Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Nitrosalicylaldehyde benzoylhydrazone

Hapipah M. Ali, Subramaniam Puvaneswary and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.113
Data-to-parameter ratio = 7.5

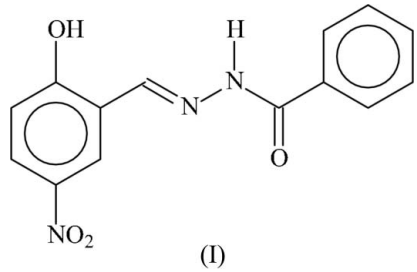
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Nearly planar molecules of the title compound, $C_{11}H_{11}N_3O_4$, are linked by O—H···O hydrogen bonds to form zigzag chains along the *c* axis; adjacent chains are, in turn, linked by weaker N—H···O hydrogen bonds to form layers perpendicular to the *a* axis of the orthorhombic unit cell.

Received 16 September 2005
Accepted 23 September 2005
Online 28 September 2005

Comment

The crystal structure of salicylaldehyde benzoylhydrazone features an intramolecular hydrogen bond between the donor OH group of the salicylaldehyde moiety and the acceptor =N—NH— group of the benzhydrazidyl moiety (Lyubchova *et al.*, 1995); the short hydrogen bond permits the amino nitrogen of the =N—NH— unit to interact with the –C=O– unit of an adjacent molecule. Such a hydrogen-bonding scheme is also found in 5-chlorosalicylaldehyde benzoylhydrazone (Ali *et al.*, 2005).



(I)

In the title compound, (I), the much stronger electron-withdrawing nitro group reduces the donor ability of the OH group, and in 5-nitrosalicylaldehyde benzoylhydrazone

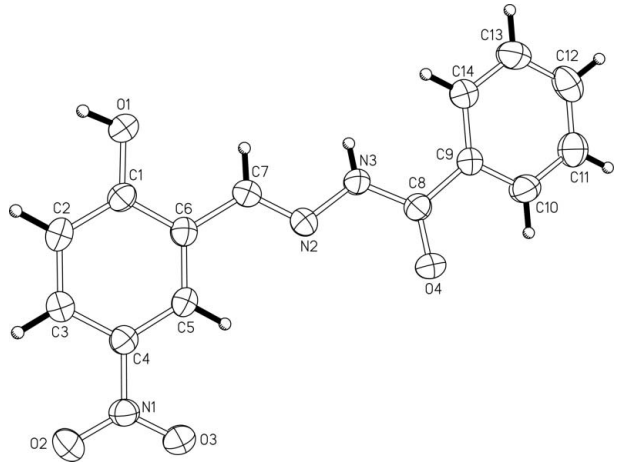


Figure 1
ORTEPII (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

(Fig. 1), the salicylaldehyde portion is rotated so that its OH group forms an intermolecular hydrogen bond with the amide O atom of an adjacent molecule, the hydrogen bond giving rise to a chain motif (Fig. 2). In contrast, the hydrogen bonds involving the amine N atom are weaker (Table 1); these N—H···O bonds lead to the formation of layers.

Experimental

Benzhydrazide (0.16 g, 1.2 mmol) and 5-nitrosalicylaldehyde (0.20 g, 1.2 mol) were refluxed in ethanol (20 ml) for 2 h. The solvent was removed and the product recrystallized from pyridine.

Crystal data

$C_{14}H_{11}N_3O_4$
$M_r = 285.26$
Orthorhombic, $Pca2_1$
$a = 30.224$ (2) Å
$b = 4.8017$ (3) Å
$c = 8.7257$ (6) Å
$V = 1266.3$ (2) Å^3
$Z = 4$
$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2819 reflections
$\theta = 2.3$–$26.7°$
$\mu = 0.11$ mm^{-1}
$T = 295$ (2) K
Plate, yellow
0.39 × 0.30 × 0.09 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
7031 measured reflections
1478 independent reflections
1226 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.0°$
$h = -38 \rightarrow 36$
$k = -5 \rightarrow 6$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.113$
$S = 1.17$
1478 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O4^{i}	0.85 (1)	1.86 (1)	2.700 (3)	170 (3)
N3—H3n···O3^{i}	0.85 (1)	2.48 (2)	3.178 (3)	140 (3)
N3—H3n···O4^{ii}	0.85 (1)	2.48 (3)	3.106 (4)	131 (3)

Symmetry codes: (i) $-x+\frac{1}{2}, y+1, z-\frac{1}{2}$; (ii) $x, y+1, z$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. C-bound H atoms were placed at calculated positions (C—H = 0.93 Å) and included in the refinement in the riding-model approximation with U_{iso}(H) values set at $1.2U_{eq}$(C). N- and O-bound H atoms were located in a difference Fourier map and refined with a distance restraint of N—H = O—H = 0.85 (1) Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*.

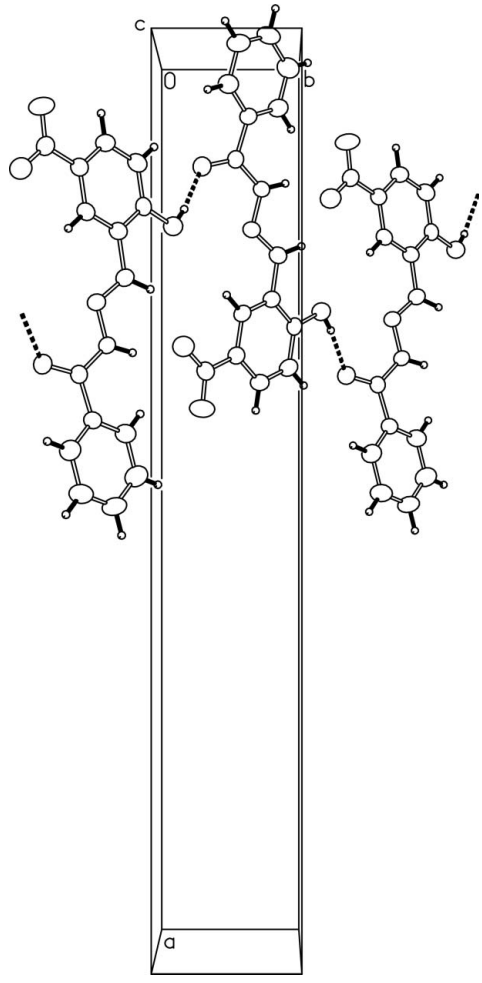


Figure 2
ORTEPII (Johnson, 1976) of the hydrogen-bonded (dashed lines) chain structure in (I). The weaker N—H···O interactions that link adjacent chains into layers are not shown.

The authors thank the Scientific Advancement Grant Allocation (No. 66–02-03–0046/Oracle 815–0046) and the University of Malaya for supporting this study. We also thank Mr Xiao-Long Feng of Sun Yat-Sen University for the diffraction measurements.

References

Ali, M. H., Puvaneswary, S. & Ng, S. W. (2005). *Acta Cryst.* E**61**, o2415–o2416.
Bruker (2000). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lyubchova, A., Cossé-Barbi, A., Doucet, J. P., Robert, F., Souron, J.-P. & Quarton, M. (1995). *Acta Cryst.* C**51**, 1893–1895.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.