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{Tris[2-(2-pyridylmethyleneamino-κ^2N,N')-ethyl]amine}cadmium(II) bis(perchlorate)

Hong-Shan He

Department of Applied Chemistry, Huaqiao University, Quanzhou 362011, People's Republic of China, and Department of Chemistry, Biochemistry and Molecular Biology, North Dakota State University, Fargo, ND 58105, USA

Correspondence e-mail:
hongshan.he@ndsu.edu

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In the title compound, $[Cd(C_{24}H_{27}N_7)](ClO_4)_2$, the Cd^{2+} ion is coordinated by six N atoms from the tripodal organic ligand in the form of a trigonal antiprism.

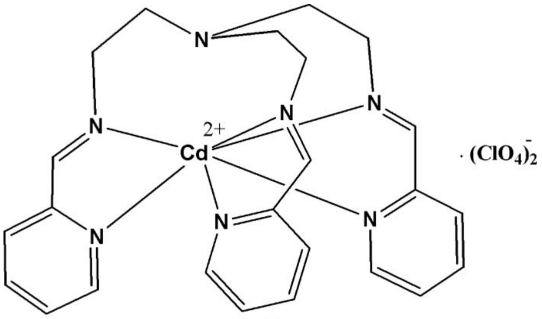
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.036
wR factor = 0.113
Data-to-parameter ratio = 15.3

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

Comment

The synthesis of metal complexes of [tris(2-pyridylmethyleneaminoethyl)amine], py$_3$tren, was first studied by Wilson & Norman (1968). Subsequent structural studies on the Mn^{2+}, Co^{2+}, Zn^{2+}, Cu^{2+} (Kirchner *et al.*, 1987) and Fe^{2+} (Brewer *et al.*, 2004; Morgenstern-Badarau *et al.*, 2000) py$_3$tren complexes have shown that the py$_3$tren ligand chelates the metal ion, forming stable complexes with the coordination of up to seven N atoms. In the title compound, (I) (Fig. 1), the metal ion is coordinated by three imine and three pyridine N atoms. The tripodal bridging amine N atom is situated 2.834 (3) Å from the metal center, indicating a moderate non-bridging interaction, as supported by *ab-initio* molecular orbital studies of the py$_3$tren cadmium complex (Jäntti *et al.*, 1998) The three imine and the three pyridine N atoms form two triangles between which the metal ion is located. These two triangles, nearly parallel to each other, are staggered by almost 39°, thus the coordination geometry can be described as a trigonal antiprism. The bite angles, N—Cd—N, are 69.97 (12), 70.99 (12), and 69.95 (9)° for N2—Cd1—N3, N4—Cd1—N5 and N6—Cd1—N7, respectively. The bite distances, N···N, are 2.719 (5), 2.726 (5) and 2.732 (4) Å, respectively, for N2···N3, N4···N5 and N6···N7.



(I)

Experimental

A magnetically stirred solution of tren (0.146 g, 1.0 mmol) dissolved in 6 ml of freshly dried methanol was added to 0.32 g (3.0 mmol) pyridine-2-carboxaldehyde dissolved in 4 ml of dried methanol. The solution was refluxed for 4 h and cadmium perchlorate hexahydrate (0.419 g, 1.0 mmol) dissolved in 3 ml of methanol was then added dropwise. The mixture was refluxed for a further 2 h, then the solvent was reduced to *ca* 5 ml on a rotary evaporator; the solid was collected by filtration, washed with methanol and dried under vacuum (0.53 g, yield 74%). A single crystal suitable for X-ray diffraction analysis was obtained by slow evaporation of an acetonitrile solution of the complex over two weeks at room temperature.

Crystal data

$[Cd(C_{24}H_{27}N_7)](ClO_4)_2$
$M_r = 724.83$
Monoclinic, $C2/c$
$a = 28.4372$ (3) Å
$b = 10.8977$ (1) Å
$c = 19.6203$ (2) Å
$\beta = 101.140$ (1)°
$V = 5965.77$ (10) Å^3
$Z = 8$
$D_x = 1.614$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.97$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 1K CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.760$, $T_{\max} = 0.830$
66248 measured reflections
5866 independent reflections
4911 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.113$
$S = 1.02$
5866 reflections
384 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 9.6103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.05$ e Å^{-3}
$\Delta\rho_{\min} = -0.47$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00032 (7)

H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C—H bond lengths of 0.93 (aromatic CH) or 0.97 Å (CH_2), and isotropic displacement parameters equal to 1.2 times $U_{\rm eq}$ of the parent atom. The highly anisotropic displacement ellipsoids of one of the perchlorate ions are probably a consequence of some disorder of this anion. The deepest residual density hole is located 1.39 Å from atom O8.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

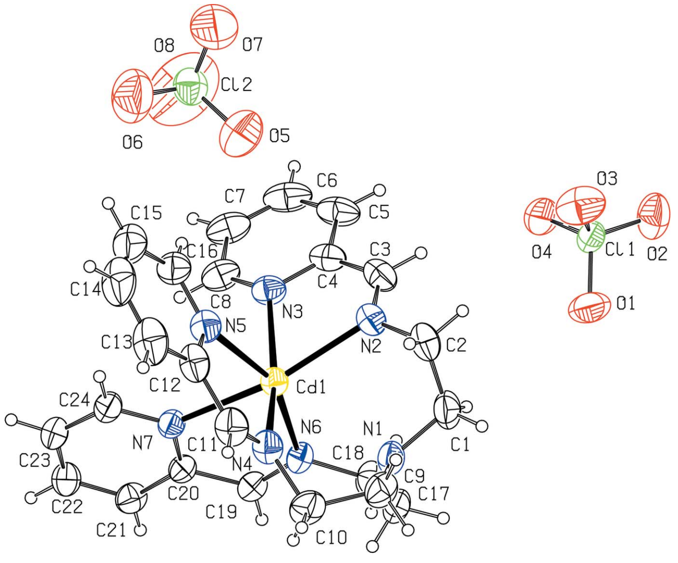


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are drawn as small circles of arbitrary radius.

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