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Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{N}-\text{C}) = 0.007$ Å
 R factor = 0.032
 wR factor = 0.085
Data-to-parameter ratio = 11.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Poly[[bis(μ_2 -3,5-diamino-1,2,4-triazole)-
 $\kappa^2\text{N}^2:\text{N}^4$; $\kappa^2\text{N}^4:\text{N}^2$ - μ_2 -sulfato- $\kappa^2\text{O}:\text{O}'$ -cadmium(II)]
monohydrate]

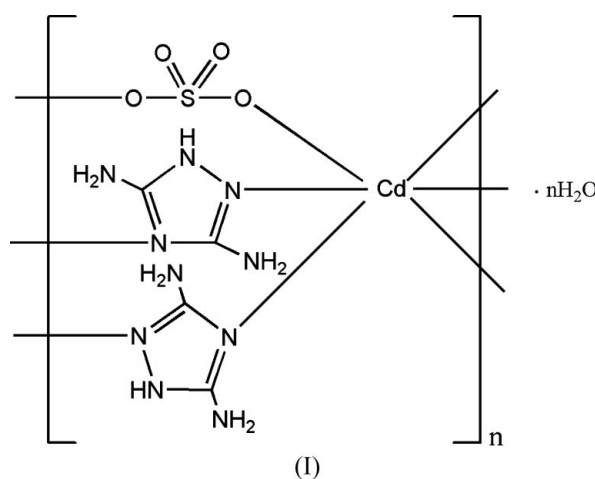
The geometry of the Cd atom in the polymeric title complex, $\{[\text{Cd}(\text{SO}_4)(\text{C}_2\text{H}_5\text{N}_3)_2]\cdot\text{H}_2\text{O}\}_n$, is six-coordinate distorted octahedral, defined by two O-atom donors derived from two sulfate dianions and four N atoms from four different 3,5-diamino-1,2,4-triazole ligands. The sulfate dianions interact with Cd^{II} atoms, forming infinite strands running in the c -axis direction. The three-dimensional supramolecular structure is stabilized by $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ interactions. The two independent water molecules lie on different twofold axes and interact with the network through hydrogen bonds.

Received 1 July 2006

Accepted 26 July 2006

Comment

3,5-Diamino-1,2,4-triazole, guanazole, has been actively investigated as a versatile ligand that can yield polynuclear metal complexes with interesting magnetic properties (Antolini *et al.*, 1990, 1991). It can also be used in the synthesis of metal-organic compounds that exhibit tubular architectures (Goforth *et al.*, 2005). Among the cadmium complexes with guanazole whose structures are known is $[\text{Cd}(\text{SCN})_2(\text{C}_2\text{H}_5\text{N}_3)_2]$ (Desseyn *et al.*, 1990). In this structure, the metal centers are linked by $\text{N}2/\text{N}4$ bridges, forming a two-dimensional network. Our interest is currently focused on the thermochemistry of transition-metal complexes bearing triazole ligands, and this led to the investigation of the title complex, (I).



Each Cd^{II} ion in (I) (Fig. 1) is in a six-coordinate distorted octahedral environment (Table 1). Four N atoms from four N-heterocycles fill the equatorial positions, and two O atoms from two sulfate dianions occupy the axial positions. The interactions between Cd and sulfate dianions are relatively strong and lead to the formation of infinite strands of

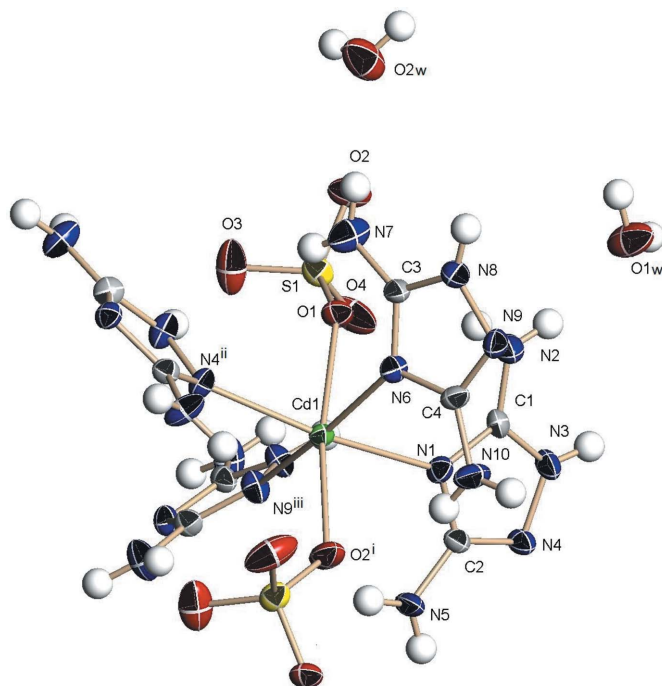


Figure 1

The coordination geometry for the Cd center in (I). Displacement ellipsoids are drawn at the 50% probability level [symmetry codes: (i) $\frac{1}{2} - y, x, z - \frac{1}{4}$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (iii) $\frac{1}{2} - x, \frac{1}{2} + y, z$].

$\{\text{Cd} \cdots \text{SO}_4\}_n$ synthons along the c axis. The guanazole ligands link the infinite strands into a three-dimensional network. The two independent water molecules lie on different twofold axes; they interact with the network through hydrogen bonds. The resulting three-dimensional assembly is thus stabilized by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds, as detailed in Table 2.

Experimental

Single crystals of (I) were grown by slow diffusion of the guanazole ligand (0.2 mmol, 0.0198 g) in methanol (5 ml) into an aqueous solution (5 ml) of $\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ (0.033 mmol, 0.0256 g). Colorless single crystals suitable for single-crystal X-ray diffraction analysis were obtained after two weeks. Analysis calculated for $\text{C}_4\text{H}_{12}\text{CdN}_{10}\text{O}_5\text{S}$: C 11.31, H 2.85, N 32.98%; found: C 11.62, H 2.94, N 32.54%.

Crystal data

$[\text{Cd}(\text{SO}_4)(\text{C}_2\text{H}_5\text{N}_5)_2] \cdot \text{H}_2\text{O}$
 $M_r = 424.70$
 Tetragonal, $I4_1cd$
 $a = 13.446$ (1) Å
 $c = 27.980$ (2) Å
 $V = 5058.5$ (5) Å³
 $Z = 16$

$D_x = 2.231$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 1.94$ mm⁻¹
 $T = 295$ (2) K
 Block, colorless
 $0.47 \times 0.19 \times 0.14$ mm

Data collection

Bruker APEX-II area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.463, T_{\max} = 0.773$
 11734 measured reflections
 2121 independent reflections
 1966 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.085$
 $S = 1.08$
 2121 reflections
 191 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 5.4574P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 2.06 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.10 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 986 Friedel pairs
 Flack parameter: 0.04 (3)

Table 1

Selected geometric parameters (Å, °).

Cd1—O1	2.354 (7)	Cd1—N4 ⁱⁱ	2.341 (4)
Cd1—O2 ⁱ	2.308 (7)	Cd1—N6	2.344 (4)
Cd1—N1	2.377 (4)	Cd1—N9 ⁱⁱⁱ	2.355 (4)
O1—Cd1—O2 ⁱ	171.3 (2)	O2 ⁱ —Cd1—N9 ⁱⁱⁱ	93.5 (2)
O1—Cd1—N1	84.1 (2)	N1—Cd1—N4 ⁱⁱ	168.1 (2)
O1—Cd1—N4 ⁱⁱ	85.2 (2)	N1—Cd1—N6	90.9 (1)
O1—Cd1—N6	81.6 (2)	N1—Cd1—N9 ⁱⁱⁱ	86.1 (1)
O1—Cd1—N9 ⁱⁱⁱ	90.3 (2)	N4 ⁱⁱ —Cd1—N6	92.8 (1)
O2 ⁱ —Cd1—N1	88.4 (2)	N4 ⁱⁱ —Cd1—N9 ⁱⁱⁱ	88.7 (1)
O2 ⁱ —Cd1—N4 ⁱⁱ	102.6 (2)	N6—Cd1—N9 ⁱⁱⁱ	171.6 (2)
O2 ⁱ —Cd1—N6	94.3 (2)		

Symmetry codes: (i) $-y + \frac{1}{2}, x, z - \frac{1}{4}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z$; (iii) $-x + \frac{1}{2}, y + \frac{1}{2}, z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
N2—H2a \cdots O1	0.85	2.41	2.979 (6)	125
N2—H2b \cdots O1w	0.85	2.18	3.02 (1)	168
N3—H3 \cdots O3 ^{iv}	0.85	2.07	2.837 (6)	150
N5—H5a \cdots O3 ^v	0.85	2.05	2.857 (6)	157
N5—H5b \cdots O2 ⁱ	0.85	2.33	3.039 (7)	142
N7—H7a \cdots O2w	0.85	2.17	2.99 (1)	162
N7—H7b \cdots O1	0.85	2.25	2.803 (8)	123
N8—H8 \cdots O4 ^{vi}	0.85	2.13	2.783 (6)	133
N10—H10a \cdots O4 ^{vii}	0.85	2.02	2.826 (6)	159
N10—H10b \cdots O2 ⁱ	0.85	2.32	3.106 (9)	154
O1w—H1w \cdots N5 ^{viii}	0.85	2.31	3.000 (5)	138
O2w—H2w \cdots N10 ^{viii}	0.85	2.18	2.946 (6)	150

Symmetry codes: (i) $-y + \frac{1}{2}, x, z - \frac{1}{4}$; (iv) $x + \frac{1}{2}, -y + \frac{1}{2}, z$; (v) $-y + 1, -x + \frac{1}{2}, z - \frac{1}{4}$; (vi) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (vii) $y, x - \frac{1}{2}, z - \frac{1}{4}$; (viii) $y, -x + \frac{1}{2}, z + \frac{1}{4}$.

C-bound H atoms were positioned geometrically ($\text{C}-\text{H} = 0.97$ Å) and were included in the refinement in the riding-model approximation. The water H atoms were placed at chemically sensible positions on the basis of hydrogen-bonding interactions, with $\text{O}-\text{H} = 0.85$ Å. The displacement parameters of all H atoms were set at $1.2U_{\text{eq}}(\text{C}, \text{O})$. The final difference Fourier map had a large residual electron-density peak about 1 Å from the Cd atom and a deep hole at about 1 Å from the S1 atom, but was otherwise featureless.

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

We thank the Northwest University for the diffraction measurements. We also thank Shaanxi Normal University and the University of Malaya for supporting this study.

References

- Antolini, L., Fadretti, A. C., Gatteschi, D., Giusti, A. & Sessoli, R. (1990). *Inorg. Chem.* **29**, 143–145.
- Antolini, L., Fadretti, A. C., Gatteschi, D., Giusti, A. & Sessoli, R. (1991). *Inorg. Chem.* **30**, 4860–4863.
- Bruker (2004). *SAINT* (Version 7.06A) and *SMART* (Version 7.06A). Bruker AXS Inc., Madison, Wisconsin, USA.
- Desseyn, H. O., Fabretti, A. C. & Malavasi, W. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 355–362.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Goforth, A. M., Su, C. Y., Hipp, R., Macquart, R. B., Smith, M. D. & zur Loye, H. C. (2005). *J. Solid State Chem.* **78**, 2511–2518.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.