C2—C3	1.375 (8)	C19—C18	1.36 (2)
C3—C6	1.495 (9)	C19—C22^{ii}	1.53 (2)
C4—C3	1.378 (9)	C20—C19	1.32 (1)
N1—Cd—N2	91.1 (2)	C9—C8—C7	124.0 (7)
N1—C1—C2	124.1 (6)	C10—N2—Cd	120.3 (4)
N2—C10—C9	123.7 (6)	C10—C9—C8	121.6 (7)
N2—C11—C12	122.7 (8)	C11—N2—Cd	123.6 (5)
N3—C13—Ni	178.2 (6)	C11—N2—C10	116.2 (7)
N3—Cd—N1	90.2 (2)	C12—C8—C7	121.4 (7)
N3—Cd—N2	90.9 (2)	C12—C8—C9	114.5 (7)
N4—C14—Ni	179.1 (5)	C13—Ni—C14	89.0 (2)
C1—N1—Cd	121.7 (4)	C13—N3—Cd	164.2 (5)
C1—N1—C5	114.7 (6)	C16—C15—C20	124.5 (7)
C2—C3—C4	115.1 (6)	C16—C17—C18	123.4 (9)
C2—C3—C6	124.9 (6)	C17—C16—C15	114.3 (8)
C3—C2—C1	120.3 (6)	C17—C18—C19	121.1 (9)
C3—C6—C7^{i}	117.4 (6)	C18—C19—C22^{ii}	119.7 (8)
C4—C3—C6	120.0 (6)	C19—C20—C15	116.8 (7)
C4—C5—N1	124.3 (7)	C20—C19—C18	119.8 (8)
C5—N1—Cd	123.5 (4)	C20—C19—C22^{ii}	120.5 (8)
C5—C4—C3	121.5 (6)	C21—C15—C16	117.4 (9)
C8—C7—C6^{iii}	115.4 (6)	C21—C15—C20	118.0 (9)
C8—C12—C11	121.2 (7)		
N1—C1—C2—C3	−0.5 (9)	C1—C2—C3—C6	176.5 (7)
N1—C5—C4—C3	−1.8 (9)	C4—C3—C6—C7^{i}	−164.3 (7)
Cd—N1—C1—C2	−178.0 (5)	C5—C4—C3—C6	−175.6 (7)
Cd—N1—C5—C4	179.1 (6)	C6^{iii}—C7—C8—C9	−126.8 (8)
Cd—N2—C11—C12	−177.1 (8)	C7—C8—C9—C10	−177.9 (7)
Cd—N2—C10—C9	178.5 (6)	C7—C8—C12—C11	179.3 (9)

Symmetry codes: (i) $x, y-1, z$; (ii) $1-x, -y, -z$; (iii) $x, 1+y, z$.

Data collection and cell refinement were performed using *CAD*-4-*PC* software (Enraf–Nonius, 1993). *MolEN* (Fair, 1990) was used for data reduction. Absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Table 4.2.4.2). The structure was solved by Patterson heavy-atom methods using *SHELXS*86 (Sheldrick, 1990), which revealed the positions of the Cd and Ni atoms. Refinement was carried out by full-matrix least-squares methods using *MolEN* (Fair, 1990). H atoms, except those of the *Guest* molecule, were located from difference Fourier maps and refined isotropically. The H atoms of the *Guest* molecule were placed geometrically and refined by the riding method. The C15—C21 bond length was restrained to have a reasonable value. Positional disorder of the *Guest* atoms was not found. Molecular graphics were prepared using *ORTEP*II (Johnson, 1976) and the material for publication was prepared with *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1143). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Tetraaquabis(*N*,*N*′,*N*″-tricyanomelamine)cobalt(II) Hexahydrate

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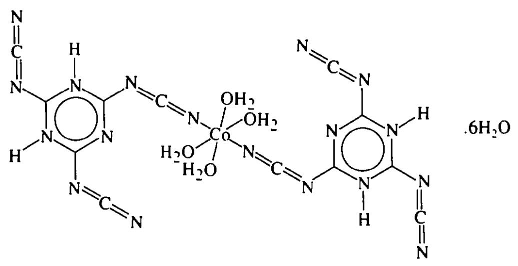
Abstract

The crystal structure of *trans*-tetraaquabis[1,3,5-triazine-2,4,6-tricarbodiimidato(1−)-*N*]cobalt(II) hexahydrate, $[Co(C_6H_2N_9)_2(H_2O)_4].6H_2O$, shows the cobalt(II) metal ion to be octahedrally coordinated by two *trans* doubly-protonated tricyanomelamine ligands and four water molecules. Hydrogen bonding between ligands yields a two-dimensional sheet structure.

Comment

Recent work has demonstrated that new extended networks may be constructed by linking metal centres through rod-like or slab-like ligands (Robson *et al.*, 1992). The trianion of tricyanomelamine, $tcmel^{3-}$, is a robust unit which is expected to offer a rich coordination chemistry given the number and variety of potential donor sites. Accordingly, the ligand was considered an ideal candidate for the construction of metal–ligand polymers. As part of a preliminary investigation of the ligand and its coordination chemistry, a cobalt(II) complex of the doubly-protonated ligand, H_2tcmel^-, was isolated as the 1:2 complex $[Co^{II}(H_2tcmel)_2(H_2O)_4].6H_2O$, (I).



(I)

The Co^{II} atom lies on a centre of symmetry and is octahedrally coordinated by two *trans* H_2tcmel^- ligands as shown in Fig. 1. Although the trianion possesses potential threefold symmetry, the ligands in the structure adopt a lower-symmetry arrangement. As indicated in Fig. 2, each H_2tcmel^- ligand forms hydrogen bonds to two centrosymmetrically related ligands resulting in the formation of 8 and 12-membered rings. From a topological viewpoint, each H_2tcmel^- ligand may be considered as a triply-connecting centre forming links to three other ligands through hydrogen bonds and coordinate bonds. The resulting sheet possesses the same connectivity as a two-dimensional hexgaonal grid. Hydrogen bonding involving lattice water extends throughout the structure.

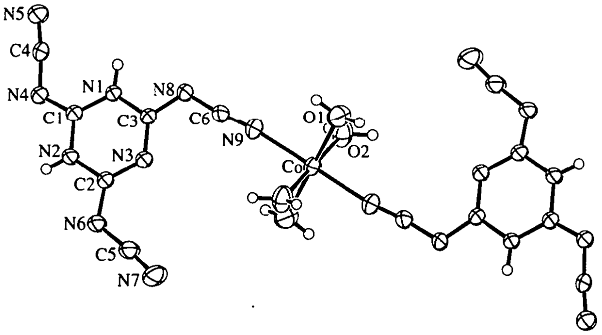


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of compound (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are of arbitrary size.

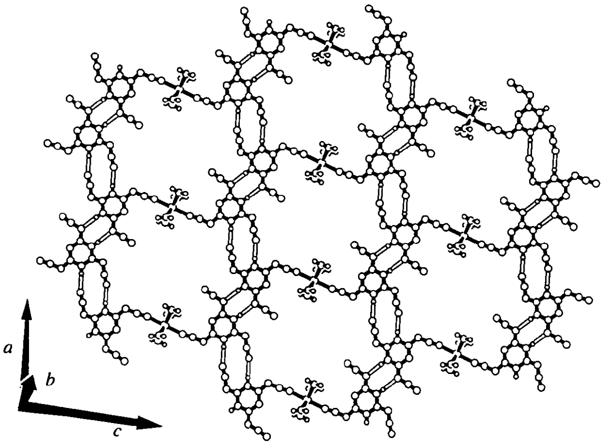


Fig. 2. *ORTEP*II (Johnson, 1976) diagram showing the hydrogen bonding between molecules. Atoms are of arbitrary size and hydrogen bonds are indicated by open lines.

Despite the relatively planar nature of the ligand, the Co atom lies 0.684 (1) Å out of the plane of the ligands. Analysis of the ligand geometry indicates extensive delocalization.

Experimental

The hydrated salt $Na_3tcmel.3H_2O$ was prepared according to the literature method of Strassberger & Wendlberger (1961). Crystals of (I) were obtained by carefully layering an ethanolic solution of $Co(NO_3)_2.6H_2O$ (14.6 mg in 14 ml) on an aqueous solution of $Na_3tcmel.3H_2O$ (15.1 mg in 4.0 ml) acidified with HBF_4 (8.9×10^{-3} M). After 12 h, pale orange crystals suitable for crystallographic analysis formed at the bottom of the aqueous layer.

Crystal data

$[Co(C_6H_2N_9)_2(H_2O)_4].6H_2O$
$M_r = 639.37$
Triclinic
$P\overline{1}$
$a = 6.663 (2)$ Å
$b = 6.861 (3)$ Å
$c = 15.721 (2)$ Å
$\alpha = 93.16 (3)°$
$\beta = 94.31 (2)°$
$\gamma = 111.13 (3)°$
$V = 665.8 (4)$ Å^3
$Z = 1$
$D_x = 1.595$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10.5–31.5°$
$\mu = 5.805$ mm^{-1}
$T = 293 (2)$ K
Parallelepiped
$0.5 \times 0.5 \times 0.2$ mm
Pale orange

Data collection

Enraf–Nonius CAD-4*F* diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical, indexed crystal faces
$T_{min} = 0.1312$, $T_{max} = 0.3927$
3474 measured reflections
2743 independent reflections

2728 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.020$
$\theta_{max} = 74.90°$
$h = -1 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -19 \rightarrow 19$
3 standard reflections frequency: 120 min intensity decay: insignificant

Refinement

Refinement on F^2
$R(F) = 0.0355$
$wR(F^2) = 0.1006$
$S = 1.084$
2743 reflections
232 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.1047P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.511$ e Å^{-3}
$\Delta\rho_{min} = -0.414$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0136 (14)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Co	0	0	0	0.03247 (15)
N1	0.1254 (2)	−0.1208 (2)	−0.40919 (9)	0.0291 (3)
N2	−0.2178 (2)	−0.3233 (2)	−0.46806 (9)	0.0318 (3)
N3	−0.1666 (2)	−0.1890 (2)	−0.32340 (9)	0.0305 (3)
N4	0.0532 (2)	−0.2706 (2)	−0.55340 (9)	0.0343 (3)
N5	0.4308 (3)	−0.1303 (3)	−0.58812 (11)	0.0448 (4)
N6	−0.5071 (2)	−0.4085 (2)	−0.39117 (10)	0.0348 (3)
N7	−0.6882 (3)	−0.4157 (4)	−0.25941 (13)	0.0595 (5)
N8	0.1924 (2)	0.0130 (2)	−0.27037 (9)	0.0335 (3)
N9	0.0941 (3)	0.0406 (3)	−0.12382 (10)	0.0410 (4)
C1	−0.0045 (2)	−0.2361 (2)	−0.47826 (10)	0.0276 (3)
C2	−0.2961 (3)	−0.3043 (3)	−0.39084 (10)	0.0285 (3)
C3	0.0444 (3)	−0.0998 (3)	−0.33228 (10)	0.0275 (3)
C4	0.2583 (3)	−0.1910 (3)	−0.56789 (10)	0.0321 (4)
C5	−0.5942 (3)	−0.4071 (3)	−0.31829 (13)	0.0386 (4)
C6	0.1288 (3)	0.0247 (3)	−0.19346 (11)	0.0322 (4)
O1	0.1928 (3)	0.3137 (2)	0.04206 (11)	0.0513 (4)
O2	0.2639 (3)	−0.0793 (3)	0.03380 (10)	0.0512 (4)
O3	0.3538 (2)	−0.0720 (3)	0.20893 (9)	0.0459 (3)
O4	−0.3540 (3)	0.3773 (3)	0.06728 (12)	0.0583 (4)
O5	0.1018 (4)	−0.4960 (3)	0.1794 (2)	0.0792 (6)

Table 2. *Selected geometric parameters (Å, °)*

Co—O2	2.062 (2)	N4—C1	1.305 (2)
Co—N9	2.092 (2)	N4—C4	1.319 (2)
Co—O1	2.107 (2)	N5—C4	1.149 (2)
N1—C1	1.351 (2)	N6—C5	1.324 (2)
N1—C3	1.382 (2)	N6—C2	1.327 (2)
N2—C1	1.355 (2)	N7—C5	1.148 (3)
N2—C2	1.375 (2)	N8—C6	1.319 (2)
N3—C2	1.331 (2)	N8—C3	1.319 (2)
N3—C3	1.337 (2)	N9—C6	1.143 (2)
O2—Co—N9^{i}	90.39 (7)	N4—C1—N1	127.07 (14)
O2—Co—N9	89.62 (7)	N4—C1—N2	116.91 (14)
O2—Co—O1	88.10 (8)	N1—C1—N2	116.01 (14)
N9—Co—O1	91.57 (7)	N6—C2—N3	124.72 (15)
O2—Co—O1^{i}	91.90 (8)	N6—C2—N2	113.98 (15)
N9—Co—O1^{i}	88.43 (7)	N3—C2—N2	121.30 (14)
C1—N1—C3	121.43 (14)	N8—C3—N3	124.33 (15)
C1—N2—C2	122.01 (14)	N8—C3—N1	114.14 (14)
C2—N3—C3	117.62 (14)	N3—C3—N1	121.53 (14)
C1—N4—C4	120.23 (14)	N5—C4—N4	173.7 (2)
C5—N6—C2	117.98 (15)	N7—C5—N6	173.6 (2)
C6—N8—C3	117.23 (14)	N9—C6—N8	173.4 (2)
C6—N9—Co	167.8 (2)		

Symmetry code: (i) $-x, -y, -z$.

The structure was solved using the *TREF* routine in *SHELXS*86 (Sheldrick, 1990). Refinement was performed using *SHELXL*93 (Sheldrick, 1993). All non-H atoms were refined with anisotropic displacement parameters. H atoms were refined isotropically. The water H atoms were refined with a common O—H separation. The tables were produced using *SHELXL*93.

The authors are grateful to the Australian Research Council for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(creatinine)platinum(II) Dithiocyanate

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Abstract

The structure of the title compound, tetrakis(2-amino-1,5-dihydro-1-methyl-4*H*-imidazol-4-one-N^3)platinum(II) dithiocyanate, $[Pt(C_4H_7N_3O)_4](SCN)_2$, consists of $[Pt(creat)_4]^{2+}$ (creat is 2-imino-1-methyl-4-imidazolidinone) complex cations possessing approximate D_2 symmetry and thiocyanate anions. The Pt atom is coordinated in a square-planar manner by four creatinine ligands bonded through their endocyclic N atoms. The Pt—N bond distances range from 1.980 (6) to 2.034 (7) Å. The creatinine molecules are planar and participate in inter- and intramolecular hydrogen bonds. The ligands are almost perpendicular to the equatorial PtN_4 plane.

Comment

Recently, a number of structural papers on different Pt^{II}–creatinine complexes have appeared (Mitewa, Gencheva, Bontchev & Macíček, 1988; Macíček, Angelova, Gencheva, Mitewa & Bontchev, 1988; Beurskens, Perales, Martin-Gil & Martin-Gil, 1988; Beja, Paixao, Martin-Gil & Salgado, 1991; Gencheva *et al.*, 1992). It was found that changing the reaction conditions led to the formation of different complex species, some of them being paramagnetic metal–metal-bonded and creatinine-bridged oligomers involv-