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Diethanolbis[4-(2-furoyl)-3-methyl-1-phenyl-1*H*-pyrazol-5-onato]zinc(II)

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Key indicators

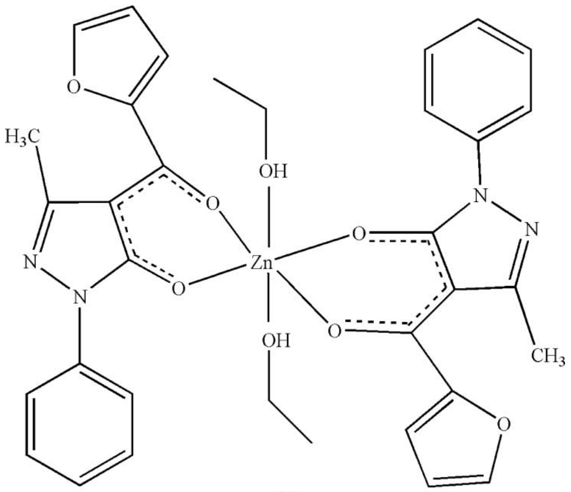
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.095
Data-to-parameter ratio = 17.2

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

The Zn atom in the title complex, $[Zn(C_{15}H_{11}N_2O_3)_2(C_2H_5OH)_2]$, lies on an inversion center and has a slightly distorted octahedral coordination environment, with four O atoms of the pyrazolone rings in the equatorial plane and two ethanol O atoms in axial positions. The structure displays O—H···N hydrogen bonding.

Comment

4-Acyl-5-pyrazolones are an interesting class of β-diketones, containing a pyrazole fused to a chelating arm. Metal complexes with these ligands are utilized in analytical chemistry and the isolation of metal ions (Nishihama *et al.*, 2001). A derivative of the 4-acyl-5-pyrazolones, 1-phenyl-3-methyl-4-(2-furoyl)-5-pyrazolone (HPMFP), was first studied in 1983 (Dong *et al.*, 1983) and in recent years, both HPMFP and its metal complexes have been found to possess antibacterial activity (Li *et al.*, 2000). The metal complexes also show analgesic activity (Liu *et al.*, 1980; Li *et al.*, 1997). Interest in understanding the coordination modes in such ligands led us to synthesize the title Zn^{II} complex, (I), and we report its crystal structure here.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. The Zn atom is located on an inversion center. It forms a distorted octahedral ZnO_6 coordination geometry. The two ethanol molecules are mutually *trans*, while the two bidentate 1-phenyl-3-methyl-4-(2-furoyl)-5-pyrazolone ligands adopt a deprotonated enol form, chelating to the Zn atom *via* their O atoms. Similar coordination is observed in other Cd (Xu *et al.*, 2004), Zn (Wang *et al.*, 2001) and Co structures (Shi *et al.*,

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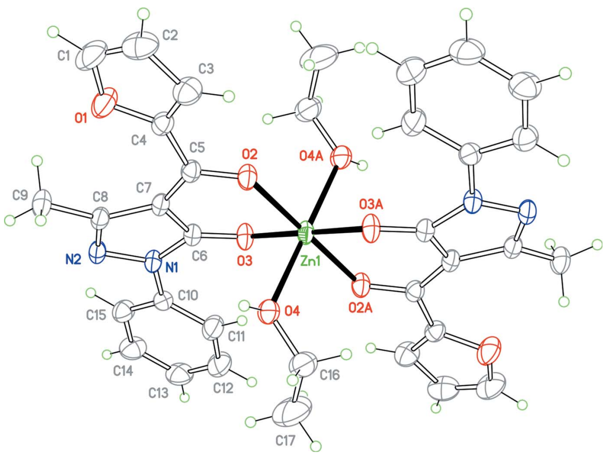

Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids [symmetry code: (A) $1 - x$, $1 - y$, $1 - z$].

2005). The two Zn—O bond lengths in the equatorial plane differ by *ca* 0.08 Å (Table 1). O—H···N hydrogen bonds (Table 2) involving the pyrazolonate N atoms link adjacent molecules to form chains in approximately orthogonal directions.

Experimental

HPMFP was synthesized according to the method proposed by Jensen (1959) (yield 73%, m.p. 374–375 K). A mixture of a 10 ml $Zn(CHCOO)_2\cdot 2H_2O$ (1 mmol, 0.2195 g) ethanol solution and a 10 ml HPMFP (2 mmol, 0.5365 g) ethanol solution was refluxed over a water bath. After vigorous stirring for 4 h at 348–353 K, a yellow product precipitated. This was filtered off, washed with cold anhydrous ethanol several times and dried in air. Pale-yellow single crystals suitable for X-ray analysis were obtained by slow cooling of a warmed anhydrous ethanol solution at room temperature.

Crystal data

$[Zn(C_{15}H_{11}N_2O_3)_2(C_2H_6O)_2]$
$M_r = 692.04$
Orthorhombic, *Pbca*
$a = 15.366$ (3) Å
$b = 9.2799$ (19) Å
$c = 22.879$ (5) Å
$V = 3262.4$ (12) Å^3

$Z = 4$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.81$ mm^{-1}
$T = 295$ (2) K
Prism, pale yellow
0.38 × 0.26 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.748$, $T_{\max} = 0.855$

29532 measured reflections
3728 independent reflections
2907 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.095$
$S = 1.07$
3728 reflections
217 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.7953P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O3	1.9992 (12)	N1—C6	1.364 (2)
Zn1—O2	2.0831 (11)	N1—N2	1.3938 (17)
Zn1—O4	2.1525 (12)	N1—C10	1.419 (2)
O2—C5	1.2627 (19)	N2—C8	1.313 (2)
O3—C6	1.2637 (18)	C5—C7	1.416 (2)
O4—C16	1.423 (2)	C6—C7	1.430 (2)
O3—Zn1—O2$^{\rm i}$	92.83 (5)	O3—Zn1—O4$^{\rm i}$	90.15 (5)
O2—Zn1—O4	90.67 (5)	O4—Zn1—O4$^{\rm i}$	180

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H18···N2$^{\rm ii}$	0.839 (10)	1.899 (10)	2.7339 (17)	173 (2)

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

The hydroxyl H atom was located in a difference Fourier map and refined as riding, with O—H distance restraints of 0.85 (1) Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$. Other H atoms were placed in calculated positions, with C—H = 0.93 for phenyl, 0.96 for methyl and 0.97 Å for methylene H atoms, and refined as riding, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for phenyl and methylene H, and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H.

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: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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