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Pentaamminenitrosylchromium(III) dichloride

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(O–N) = 0.008 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.117
Data-to-parameter ratio = 17.7

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

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The cation in the title compound, $[Cr(NO)(NH_3)_5]Cl_2$, is distorted octahedral with a linear Cr—N—O moiety. The short Cr—N(nitrosyl) distance of 1.692 (7) Å indicates significant multiple bonding between these atoms. The Cr atom, the NO group and the N atom *trans* to NO have *m*2*m* symmetry and the remaining N and Cl atoms have *m* symmetry.

Comment

This report is the first example of an X-ray structural study of a pentaamminenitrosylchromium complex, $[Cr(NO)(NH_3)_5]^{2+}$, even though chromium complexes with Cl, Br, NO_3, ClO_4, $B(C_6H_5)_4$ and $OC_6H_2(NO_2)_3$ ligands have been characterized (Mori *et al.*, 1963; Griffith, 1963; Kobayashi *et al.*, 1969). The structure of the related compound $[Cr(NO)(H_2O)_5]SO_4$ (Ardon & Cohen, 1993) has been reported. The results of *ab initio* calculations on $[Cr(NO)(H_2O)_5]^{2+}$ have also been reported (Shim *et al.*, 1995).

The title compound, $[Cr(NO)(NH_3)_5]Cl_2$ (Fig. 1 and Table 1), (I), was found to crystallize in the orthorhombic space group *Cmcm* with the Cr atom, the NO group and the N2 atom, *trans* to NO, having *m*2*m* symmetry imposing disorder in the $N2H_3$ ammine group. The remaining N atoms and the Cl atom are at sites with *m* symmetry.

The Cr^{III} centre exists in a slightly distorted octahedral geometry with a strictly linear Cr—N—O arrangement (from symmetry). The N(nitrosyl)—Cr—N(ammonia, *cis* to NO) angles are 94.2 (1) and 92.8 (1)° so that the Cr—N(ammonia, *cis* to NO) bonds are slightly bent towards the ammonia molecule in the *trans* position to NO.

The short Cr—N(nitrosyl) distance of 1.692 (7) Å indicates the presence of multiple bonding between these atoms. A short Cr—N(nitrosyl) distance of 1.682 (2) Å was also reported for $[Cr(NO)(H_2O)_5]^{2+}$ (Ardon & Cohen, 1993). The Cr—N(ammonia) distances in $[Cr(NH_3)_6]^{3+}$ are 2.070 (8) Å (Clegg, 1982) and 2.085 (10) Å (average; Dieterich & Stráhle, 1993). These are close to those of Cr—N(ammonia, *cis* to NO) in $[Cr(NO)(NH_3)_5]^{2+}$.

The O(nitrosyl) atom appears to form hydrogen bonds with both H3 and H4 of adjacent ammonia molecules, with distances and angles as shown in Table 2.

Experimental

The preparation of $[Cr(NO)(NH_3)_5]Cl_2$ was carried out according to the literature method (Mori *et al.*, 1963). Crystals of $[Cr(NO)(NH_3)_5]Cl_2$ were obtained by recrystallization from aqueous hydrochloric acid.

Crystal data

$[Cr(NO)(NH_3)_5]Cl_2$
$M_r = 238.06$
Orthorhombic, *Cmcm*
$a = 10.0236$ (9) Å
$b = 9.098$ (3) Å
$c = 10.357$ (1) Å
$V = 944.5$ (5) Å^3
$Z = 4$
$D_x = 1.674$ Mg m^{-3}
$D_m = 1.62$ Mg m^{-3}
D_m measured by flotation
Mo $K\alpha$ radiation
Cell parameters from 1131 reflections
$\theta = 3.0$–27.5°
$\mu = 1.73$ mm^{-1}
$T = 273$ K
Prism, orange
0.20 × 0.10 × 0.10 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (*TEXSAN*; Molecular Structure Corporation & Rigaku Corporation, 1999)
$T_{min} = 0.587$, $T_{max} = 0.707$
5457 measured reflections
601 independent reflections
389 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.084$
$\theta_{max} = 27.2^\circ$
$h = -11 \rightarrow 12$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.117$
$S = 0.83$
601 reflections
34 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.034P^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—N1	1.697 (7)	Cr1—N4	2.087 (4)
Cr1—N2	2.126 (7)	Cr1—N4^{ii}	2.087 (4)
Cr1—N3	2.085 (4)	O1—N1	1.179 (8)
Cr1—N3^{i}	2.085 (4)		
N1—Cr1—N2	180.0	N3—Cr1—N3^{i}	171.4 (3)
N1—Cr1—N3	94.3 (1)	N3—Cr1—N4	89.79 (1)
N1—Cr1—N4	92.9 (1)	N4—Cr1—N4^{ii}	174.3 (3)
N2—Cr1—N3	85.7 (1)	Cr1—N1—O1	180.0
N2—Cr1—N4	87.1 (1)		

Symmetry codes: (i) $1-x, y, z$; (ii) $x, y, \frac{3}{2}-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1^{i}	0.95	2.75	3.302 (6)	118
N3^{ii}—H3^{ii}···O1^{iii}	0.95	2.75	3.302 (6)	118
N4—H6···O1^{iv}	0.95	2.89	3.211 (5)	101
N4—H6^{ii}···O1^{iv}	0.95	2.89	3.211 (5)	101
N4^{v}—H6^{v}···O1^{vi}	0.95	2.89	3.211 (5)	101
N4^{v}—H6^{vii}···O1^{vi}	0.95	2.89	3.211 (5)	101

Symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, z$; (ii) $1-x, y, z$; (iii) $\frac{1}{2}+x, y-\frac{1}{2}, z$; (iv) $x, 1-y, \frac{1}{2}+z$; (v) $x, y, \frac{3}{2}-z$; (vi) $x, 1-y, 1-z$; (vii) $1-x, y, \frac{3}{2}-z$.

All the H atoms were fixed in the refinement with isotropic displacement parameters set at 1.2 times the value of the equivalent isotropic displacement parameter of their carrier atom. Since the N2 atom is at a site with *m*2*m* symmetry, the six disordered H atoms of the ammonia molecule have been developed by mirror symmetry.

Data collection: *CRYSTALCLEAR* (Rigaku, 1996); cell refinement: *CRYSTALCLEAR*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

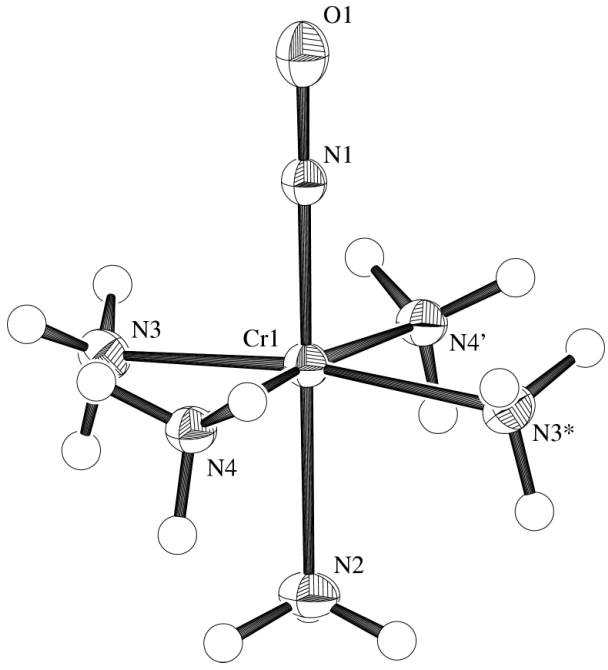

Figure 1
The structure of the $[Cr(NO)(NH_3)_5]^{2+}$ cation. Displacement ellipsoids are plotted at the 50% probability level (Johnson, 1976). For clarity, only three out of the six disordered H atoms of the ammonia molecule *trans* to NO developed by mirror symmetry are shown.

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References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Ardon, M. & Cohen, S. (1993). *Inorg. Chem.* **32**, 3241–3243.
Clegg, W. (1982). *J. Chem. Soc. Dalton Trans.* pp. 593–595.
Dieterich, S. & Stráhle, J. (1993). *Z. Naturforsch. Teil B*, **48**, 1574–1580.
Griffith, P. W. (1963). *J. Chem. Soc.* pp. 3286–3291.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kobayashi, H., Tujikawa, I., Mori, M. & Yamamoto, Y. (1969). *Bull. Chem. Soc. Jpn*, **42**, 709–715.
Molecular Structure Corporation & Rigaku Corporation (1999). *TEXSAN*. Version 1.11. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Corporation, Akishima, Tokyo, Japan.
Mori, M., Ueshiba, S. & Kawaguchi, S. (1963). *Bull. Chem. Soc. Jpn*, **36**, 796–798.
Rigaku (1996). *CRYSTALCLEAR*. Rigaku Corporation, Akishima-shi, Tokyo, Japan.
Shim, I., Gingerich, A. K., Mandix, K. & Feng, X. (1995). *Inorg. Chim Acta*, **229**, 455–460.