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#### Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

$R$  factor = 0.032

w $R$  factor = 0.091

Data-to-parameter ratio = 6.9

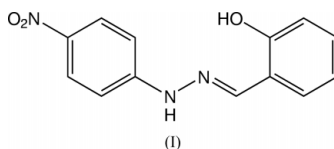
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Salicylaldehyde 4-nitrophenylhydrazone

Crystals of the title compound,  $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_3$ , were obtained from a condensation reaction of salicylaldehyde and 4-nitrophenylhydrazine. The molecule exhibits a planar structure, with the 4-nitro group slightly inclined to the plane. The molecules link to each other *via* intermolecular hydrogen bonding between the imino and nitro groups.

### Comment

Some phenylhydrazone derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*, 1993). In order to investigate the relationship of the biological activity and the molecular structure, a series of new phenylhydrazone derivatives has been synthesized and structures determined in our laboratory (Shan *et al.*, 2002).



The molecular structure of (I) is shown in Fig. 1. The molecule has an approximately planar structure, except for the N1-nitro group which is slightly tilted due to intermolecular hydrogen bonding, the dihedral angle between the nitro group and the attached benzene ring being  $10.6(2)^\circ$ .

The compound crystallizes in the *E* configuration, with the phenol group and the nitrophenyl group on opposite sides of the  $\text{C}7=\text{N}3$  double bond. This agrees with the configuration found in phenylhydrazone derivatives reported previously (Shan *et al.*, 2002; Drumright *et al.*, 1990).

An intramolecular hydrogen bond is formed between the hydroxy group and atom N3. An intermolecular hydrogen bond is observed between the imino and nitro groups, as shown in Fig. 1.

### Experimental

4-Nitrophenylhydrazine (0.31 g, 0.2 mmol) was dissolved in ethanol (10 ml), then  $\text{H}_2\text{SO}_4$  solution (98%, 0.5 ml) was slowly added to the ethanol solution with stirring. The solution was heated at about 333 K for several minutes until the solution cleared. Salicylaldehyde (0.24 g, 0.2 mmol) was added dropwise to the solution with continuous stirring, and the mixture was kept at about 333 K for half an hour. When the solution had cooled to room temperature an orange microcrystalline appeared. The powder was separated from the solution and washed with water three times. Recrystallization was performed twice, with chloroform and acetone, respectively, to obtain well-shaped single crystals.

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## Crystal data

$C_{13}H_{11}N_3O_3$   
 $M_r = 257.25$   
 Monoclinic,  $Pc$   
 $a = 5.0052$  (11) Å  
 $b = 9.9004$  (16) Å  
 $c = 12.1749$  (12) Å  
 $\beta = 92.199$  (15)°  
 $V = 602.86$  (17) Å<sup>3</sup>  
 $Z = 2$

$D_x = 1.417$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 24 reflections  
 $\theta = 8.6$ – $12.2$ °  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Plate, orange  
 $0.58 \times 0.38 \times 0.30$  mm

## Data collection

Rigaku AFC-7S diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1328 measured reflections  
 1190 independent reflections  
 774 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.016$

$\theta_{max} = 26.0$ °  
 $h = 0 \rightarrow 6$   
 $k = 0 \rightarrow 12$   
 $l = -15 \rightarrow 15$   
 3 standard reflections every 150 reflections  
 intensity decay: 1.9%

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.091$   
 $S = 1.05$   
 1190 reflections  
 172 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.0138P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.009$   
 $\Delta\rho_{max} = 0.13$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.15$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

O1–N1	1.229 (4)	N2–C1	1.364 (4)
O2–N1	1.224 (4)	N2–N3	1.364 (3)
O3–C9	1.359 (4)	N3–C7	1.274 (4)
N1–C4	1.436 (4)	C7–C8	1.468 (4)
C1–N2–N3	119.9 (3)	C7–N3–N2	117.7 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O3–H9 <sup>ii</sup> ···N3	0.96	1.82	2.664 (3)	145
N2–H1 <sup>i</sup> ···O2 <sup>i</sup>	0.86	2.36	3.215 (4)	173

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

In the absence of significant anomalous scattering and of any Friedel pairs, the Flack (1983) parameter was indeterminate. All H atoms on C and N atoms were placed in calculated positions, with C–H = 0.93 Å and N–H = 0.86 Å, and were included in the final cycles

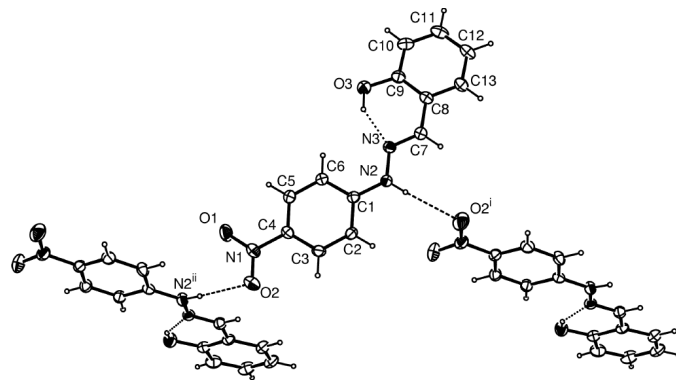


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonding. [Symmetry codes: (i)  $x - 1, -y, z - \frac{1}{2}$ ; (ii)  $x + 1, -y, z + \frac{1}{2}$ ]

of refinement in the riding mode, with  $U_{iso}(H) = 1.2U_{eq}$  of the carrier atoms. The hydroxy H atom was located in a difference Fourier map and was included in the final cycles of refinement with fixed positional parameters and  $U_{iso}(H) = 0.08$  Å<sup>2</sup>.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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