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trans-Chlorobis(ethylenediamine-*N*,*N*′)nitrocobalt(III) perchlorate

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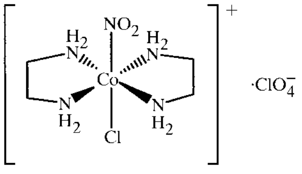
In the title compound, *trans*-$[CoCl(NO_2)(C_2H_8N_2)_2]ClO_4$, there are two independent Co^{III} complexes with a distorted octahedral coordination, and they show an orientational disorder such that the positions of the nitro and chloro ligands are exchanged. As a result, the averaged structure has inversion centres at the Co atoms. The perchlorate-O atoms 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). Although the title crystal, (I), is photostable, the structure was determined as part of a study on photoisomerization.



(I)

The crystals of *trans*-$[Co(en)_2Cl(NO_2)]PF_6$ (en is ethylenediamine) were also prepared in the present study to indicate that the structure is isomorphous with (I); triclinic, space group $P\overline{1}$, with $a = 8.745$ (1), $b = 12.791$ (1), $c = 6.489$ (1) Å, $\alpha = 101.18$ (1), $\beta = 103.36$ (1), $\gamma = 79.44$ (1)°, $V = 685.4$ (1) Å^3 and $Z = 2$ at 297 K.

Experimental

The title compound was prepared from the nitrate salt (Adell, 1971) by anion exchange. Crystals of (I) were grown from an aqueous solution.

Crystal data

$[CoCl(NO_2)(C_2H_8N_2)_2]ClO_4$
$M_r = 360.04$
Triclinic, $P\overline{1}$
$a = 8.349$ (2) Å
$b = 12.644$ (3) Å
$c = 6.556$ (3) Å
$\alpha = 103.19$ (3)°
$\beta = 105.11$ (3)°
$\gamma = 77.30$ (2)°
$V = 641.0$ (4) Å^3
$Z = 2$
$D_x = 1.865$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.6$–15.0°
$\mu = 1.783$ mm^{-1}
$T = 298$ (1) K
Prismatic, orange–red
0.5 × 0.4 × 0.3 mm

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.517$, $T_{max} = 0.586$
4041 measured reflections
3734 independent reflections
3409 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 30.0°$
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 17$
$l = 0 \rightarrow 9$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.042$
$wR(F^2) = 0.112$
$S = 1.18$
3734 reflections
264 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0430P)^2 + 0.6970P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

Co1—Cl3	2.277 (4)	Co2—Cl4	2.297 (7)
Co1—N20	1.91 (2)	Co2—N23	1.947 (8)
Co1—N21	1.956 (2)	Co2—N24	1.956 (2)
Co1—N22	1.952 (2)	Co2—N25	1.956 (2)

The Co1 and Co2 atoms lie on centres of symmetry. A split-site model was applicable for the positional disorder of the nitro and chloro ligands. In the complex involving the Co1 atom, the nitro–O atoms are further disordered over two sites, the O6/O7 and O8/O9 atoms, with 35 and 15% probabilities, respectively. The minor O8 and O9 atoms were refined isotropically. The Co1—N20 bond distance was restrained to be 1.95 Å (s.u. = 0.001 Å) and the nitro N20—O distances to be 1.24 Å (s.u. = 0.001 Å). There is a positional disorder of the ethylenediamine C atoms, which were split into two sites with 50% probability each (atoms C26—C29). In the complex involving the Co2 atom, a large anisotropic displacement parameter of Cl4 suggested positional disorder. However, the split-site model of the Cl position was not applicable. All H-atom positional parameters were calculated geometrically and fixed with $U_{iso}(H) = 1.2U_{eq}$(parent atom). The perchlorate O atoms have positional disorder (atoms O12–O19), suggesting that the perchlorate ion has two possible orientations with 50% probability each.

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); 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); software used to prepare material for publication: *TEXSAN*.

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