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Structure of Pentaammineaquacobalt(III) Hexacyanochromate(III), $[Co(NH_3)_5(H_2O)][Cr(CN)_6]$, by Neutron Diffraction at 2·0 K

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Abstract

M_r = 370·2, rhombohedral, $R\bar{3}$, a = 7·282 (2) Å, α = 97·71 (2)°, V = 374·6 (2) Å^3, Z = 1, D_m(295 K) = 1·59 (1), D_x = 1·64 g cm^{-3}, neutron diffraction, λ = 1·1760 (2) Å, μ = 2·05 cm^{-1}, F(000) = 27·4 × 10^{-14} m, T = 2·0 K, $R(F)$ = 0·029 for 643 unique reflections, χ = 2·17. The water molecule is dis-

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ordered over the six 'ammine' sites of the cation with its H atoms occupying two of the three sites of the ammonia protons. As a consequence of the disordered water molecule the cyanide N atom is also disordered over two sites 0·32 (2) Å apart with site occupancies of 0·84 (3) and 0·15 (3). Both the CrC_6 and Co'N'$_6$ octahedra are, as found previously, highly regular with bond lengths of 2·064 (1) Å for Cr—C and 1·964 (1) Å for Co—'N' and bond angles of 90·36 (5)° for C—Cr—C and 89·11 (5)° for 'N'—Co—'N'.

Experimental

Large single crystals of pentaamminecobalt(III) hexacyanochromate(III) were grown by repeated seeding of freshly filtered 0·01 *M* aqueous solutions of pentaammineaquacobalt(III) perchlorate and potassium hexacyanochromate(III) held at 278 K.

Intensity data from two crystals of approximately hexagonal prismatic habit were collected on the D15 lifting-arm normal-beam diffractometer at the Institute Laue–Langevin, Grenoble. Crystal (1) was mounted with the rotation axis approximately along the hexagonal *b* axis and (2) with the rotation axis approximately along the hexagonal *c* axis. The temperature was maintained at 2·0 K during the 10 day duration of the two experiments. Crystal data information is given in Table 1. Hexagonal cell parameters were obtained from least-squares refinement of the angular parameters of 26 reflections with $35\cdot2 < 2\theta < 97\cdot2^\circ$. 1033 reflections were measured in the first crystal orientation and 363 in the second orientation. $2\theta/\omega$ scans were employed and the data collected to $\sin\theta/\lambda < 0\cdot77$ Å^{-1} with $|h| < 16$, $|k| < 8$, $|l| < 16$. Some reflections were omitted due to lifting-arm restrictions and the limitations of normal-beam geometry. One standard reflection measured after every 40 reflections showed no systematic variation with time. Scaled squares of structure-factor moduli were obtained using the profile analysis and Lorentz correction program *COLL5N* (Lehmann & Larsen, 1974). These intensity data were corrected analytically for absorption using a modified version of *XTAL* (Hall & Stewart, 1989). The effective absorption coefficient was estimated by adding the incoherent contribution to the absorption cross section. After averaging of equivalents, 579 and 196 unique reflections were obtained for crystals (1) and (2) respectively (Table 1). The agreement factor between the 107 reflections common to both crystals, $\sum|F_b^2 - F_c^2|/\sum(F_b^2 + F_c^2)/2$, was 0·035.

Table 1. *Crystal data for pentaammineaquacobalt*(III) *hexacyanochromate*(III)

	Neutron 2·0 K	Neutron 11·5 K^{i}	X-ray 120 K^{ii}	X-ray 295 K^{ii}
M_r	370·2	370·2	370·2	370·2
Space group	$R\bar{3}$	$R\bar{3}$	$R\bar{3}$	$R\bar{3}$
Unit cell (hexagonal setting)				
a (Å)	10·968 (3)	11·00 (1)	10·986 (4)	11·027 (3)
c (Å)	10·788 (3)	10·91 (1)	10·817 (4)	10·912 (3)
V (Å^3)	1123·9 (5)	1143 (3)	1130·7 (9)	1149·0 (6)
Z	3	3	3	3
Unit cell (rhombohedral setting)				
a (Å)	7·282 (2)	7·318 (8)	7·296 (3)	7·332 (2)
α ( )	97·71 (2)	97·5 (1)	97·68 (3)	97·52 (2)
V (Å^3)	374·6 (2)	381 (1)	376·9 (3)	383·0 (2)
Z	1	1	1	1
D_m (295 K) (g cm^{-3})				1·59
D_x (g cm^{-3})	1·64	1·61	1·63	1·60
Dimensions (cm)*				
$(\bar{1}20)$ $(1\bar{2}0)$	0·094, 0·125			
$(2\bar{1}0)$–$(\bar{2}10)$	0·132, 0·125			
(110)–$(\bar{1}\bar{1}0)$	0·132, 0·125			
(001) $(00\bar{1})$	0·274, 0·320			
Transmission factors*	0·61 to 0·79, 0·77 to 0·81			
μ (cm^{-1})	2·05			
R_{int}*	0·0276, 0·0183			
$\sum\sigma(F^2)/\sum F^2$*	0·0209, 0·0151			
Reflections measured*	1033, 363			
Unique reflections*	579, 196			

Notes: (i) Figgis, Leung & Schultz (1985); (ii) Figgis & Reynolds (1985).

* Data listed are for crystal (1) followed by crystal (2) (see text). Face indices are for the hexagonal setting.

Table 2. *Fractional atomic coordinates* [$\times 10^4$ *except* N(1*b*) $\times 10^3$; *hexagonal setting*] *and equivalent isotropic temperature factors for pentaammineaquacobalt*(III) *hexacyanochromate*(III) *at* 2·0 K

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j \text{ (Fischer \& Tillmanns, 1988).}$$

		x	y	z	U_{eq} (Å^2)
Co	3(*b*)	0	0	$\frac{1}{2}$	0·008 (1)
'N'(2)^{i}	18(*f*)	1281 (1)	1575 (1)	6068 (1)	0·0110 (2)
H(1)	18(*f*)	917 (2)	2212 (2)	6322 (3)	0·043 (1)
H(2)^{ii}	18(*f*)	1466 (3)	1251 (2)	6897 (2)	0·036 (1)
H(3)	18(*f*)	2262 (2)	2195 (2)	5702 (2)	0·031 (1)
Cr	3(*a*)	0	0	0	0·010 (1)
C(1)	18(*f*)	1452 (1)	1617 (2)	1098 (1)	0·0149 (2)
N(1*a*)^{iii}	18(*f*)	2239 (2)	2558 (5)	1703 (5)	0·0198 (5)
N(1*b*)^{iii}	18(*f*)	218 (1)	233 (2)	191 (1)	0·010 (2)

Notes: (i) *b*['N'(2)] = 8·71 (4) fm; (ii) *p*[H(2)] = 0·841 (9); (iii) *p*[N(1*a*)] = 0·84 (3), *p*[N(1*b*)] = 0·15 (3).

The structure was refined using the program *ASRED* (Figgis, Reynolds & Williams, 1980) starting with the positional and anisotropic thermal parameters of the 11·5 K neutron structure (Figgis, Leung & Schultz, 1985). Both data sets were refined together on $|F^2|$ employing weights of $w = [\sigma(F^2)]^{-2}$, with separate scale, isotropic type I extinction (Becker & Coppens, 1974, 1975) and multiple-scattering corrections (Le Page & Gabe, 1979), but with common positional and anisotropic thermal parameters. For the refinement, $\sigma(F^2)$ of the reflections used as standards was adjusted to be equal to that of a similar reflection measured with the average number of equivalents. Extinction effects were negligible in the first crystal and small in the second, the

Table 3. *Principal bond lengths* (Å) *and angles* (°) *and hydrogen bonds* (Å, °) *in pentaamminеaquacobalt*(III) *hexacyanochromate*(III) *at* 2·0, 11·5 (*neutron*) *and* 120 K (*X-ray*)

The 2·0 K results are given first with the 11·5 and 120 K results immediately below. The quotation marks around the ammonia N atom, ‘N’(2), emphasize that it is an average of five N and one O atoms. N(1*a*) and N(1*b*) are two positions of the disordered cyanide nitrogen N(1), 0·32 (2) Å apart and of occupancy 0·84 (3) and 0·15 (3) respectively.

Co—‘N’(2)	1·964 (1) 1·975 (2) 1·957 (1)	‘N’(2)—Co—‘N’(2)	89·11 (5) 88·7 (1) 89·5 (1)
‘N’(2)—H(1)	1·000 (3) 0·989 (5) 0·85 (2)	Co—‘N’(2)—H(1)	114·5 (2) 114·5 (4) 114 (1)
‘N’(2)—H(2)	1·020 (3) 1·016 (8) 0·85 (2)	Co—‘N’(2)—H(2)	112·8 (1) 114·0 (4) 114 (1)
‘N’(2)—H(3)	1·022 (2) 1·017 (4) 0·79 (2)	Co—‘N’(2)—H(3)	114·3 (2) 114·2 (4) 114 (1)
		H(1)—‘N’(2)—H(2)	102·8 (3) 102·5 (8) 100 (2)
		H(1)—‘N’(2)—H(3)	107·2 (2) 106·1 (5) 105 (2)
		H(2)—‘N’(2)—H(3)	104·1 (2) 104·3 (6) 109 (2)
Cr—C(1)	2·064 (1) 2·074 (3) 2·068 (1)	C(1)—Cr—C(1)	90·36 (5) 90·1 (1) 90·3 (1)
C(1)—N(1)*	1·160 (4), 1·18 (1) 1·170 (3) 1·147 (1)	Cr—C(1)—N(1)	177·1 (3), 166·2 (8) 177·5 (2) 177·5 (2)

Hydrogen bonds

(*a*) Interactions between N(1) and the $[Co(NH_3)_5(H_2O)]^{3+}$ ion at $(\frac{1}{3}, \frac{2}{3}, \frac{1}{6})$

N(1)···H(1)	2·471 (6), 2·78 (2) 2·45 (1)	‘N’(2)—H(1)···N(1)	122·3 (3), 121·1 (4) 123·4 (7)
N(1)···H(3)$^{\rm i}$	1·898 (5), 2·04 (2) 1·933 (5) 2·16 (2)	‘N’(2)—H(3)···N(1)	168·5 (3), 167·1 (7) 168·3 (5) 165 (2)

(*b*) Interactions between N(1) and the $[Co(NH_3)_5(H_2O)]^{3+}$ ion at $(0, 0, \frac{1}{2})$

N(1)···H(1)$^{\rm iii}$	2·534 (7), 2·23 (2) 2·54 (1)	‘N’(2)—H(1)···N(1)	129·9 (2), 127·7 (5) 128·4 (7)
N(1)···H(2)$^{\rm iv}$	2·447 (5), 2·27 (1) 2·45 (1)	‘N’(2)—H(2)···N(1)	155·7 (3), 153·1 (6) 154·3 (8)

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

* Values given are for N(1*a*), N(1*b*).

largest correction applied being 1·07. Neutron scattering lengths were taken from the compilation by Sears (1984).

The disorder of the water molecule was examined by allowing the ammonia hydrogen populations and the nitrogen scattering length to refine. The populations of H(1) and H(3) were found to be unity to within experimental errors [0·990 (9)] and were fixed at unity in subsequent refinements. Refinement, including the populations of N and H(2), proceeded until all shift/e.s.d. ratios were <0·1 with $R(F^2) = 0{\cdot}043$, $wR(F^2) = 0{\cdot}048$, $R(F) = 0{\cdot}034$ (3σ cut) and $\chi = 2{\cdot}42$. The difference Fourier map was essentially featureless except for a positive peak of 1·7 fm Å^{-3} near the cyanide N atom. This peak could be removed by assuming the N atom to be disordered over two adjacent sites, N(1*a*) and N(1*b*), *ca* 0·3 Å apart and with approximate occupancies of $\frac{5}{6}$ and $\frac{1}{6}$ respectively. Allowing the positional, thermal parameters (isotropic for the minor site) and occupancies of the two sites to refine independently produced a significant improvement in the fit. In the final cycle all shift/e.s.d. ratios were <0·02 with $R(F^2) = 0{\cdot}039$, $wR(F^2) = 0{\cdot}044$, $R(F) = 0{\cdot}029$ and $\chi = 2{\cdot}17$. The resulting difference Fourier map showed no features >0·8 fm Å^{-3} and was essentially featureless. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 2. The principal interatomic distances and angles obtained using the *XTAL* system of programs (Hall & Stewart, 1989) are given in Table 3.*

Discussion

The present neutron diffraction study provides a significantly improved description of the pentaamminеaquacobalt(III) hexacyanochromate(III) structure relative to the previous X-ray (Figgis & Reynolds, 1985) and neutron diffraction studies (Figgis, Leung & Schultz, 1985). In the neutron case the improved accuracy is reflected in the generally smaller errors in positional and thermal parameters and the lower agreement factors [$R(F) = 0{\cdot}029$, *cf.* 0·071 for the 11·5 K study] of the present study. All the major features of the earlier studies are reproduced but with an improved model for the disorder.

The rhombohedral cell dimensions of the present structure [$a = 7{\cdot}282$ (2) Å, $\alpha = 97{\cdot}71$ (2)°] are consistent with those expected from a linear extrapolation of the 120 K [7·296 (3) Å, 97·68 (3)°] and 295 K [7·332 (2) Å, 97·52 (2)°] values. This is in contrast with the values found in the 11·5 K neutron diffraction study [7·318 (8) Å, 97·5 (1)°] which are intermediate between the 120 and 295 K results.

The model for the disorder of the water molecule used in the previous studies is seen to be basically correct. Refinement of the H(2) population resulted in a value of 0·841 (9) which compares favourably

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* Lists of anisotropic thermal parameters, and $F(\text{obs})^2$, $F(\text{cal})^2$ and $\sigma(F^2)$ have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54385 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

with 0·833, the value expected for the complete ordering of the water molecule protons into the H(1) and H(3) ammonia sites. The N(2) scattering length, which was refined to allow for the superposition of the water O atom on the ammine nitrogen site, reduced from the 9·36 fm for a 'pure' nitrogen to 8·71 (4) fm. That value compares favourably with 8·76 (2) fm, the mean scattering length for five N and one O atoms. The success of the model of the disorder of the water molecule is also reflected in the hydrogen thermal parameters. Whereas the isotropic thermal parameter for H(2) may be expected to be the outlier, its value lies intermediate between that for H(1) and H(3). The success is further supported by the essentially featureless difference Fourier in the vicinity of the cation.

A significant difference between this study and previous structure determinations is the appearance of disorder in the cyanide-N-atom position. This disorder can be modelled by assuming the N atom occupies two adjacent sites. Allowing the positional and thermal (isotropic for the minor site) parameters and the occupancies of the two sites to refine independently resulted in positions 0·32 (2) Å apart with populations of 0·84 (3) and 0·15 (3). These occupancies compare well with the 0·83 ($=\frac{5}{6}$) and 0·17 ($=\frac{1}{6}$) expected if the disorder is a direct consequence of the water molecule disorder and further supports the model for that.

The average thermal motion of the non-H atoms at 2·0 K measured as $U_{\rm iso}$ ($=\frac{1}{3}\sum U_{ii}$) is 0·61 (5) that observed in the 120 K X-ray study. Although this value is in good agreement with the value 0·59 predicted by a linear extrapolation of the 120 and 295 K results, the actual values of the thermal tensor elements are more than 50% higher than found in similar low-temperature neutron studies (*e.g.* Brown, Figgis & Forsyth, 1987; Figgis, Kucharski & Forsyth, 1990, and references therein). The good linearity and high values at low temperatures is indicative of the disorder required for the structure to conform to the space group $R\bar{3}$.

The hydrogen bonding in the compound has been discussed in detail (Figgis, Leung & Schultz, 1985). The cyanide N atom experiences one strong [N(1)···H(3)] and one weak [N(1)···H(1)] hydrogen-bond interaction with the cation at ($\frac{1}{3}$, $\frac{2}{3}$, $\frac{1}{6}$), and two weak interactions [N(1)···H(1), H(2)] with the cation at (0, 0, $\frac{1}{2}$). The nitrogen site N(1*a*) shows a stronger interaction with the cation at ($\frac{1}{3}$, $\frac{2}{3}$, $\frac{1}{6}$) than does N(1*b*) (Table 3). In addition one of the weak interactions is with H(2), the hydrogen site unoccupied when a water molecule is present, so that a possible mechanism for the disorder in the cyanide nitrogen is through this hydrogen-bond interaction.

The bond lengths and angles deduced are generally more precise than from previous studies, the only exception being those involving the disordered cyanide N atom, and even these are of comparable precision. The CoN_6 and CrC_6 octahedra are, as found previously, highly regular, with only very small differences (especially in the latter) from ideal geometry. The non-H bond lengths and angles are in better agreement with the 120 K X-ray structure than are those of the 11·5 K neutron structure (Table 3). However the differences are only slight and can almost certainly be attributed to the inconsistency in the cell dimensions noted above. The hydrogen-bond lengths and angles are essentially the same as those found in the 11·5 K neutron structure determination.

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