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***trans*-Chlorobis(ethylenediamine-*N,N'*)nitrocobalt(III) nitrate**

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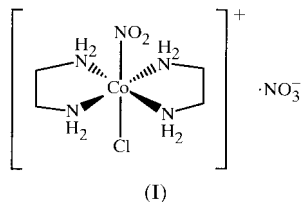
Data validation number: IUC0000311

In the title compound, *trans*-[CoCl(NO₂)(C₂H₈N₂)₂]⁺NO₃⁻, a distorted octahedral Co^{III} complex shows an orientational disorder such that the positions of the nitro and chloro ligands are exchanged. The occupation factors of the major and minor orientations are 84 and 16%, respectively. The O atoms of the nitrate ion are disordered over two sites.

Comment

Certain nitrocobalt(III) complexes show nitro-to-nitrito linkage isomerization in the solid state by irradiation of visible light (Adell, 1971; Grenthe & Nordin, 1979; Masciocchi *et al.*, 1994). Although the title crystal, (I), is photostable, the structure was determined as part of a study on photoisomerization.

In (I), the complex cation has two possible orientations. Furthermore, the O atoms of nitrate ion are disordered over two sites. A similar disorder of NO₃⁻ was observed in crystals



of *trans*-[Co(en)₂Cl₂]⁺NO₃⁻ (en is ethylenediamine; Kita *et al.*, 1994), but not in *trans*-[Co(en)₂(NO₂)₂]⁺NO₃⁻ (Bortin, 1976).

The crystals of *trans*-[Co(en)₂Cl(NO₂)]CF₃SO₃ were also prepared in the present study to indicate that there is an orientational disorder of the complex cation: monoclinic, space group *P*2₁/*c*, *a* = 6.638 (4), *b* = 17.222 (4), *c* = 12.725 (3) Å, β = 101.64 (3)°, *V* = 1424.8 (8) Å³ and *Z* = 4 at 300 K.

Experimental

The title compound was prepared by a literature method (Adell, 1971). Crystals of (I) were grown from a hot aqueous solution by slow cooling.

Crystal data

[CoCl(NO₂)(C₂H₈N₂)₂]⁺NO₃⁻
M_r = 322.59
 Monoclinic, *P*2₁/*c*
a = 6.601 (2) Å
b = 12.721 (2) Å
c = 14.072 (1) Å
 β = 97.06 (1)°
V = 1172.7 (3) Å³
Z = 4

D_x = 1.827 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 13.9–14.9°
 μ = 1.714 mm⁻¹
T = 298 (1) K
 Prismatic, orange-red
 0.5 × 0.5 × 0.2 mm

Data collection

Rigaku AFC-7R diffractometer
 θ–2θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
T_{min} = 0.605, *T_{max}* = 0.710
 2930 measured reflections
 2703 independent reflections
 2494 reflections with *I* > 2σ(*I*)

R_{int} = 0.006
θ_{max} = 27.5°
h = 0 → 9
k = 0 → 17
l = –18 → 18
 3 standard reflections
 every 150 reflections
 intensity decay: none

Refinement

Refinement on *F*²
R(*F*) = 0.032
wR(*F*²) = 0.082
S = 1.10
 2703 reflections
 205 parameters
 H-atom parameters not refined

w = 1/[σ²(*F_o*²) + (0.0406*P*)² + 1.0531*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.75 e Å⁻³
 Δρ_{min} = –0.67 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Co1–Cl2	2.2801 (8)	Co1–N20	1.961 (2)
Co1–N17	1.888 (4)	Co1–N21	1.951 (2)
Co1–N18	1.955 (2)	O8–N17	1.235 (5)
Co1–N19	1.952 (2)	O9–N17	1.240 (4)
Cl2–Co1–N17	179.3 (1)	O8–N17–O9	120.6 (4)

A split-site model was applicable for the positional disorder of the nitro and chloro ligands. The site-occupation factor of the major orientation (atoms N17, O8, O9, and Cl2) was fixed to 84% after some refinements. In the minor orientation, nitro-O atoms are further disordered over two sites with 8% probability each (atoms O4–O7). The Co1–N16 bond distance was restrained to be 1.90 Å (s.u. 0.01 Å) and the nitro N16–O distances to be 1.24 Å (s.u. 0.01 Å). The minor Cl3, O4–O7 and N16 atoms were refined isotropically. All H-atom positional parameters were calculated geometrically and fixed with *U_{iso}*(H) = 1.2*U_{eq}*(parent atom). The nitrate O atoms also show positional disorder, suggesting that there are two possible orientations of the ion with 50% probability each.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

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