Crystal structure of 2,6-dichlorobenzaldehyde, $C_7H_4Cl_2O$

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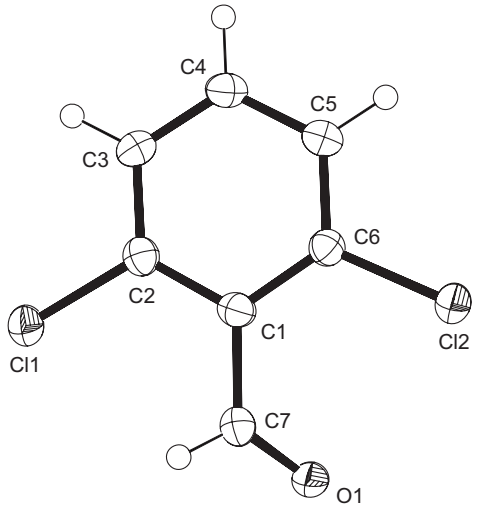

Abstract

$C_7H_4Cl_2O$, monoclinic, $P12_1/c1$ (no. 14), a = 3.837(1) Å, b = 13.633(4) Å, c = 13.117(1) Å, β = 91.230(7)°, V = 686.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.159, T = 103 K.

Source of material

The compound was isolated as a side product of a reaction involving secondary propargylic alcohols [1]. It is also available commercially from Sigma Inc.

Experimental details

All hydrogen atoms were located from the Fourier difference map, but in order to have a favorable N_{gt}/N_{param} ratio, the hydrogen atoms of the benzene ring were placed in calculated positions and refined using a riding-model approximation with C—H bond lengths of 0.95 Å. Positional and isotropic displacement parameters were only refined for the H atom of the aldehyde group.

Discussion

In the crystal structure of 2,6-dichlorobenzaldehyde the benzene ring and chlorine atoms form a plane with an r.m.s. deviation from planarity of 0.0252 Å. The aldehyde group is twisted from planarity with values of the torsion angles ∠C6–C1–C7–O1 being –27.3(4)° and ∠C2–C1–C7–O1 being 152.6 (3)°. The corresponding values for crystal structures containing unsubstituted benzaldehyde molecules and deposited in the Cambridge Structural Database [2] range from 9.5° to 0.08° and 171.3° to 180.0°, respectively. There are only nine structures of benzaldehyde halogen derivatives in which two halogen atoms are both in *ortho* position relative to the —CHO group. The arrangement of the benzene ring and the aldehyde group varies from parallel to perpendicular. The most similar conformation to the one described in this paper is reported for the structure of 2,4,6-tribromo-5-hydroxybenzaldehyde [3], where the torsion angles for one from two molecules in the asymmetric unit are –27.7° and 152.2°. In contrast, the structure of a compound with a close chemical similarity to the reported aldehyde, 2,6-dichlorobenzoic acid [4], has its carboxyl group in a nearly perpendicular position to the plane of benzene ring with corresponding torsion angles of –88.1(3)° and 91.8(3)°. The most important features for the packing of 2,6-dichlorobenzaldehyde are stacking and short-contact interactions. Stacking is observed along [001] and the distance between planes of neighboring rings is 3.426 Å. The closest stacking distances exist between the following atoms: C6···C1 at x–1,y,z, C4···C5 at x–1,y,z and C6···Cl2 at x–1,y,z. They are equal 3.434(4) Å, 3.454(4) Å and 3.527(3) Å, respectively. There are three types of weak interactions in which distances between atoms are at least 0.13 Å shorter than the van der Waals distances. One of them is O1···C7(x+1,y,z), and the respective distance is 3.050(4) Å. The hydrogen atom H3(1+x,½–y,½+z) participates in other two weak interactions with the O1 and Cl2 atoms. The distances O1···H3(1+x,½–y,½+z) and Cl2···H3(1+x,½–y,½+z) are 2.516 Å and 2.814 Å, respectively, while the angles O1···H3–C3(1+x,½–y,½+z) and Cl2···H3–C3(1+x,½–y,½+z) have values of 133.8° and 156.9°, respectively. The weak interaction O1···H3–C3(1+x,½–y,½+z) is similar to this observed in a crystal structure of 2-nitrobenzaldehyde [5,6].

Table 1. Data collection and handling.

Crystal:	light yellow prism, size 0.05 × 0.10 × 0.20 mm
Wavelength:	Cu K_α radiation (1.54179 Å)
μ:	78.23 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω with χ offset
$2\theta_{max}$:	136.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1244, 1181
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > -3\ \sigma(I_{obs})$, 1088
$N(param)_{refined}$:	95
Programs:	SHELXS-97 [7], HKL-3000SM [8], SHELXL-97 [9], ORTEP-III [10], ORTEP-3 [11]

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

Atom	Site	x	y	z	U_{iso}
H(4)	4*e*	–0.1286	0.4203	0.2673	0.032
H(5)	4*e*	0.1507	0.4626	0.4211	0.029
H(3)	4*e*	–0.2911	0.2580	0.2353	0.031
H(1)	4*e*	0.004(8)	0.097(3)	0.534(2)	0.025(9)

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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*	0.3486(2)	0.36301(6)	0.59483(5)	0.0301(5)	0.0253(7)	0.0242(5)	–0.0005(2)	–0.0045(3)	–0.0028(3)
Cl(1)	4*e*	–0.2381(2)	0.08431(6)	0.34449(5)	0.0309(5)	0.0242(7)	0.0291(5)	–0.0016(3)	–0.0047(3)	–0.0032(3)
C(1)	4*e*	0.0685(6)	0.2258(2)	0.4640(2)	0.023(1)	0.021(2)	0.026(2)	0.000(1)	0.001(1)	0.003(1)
O(1)	4*e*	0.3833(5)	0.1456(2)	0.6002(2)	0.038(1)	0.020(2)	0.026(1)	0.0015(8)	–0.0077(9)	0.0008(9)
C(4)	4*e*	–0.0802(7)	0.3712(3)	0.3170(2)	0.030(1)	0.023(2)	0.026(2)	0.004(1)	0.001(1)	0.004(1)
C(6)	4*e*	0.1596(7)	0.3238(2)	0.4801(2)	0.022(1)	0.022(2)	0.022(1)	0.001(1)	0.0005(9)	–0.001(1)
C(2)	4*e*	–0.1054(7)	0.2037(2)	0.3709(2)	0.022(1)	0.024(2)	0.026(1)	–0.001(1)	0.001(1)	–0.002(1)
C(5)	4*e*	0.0866(7)	0.3964(3)	0.4080(2)	0.026(1)	0.022(2)	0.026(2)	0.001(1)	0.003(1)	0.003(1)
C(7)	4*e*	0.1447(8)	0.1464(2)	0.5382(2)	0.028(1)	0.022(2)	0.025(1)	–0.002(1)	–0.001(1)	–0.002(1)
C(3)	4*e*	–0.1770(7)	0.2750(3)	0.2978(2)	0.027(1)	0.028(2)	0.021(1)	0.002(1)	–0.002(1)	0.002(1)

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