

{Tris[2-(1*H*-imidazol-2-ylmethyleneamino- κ^2 N,N')-ethyl]amine}cobalt(II) bis(perchlorate) hemihydrate**Hong-Shan He**

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.013$ Å
 H-atom completeness 96%
 R factor = 0.077
 wR factor = 0.195
 Data-to-parameter ratio = 12.7

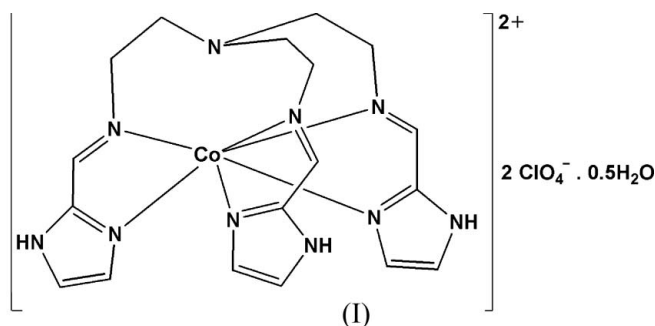
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $[\text{Co}(\text{C}_{18}\text{H}_{23}\text{N}_{10})_2](\text{ClO}_4)_2 \cdot 0.5\text{H}_2\text{O}$, the Co^{II} ion is coordinated by six N atoms from the tripodal organic ligand in a distorted square-bipyramidal geometry. There are two cations, four anions and one water molecule in the asymmetric unit.

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Comment

Previously, we have reported the crystal structure of the Mg^{II} complexes of tris(2-(4-imidazolylmethyleneaminoethyl)amine) (He *et al.*, 2004). The results showed that the tripodal ligand chelates the metal ion, forming stable complexes with coordination by up to six N atoms. We now present the crystal structure of the title compound, (I), a Co^{II} complex of the same amine.



Complex (I) crystallizes in the monoclinic system with two independent cations, four anions and one water molecule in the asymmetric unit (Fig. 1). For the Co1 cation, the Co^{II} ion is coordinated by three imine and three imidazolyl N atoms. The geometry is distorted square-bipyramidal, with atoms N2, N6, N8 and N9 in equatorial positions, and N3 and N5 in axial positions. The tripodal bridging amine N atom is situated 3.332 (7) Å from the metal center, indicating a weak non-bridging interaction. The bite angles are given in Table 1. The bite distances, $\text{N}\cdots\text{N}$, are 2.484 (10), 2.525 (9) and 2.521 (10) Å, for $\text{N}2\cdots\text{N}3$, $\text{N}5\cdots\text{N}6$ and $\text{N}8\cdots\text{N}9$, respectively.

The coordination environment around Co2 is almost identical to that of Co1, except for slight variations in the bond distances and angles.

Experimental

The title compound was prepared according to the literature method of He *et al.* (2004). An acetonitrile solution of the compound was kept at room temperature and the solvent evaporated slowly. Crystals of

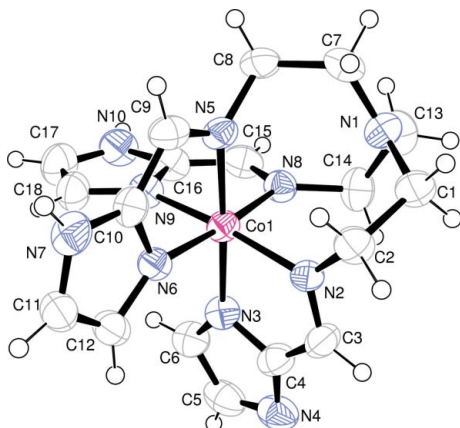


Figure 1
View of the Co^I cation of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

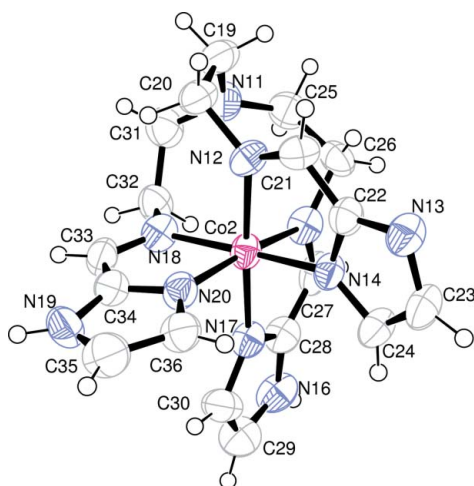


Figure 2
View of the Co^{II} cation of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(I) suitable for X-ray diffraction analysis were obtained one month later.

Crystal data

[Co(C₁₈H₂₃N₁₀)₂](ClO₄)₂·0.5H₂O
M_r = 645.30
 Monoclinic, *P*₂₁/*n*
a = 9.1219 (18) Å
b = 19.005 (4) Å
c = 28.251 (6) Å
 β = 95.28 (3)°

V = 4876.9 (17) Å³
Z = 8
 Mo Kα radiation
 μ = 0.99 mm⁻¹
T = 293 (2) K
 0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
*T*_{min} = 0.826, *T*_{max} = 0.826

37068 measured reflections
 9021 independent reflections
 4571 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.100

Refinement

R[*F*² > 2σ(*F*²)] = 0.077
wR(*F*²) = 0.195
S = 1.05
 9021 reflections

713 parameters
 H-atom parameters constrained
 Δρ_{max} = 0.69 e Å⁻³
 Δρ_{min} = -0.82 e Å⁻³

Table 1

Selected bond angles (°).

N3—Co1—N2	82.2 (3)	N12—Co2—N14	82.8 (3)
N9—Co1—N8	83.0 (3)	N15—Co2—N17	82.7 (3)
N6—Co1—N5	83.2 (3)	N18—Co2—N20	82.5 (3)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N7—H7...N4 ⁱ	0.86	1.92	2.663 (11)	144
N10—H10...O1 ⁱⁱ	0.86	2.06	2.816 (10)	146
N10—H10...O42 ⁱⁱ	0.86	2.38	2.985 (11)	128
N16—H16...N13 ⁱ	0.86	1.91	2.654 (11)	144
N19—H19...O33 ⁱⁱⁱ	0.86	2.46	3.035 (11)	125
N19—H19...O1	0.86	2.09	2.873 (11)	151

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

H atoms were positioned geometrically and refined in riding mode, with C—H = 0.96 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H, C—H = 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) for aromatic H, and N—H = 0.86 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) for amine H. The two H atoms on O1 could not be located in a difference map.

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).

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