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Bis[1-hydroxy-4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-4,5-dihydro-1*H*-imidazole]-nitratocobalt(II) nitrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
wR factor = 0.078
Data-to-parameter ratio = 18.1

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

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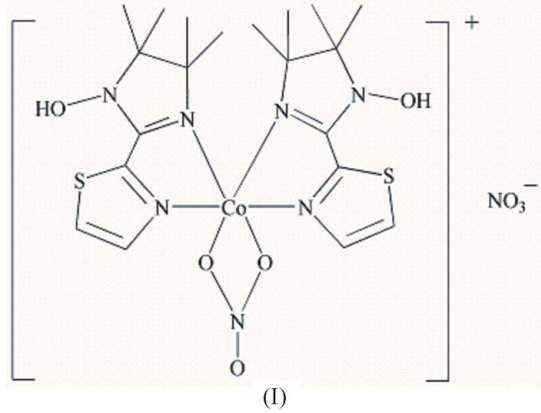
The title crystal structure, $[Co(NO_3)(C_{10}H_{15}N_3OS)_2]NO_3$, consists of Co^{II} complex cations and NO_3^- anions, located on different twofold axes and linked to each other *via* O—H···O hydrogen bonding. The Co^{II} complex assumes a distorted octahedral coordination geometry, formed by a chelating nitrate and two ImTh molecules (ImTh is 1-hydroxy-4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-4,5-dihydro-1*H*-imidazole). Within the ImTh molecule, the N—O bond distance of 1.386 (2) Å is much longer than the N—O bonds found in related complexes with the nitroxide radical.

Comment

Nitroxide radicals have attracted much attention in the past decade because of their possible magnetic properties (Fettouhi *et al.*, 2003; Fegy *et al.*, 1998; Wang *et al.*, 2003; Luneau *et al.*, 1998). In the process of preparing the Co^{II} complex with an imine nitroxide radical, we obtained the title complex, (I), in which the nitroxide radical was reduced due to an acidic impurity. Similar reduction phenomena have been reported previously (Jiang *et al.*, 1998; Li *et al.*, 2001). We present here the crystal structure of (I).



(I)

The crystal structure of (I) consists of Co^{II} complex cations and uncoordinated NO_3^- anions (Fig. 1). The Co^{II} complex lies on a twofold axis and displays a distorted octahedral coordination geometry, formed by a chelating nitrate anion and two chelating ImTh molecules (ImTh is 1-hydroxy-4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-4,5-dihydro-1*H*-imidazole). The N3—O1 bond distance (Table 1) is much longer than those (ranging from 1.25 to 1.31 Å) found in corresponding complexes incorporating nitroxide radicals (Awaga *et al.*, 1992; Cogne *et al.*, 2000).

The uncoordinated NO_3^- anion, lying on another twofold axis, links to the Co^{II} complex cation *via* O—H···O hydrogen bonding (Table 2).

Experimental

ImThO was prepared according to the literature method (Ullman *et al.*, 1970). ImThO (0.11 g, 0.5 mmol) was added to an ethanol solution (15 ml) of $Co(NO_3)_2{\cdot}6H_2O$ (0.073 g, 0.25 mmol). The mixture was stirred for 2 h at room temperature and then filtered. The filtrate was kept in an atmosphere of diethyl ether vapor at room temperature. Single crystals of (I) were obtained after 10 d.

Crystal data

$[Co(NO_3)(C_{10}H_{15}N_3OS)_2]NO_3$
$M_r = 633.59$
Orthorhombic, $P2_12_12$
$a = 13.1701$ (8) Å
$b = 10.4625$ (6) Å
$c = 10.6961$ (7) Å
$V = 1473.84$ (16) Å^3
$Z = 2$
$D_x = 1.428$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5723 reflections
$\theta = 2.5$–$25.9°$
$\mu = 0.78$ mm^{-1}
$T = 295$ (2) K
Platelet, red
0.31 × 0.23 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.797$, $T_{max} = 0.925$
13046 measured reflections
3326 independent reflections
2897 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$
$h = -16 \rightarrow 17$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.078$
$S = 1.04$
3326 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.1802P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack (1983), 1385 Friedel Pairs
Flack parameter: 0.019 (15)

Table 1
Selected geometric parameters (Å, °).

Co1—N1	2.1461 (18)	Co1—O3	2.1894 (19)
Co1—N2	2.0676 (18)	O1—N3	1.386 (2)
N1—Co1—N1^{i}	179.30 (11)	N2^{i}—Co1—N2	104.94 (10)
N1—Co1—N2	79.21 (7)	N2^{i}—Co1—O3	98.48 (8)
N1—Co1—N2^{i}	100.35 (7)	N2—Co1—O3	156.26 (7)
N1—Co1—O3	92.95 (8)	O3—Co1—O3^{i}	58.53 (11)
N1—Co1—O3^{i}	87.67 (8)		

Symmetry code: (i) $-x+1, -y, z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1'···O5^{ii}	0.82	1.85	2.6227 (19)	158

Symmetry code: (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+1$.

Methyl H and hydroxy H atoms were placed in calculated positions, with C—H = 0.96 and O—H = 0.82 Å, and refined with free torsion angles to fit the electron density; $U_{iso}(H) = 1.5U_{eq}$(carrier). Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined in the riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$.

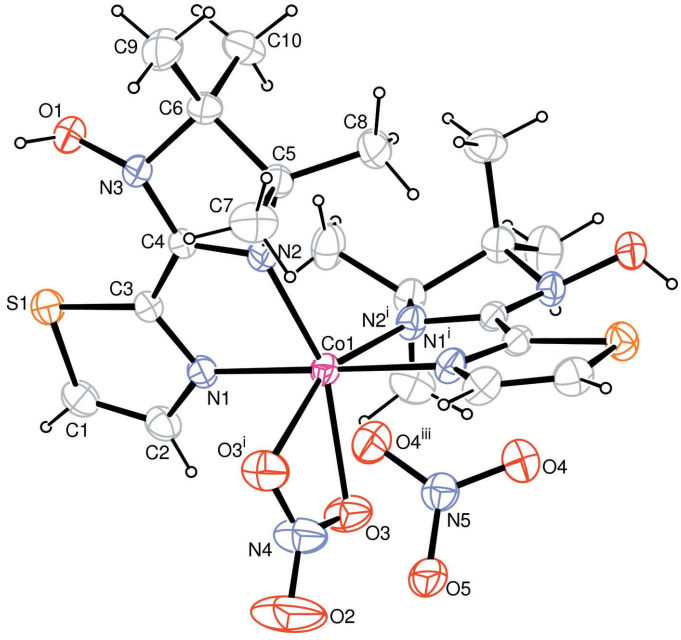


Figure 1
The molecular structure of (I), with 25% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry codes: (i) $-x+1, -y, z$; (iii) $-x+1, -y+1, z$].

Data collection: *SMART* (Bruker 2002); cell refinement: *SAINT* (Bruker 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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