

## 2'-Acetyl-2-hydroxynaphthohydrazide

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## Key indicators

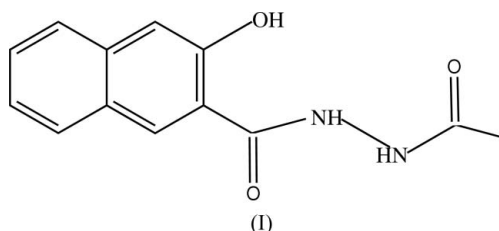
Single-crystal X-ray study  
 $T = 298$  K  
Mean  $\sigma(C-C) = 0.004$  Å  
 $R$  factor = 0.054  
 $wR$  factor = 0.173  
Data-to-parameter ratio = 16.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title compound,  $C_{13}H_{12}N_2O_3$ , has been synthesized and characterized by X-ray diffraction and FT-IR spectroscopic analysis. The approximately planar molecule is in an *E* configuration with respect to the N–N bond [torsion angle  $-176.6$  ( $2^\circ$ )]. In the crystal structure, each hydrazide adopts the ketoamine form to link adjacent molecules by the maximum possible number of O–H $\cdots$ O and N–H $\cdots$ O intermolecular hydrogen bonds. A one-dimensional chain framework is formed by this hydrogen bonding.

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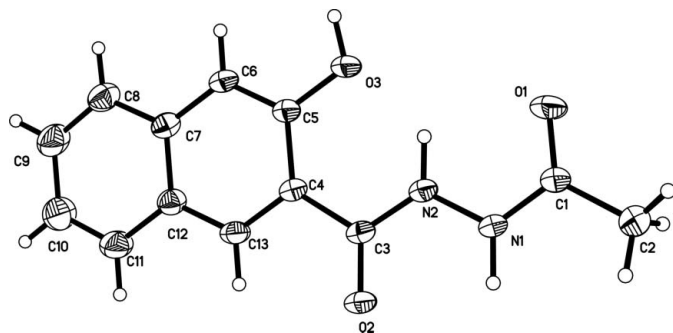
## Comment

Although hydrazide complexes have been extensively studied (Lin *et al.*, 2002; Kwak *et al.*, 1998), there are few examples utilizing the related 3-hydroxy-2-naphthoylhydrazide (Dogan *et al.*, 1998).



The crystal structure of the title compound, (I), is shown in Fig. 1. An *E* configuration with respect to the N–N bond is observed, with a C–N–N–C torsion angle of  $-176.6$  ( $2^\circ$ ). The C1–O1 and C3–O2 distances are 1.233 (3) and 1.226 (2) Å, respectively, typical of double bonds (Sakarnoto *et al.*, 1989; Yan *et al.*, 1993). The C3–N2, C1–N1 and N1–N2 bond distances are typical for a single bond, at 1.331 (3), 1.328 (3) and 1.384 (3) Å, respectively, and these are in agreement with the values found in the analogous compound, *N'*-(2-hydroxy-3-methoxyphenylmethylidene)-3-pyridinecarbohydrazide (Galić *et al.*, 2001). These values suggest that this aroylhydrazide exists in the ketoamine form.

The molecule of the title compound is approximately planar. Two dihedral angles indicating a slight deviation from planarity are  $2.67$  ( $16^\circ$ ) for the naphthalene group and O3/C5/C4/C3/O2, and  $4.74$  ( $15^\circ$ ) for the naphthalene group and C3/O3/N2/N1/C1/O1. The intramolecular hydrogen bond involving the hydroxy group and the N atom on the hydrazine chain stabilizes such a conformation (Table 1). The maximum possible number of intermolecular hydrogen bonds are formed between neighbouring molecules of (I), as shown in Fig. 2. Each molecule provides one O(acyl) and one N(hydrazine) as acceptor and donor to link to one adjacent molecule *via* two N–H $\cdots$ O hydrogen bonds. The remaining two O atoms, O(acyl) and O(naphthol), as acceptor and



**Figure 1**  
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

donor, respectively, connect to another molecule *via* two O—H···O hydrogen bonds (Table 1). Thus, the molecules are assembled into one-dimensional chains.

## Experimental

Acetic anhydride (3.41 g, 33.4 mmol) and 3-hydroxyl-2-naphthoylhydrazide (6.75 g, 33.4 mmol) were added to chloroform (60 ml) at 273 K. The reaction mixture was slowly warmed to 298 K and stirred for 4 h. After standing overnight in the refrigerator, the resulting white precipitate was filtered off and rinsed with chloroform and diethyl ether (m. p. 541–543 K). The product (0.25 mmol) was dissolved in a mixture of dimethylformamide and methanol (5 ml; 1:1 *v/v*). A sample of white crystals of (I) was isolated after evaporating the resulting solution for about one week at room temperature. FT-IR spectra ( $\nu$ ,  $\text{cm}^{-1}$ ): 3428 (*bs*), 3198 (*bs*), 3057 (*w*), 1633 (*s*), 1602 (*vs*), 1491 (*s*), 1359 (*m*), 1273 (*m*), 1252 (*m*), 1229 (*m*), 1210 (*s*), 1085 (*m*), 1004 (*m*), 910 (*w*), 876 (*m*), 757 (*m*), 592 (*m*).

### Crystal data

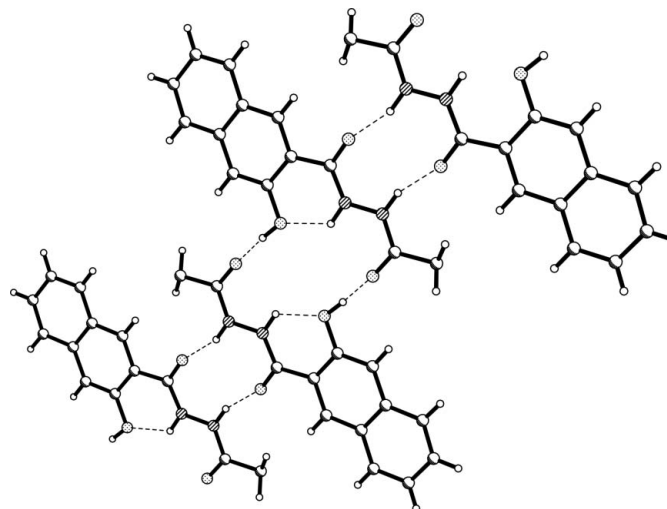
$\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$	$D_x = 1.351 \text{ Mg m}^{-3}$
$M_r = 244.25$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1313 reflections
$a = 6.266 (2) \text{ \AA}$	$\theta = 2.6\text{--}24.1^\circ$
$b = 22.987 (8) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 8.503 (3) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\beta = 101.329 (5)^\circ$	Block, white
$V = 1200.9 (7) \text{ \AA}^3$	$0.32 \times 0.18 \times 0.12 \text{ mm}$
$Z = 4$	

### Data collection

Siemens SMART CCD area-detector diffractometer	2934 independent reflections
$\varphi$ and $\omega$ scans	1283 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.043$
$T_{\text{min}} = 0.969$ , $T_{\text{max}} = 0.988$	$\theta_{\text{max}} = 28.4^\circ$
7738 measured reflections	$h = -8 \rightarrow 7$
	$k = -30 \rightarrow 16$
	$l = -10 \rightarrow 11$

### Refinement

Refinement on $F^2$	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.079P)^2]$
$wR(F^2) = 0.173$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{\text{max}} = 0.047$
2934 reflections	$\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
176 parameters	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$



**Figure 2**  
The crystal packing of the title compound, viewed along the *c* axis.

**Table 1**

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$\text{N2--H16}\cdots\text{O3}$	0.94 (2)	1.91 (2)	2.615 (3)	130 (2)
$\text{O3--H14}\cdots\text{O1}^{\text{i}}$	0.94 (3)	1.73 (3)	2.658 (3)	172 (2)
$\text{N1--H15}\cdots\text{O2}^{\text{ii}}$	0.97 (3)	1.90 (3)	2.829 (3)	161 (2)

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

Atoms H14, H15 and H16 were located in a difference Fourier map and thereafter refined isotropically. All other H atoms were positioned geometrically and allowed to ride on their parent atoms, with  $\text{C}(sp^2\text{ hybrid})\text{--}H = 0.93 \text{ \AA}$  and  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ , and  $\text{C}(\text{methyl})\text{--}H = 0.96 \text{ \AA}$  and  $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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