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# Tris(ethylenediamine-$\kappa^2N,N'$)cadmium(II) dinitrate

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The title compound, $[Cd(C_2H_8N_2)_3](NO_3)_2$, crystallizes with distorted $Cd(en)_3^{2+}$ octahedra. Extensive hydrogen bonding is present between the ethylenediamine and nitrate groups.

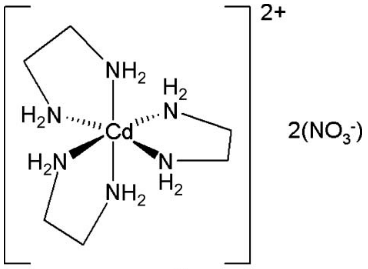
**Key indicators**

Single-crystal synchrotron study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.005 Å
Disorder in solvent or counterion
$R$ factor = 0.049
$wR$ factor = 0.118
Data-to-parameter ratio = 24.2

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

## Comment

Six-coordinate cadmium(II) complexes are intriguing systems because of the competition between pseudo-octahedral and trigonal–prismatic geometry. The $4d^{10}$ configuration of the cation allows a study of the geometric effects of altering the ligand set without competing ligand field effects (Holloway & Melnik, 1995).



(I)

The title compound, $[Cd(en)_3](NO_3)_2$ (en = ethylenediamine), (I) (Fig. 1), contains roughly octahedral $Cd(en)_3^{2+}$ units with each ethylenediamine bidentate to $Cd^{2+}$, and is charge-balanced by two nitrate anions. The relatively small 'bite angle' of the ethylenediamine (Table 1) means there is a significant distortion away from ideal octahedral geometry, similar to analogous compounds (Mahadevan *et al.*, 1985; Yuge *et al.*, 1995).

The Cd—N distances (Table 1) all lie between 2.347 (3) and 2.400 (3) Å, in good agreement with similar compounds (Mahadevan *et al.*, 1985; Yuge *et al.*, 1995). While the space

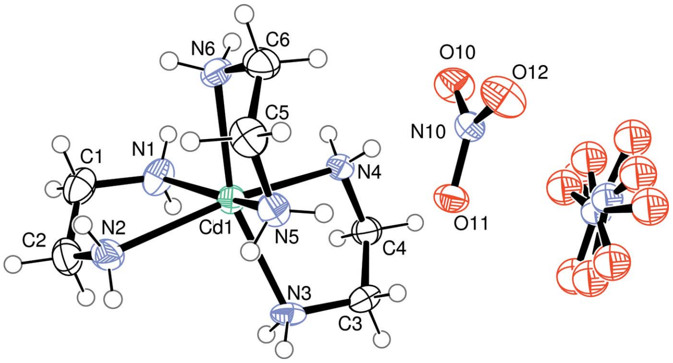


**Figure 1**
A view of (I), with displacement ellipsoids drawn at the 50% probability level. The disordered nitrate group is unlabelled.

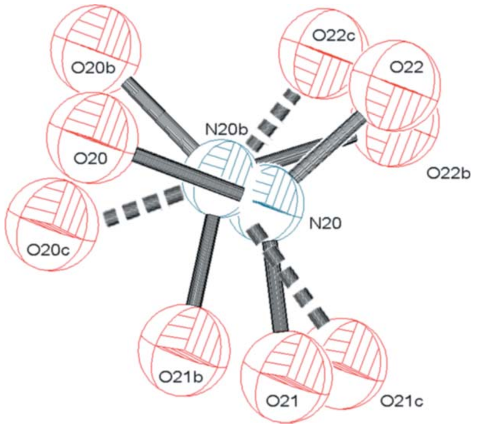


**Figure 2**
The three orientations of the disordered nitrate group in (I). Displacement ellipsoids are drawn at the 50% probability level.

group is centrosymmetric, it is important to note that the $Cd^{2+}$ ion does not lie on an inversion centre. The unit cell thus contains both L- and D-$Cd(en)_3^{2+}$ cations.

The asymmetric unit of (I) (Fig. 2) contains two nitrate anions. One nitrate is well ordered (N20/O20/O21/O22), but there is significant disorder in the second and it was modelled as split over three orientations in the ratio 0.477 (8):0.331 (6): 0.195 (7). A large number of N—H···O hydrogen bonds exist between the $Cd(en)_3^{2+}$ and nitrate units. For the well ordered nitrate, the N···O distances lie in the range 2.976 (4)–3.163 (4)Å (Table 2). N···O distances for those to the second (disordered) nitrate lie in the range 2.828 (4)–3.444 (4)Å.

## Experimental

Cadmium nitrate tetrahydrate (0.0308 g, 0.1 mmol) was dissolved in propan-2-ol (15 ml) in a 50 ml beaker. Ethylenediamine (1 ml, 0.0167 mol) was placed in a sealed plastic vial. The top of the vial was pierced with a needle and the vial placed in the beaker. The beaker was sealed with Parafilm. Slow diffusion of ethylenediamine into the alcoholic solution led to colourless crystals after a period of a few weeks. Reagents were purchased from the Aldrich Chemical Company and used without further purification.

### *Crystal data*

$[Cd(C_2H_8N_2)_3](NO_3)_2$
$M_r = 416.73$
Monoclinic, $I2/a$
$a = 15.2574$ (12) Å
$b = 11.0441$ (8) Å
$c = 19.155$ (2) Å
$\beta = 90.246$ (1)°
$V = 3227.6$ (5) Å$^3$
$Z = 8$
$D_x = 1.715$ Mg m$^{-3}$

Synchrotron radiation
$\lambda = 0.6711$ Å
Cell parameters from 2733 reflections
$\theta = 2.9$–$23.0°$
$\mu = 1.39$ mm$^{-1}$
$T = 150$ (2) K
Block, colourless
0.02 × 0.01 × 0.01 mm

### *Data collection*

Bruker APEXII diffractometer
Narrow-frame $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.647$, $T_{max} = 1.00$
20814 measured reflections
5813 independent reflections

4525 reflections with $I > 2\sigma(I)$
$R_{int} = 0.076$
$\theta_{max} = 31.1°$
$h = -22 \rightarrow 23$
$k = -16 \rightarrow 16$
$l = -29 \rightarrow 28$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.118$
$S = 1.07$
5813 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 0.6151P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.03$ e Å$^{-3}$
$\Delta\rho_{min} = -0.69$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cd1—N1 | 2.364 (3) | N3—C3 | 1.469 (5) |
| Cd1—N2 | 2.400 (3) | N4—C4 | 1.474 (4) |
| Cd1—N3 | 2.362 (3) | N5—C5 | 1.461 (5) |
| Cd1—N4 | 2.347 (3) | N6—C6 | 1.466 (5) |
| Cd1—N5 | 2.355 (3) | C2—C1 | 1.499 (6) |
| Cd1—N6 | 2.382 (3) | C3—C4 | 1.514 (5) |
| N1—C1 | 1.473 (5) | C5—C6 | 1.515 (5) |
| N2—C2 | 1.476 (5) | | |
| N1—Cd1—N2 | 74.45 (12) | N5—Cd1—N6 | 74.52 (11) |
| N3—Cd1—N4 | 75.38 (10) | | |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H4*D*···O10 | 0.85 (2) | 2.31 (2) | 3.163 (4) | 175 (3) |
| N5—H5*C*···O11 | 0.84 (2) | 2.21 (2) | 3.026 (4) | 166 (4) |
| N4—H4*C*···O10$^{i}$ | 0.84 (2) | 2.46 (3) | 2.991 (3) | 122 (3) |
| N6—H6*D*···O10$^{i}$ | 0.86 (2) | 2.29 (3) | 3.050 (4) | 148 (4) |
| N2—H2*D*···O12$^{ii}$ | 0.85 (2) | 2.21 (2) | 2.976 (4) | 150 (3) |
| N6—H6*C*···O12$^{ii}$ | 0.84 (2) | 2.29 (3) | 3.022 (4) | 146 (4) |
| N3—H3*D*···O11$^{iii}$ | 0.84 (2) | 2.31 (3) | 3.047 (4) | 147 (4) |

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

The non-standard setting of $C2/c$ employed ($I2/a$) was chosen as the *C*-centred setting has a rather oblique $\beta$ angle (128.39°). H atoms attached to the ethylenediamine C atoms were positioned geometrically and refined with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bonded to the amine N atoms were located in a difference map. The positions and isotropic displacement parameters of these were refined, but the N—H bond lengths were restrained to 0.86 (2) Å. The disordered nitrate was modelled over three positions. All of the O atoms were refined with a single isotropic displacement parameter. Initially three positions were refined for the N atoms, but this refinement proved unstable. Two N-atom positions were therefore employed, with a single iostropic displacement parameter. N—O bond lengths in the three orientations were restrained to be equal, with an s.u. of 0.02Å. The maximum residual electron density is located 0.57 Å from O21*B*.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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