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5-Nitrosalicylaldehyde (2-hydroxybenzoyl)-hydrazone

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Key indicators

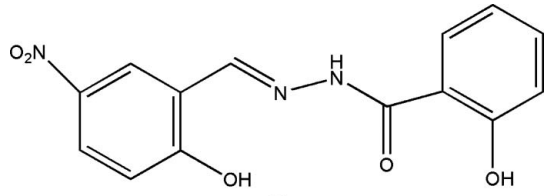
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.131
Data-to-parameter ratio = 14.1

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

The molecule of the title compound, $C_{14}H_{11}N_3O_5$, is approximately planar, the dihedral angles between the two aromatic rings being 4.63 (7)°. O—H···N, N—H···O and O—H···O hydrogen bonds and π–π stacking interactions help to consolidate the crystal packing.

Comment

Considerable attention has been paid to the chemistry of aroylhydrazones and their complexes (Bu *et al.*, 2001; Liao *et al.*, 2000; Tai *et al.*, 2003; Xue *et al.*, 2006). These compounds may serve as potential chelating agents (Fun *et al.*, 1996; Lu *et al.*, 1996) and possess biological activity (Liao *et al.*, 2000, Yang & Pan, 2004). Here we report the synthesis and crystal structure of the title compound, (I) (Fig. 1), obtained by the condensation of 5-nitrosalicylaldehyde and salicylhydrazide.



(I)

The molecule of (I) is approximately planar, the dihedral angles between the aromatic ring *A* (atoms C1–C6) and aromatic ring *B* (C9–C14) and between ring *A* and the nitro group being 4.63 (7) and 7.6 (2)°, respectively. The torsion angle C1—C2—C7—N1 is −3.2 (2)°.

As illustrated in Figs. 2 and 3, there are weak π–π interactions between neighboring hydrazone molecules along the *a* axis. The distance between the aromatic ring *A* of one molecule and the neighboring aromatic ring *B* of a molecule related by the symmetry operation $(1 - x, -y, -z)$ is 3.97 Å, and the dihedral angle between the two rings is 4.63 (7)°. A similar stacking phenomenon was observed in 3-hydroxysalicylaldehyde 2-furoylhydrazone (Ali *et al.*, 2005).

Various hydrogen bonds occur in (I) (Table 1), including two intramolecular interactions. These H atoms are also involved in intermolecular hydrogen bonds to O-atom acceptors. The amino (N2) H atom makes an intermolecular hydrogen bond to the O4 atom of the nitro group of an adjacent molecule.

Experimental

An equimolar mixture of salicylhydrazide (15 mmol) and 5-nitrosalicylaldehyde (15 mmol) in ethanol (30 ml) was refluxed in a round-

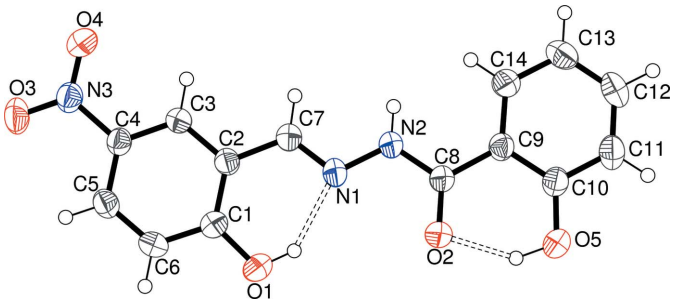


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids (arbitrary spheres for the H atoms). Hydrogen bonds are indicated by dashed lines.

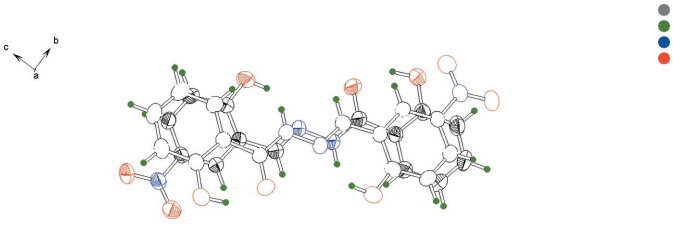


Figure 2
A plot illustrating the π–π stacking of molecules of (I). The molecule containing the unshaded atoms is generated by the symmetry operation $(1 - x, -y, -z)$.

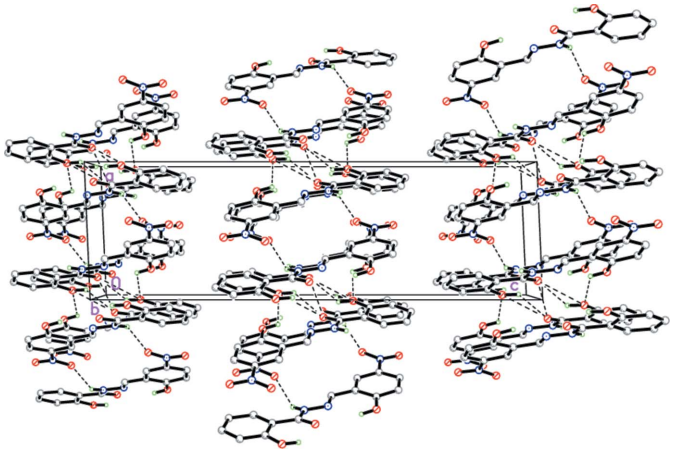


Figure 3
Packing of (I), showing the intermolecular hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

bottomed flask for 3 h. The solution was then filtered to remove some undissolved solids. Colourless crystals of (I) were obtained in the filtrate after 6 days of evaporation at room temperature. Analysis calculated for $C_{14}H_{11}N_3O_5$: C 55.82, H 3.68, N 13.95, O 26.55%; found: C 55.66, H 3.72, N 14.26, O 25.98%.

Crystal data

$C_{14}H_{11}N_3O_5$
M_r = 301.26
Monoclinic, $P2_1/c$
a = 7.645 (3) Å
b = 6.751 (3) Å
c = 24.994 (11) Å
β = 92.473 (9)°
V = 1288.6 (9) Å^3
Z = 4
D_x = 1.553 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Chip, colorless
0.40 × 0.15 × 0.10 mm

Data collection

Rigaku Weissenberg IP diffractometer
ω scans
Absorption correction: ψ scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.978, T_{max} = 0.988
11290 measured reflections
2939 independent reflections
2101 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.131
S = 1.04
2939 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 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
N2—H2B···O4^{i}	0.86	2.34	3.070 (2)	143
O1—H1B···N1	0.89	1.94	2.686 (2)	140
O1—H1B···O5^{ii}	0.89	2.55	2.9822 (18)	111
O5—H5B···O2	0.95	1.79	2.5960 (17)	141
O5—H5B···O2^{ii}	0.95	2.21	2.873 (2)	126

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

The N– and O-bound H atoms were located in a difference map and then refined as riding in their as-found relative positions. The other H atoms were placed in idealized positions (C—H = 0.93 Å) and refined as riding. A U_{iso} value was freely refined for all H atoms.

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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