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3-(Hydroxyimino)butan-2-one 4-methoxy-benzoylhydrazone

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Key indicators

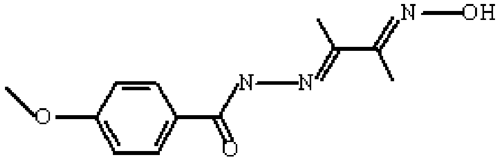
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.133
Data-to-parameter ratio = 16.8

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

Excluding H atoms, the title molecule, $C_{12}H_{15}O_3N_3$, is approximately planar. Glide-related molecules are linked by intermolecular O—H···O hydrogen bonds into a chain structure running along [101]. The crystal packing is further stabilized by π–π interactions.

Comment

Hydrazone complexes, in which the hydrazone ligands are formed by condensing hydrazine with β-diketones, salicylaldehydes and their derivatives, have been extensively studied over the past few decades (Aruffo *et al.*, 1982; Gao *et al.*, 1998; Liu & Gao, 1998; Huo, Gao, Liu, Zhao & Ng, 2004). However, there is little information about the structures of complexes based on the hydrazone ligand formed by diacetyl monoxime. Recently, we have reported some mononuclear Zn^{II} and Ni^{II} and dinuclear Cu^{II} complexes including the diacetyl monoxime benzoylhydrazone ligand (Gao, Huo, Liu *et al.*, 2004; Gao, Huo, Zhao & Ng, 2004; Huo, Gao, Liu, Wang & Zhao, 2004; Huo, Gao, Zhao *et al.*, 2004; Huo, Lu, Gao & Zhao, 2004; Huo, Lu, Gao, Zhao & Ng, 2004). In order to gain more insight into this kind of hydrazone ligand, we synthesized the title compound, (I), by the condensation reaction of diacetyl monoxime and (4-methoxybenzoyl)hydrazine in ethanol solution.



(I)

Excluding H atoms, the molecule of (I) (Fig. 1) is nearly planar [r.m.s. deviation 0.07 (3) Å], with O2 deviating by a maximum of 0.177 (1) Å. The observed planarity can be

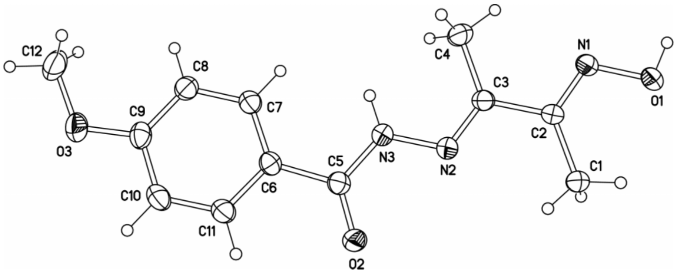


Figure 1
ORTEPII (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

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attributed to the highly conjugated π system and is also evident from the variations in C—N, C═N and C═O bond lengths (Table 1). The hydroxy H atom of diacetyl monoxime forms an intermolecular hydrogen bond with acyloxy atom O2, giving rise to a hydrogen-bonded chain structure along [101] (Table 2 and Fig. 2). In the crystal packing, the benzene rings of adjacent chains are stacked 3.459 (1) Å apart, an optimum arrangement for π–π stacking interactions.

Experimental

An ethanol solution (20 ml) of diacetyl monoxime (5.06 g, 0.05 mol) was added dropwise to an ethanol solution (100 ml) of 4-methoxybenzoylhydrazine (8.30 g, 0.05 mol); glacial acetic acid (1 ml) was then added. The mixture was refluxed for 2.5 h. Yellow crystals were isolated from the filtered solution after several days. Analysis calculated for $C_{12}H_{15}N_3O_3$: C 57.82, H 6.07, N 16.86%; found: C 57.78, H 6.01, N 16.82%.

Crystal data

$C_{12}H_{15}N_3O_3$
$M_r = 249.27$
Monoclinic, $P2_1/n$
$a = 6.8829$ (14) Å
$b = 23.589$ (5) Å
$c = 7.7372$ (15) Å
$\beta = 95.66$ (3)°
$V = 1250.1$ (4) Å^3
$Z = 4$
$D_x = 1.324$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10 756 reflections
$\theta = 3.1$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 296$ (2) K
Prism, yellow
0.39 × 0.26 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.969$, $T_{\max} = 0.978$
12 054 measured reflections
2847 independent reflections
2092 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 8$
$k = -30 \rightarrow 30$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 1.07$
2847 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.0771P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.2810 (16)	O1—N1	1.3978 (15)
N2—C3	1.2852 (16)	O2—C5	1.2280 (15)
N2—N3	1.3767 (15)	O3—C9	1.3605 (17)
N3—C5	1.3537 (17)	O3—C12	1.422 (2)
N1—C2—C1	125.22 (12)	O3—C9—C8	124.68 (13)
N1—C2—C3	113.87 (11)	O3—C9—C10	115.27 (13)
N2—C3—C2	115.77 (11)	C2—N1—O1	113.16 (10)
N2—C3—C4	125.07 (12)	C3—N2—N3	115.85 (10)
N3—C5—C6	116.05 (11)	C5—N3—N2	120.43 (11)
O2—C5—N3	122.42 (12)	C9—O3—C12	118.29 (13)
O2—C5—C6	121.49 (12)		

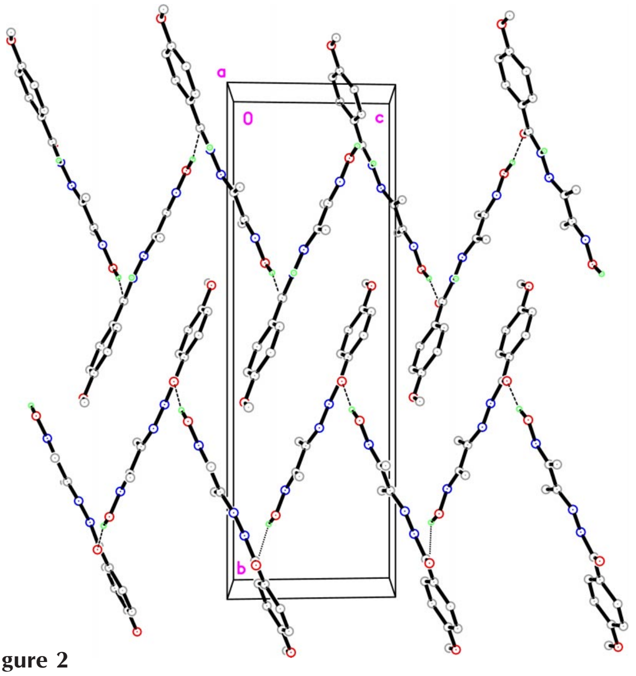

Figure 2
View, along the *a* axis, of the hydrogen-bonded chains along [101]. Hydrogen bonds are shown as dashed lines. For clarity, H atoms attached to C atoms have been omitted.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H14···O2$^{\rm i}$	0.85 (1)	1.84 (1)	2.683 (1)	168 (2)

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$.

H atoms bound to C and N atoms were placed in calculated positions [C—H = 0.93 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N) for aromatic and amide H atoms; C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms] and were included in the refinement in the riding-model approximation. The H atom of the oxime O atom was located in a difference Fourier map and refined with the O—H distance restrained to 0.85 (1) Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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*.

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