Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Chlorosalicylaldehyde benzoylhydrazone

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.085
Data-to-parameter ratio = 15.9

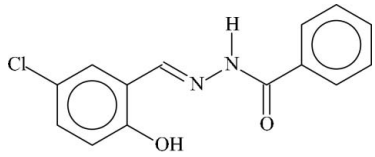
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Molecules of 5-chlorosalicylaldehyde benzoylhydrazone, $C_{14}H_{11}ClN_2O_2$, are linked by an intermolecular amino–carbonyl hydrogen bond into a linear chain that runs along the *a* axis of the orthorhombic unit cell.

Received 28 June 2005
Accepted 30 June 2005
Online 6 July 2005

Comment

The title compound, (I), which crystallizes in the non-centrosymmetric space group $Pna2_1$, has a chlorine substituent at the 5-position of the salicylaldehyde benzene ring of a salicylaldehyde benzoylhydrazone (Fig. 1). In the parent compound, *viz*. salicylaldehyde benzoylhydrazone, originally described in the non-standard setting $Pc2_1n$ (Lyubchova *et al.*, 1995), molecules are linked by a hydrogen bond from the amino group of one molecule to the hydroxy group of another; the chain motif is better regarded as a helix arising from propagation by a twofold screw axis. The introduction of a chlorine substituent to a position *para* to the hydroxy group lowers the electron-donating ability of the hydroxy O atom in (I); the hydroxy group engages in intramolecular hydrogen bonding only. In the crystal structure, a linear chain motif results from an intermolecular hydrogen-bonding interaction between the amine and carbonyl groups (Fig. 2 and Table 1).



(I)

Experimental

5-Chlorosalicylaldehyde (0.20 g, 1.3 mmol) and benzoylhydrazine (0.17 g, 1.3 mmol) were refluxed in a small volume of ethanol for an

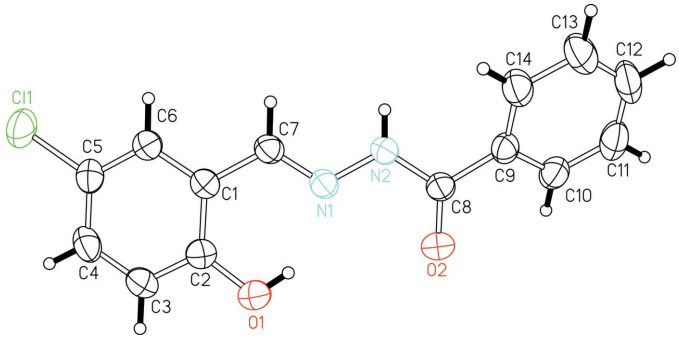


Figure 1
ORTEPII plot (Johnson, 1976) of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

hour. The solvent was removed and the solid product recrystallized from ethyl acetate to give crystals of the title Schiff base, (I).

Crystal data

$C_{14}H_{11}ClN_2O_2$
M_r = 274.70
Orthorhombic, $Pna2_1$
a = 9.496 (3) Å
b = 9.895 (3) Å
c = 13.619 (5) Å
V = 1279.6 (7) Å^3
Z = 4
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11373 reflections
θ = 3.0–27.5°
μ = 0.30 mm^{-1}
T = 295 (2) K
Block, yellow
0.39 × 0.28 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.747, T_{max} = 0.940
11373 measured reflections
2874 independent reflections
2744 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°
$h = -10 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.085
S = 1.07
2874 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.0336P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.045 (4)
Absolute structure: Flack (1983), 1370 Friedel pairs
Flack parameter: 0.01 (5)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···N1	0.83 (2)	1.87 (2)	2.588 (2)	144 (2)
N2—H2n···O2^{i}	0.84 (2)	2.09 (2)	2.911 (2)	167 (2)

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z$.

The carbon-bound H atoms were positioned geometrically (C—H = 0.93 Å) and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(parent atom). The hydroxy and amino H atoms were located in a difference Fourier map and were refined with a distance restraint of O—H = N—H = 0.85 (1) Å.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

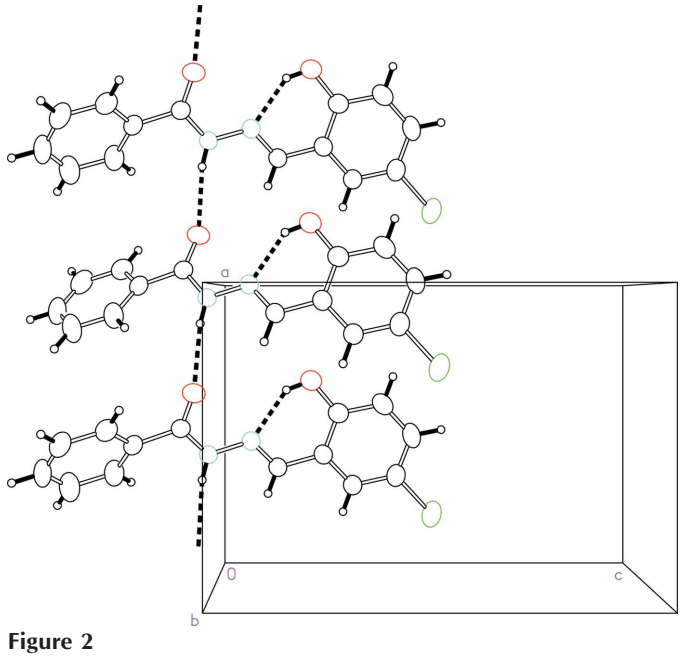


Figure 2
ORTEPII plot (Johnson, 1976) of the hydrogen-bonded chain structure in (I). Hydrogen bonds are drawn as dashed lines.

The authors thank the Ministry of Science, Technology and the Environment for supporting this study (grant No. IPRA 33–02-03–3055), and Professor Gao Shan of Heilongjiang University for carrying out the diffraction measurements.

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