

## SHORT-FORMAT PAPERS

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*Acta Cryst.* (1987), C43, 995–996

**cis-Tetraammineaquachlorochromium(III) Chloride**

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**Abstract.** [CrCl(H<sub>2</sub>O)(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub>,  $M_r = 244.47$ , orthorhombic, *Pnma*,  $a = 13.205$  (2),  $b = 10.506$  (2),  $c = 6.760$  (1) Å,  $V = 937.9$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.731$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 20.0$  cm<sup>-1</sup>,  $F(000) = 500$ ,  $T = 293$  K,  $R_F = 2.86\%$  for 1295 observed reflections. The title complex crystallizes with imposed mirror-plane symmetry; the cation has regular octahedral geometry [the maximum deviation from octahedral angles is 3.0 (2)°] and is strongly associated with the chloride anions through the coordinated H<sub>2</sub>O molecule, Cl(2)···O(1) = 3.112 (2), Cl(2)···H(O1) = 2.17 (1) Å.

**Experimental.** Synthesized by published procedure (Mori, 1953) and recrystallized from dilute aqueous HCl. Deep-red, 0.24 × 0.26 × 0.38 mm, epoxied to glass fiber. Nicolet R3m/μ, graphite monochromator,

unit cell from the angular settings of 25 reflections ( $27 \leq 2\theta \leq 32^\circ$ ). 1752 reflections,  $4 \leq 2\theta \leq 62^\circ$ , collected for  $-19 \leq h \leq 0$ ,  $0 \leq k \leq 16$ ,  $0 \leq l \leq 10$  at  $4^\circ \text{ min}^{-1}$  using  $\theta/2\theta$  scans. Three standard reflections varied less than 1%, measured every 97 reflections, no absorption correction (transmission 0.91 to 1.00). 1399 unique reflections, 170 rejected with  $F_o \leq 2.5\sigma(F_o)$ . Systematic absences (0kl,  $k+l$  odd; hk0,  $h$  odd) showed either *Pnma* or *Pn2<sub>1</sub>a*. *E* statistics suggested centrosymmetric, judged correct from sensible solution and refinement. Patterson solution, blocked-cascade least squares on  $F$ ,  $w^{-1} = \sigma^2(F) + 0.001F$ , complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). All H atoms located and isotropically refined,  $U$  fixed at 0.08 Å<sup>2</sup>; all non-H atoms anisotropically refined, 72 parameters.  $R_F = 2.86\%$ ,  $wR_F = 3.46\%$ , slope of normal probability plot = 0.918, max. final  $\Delta/\sigma = 0.035$ , max.  $\Delta\rho = 0.40$  e Å<sup>-3</sup> [0.89 Å Cl(1)], min.  $\Delta\rho = -0.35$  e Å<sup>-3</sup>. *SHELXTL* program package (Sheldrick,

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Table 1. Atomic coordinates ( $\times 10^4$ ) and isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>)

	x	y	z	$U_{eq}/U_{iso}$
Cr	961 (1)	7500	6755 (1)	20 (1)*
Cl(1)	-403 (1)	7500	4611 (1)	29 (1)*
Cl(2)	3509 (1)	163 (1)	6730 (1)	33 (1)*
O(1)	1937 (2)	7500	4421 (3)	34 (1)*
H(O1)	1866 (26)	8236 (18)	3618 (37)	80
N(1)	987 (1)	9469 (1)	6806 (2)	29 (1)*
N(2)	2216 (2)	7500	8551 (4)	34 (1)*
N(3)	6 (2)	7500	9177 (4)	34 (1)*
H(2a)	2863 (14)	7500	7960 (47)	80
H(2b)	2126 (19)	6848 (20)	9488 (32)	80
H(3a)	404 (25)	7500	10365 (37)	80
H(3b)	-383 (19)	6732 (18)	9115 (37)	80
H(1a)	320 (9)	9802 (25)	7068 (34)	80
H(1b)	1208 (17)	9756 (23)	5530 (18)	80
H(1c)	1446 (14)	9773 (26)	7799 (25)	80

\* Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

Table 2. Bond distances (Å) and angles (°) for *cis*-[CrCl(H<sub>2</sub>O)(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub>

Cr–Cl(1)	2.313 (1)	Cr–O(1)	2.037 (2)
Cr–N(1)	2.069 (2)	Cr–N(2)	2.054 (2)
Cr–N(3)	2.067 (2)	Cr–N(1a)	2.069 (2)
O(1)–H(O1)	0.949 (22)	N–H (av.)	0.95 (1)
Cl(1)–Cr–O(1)	90.4 (1)	Cl(1)–Cr–N(1)	91.3 (1)
O(1)–Cr–N(1)	90.1 (1)	Cl(1)–Cr–N(2)	177.4 (1)
O(1)–Cr–N(2)	87.0 (1)	N(1)–Cr–N(2)	88.7 (1)
Cl(1)–Cr–N(3)	91.2 (1)	O(1)–Cr–N(3)	178.4 (1)
N(1)–Cr–N(3)	89.8 (1)	N(2)–Cr–N(3)	91.4 (1)
Cl(1)–Cr–N(1a)	91.3 (1)	O(1)–Cr–N(1a)	90.1 (1)
N(1)–Cr–N(1a)	177.3 (1)	N(2)–Cr–N(1a)	88.7 (1)
N(3)–Cr–N(1a)	89.8 (1)	Cr–N–H (av.)	110 (1)
H–N–H (av.)	110 (1)	Cr–N–H (range)	107 (1)–119 (1)
H–N–H (range)	109 (1)–114 (1)	Cr–O(1)–H(O1)	112 (2)

Symmetry-generated atoms: x, 1–5–y, z.

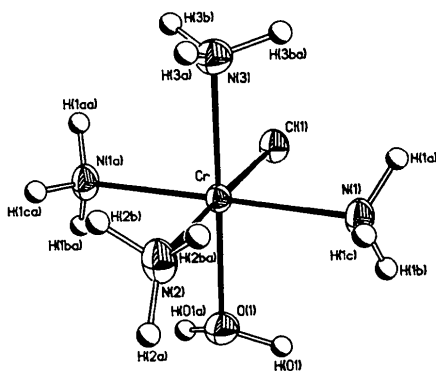


Fig. 1. Cation structure and labeling scheme for  $[\text{CrCl}(\text{H}_2\text{O})(\text{NH}_3)_4]\text{Cl}_2$ . Symmetry-generated species derived by the transformation  $x, 1.5-y, z$ .

1986).<sup>\*</sup> Atomic parameters are given in Table 1, bond distances and angles in Table 2. The cation structure is in Fig. 1 and the unit-cell packing is shown in Fig. 2.

**Related literature.** Methods for the synthesis of the title complex and closely related complexes have been systematically characterized (Kyuno, Kamada & Tanaka, 1967). Solvent interchange (Jackson, Vowles & Fee, 1977) and luminescence (Forster, Rund & Fucaloro, 1984) have been studied. Two reviews are relevant; one on the aqueous chemistry of  $\text{Cr}^{\text{III}}$  complexes (Earley & Cannon, 1965) and the other devoted to ammine complexes of  $\text{Cr}^{\text{III}}$  (Garner & House, 1970). Very few simple  $\text{Cr}^{\text{III}}$  coordination complexes have been crystallographically charac-

<sup>\*</sup> Lists of anisotropic thermal parameters, observed and calculated structure factors and bond distances and angles involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43649 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Low-Temperature Structure of 3,4;3',4'-Bis(ethylenedithio)-2,2',5,5'-tetrathiafulvalene Diiodaurate(I) (2:1), $\beta$ -(BEDT-TTF)<sub>2</sub>AuI<sub>2</sub>

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**Abstract.**  $[\text{C}_{10}\text{H}_8\text{S}_8]_2[\text{AuI}_2]$ ,  $M_r = 1220.1$ , triclinic,  $P\bar{1}$ ,  $a = 6.570(1)$ ,  $b = 8.891(1)$ ,  $c = 15.299(3)$  Å,  $\alpha =$

<sup>\*</sup> Student undergraduate research participants sponsored by Argonne Division of Educational Programs.

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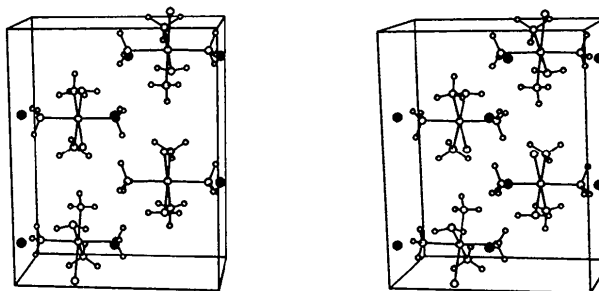


Fig. 2. Unit-cell packing diagram for  $[\text{CrCl}(\text{H}_2\text{O})(\text{NH}_3)_4]\text{Cl}_2$ . Chloride ions are shown as solid circles.

terized; for  $[\text{CrCl}_2(\text{H}_2\text{O})]\text{Cl} \cdot 2\text{H}_2\text{O}$  (Morosin, 1966) a Cr–Cl distance of 2.286 Å and Cr–O distances of 2.002 and 2.013 Å are quoted without e.s.d.'s.  $\text{H}_2\text{O} \cdots \text{Cl}(\text{free})$  non-bonded distances of 3.061, 3.112 and 3.197 Å are very similar to those we report.

The crystals were the gift of Dr John V. Rund, University of Arizona.

### References

- EARLEY, J. E. & CANNON, R. D. (1965). *Transition Met. Chem.* **1**, 33–94.  
 FORSTER, L. S., RUND, J. V. & FUCALORO, A. F. (1984). *J. Phys. Chem.* **88**, 5012–5017.  
 GARNER, C. S. & HOUSE, D. A. (1970). *Transition Met. Chem.* **6**, 59–284.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
 JACKSON, W. G., VOWLES, P. D. & FEE, W. W. (1977). *Inorg. Chim. Acta*, **22**, 111–130.  
 KYUNO, E., KAMADA, M. & TANAKA, N. (1967). *Bull. Chem. Soc. Jpn*, **40**, 1848–1854.  
 MORI, M. (1953). *Nippon Kagaku Zasshi*, **74**, 253–259.  
 MOROSIN, B. (1966). *Acta Cryst.* **21**, 280–282.  
 SHELDRIK, G. (1986). *SHELXTL Program Library*, version 5.1. Nicolet XRD Corporation, Madison, Wisconsin.