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2-Hydroxyacetophenone 3-hydroxy-2-naphthoylhydrazone

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.137
Data-to-parameter ratio = 13.8

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

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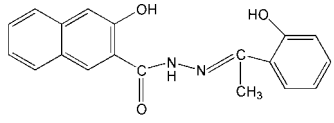
The molecule of the title compound, $C_{19}H_{15}N_2O_3$, an aroylhydrazone, is almost planar. The C═O bond length of 1.219 (2) Å suggests that the title compound is in the keto form. The C═N double bond has a length of 1.280 (2) Å. The crystal structure is stabilized by N—H···O, O—H···O and O—H···N hydrogen bonds.

Comment

During the past two decades, considerable attention has been paid to the chemistry of aroylhydrazones and their complexes with metal ions (Bu *et al.*, 2001; Liao *et al.*, 2000; Tai *et al.*, 2003); these compounds may serve as potential chelating agents (Fun *et al.*, 1996; Lu *et al.*, 1996) and possess biological activity (Liao *et al.*, 2000). As part of our studies on the synthesis and characterization of aroylhydrazone derivatives, we report the crystal structure of 2-hydroxyacetophenone 3-hydroxy-2-naphthoylhydrazone, (I).



(I)

In the title compound, (I), the C═O bond length is 1.219 (2) Å, indicating that the molecule is in the keto form. The configuration of the C12—N2 bond is *E* (Fig. 1). The bond distances and angles in (I) are normal and the molecule is almost planar. As the distances for C12═N2 and C11═O2 are 1.280 (2) and 1.219 (2) Å, respectively, typical for double bonds, this is a novel kind of aroylhydrazone. In the crystal structure, the molecules are stabilized by N—H···O, O—H···O and O—H···N hydrogen bonds (Table 1 and Fig. 2).

Experimental

All commercially available reagents were used as supplied. The title compound was prepared as follows: a solution of 2-hydroxyacetophenone (10 mmol) in ethanol (5 ml) was added to a solution of

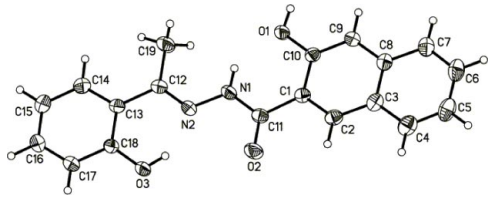


Figure 1
View of (I), with the atom-labelling scheme and 30% probability displacement ellipsoids.

2-hydroxy-3-naphthoylhydrazine (10 mmol) in ethanol (30 ml). The reaction mixture was refluxed for 3 h with stirring, then the resulting pale-yellow precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 82%). Analysis calculated for $C_{19}H_{16}N_2O_3$: C 71.17, H 4.99, N 8.74%; found: C 71.20, H 4.80, N 8.66%; IR (KBr, cm^{-1}): 1661 (C═O), 1642 (C═N). A methanol solution of the title compound was slowly evaporated and pale-yellow crystals were obtained after three weeks.

Crystal data

$C_{19}H_{16}N_2O_3$
M_r = 320.34
Monoclinic, $P2_1/n$
a = 8.6329 (12) Å
b = 8.0641 (11) Å
c = 22.621 (3) Å
β = 99.920 (3)°
V = 1551.2 (4) Å^3
Z = 4
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1742 reflections
θ = 2.4–25.5°
μ = 0.09 mm^{-1}
T = 273 (2) K
Block, light yellow
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8027 measured reflections
3039 independent reflections
2226 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°
$h = -10 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.137
S = 1.02
3039 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.3335P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.038
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.86	1.90	2.6026 (18)	137
O1—H1···O3^{i}	0.82	1.89	2.6931 (17)	166
O3—H3···N2	0.82	1.77	2.4936 (18)	146

Symmetry code: (i) $1 + x, y, z$.

H atoms were positioned geometrically and treated as riding, with C—H = 0.96 (methyl atoms) and 0.93 Å (other atoms), O—H = 0.82 Å, and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{iso}(C_{methyl},O).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

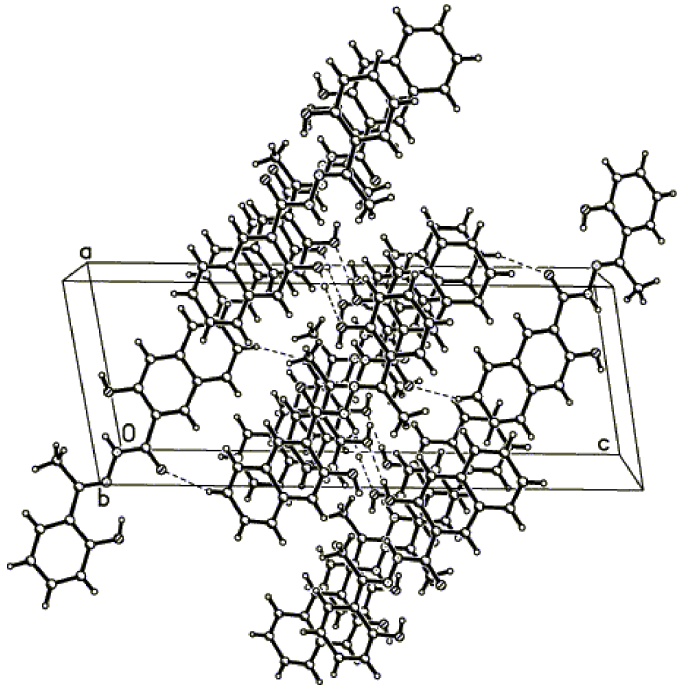


Figure 2
A view of the crystal packing. Hydrogen bonds are indicated by dashed lines.

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References

Bruker (2000). *SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bu, X. H., Gao, Y. X., Chen, W., Liu, H. & Zhang, R. H. (2001). *J. Rare Earths*, **19**, 70–73.
Fun, H.-K., Sivakumar, K., Lu, Z.-L., Duan, C.-Y., Tian, Y.-P. & You, X.-Z. (1996). *Acta Cryst.* C**52**, 1505–1507.
Liao, Z.-X., Ma, X.-Y., Shi, Z.-X. & Chen, Y.-Z. (2000). *Pol. J. Chem.* **8**, 1191–1194.
Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Fun, H.-K. & Sivakumar, K. (1996). *Acta Cryst.* C**52**, 1507–1509.
Tai, X.-S., Yin, X.-H., Tan, M.-Y. & Li, Y.-Z. (2003). *Acta Cryst.* E**59**, o681–o682.