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(Acetato-κO)(diacetyl monoxime benzoylhydrazonato-$\kappa^3 O,O',N$)(quinoline-κN)zinc(II) quinoline hemisolvate

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In the title complex, $[Zn(C_{11}H_{12}N_3O_2)(C_2H_3O_2)(C_9H_7N)]\cdot0.5C_9H_7N$, the Zn^{II} atom is five-coordinate, bonded to one acetate O atom, one O atom and two N atoms from the tridentate hydrazone HL^- ligand (H_2L is diacetyl monoxime benzoylhydrazone), and one N atom from the quinoline molecule. The uncoordinated quinoline molecule is disordered and lies on a center of inversion. The presence of π–π stacking interactions between the coordinated quinoline ligands results in a chain structure along the *a* axis.

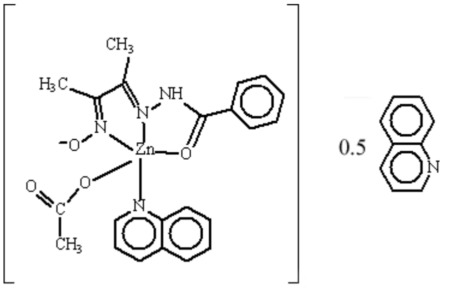
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.051
wR factor = 0.135
Data-to-parameter ratio = 18.0

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

Comment

Transition metal–hydrazone complexes, in which the hydrazone ligands are formed by condensing hydrazine with β-diketones, salicylaldehydes and their derivatives, have been extensively studied in the past few decades (Aruffo *et al.*, 1982; Gao *et al.*, 1998; Liu & Gao, 1998). However, only one structure of the *N*-(diacetyl monoxime)-*N*′-(benzoyl)hydrazone ligand with copper (Chumakov *et al.*, 1979) has been reported to date. Recently, we obtained the title complex, $[HL(C_9H_7N)(OOCCH_3)Zn]\cdot0.5C_9H_7N$ (H_2L is diacetyl monoxime benzoylhydrazone), (I) (Fig. 1), by the reaction of zinc(II) acetate dihydrate, quinoline and *N*-(diacetyl monoxime)-*N*′-(benzoyl)hydrazone in methanol solution.



(I)

The asymmetric unit of (I) consists of a Zn^{II} complex, $[HL(C_9H_7N)(OOCCH_3)Zn]$, and half of a free quinoline molecule. The free quinoline molecule, which is located on a center of inversion, is disordered (see *Experimental*). The uncoordinated hydrazide atom, N1, remains protonated, so that the hydrazone ligand bears a formal charge of −1. The Zn^{II} ion is five-coordinate, the coordination involving atoms O1, N2 and N3 from the tridentate hydrazone HL^- ligand, atom O3 from the acetate group and atom N4 from the quinoline molecule. The bond angles around the central Zn^{II} ion range from 72.1 (1) to 147.34 (9)°, which suggests that the coordination polyhedron of the central Zn atom is intermediate between trigonal bipyramidal and square pyramidal.

The C10—N3, C8—N2 and C1—O1 bond lengths are 1.278 (4), 1.272 (4) and 1.267 (3) Å, respectively. There exist π–π stacking interactions between the coordinated quinoline ligands [3.541 (4) Å], thus forming a chain structure along the *a* axis (Fig. 2).

Experimental

The ligand *N*-(diacetyl monoxime)-*N*′-(benzoyl)hydrazone was synthesized by condensing benzoylhydrazine with equimolar diacetyl monoxime in ethanol. A methanol solution containing zinc(II) acetate dihydrate (1 mmol) and quinoline (1 ml) was added slowly, dropwise, to a methanol solution (15 ml) of diacetyl monoxime benzoylhydrazone (1 mmol). The resulting mixture was refluxed with stirring for 0.5 h, then cooled to room temperature and filtered. Yellow crystals were isolated from the solution over 2 d. Analysis calculated for $C_{53}H_{51}N_9O_8Zn_2$: C 59.34, H 4.79, N 11.75%; found: C 59.52, H 4.57, N 11.83%.

Crystal data

$[Zn(C_{11}H_{12}N_3O_2)(C_2H_3O_2)-(C_9H_7N)]_2\cdot C_9H_7N$
M_r = 536.39
Triclinic, $P\overline{1}$
a = 7.383 (2) Å
b = 12.286 (3) Å
c = 13.910 (3) Å
α = 85.97 (3)°
β = 83.51 (3)°
γ = 84.07 (3)°
V = 1244.8 (5) Å^3
Z = 2
D_x = 1.431 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4813 reflections
θ = 3.3–27.4°
μ = 1.03 mm^{-1}
T = 293 (2) K
Prism, yellow
0.35 × 0.27 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.715, T_{max} = 0.837
12 128 measured reflections
5674 independent reflections
4301 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°
$h = -9 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.135
S = 1.04
5674 reflections
315 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.4688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—N2	2.047 (2)	N1—N2	1.373 (4)
Zn1—N3	2.220 (3)	N2—C8	1.278 (4)
Zn1—N4	2.052 (2)	N3—C10	1.272 (4)
Zn1—O1	2.074 (2)	O1—C1	1.267 (3)
Zn1—O3	1.950 (2)	O2—N3	1.386 (3)
N2—Zn1—N3	72.1 (1)	O1—Zn1—N3	147.34 (9)
N2—Zn1—N4	125.3 (1)	O3—Zn1—N2	127.9 (1)
N2—Zn1—O1	75.73 (9)	O3—Zn1—N3	97.5 (1)
N4—Zn1—O1	102.17 (9)	O3—Zn1—N4	106.7 (1)
N4—Zn1—N3	100.87 (9)	O3—Zn1—O1	97.68 (9)

The uncoordinated quinoline molecule is disordered over a center of inversion, and it was refined as a rigid naphthalene molecule. As the displacement parameters of the atoms were large, the quinoline molecule was then refined as two molecules, each of 0.25 occupancy. The N atoms in the quinoline rings (N6/C23–C31 and N7/C32–C40) were arbitrarily assigned. The displacement parameters were not refined. H atoms on C and N atoms were placed in calculated positions, with C—H = 0.93 (aromatic) and 0.96 Å (methyl), and N—H = 0.86 Å (imino group), and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $U_{iso}(H) = 1.5U_{eq}(C)$ (methyl atoms). The torsion angles of the methyl groups were refined.

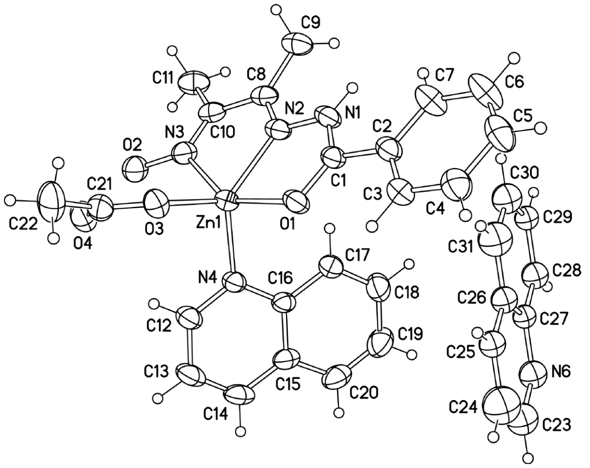


Figure 1
View of (I), with 30% probability displacement ellipsoids. H atoms are represented by spheres of arbitrary radii, and one of the disordered uncoordinated quinoline orientations has been omitted for clarity.

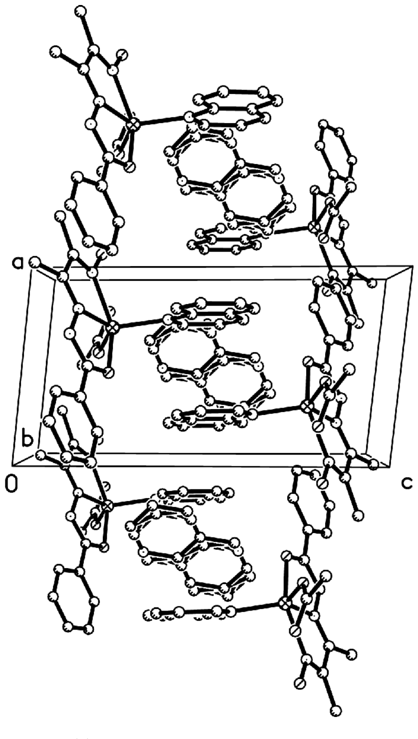


Figure 2
Packing diagram of (I). H atoms have been omitted.

Data collection: *RAID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku Corporation, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics:

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*ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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