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trans-Dinitro(1,4,8,11-tetraazacyclotetradecane-*N*,*N*′,*N*″,*N*‴)cobalt(III) perchlorate

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Key indicators

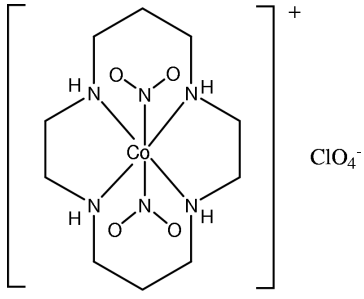
Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.035
wR factor = 0.108
Data-to-parameter ratio = 9.4

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

In the title compound, *trans*-$[Co(NO_2)_2(cyclam)]ClO_4$, (I), where cyclam is 1,4,8,11-tetraazacyclotetradecane ($C_{10}H_{24}N_4$), the Co^{III} complex has a distorted octahedral coordination. The O atoms of each nitro ligand are disordered over two sites with 65:35% occupancy.

Comment

In the title compound, (I), the Co—NO_2 bond distances are 1.962 (5) and 1.968 (5) Å. The Co—N bond distances of the cyclam are 1.974 (5)–1.986 (4) Å.



(I)

Experimental

The title compound, (I), was obtained as a by-product in the synthesis of *trans*-$[Co(cyclam)(NCS)(NO_2)]ClO_4$ from the reaction of *trans*-$[Co(cyclam)(NCS)Cl]ClO_4$ (0.5 mmol) with $NaNO_2$ (1.5 mmol) in 30 ml 0.03 M HNO_3. Crystals of (I) were grown from a dimethyl sulfoxide solution by a diffusion of diethyl ether vapour.

Crystal data

$[Co(NO_2)_2(C_{10}H_{24}N_4)]ClO_4$
M_r = 450.72
Orthorhombic, $P2_12_12_1$
a = 13.299 (3) Å
b = 19.820 (1) Å
c = 6.6531 (7) Å
V = 1753.7 (5) Å^3
Z = 4
D_x = 1.707 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.3–13.9°
μ = 1.19 mm^{-1}
T = 297 (1) K
Plate, orange
0.40 × 0.25 × 0.05 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
T_{min} = 0.752, T_{max} = 0.942
3312 measured reflections
2554 independent reflections
2245 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°
$h = -17 \rightarrow 8$
$k = 0 \rightarrow 25$
$l = -4 \rightarrow 8$
3 standard reflections
every 150 reflections
intensity decay: none

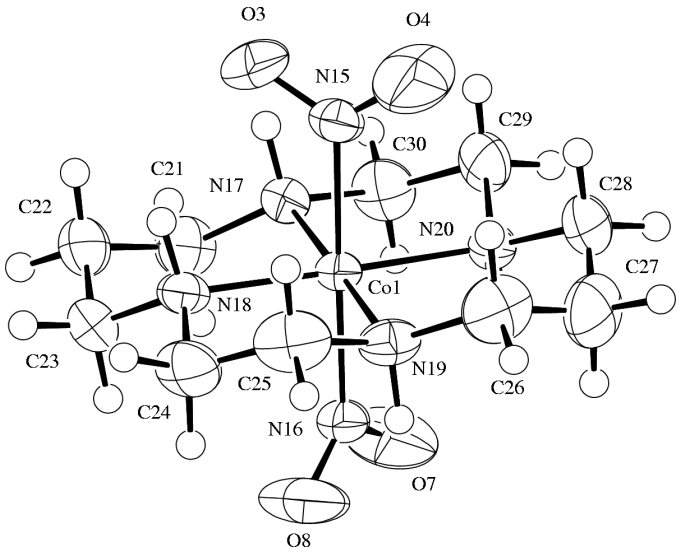


Figure 1
The structure of the complex cation in (I). Displacement ellipsoids are plotted at the 50% probability level. The disordered nitro O atoms with 35% occupancy have been omitted for clarity.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0410P)^2 + 3.4475P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R(F) = 0.035$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.108$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.55$ e Å^{-3}
2554 reflections	Absolute structure: (Flack, 1983), 225 Friedel pairs
271 parameters	Flack parameter = −0.05 (4)
H-atom parameters constrained	

X-ray intensities were measured for $\pm h,+k,\pm l$ ($\theta < 15^\circ$) and $-h,+k,+l$ ($15 < \theta < 27.5^\circ$). The O atoms of the nitro ligands show positional disorder (atoms O3–O10), suggesting that there are two possible orientations with 65:35% occupancy for each NO_2 group. All H-atom positional parameters were calculated geometrically and fixed with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *WinAFC* (Rigaku Corporation, 1999); cell refinement: *WinAFC*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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