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Bis[4-benzoyl-5-methyl-2-phenyl-2*H*-pyrazol-3(4*H*)-onato-κ^2O,O']bis(*N*,*N*-dimethylformamide-κO)cadmium(II)

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In the structure of the title complex, $[Ni(C_{17}H_{13}N_2O_2)_2(C_3H_7NO)_2]$ or $[Ni(PMBP)_2(DMF)_2]$, where HPMBP is 4-benzoyl-5-methyl-2-phenyl-2*H*-pyrazol-3(4*H*)-one, the Cd^{II} atom, which lies on an inversion centre, is in a distorted octahedral coordination environment. The metal is coordinated by four O atoms from the two symmetry-related chelating $PMBP^-$ ligands and O atoms from the two symmetry-related DMF ligands.

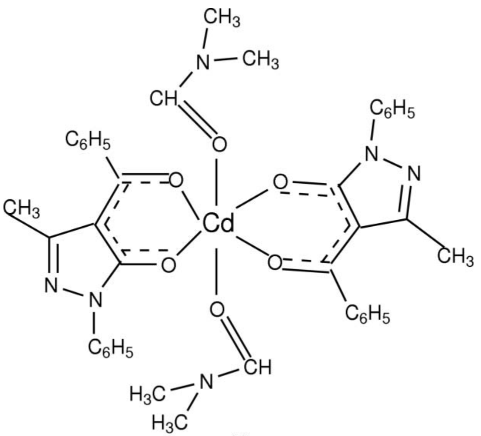
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.110
Data-to-parameter ratio = 17.6

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

Comment

Many β-diketonate complexes such as those of acetylacetonate, hexafluoroacetonate, 1,1,1-trifluoro-3-(2-thieny)acetonate and benzoylacetonate (Dong *et al.*, 1999; Li, *et al.*, 1999, 2003) have been reported. 4-Benzoyl-5-methyl-2-phenyl-2*H*-pyrazol-3(4*H*)-one (HPMBP) has also been widely studied as an extractant and chelating agent for metal ions (Okafor, 1981; Barkat *et al.*, 2004). Recently $PMBP^-$ metal complexes have attracted attention because of their potential biological activity, for example, as antibacterial and antiviral agents (Xu *et al.*, 2003). Several $PMBP^-$ metal complexes were also structurally characterized (Miao *et al.*, 1991; Xu *et al.*, 2003; Shen & Yuan, 2004).



(I)

We report here the preparation and the crystal structure of the title complex, $[Cd(PMBP)_2(DMF)_2]$, (I) (Fig. 1). The molecule has a centre of symmetry at the Cd^{II} atom, which has a distorted octahedral environment. The metal ion is coordinated by four O atoms from two symmetry-related chelating bidentate $PMBP^-$ ligands that make up the equatorial plane and O atoms from two mutually *trans* symmetry-related DMF molecules in axial sites. The Cd1—O3 bond length to the DMF ligand is 2.309 (3) Å, slightly longer than the Cd—O distances [2.206 (3) and 2.277 (3) Å] to the bidentate $PMBP^-$ ligands in

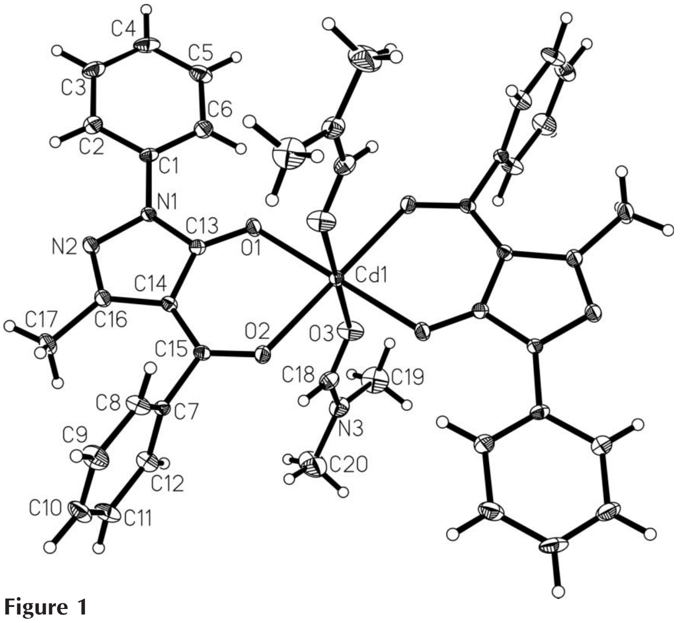


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are related by the symmetry code (−*x*, −*y*, −*z*).

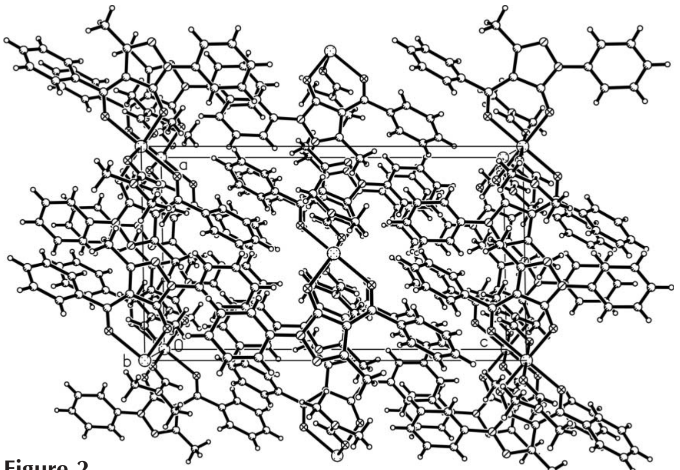


Figure 2
The crystal packing in (I).

the equatorial plane (Table 1). The *cis* O—Cd—O angles range from 84.73 (9) to 95.27 (9)°. The N1—N2, N1—C13, C13—C14 and C14—C16 bond lengths in the pyrazole ring are in the range 1.373 (5)–1.435 (5) Å, suggesting some delocalization. However, the shortest bond, N2—C16, in the pyrazole ring [1.306 (5) Å] can be assigned as a double bond. The other C—C and C—O distances in the ligand system suggest a significant degree of conjugation throughout the PMBP$^-$ ligand.

The pyrazole ring is essentially planar, with a maximum deviation of 0.006 (2) Å for C13. Atoms O1, O2 and C13–C15 are also approximately coplanar [maximum deviation = 0.033 (3) Å for C15]. The dihedral angle between the O1/O2/C13—C15 plane and the pyrazole ring is 1.7 (2)°, suggesting some π delocalization in the β-diketonate enol ring. The two phenyl rings in the ligand are not coplanar with the pyrazole system; the dihedral angles between the pyrazole plane and the C1–C6 and C7–C12 phenyl planes are 14.8 (2) and 88.77 (14)°, respectively. The dihedral angle between the two phenyl planes is 78.13 (13)°.

No significant intermolecular interactions were found in the crystal structure, the distances between planes being close to the sums of van der Waals radii (Fig. 2).

Experimental

An aqueous solution (10 ml) of $Cd(NO_3)_2 \cdot 3H_2O$ (0.290 g, 1.0 mmol) was added to a DMF solution (10 ml) of HPMBP (0.556 g, 2.0 mmol). The pH of the solution was adjusted to 6 with NaOH and the reagents were stirred for 30 min at room temperature. Well shaped colourless single crystals suitable for X-ray diffraction analysis were obtained from the filtrate after about one week at room temperature. Analysis found: C 58.97, H 4.94, N 10.29%; calculated for $C_{40}H_{40}CdN_6O_6$: C 59.08, H 4.96, N 10.34%.

Crystal data

$[Cd(C_{17}H_{13}N_2O_2)_2(C_3H_7NO)_2]$
$M_r = 813.18$
Monoclinic, $P2_1/n$
$a = 10.4835$ (19) Å
$b = 9.4768$ (18) Å
$c = 18.660$ (4) Å
$\beta = 90.981$ (4)°
$V = 1853.6$ (6) Å^3
$Z = 2$
$D_x = 1.457$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6769 reflections
$\theta = 3.1$–27.5°
$\mu = 0.65$ mm^{-1}
$T = 193$ (2) K
Block, colourless
0.34 × 0.21 × 0.20 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (North *et al.*, 1968)
$T_{min} = 0.838$, $T_{max} = 0.883$
20261 measured reflections
4248 independent reflections
3899 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 13$
$k = -12 \rightarrow 12$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.110$
$S = 1.09$
4248 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.012P)^2 + 9.086P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—O1	2.206 (3)	N1—N2	1.401 (4)
Cd1—O2	2.277 (3)	N1—C1	1.420 (5)
Cd1—O3	2.309 (3)	N2—C16	1.306 (5)
O1—C13	1.267 (4)	C13—C14	1.431 (5)
O2—C15	1.255 (4)	C14—C15	1.416 (5)
O3—C18	1.229 (5)	C14—C16	1.435 (5)
N1—C13	1.373 (5)		
O1^i—Cd1—O1	180	O2—Cd1—O3^i	90.26 (11)
O1^i—Cd1—O2	95.27 (9)	O1—Cd1—O3	92.81 (12)
O1—Cd1—O2	84.73 (9)	O2—Cd1—O3	89.74 (11)
O2—Cd1—O2^i	180	O3^i—Cd1—O3	180
O1—Cd1—O3^i	87.19 (12)		

Symmetry code: (i) −*x*, −*y*, −*z*.

H atoms were placed in idealized positions and refined as riding, with C—H distances of 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms and the H atom attached to C18, and C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms.

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 1998); software used to prepare material for publication: *SHELXTL*.

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