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Tris(biacetyl dihydrazone-κ^2N,N')cadmium(II) bis(perchlorate) at 110 K

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.090
Data-to-parameter ratio = 14.5

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

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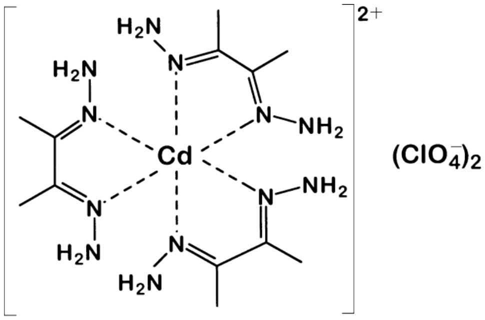
The crystal structure of the title compound, $[Cd(C_4H_{10}N_4)_3](ClO_4)_2$, has been precisely determined at *ca* 110 K. The cation is located on a $\overline{3}$ axis and is characterized by an approximate octahedral geometry, with each of the ligands occupying two coordination sites around the metal.

Comment

This structure of a 3:1 complex, (I), of biacetyl dihydrazone with cadmium perchlorate is isomorphous and isometric with that of an analogous zinc complex described in the preceeding paper (Elengoz *et al.*, 2005). It has perfect $\overline{3}$ symmetry, in which three chelating ligands occupy the octahedral coordination sites of the metal ion (Fig. 1), crystallizing in the trigonal space group $P\overline{3}c1$ with two units of the complex in the unit cell. The imine N atoms of the ligand provide the coordination sites to the central metal ion. The bond lengths observed in the isostructural complexes with cadmium (this study; see Table 1) and Zn (Elengoz *et al.*, 2005), analysed at 110 K, and with Ni (Romanenko *et al.*, 1989) analysed (with lower precision) at room temperature, are compared in Table 2.



(I)

Experimental

Compound (I) was synthesized by reacting stoichiometric amounts of cadmium diperchlorate monohydrate and biacetyl dihydrazone dissolved in hot methanol, followed by slow crystallization.

Crystal data

$[Cd(C_4H_{10}N_4)_3](ClO_4)_2$
M_r = 653.78
Trigonal, $P\overline{3}c1$
a = 9.5905 (7) Å
c = 15.2874 (8) Å
V = 1217.72 (14) Å^3
Z = 2
D_x = 1.783 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1745 reflections
θ = 2.5–27.0°
μ = 1.18 mm^{-1}
T = 110 (2) K
Prism, colourless
0.20 × 0.15 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.798$, $T_{\max} = 0.843$
8975 measured reflections
810 independent reflections
638 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\max} = 27.0°$
$h = -12 \rightarrow 12$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.090$
$S = 1.08$
810 reflections
56 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.0248P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.013$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—N3	2.325 (2)	C4—C4$^{\rm i}$	1.489 (5)
N2—N3	1.375 (3)	C4—C5	1.502 (4)
N3—C4	1.289 (3)		
N3$^{\rm ii}$—Cd1—N3	154.27 (12)	N3$^{\rm iii}$—Cd1—N3	106.63 (12)
N3$^{\rm i}$—Cd1—N3	69.82 (11)	N3$^{\rm iv}$—Cd1—N3	94.53 (7)

Symmetry codes: (i) $x - y, -y, \frac{1}{2} - z$; (ii) $y, x, \frac{1}{2} - z$; (iii) $-x, -x + y, \frac{1}{2} - z$; (iv) $-x + y, -x, z$.

Table 2
Comparison of some bond lengths (Å) in the isostructural Cd, Zn and Ni 1:3 complexes with biacetyldihydrazone.

Bond	*M* = Zn	*M* = Cd	*M* = Ni
M—N3	2.158 (2)	2.325 (2)	2.060–2.088
N2—N3	1.386 (3)	1.375 (3)	1.375–1.388
N3—C4	1.281 (3)	1.289 (3)	1.278–1.298
C4—C5	1.492 (3)	1.502 (4)	1.378–1.504
C4—C4*	1.502 (5)	1.489 (5)	1.473–1.485

The amine H atoms were located in a difference Fourier map and their displacement parameters were refined as riding in their as-found relative positions, with isotropic displacement parameters. Methyl H atoms were placed in idealized positions, with C—H = 0.98 Å, and refined using a riding model, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. The methyl group was allowed to rotate about the C—CH$_3$ bond, while preserving the C—H bond distances and tetrahedral geometry.

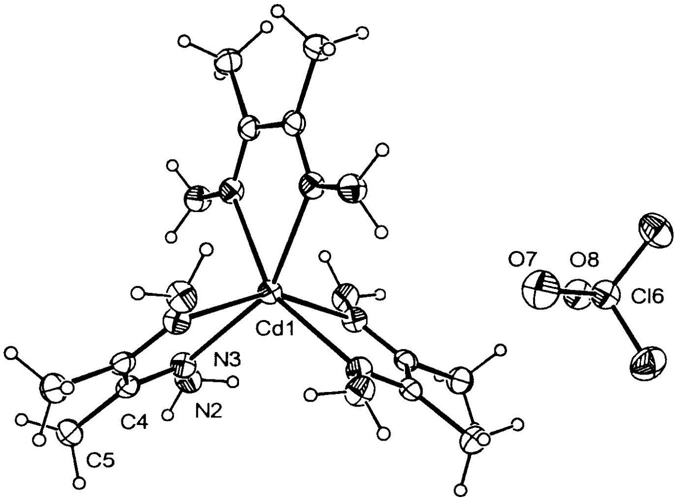


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Atom Cd1 lies on a $\overline{3}$ axis and atoms Cl6 and O8 lie on a threefold rotation axis. One of the anions has been omitted for clarity.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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