Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Acetato-κO)[*N*-(diacetyl monooxime)-*N*′-benzoylhydrazonato-$\kappa^3 O,N,N'$]-(1*H*-imidazole-κN^3)zinc(II)

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Received 22 April 2004
Accepted 23 April 2004
Online 30 April 2004

Key indicators

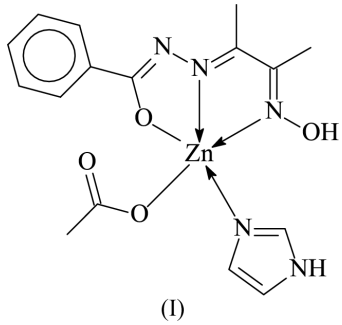
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.089
Data-to-parameter ratio = 16.9

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

In the title compound, $[Zn(C_2H_3O_2)(C_{11}H_{12}N_3O_2)(C_3H_4N_2)]$, the Zn atom is five-coordinate in a ZnN_3O_2 trigonal-bipyramidal enviroment. Adjacent molecules are linked by an intermolecular hydrogen bond into a chain running along the *a* axis of the monoclinic cell.

Comment

A previous study has documented the zinc complex of the hydrazone that was synthesized by condensing benzoylacetone with 4-methoxybenzoylhydrazine; the five-coordinate metal atom shows *trans*-trigonal geometry (Gao *et al.*, 2004). The Zn complex of *N*-(diacetylmonooxime)-*N*′-(benzoyl)-hydrazone crystallizes with imidazole, but the complex, (I), has the metal atom in a geometry that is intermediate between a square pyramid and a trigonal bipyramid (Fig. 1). The bond angles at the Zn atom of (I) range from 72.65 (7) to 148.61 (6)°.



(I)

The ligand has been characterized previously in a Cu complex (Chumakov *et al.*, 1979). The bond lengths of the

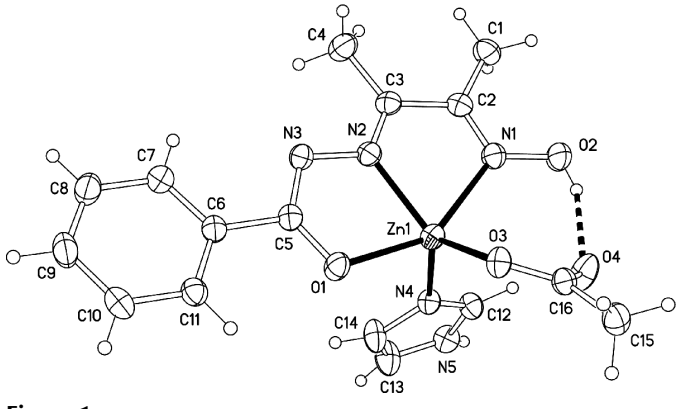


Figure 1
A view of (I), showing the atom-numbering scheme and with 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii. The dashed line indicates the intramolecular hydrogen bond.

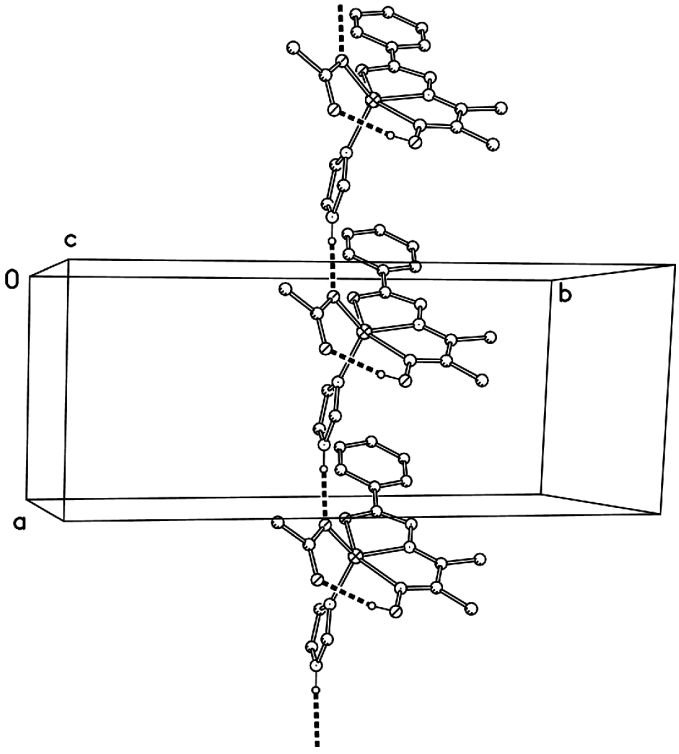


Figure 2
A view of the hydrogen-bonded chain structure of (I).

hydrazone moiety in (I) sugggest delocalization of electrons throughout. The oxime atom O2 of the hydrazone ligand forms an intramolecular O—H···O hydrogen bond with the uncoordinated acetate O atom. Another hydrogen bond gives rise to the formation of a linear chain along the *a* direction. (Table 2, Fig. 2).

Experimental

The hydrazone ligand was synthesized by condensing benzoylhydrazine with an equimolar quantity of diacetyl monooxime in ethanol. A methanol solution containing zinc(II) acetate dihydrate (1 mmol) and imidazole (1 mmol) was added to a methanol solution (25 ml) of diacetylmonooxime benzoylhydrazone (1 mmol). The resulting mixture was refluxed with stirring for 30 min, cooled slowly to room temperature and then filtered. Yellow crystals of (I) were isolated from the solution after several days. Analysis calculated for $C_{16}H_{19}N_5O_4Zn$: C 46.79, H 4.66, N 17.05%; found: C 47.52, H 4.83, N 16.92%.

Crystal data

$[Zn(C_2H_3O_2)(C_{11}H_{12}N_3O_2)(C_3H_4N_2)]$
M_r = 410.75
Monoclinic, $P2_1/c$
a = 7.731 (2) Å
b = 18.334 (4) Å
c = 12.931 (3) Å
β = 100.07 (3)°
V = 1804.6 (8) Å^3
Z = 4
D_x = 1.512 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 353 reflections
θ = 3.4–27.4°
μ = 1.39 mm^{-1}
T = 293 (2) K
Prism, yellow
0.37 × 0.25 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.626, T_{max} = 0.788
16 947 measured reflections
4122 independent reflections
3431 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -23 \rightarrow 23$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.089
S = 1.04
4122 reflections
244 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.4702P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.272 (2)	N2—N3	1.374 (2)
Zn1—N2	2.037 (2)	N2—C3	1.290 (3)
Zn1—N4	2.000 (2)	N3—C5	1.329 (3)
Zn1—O1	2.100 (2)	O1—C5	1.274 (2)
Zn1—O3	1.972 (1)	O2—N1	1.382 (2)
N1—C2	1.279 (3)	C2—C3	1.485 (3)
N2—Zn1—N1	72.65 (7)	O1—Zn1—N1	148.61 (6)
N2—Zn1—O1	76.23 (6)	O3—Zn1—N1	101.48 (7)
N4—Zn1—N1	96.71 (7)	O3—Zn1—N2	130.82 (6)
N4—Zn1—N2	112.70 (7)	O3—Zn1—N4	116.48 (6)
N4—Zn1—O1	98.95 (7)	O3—Zn1—O1	95.60 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H18···O3^{i}	0.85 (3)	1.91 (3)	2.756 (2)	176 (3)
O2—H17···O4	0.84 (3)	1.99 (3)	2.789 (2)	159 (4)

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

The H atoms on C atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and were refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(aromatic C) or $1.5U_{eq}$(methyl C). The torsion angle of the methyl groups was refined. The H atoms on the imidazole N and oxime O atoms were located in a difference Fourier map and refined with N—H and O—H distances restrained to 0.86 (1) and 0.85 (1) Å, respectively, and with $U_{iso}(H) = 1.5U_{eq}$(N,O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank the National Natural Science Foundation of China (grant No. 20101003), Heilongjiang Province Natural Science Foundation (grant No. B0007), the Educational Committee Foundation of Heilongjiang Province and Heilongjiang University for supporting this study.

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