7H_5ClO_2$

Shouwen Jin^{*I}, Feng Lin^{II}, Yuanqi Yu^{II} and Haidong He^{II}

^I Tianmu College, ZheJiang A & F University, Lin'An 311300, P. R. China

^{II} Faculty of Science, ZheJiang A & F University, Lin'An 311300, P. R. China

Received April 29, 2011, accepted and available on-line November 1, 2011; CCDC no. 1267/3553



Abstract

$C_7H_5ClO_2$, monoclinic, $P2_1$ (no. 4), $a = 3.8818(5)$ Å, $b = 5.6515(7)$ Å, $c = 15.204(2)$ Å, $\beta = 93.176(1)^\circ$, $V = 333.0$ Å³, $Z = 2$, $R_{gt}(F) = 0.050$, $wR_{ref}(F^2) = 0.131$, $T = 298$ K.

Source of material

Crystals of 5-chloro-2-hydroxybenzaldehyde were formed during an experiment to synthesize an organic acid-base adduct. A solution of 3,5-dimethylpyrazole (19.2 mg, 0.2 mmol) in 5 mL of MeOH was added to a MeOH solution (3 mL) containing 5-chloro-2-hydroxybenzaldehyde (31.3 mg, 0.2 mmol) under continuous stirring. The solution was stirred for about 1 h at room temperature, then filtered into a test tube. The filtrate was left at room temperature for several days. Light yellow block-shaped crystals were isolated after slow evaporation of the solution in air at ambient temperature. The crystals were collected and dried in air. X-ray diffraction analysis indicated that no adducts composed of the two components were formed, but crystals of the title compound.

Experimental details

All H atoms were placed in calculated positions with $d(C-H) = 0.93$ Å, and $d(O-H) = 0.82$ Å and were included in the refinement with $U_{iso}(H) = 1.2 U(C,O)$. The Flack parameter was found to be $-0.15(16)$, confirming the absolute structure [1].

Discussion

It is well known that 2-hydroxybenzaldehyde derivatives are important material in synthesizing Schiff bases. Schiff base derivatives involving 5-chloro-2-hydroxybenzaldehyde have also been reported [2,3]. 5-chloro-2-hydroxybenzaldehyde bears the donor group OH, and may form hydrogen bonds with some compounds

bearing the acceptor groups. We have studied hydrogen bonded assembly of organic acid and organic base [4].

The distance $d(O1-C1)$ is 1.217(6) Å, which is typical of C=O bond length, and $d(O2-C3) = 1.346(6)$ Å is similar to the published $d(C-O) = 1.347$ Å reported by Yang [2], but it is longer than the documented data of 1.328(4) Å, which may be contributed to the fact that in our compound there were stronger hydrogen bonding interactions than in the reported compound [3]. The usual intramolecular hydrogen bond is found between the phenol OH group and the carbonyl group to exhibit a $S_1^1(6)$ graph. There were two arrays of discrete 5-chloro-2-hydroxybenzaldehyde molecules arranged along the [010] direction. The 5-chloro-2-hydroxybenzaldehyde molecules in the same array are parallel. Two 5-chloro-2-hydroxybenzaldehyde molecules at the neighbouring arrays make a dihedral angle of ca. 60° with each other. The molecules are further inter-connected by the intermolecular O-H...O hydrogen bonds to form a zigzag chain running along the [010] direction.

Table 1. Data collection and handling.

Crystal:	colorless block, size $0.07 \times 0.45 \times 0.48$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	4.96 cm^{-1}
Diffractionmeter, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	1653, 112
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 884
$N(\text{param})_{\text{refined}}$:	91
Programs:	SHELXS-97, SHELXL-97, SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(2)	2i	−0.0270	−0.0793	0.9003	0.086
H(1)	2i	0.3707	0.4540	0.9185	0.066
H(4)	2i	−0.0950	−0.1809	0.6867	0.050
H(5)	2i	0.0993	0.0638	0.5799	0.048
H(7)	2i	0.4370	0.5283	0.7638	0.048

* Correspondence author (e-mail: jinsw@zafu.edu.cn)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cl(1)	2 <i>i</i>	0.4230(3)	0.5038(2)	0.57876(7)	0.0607(7)	0.0577(8)	0.0380(6)	−0.0009(8)	0.0079(4)	0.0081(7)
O(1)	2 <i>i</i>	0.162(1)	0.1983(7)	0.9688(2)	0.120(4)	0.065(3)	0.034(2)	0.001(3)	0.013(2)	−0.001(2)
O(2)	2 <i>i</i>	−0.0657(9)	−0.1318(7)	0.8504(2)	0.077(2)	0.056(2)	0.040(2)	−0.005(2)	0.009(2)	0.009(2)
C(1)	2 <i>i</i>	0.260(2)	0.311(1)	0.9066(3)	0.077(4)	0.051(4)	0.038(3)	0.009(3)	0.000(2)	−0.005(3)
C(2)	2 <i>i</i>	0.215(1)	0.237(1)	0.8143(3)	0.040(3)	0.041(3)	0.031(2)	0.006(2)	0.000(2)	−0.002(2)
C(3)	2 <i>i</i>	0.055(1)	0.020(1)	0.7914(2)	0.045(2)	0.037(3)	0.032(2)	0.010(3)	0.003(2)	0.001(3)
C(4)	2 <i>i</i>	0.013(1)	−0.0393(9)	0.7029(3)	0.046(2)	0.036(3)	0.042(2)	−0.003(2)	−0.004(2)	−0.005(2)
C(5)	2 <i>i</i>	0.127(1)	0.1070(9)	0.6389(3)	0.046(3)	0.046(3)	0.028(2)	0.003(2)	−0.001(2)	−0.008(2)
C(6)	2 <i>i</i>	0.285(1)	0.3203(8)	0.6622(3)	0.037(2)	0.049(3)	0.028(2)	0.008(2)	0.004(2)	0.003(2)
C(7)	2 <i>i</i>	0.330(1)	0.386(1)	0.7487(3)	0.041(3)	0.039(2)	0.039(2)	0.000(2)	−0.003(2)	−0.005(2)

References

1. Flack H. D.: On enantiomorph-polarity estimation. *Acta Crystallogr.* **A39** (1983) 876-881.
2. Yang, D. S.: 5-Chlorosalicylaldehyde salicylhydrazone. *Acta Cryst.* **E62** (2006) o1591-o1592.
3. Chohan, Z. H.; Tahir, M. N.; Shada, H. A.; Khan, I. U.: 4-(5-Chloro-2-hydroxybenzylideneamino)-*N*-(4,6-dimethylpyrimidin-2-yl)benzene-sulfonamide. *Acta Cryst.* **E64** (2008) o648.
4. Jin, S. W.; Zhang, W. B.; Liu, L.; Gao, H. F.; Wang, D. Q.; Chen, R. P.; Xu, X. L.: Hydrogen bonded supramolecular architectures of organic salts based on 5,7-dimethyl-1,8-naphthyridine-2-amine and acidic compounds. *J. Mol. Struct.* **975** (2010) 128-136.
5. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.