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Nickel Ammonium Chromate Hexahydrate†

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Abstract. $(\text{NH}_4)_2\text{Ni}(\text{CrO}_4)_2 \cdot 6\text{H}_2\text{O}$, monoclinic, $P2_1/a$, $a = 9.409$ (8), $b = 12.595$ (8), $c = 6.268$ (3) Å, $\beta = 105.86$ (1)°, $D_o = 2.013$, $D_c = 2.021$ Mg m⁻³, $Z = 2$. The structure, which is the same as that of the corresponding sulphate, has been determined by the refinement of diffractometer data to a final R value of 0.031 for 821 observed reflexions. The metal–water distances are 2.073, 2.054 and 2.022 Å.

Introduction. As a continuation of the study of the isomorphous series of Tutton's salts, $M^{II}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (Montgomery, Chastain, Natt, Witkowska & Lingafelter, 1967), the structure of a chromate analogue has been determined.

$(\text{NH}_4)_2\text{Ni}(\text{CrO}_4)_2 \cdot 6\text{H}_2\text{O}$ was prepared by the method of Briggs (1903), which gave a product composed of a rusty powder and yellow-green crystals. Recrystallization was by slow evaporation of an aqueous solution at room temperature. Attempts to prepare Zn, Co and Cu compounds were unsuccessful. A crystal $0.21 \times 0.21 \times 0.65$ mm was used for data collection (c^* axis of rotation). Accurate cell dimensions were determined by the least-squares fitting of 18 2θ values (20 to 35°; $\lambda = 0.71069$, 0.71354 and 0.70926 Å).

Intensity measurements were made manually at room temperature (296–297 K) on a Picker four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å) and a Zr filter. Reflexions were scanned in the $\omega/2\theta$ mode to a 2θ limit of 44°. The orientation of the crystal was checked twice a day using three reflexions, one of

which (332) was used as an intensity standard. 16 reflexions which had been omitted in the original crystal were measured from a second crystal and the intensities were suitably scaled by means of standard reflexions. 878 reflexions were measured, of which 57 were judged as unobserved according to the criterion that the net count must be less than σ [the standard deviation (square root) of the background count]. Intensities were corrected for Lorentz and polarization effects but not for absorption [$\mu(\text{Mo}) = 2.9$ mm⁻¹, μr for cylinder = 0.30].

Initial parameters were taken from the corresponding sulphate (Montgomery & Lingafelter, 1964) and refined by a full-matrix least-squares procedure. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, with weights determined from counting statistics. Unobserved reflexions were given zero weight in the calculations. Scattering factors for the Ni ion, Cr, O and N were taken from *International Tables for X-ray Crystallography* (1962), and those for H from Table 2 of Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections for the Ni ion were made (*International Tables for X-ray Crystallography*, 1962) in the final refinement cycles after anisotropic refinement had been completed. Two further cycles were run refining only H positions with isotropic temperature factors ($B = 5.0$ Å², initially).* A final difference Fourier map gave no indication of anomalous electron density.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33940 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Tutton's Salts. IX.

Table 1. Final coordinates ($\times 10^4$, for H $\times 10^3$) and, for H, isotropic thermal parameters, with the least-squares standard errors in parentheses

	x	y	z	B (Å ²)
Ni	0	0	0	
Cr	4074 (1)	1351 (1)	7405 (1)	
O(3)	4138 (3)	2345 (2)	5699 (4)	
O(4)	5622 (3)	706 (3)	7924 (5)	
O(5)	2713 (3)	547 (2)	6144 (4)	
O(6)	3808 (3)	1813 (2)	9732 (5)	
O(7)	1619 (3)	1052 (2)	1664 (4)	
O(8)	-1559 (3)	1066 (2)	399 (4)	
O(9)	-24 (3)	-642 (2)	2947 (4)	
N(10)	1347 (4)	3415 (3)	3524 (6)	
H(11)	74	330	232	2.9
H(12)	238	290	433	9.4
H(13)	107	330	431	6.4
H(14)	163	393	350	5.2
H(15)	179	92	277	1.2
H(16)	200	117	99	2.1
H(17)	-224	97	-33	1.7
H(18)	-134	161	32	2.9
H(19)	-87	-56	324	2.0
H(20)	15	-114	340	4.7

Table 2. Bond lengths (Å) and angles (°)

Ni—O(7)	2.073 (3)	O(7)—Ni—O(8)	88.4 (1)
Ni—O(8)	2.054 (3)	O(7)—Ni—O(9)	90.8 (1)
Ni—O(9)	2.022 (3)	O(8)—Ni—O(9)	87.9 (1)
Cr—O(3)	1.658 (3)	O(3)—Cr—O(4)	108.6 (2)
Cr—O(4)	1.621 (3)	O(3)—Cr—O(5)	108.3 (2)
Cr—O(5)	1.654 (3)	O(3)—Cr—O(6)	110.1 (2)
Cr—O(6)	1.652 (3)	O(4)—Cr—O(5)	109.0 (2)
		O(4)—Cr—O(6)	110.1 (2)
		O(5)—Cr—O(6)	110.7 (2)

Discussion. The structure follows closely that of the corresponding sulphate (Tables 1, 2, 3). The metal-water distances are 2.073, 2.054 and 2.022 Å, indicating an orthorhombic distortion of the nickel-water ion. A comparison of the corresponding nickel ammonium and nickel guanidinium (Morimoto, 1970) sulphates is given in Table 3.

The chromate ion is fairly regular. The main deviation is in the bond length Cr—O(4) which is significantly shorter than the average of the other three (0.031 Å). This is probably due to hydrogen-bonding and crystal-packing effects. Each O atom in the chromate ion [with the exception of O(4)] is hydrogen bonded to two or more atoms. A similar lengthening has been observed (Ruben, Olovsson, Zalkin & Templeton, 1973) in sodium chromate tetrahydrate where the Cr—O(4) bond is the longest (1.684 Å corr.), and has the greatest number of hydrogen bonds,

Table 3. Interatomic distances (Å) and angles (°) in double salts of nickel

	Nickel ammonium sulphate	Nickel guanidinium sulphate		Nickel ammonium chromate
		Direct	Corrected	
Ni—O(7)	2.072 (5)	2.053	2.065 (2)	2.073 (3)
Ni—O(8)	2.069 (6)	2.052	2.062 (2)	2.054 (3)
Ni—O(9)	2.033 (6)	2.064	2.070 (2)	2.022 (3)
Mean	2.058	2.056	2.066	2.050
O(7)—Ni—O(8)	88.34		89.09	88.44
O(7)—Ni—O(9)	90.13		91.93	90.80
O(8)—Ni—O(9)	89.22		86.95	87.88
Hydrogen bonds		Relative position*		
O(7)···O(5)	2.785	x, y, z		2.787 (5)
O(7)···O(6)	2.841	$x, y, z - 1$		2.827 (5)
O(8)···O(4)	2.708	$x - 1, y, z - 1$		2.720 (5)
O(8)···O(6)	2.759	$x - \frac{1}{2}, \frac{1}{2} - y, z - 1$		2.741 (5)
O(9)···O(5)	2.765	$\frac{1}{2} - x, y - \frac{1}{2}, 1 - z$		2.744 (5)
O(9)···O(3)	2.719	$-x, -y, 1 - z$		2.731 (5)
N(10)···O(3)	2.951	x, y, z		2.935 (5)
N(10)···O(3)	2.978	$x - \frac{1}{2}, \frac{1}{2} - y, z$		2.940 (5)
N(10)···O(5)	2.867	$\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$		2.816 (5)
N(10)···O(6)	2.905	$x - \frac{1}{2}, \frac{1}{2} - y, z - 1$		2.887 (5)

* Transformation relating each second atom of nickel ammonium chromate to the corresponding atom in the original asymmetric unit.

whereas Cr—O(3) (1.618 Å corr.) is the shortest and has the least number of neighbours. McGinney (1972) has calculated bond lengths and noted similar effects in anhydrous potassium chromate.

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