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Bis(4-benzoyl-3-methyl-1-phenyl-1*H*-pyrazol-5-onato)diethanolcadmium(II)

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Key indicators

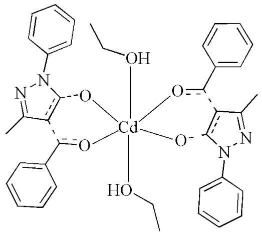
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.041
wR factor = 0.084
Data-to-parameter ratio = 13.5

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

The title complex, $[Cd(C_{17}H_{13}N_2O_2)_2(C_2H_5OH)_2]$, is centrosymmetric with a distorted octahedral coordination geometry. The pyrazolone ring is almost coplanar with the adjacent fused chelate ring [dihedral angle = 2.90 (3)°].

Comment

Many metal complexes of 4-benzoyl-3-methyl-1-phenyl-1*H*-pyrazol-5-one (PMBP) have been studied (Jensen, 1959), and it has been reported that PMBP can be used to extract lanthanides, actinides and alkaline earth metals (Liu, 1975). Moreover, in recent studies, it was discovered that chelated derivatives with lanthanides or alkaline earth metals strongly inhibit ATPase and mitochondrial enzymes in the body (Ream, 1997). In order to develop new medicines to prevent or cure some kinds of diseases associated with the metal Cd, we synthesized the complex of PMBP with Cd^{2+}, (I), and its structure is reported here.



(I)

The molecule of (I) has the Cd atom situated on an inversion center. It forms a distorted octahedral coordination geometry. The four O atoms (O1, O1^{i}, O2 and O2^{i}) of the two PMBP anions form an equatorial plane [symmetry code: (i) $1 - x, 1 - y, 1 - z$], while atoms O3 and O3^{i} from two ethanol molecules occupy the apical positions. A similar coordination was observed in other Sn (Bovio *et al.*, 1993), Zn (Wang *et al.*, 2001), and Cd structures (Zeng *et al.*, 2000). There are two sets of Cd—O bonds lengths in the equatorial plane: Cd1—O2 [2.278 (3) Å] is longer than Cd1—O1 [2.225 (3) Å]. The shorter Cd—O bonds are adjacent to the longer of the two C—O bonds, *viz*. O1—C7 [1.262 (4) Å]. This feature has been observed elsewhere (Pettnari *et al.*, 1991) and attributed to the asymmetry in the ring. The chelate ring, Cd1—O1—C7—C8—C11—O2, has a boat configuration and the deviations of atoms Cd1 and O8 from the plane are 0.1235 (13) and 0.125 (2) Å, respectively. The chelate ring plane is almost coplanar with the mean pyrazolone ring plane; the dihedral angle formed by them is 2.90 (3)°. Due to the steric hindrance, the dihedral angles of the pyrazolone ring plane with phenyl rings planes C1–C6 and C12–C17 are 14.5 (2) and 73.1 (2)°, respectively.

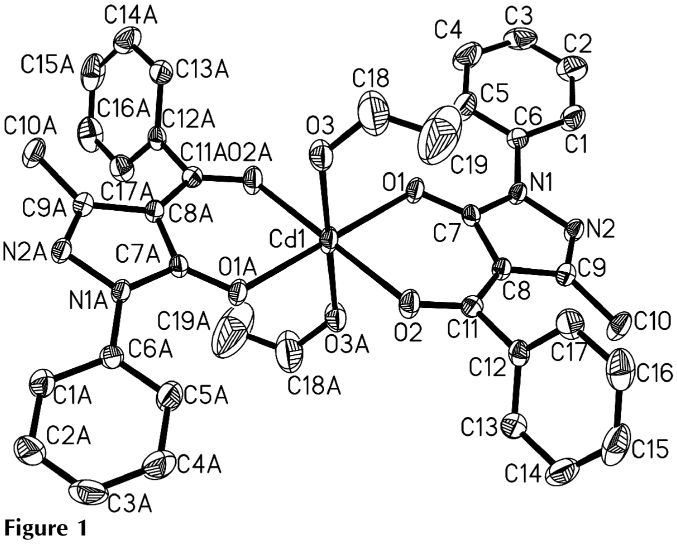


Figure 1
View of (I), with 40% probability ellipsoids [symmetry code: (*A*) $1 - x$, $1 - y$, $1 - z$.]

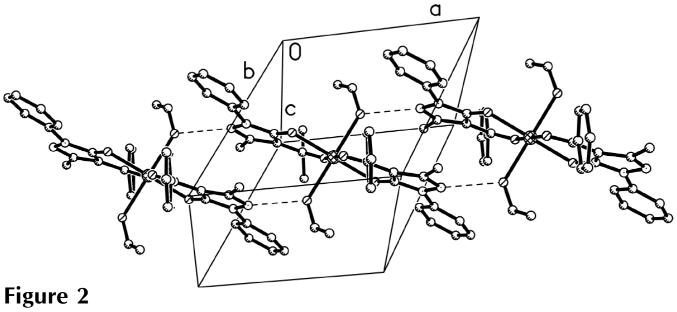


Figure 2
The molecular packing of (I). H atoms bonded to C atoms have been omitted for clarity. Dashed lines indicate the hydrogen-bonding interactions.

Experimental

A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (1 mmol) and PMBP (2 mmol) was dissolved in anhydrous hot ethanol with stirring. The solution was refluxed for 4 h at 348–353 K, and then the mixture was cooled to room temperature. The colorless product which was precipitated was filtered off and washed with cold anhydrous ethanol several times and dried in a vacuum. It was recrystallized from anhydrous ethanol.

Crystal data

$[Cd(C_{17}H_{13}N_2O_2)_2(C_2H_6O)_2]$
$M_r = 759.12$
Triclinic, $P\overline{1}$
$a = 9.130$ (3) Å
$b = 10.321$ (4) Å
$c = 10.904$ (4) Å
$\alpha = 106.791$ (6)°
$\beta = 107.621$ (5)°
$\gamma = 104.581$ (5)°
$V = 870.4$ (5) Å^3
$Z = 1$
$D_x = 1.448$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 825 reflections
$\theta = 2.6$–$26.0°$
$\mu = 0.68$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.849$, $T_{max} = 0.876$
3601 measured reflections
3055 independent reflections
2492 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 12$
$l = -12 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.084$
$S = 0.98$
3055 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—O1	2.224 (2)	O3—C18	1.396 (6)
Cd1—O2	2.278 (2)	N1—C7	1.376 (4)
Cd1—O3	2.338 (3)	N1—N2	1.399 (3)
O1—C7	1.264 (3)	N1—C6	1.427 (4)
O2—C11	1.250 (4)	N2—C9	1.316 (4)
O1—Cd1—O2	83.84 (8)	O2—Cd1—O3	93.46 (10)
O1—Cd1—O3	90.87 (9)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N2^{i}	0.800 (18)	2.05 (2)	2.839 (4)	168 (4)

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

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with $U_{iso} = 1.2U_{eq}$, except for the OH hydrogen atom, which was located in a difference Fourier map and refined isotropically. The ethanol molecule has a short C—C bond which can be attributed to large thermal motion or disorder.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SHELXTL* and *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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