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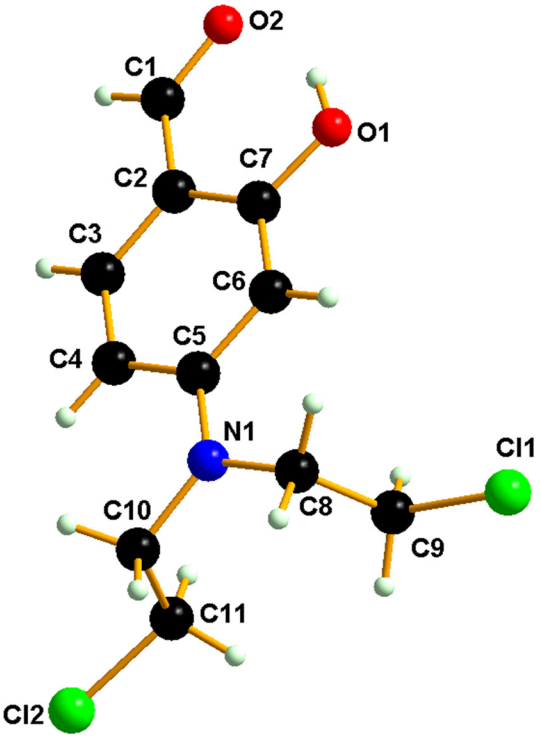
The crystal structure of 4-(bis(2-chloroethyl) amino)-2-hydroxybenzaldehyde, $C_{11}H_{13}Cl_2NO_2$



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Abstract

$C_{11}H_{13}Cl_2NO_2$, triclinic, space group $P\bar{1}$, a = 7.2061(4) Å, b = 9.5043(5) Å, c = 9.9747(6) Å, α = 70.249(2)°, β = 70.258(2)°, γ = 80.755(2)°, V = 604.34(6) Å^3, Z = 2, R_{gt}(F) = 0.0401, wR_{ref}(F2) = 0.1125, T = 293 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.18 × 0.17 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.52 mm^{-1}
Diffractometer, scan mode:	Rigaku, φ and ω scans
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13906, 2108, 0.068
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1748
$N(param)_{refined}$:	145
Programs:	Rigaku,[1] SHELX[2,3]

Table 1 contains details on crystal structure and measurement conditions.

1 Source of material

Under the protection of N_2, 1,2-dichloroethane(3.96 g, 0.04 mol) and trimethylamine (6 ml) were added to the solution of 4-amino-2-hydroxybenzaldehyde (1.371 g, 0.01 mol) in tetrahydrofuran (30 ml). Then, the above mixed solution was stirred at room temperature for 24 h. The mixture was filtered and the filtrate was evaporated to get a yellow residue, which was purified by chromatography on silica gel to get 4-(bis(2-chloroethyl)amino)-2-hydroxybenzaldehyde (1.40 g, yield 54 %) as a white solid. The single crystal suitable for X-ray diffraction was obtained by recrystallization in diethyl ether.

2 Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2.[2,3] The hydrogen atoms were placed at calculated positions as riding atoms with U_{iso}(H) set to 1.2 U_{eq}(C).

3 Discussion

As an important intermediate of organic compounds, salicylaldehyde derivatives can be used in many fields of medical to chemical applications.[9] So, the design and synthesis of novel salicylaldehyde derivatives obviously became an attractive field of research in organic chemistry. The 4-(bis(2-chloroethyl)

amino)-2-hydroxybenzaldehyde was synthesized using 4-amino-2-hydroxybenzaldehyde as the starting material. Its structure was characterized by X-ray diffraction. There is one cystallographically independent molecule (cf. the figure) in the asymmetric unit, in which all bond lengths are in normal ranges.[10–13] The bond length of C1–O2 is 1.226 (3) Å, which is slightly shorter than that of C7–O1 1.347 (3) Å. Nevertheless, the bond length of C5–N1 1.377 (3) Å is apparently shorter than that of C9–Cl1 1.781 (3) Å. The angles of O2–C1–C2, C2–C7–C6 and C5–N1–C8 are 125.1(2)°, 121.20(19)° and 120.61(18)° respectively, which demonstrated that C1, O2, O1, N1, C8, C10 and the benzene ring were nearly on the same plane. Hydrogen bonds were observed as following: O1–H1A···O2 hydrogen bond (*d* (H1A···O2) = 2.616 Å, and weak non-classical ones.

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