Table 2. *Root-mean-square displacement* (Å) *of thermal vibration*

Root-mean-square displacements obtained from $\langle u^2\rangle^{1/2} = (1/8\pi^2)^{1/2}B^{1/2}$.

	$\langle u^2\rangle^{1/2}$ at 120 K		$\langle u^2\rangle^{1/2}$ at 298 K		$(\langle u^2\rangle^{1/2}_{298} - \langle u^2\rangle^{1/2}_{120})/a \times 100$ (%)	
	Pb	X	Pb	X	Pb	X
PbS	0·083 (2)	0·086 (3)	0·128 (1)	0·126 (2)	0·76 (5)	0·66 (8)
PbSe	0·083 (1)	0·078 (1)	0·130 (1)	0·116 (1)	0·77 (3)	0·62 (3)
PbTe	0·091 (1)	0·077 (1)	0·143 (1)	0·119 (1)	0·81 (3)	0·65 (3)
GaAs*	(a = 5·654 Å)		0·096	0·087	(at room temperature)	

* Pietsch (1981).

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Structure of Ammonium *catena*-Diammine-di-μ-chromato-cuprate(II), a Compound with Bridging Chromate Groups

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Abstract. $(NH_4)_2[Cu(CrO_4)_2(NH_3)_2]$, $M_r = 365{\cdot}67$, triclinic, $P\bar{1}$, $a = 7{\cdot}362\ (2)$, $b = 6{\cdot}932\ (1)$, $c = 5{\cdot}895\ (1)$ Å, $\alpha = 112{\cdot}39\ (3)$, $\beta = 92{\cdot}79\ (2)$, $\gamma = 107{\cdot}06\ (3)^\circ$, $V = 261{\cdot}5$ Å^3, $Z = 1$, $D_m = 2{\cdot}34\ (5)$, $D_x = 2{\cdot}32$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}7107$ Å, $\mu = 4{\cdot}02$ mm^{-1}, $F(000) = 183$, $T = 293$ K. Final $R = 0{\cdot}025$ for 1896 counter reflections. The structure consists of infinite anion chains, parallel to the c axis, in which octahedral copper(II) ions (site symmetry C_i) are linked by two bridging chromium(VI) tetrahedra. The copper(II) octahedron comprises two ammonia molecules [Cu—N 1·972 (2) Å] and four chromate O atoms [Cu—O 2·022 (2) and 2·425 (1) Å, Cr—O ranges from 1·636 (1) to 1·686 (1) Å]. The anion chains are linked through hydrogen bonding from the ammonium cations.

Introduction. An undergraduate project involving the coprecipitation of copper and chromium in salts suitable for thermal decomposition into cupric chromite ($CuCr_2O_4$), led to the production of crystals thought to be $(NH_4)_2[Cu(CrO_4)_2(NH_3)_2]$. Measurement of the unit-cell dimensions indicated that these differed considerably from those of the analogous zinc compound, $(NH_4)_2[Zn(CrO_4)_2(NH_3)_2]$ [$a = 12{\cdot}85\ (2)$, $b = 5{\cdot}912\ (8)$, $c = 7{\cdot}434\ (8)$ Å, $\beta = 111{\cdot}2\ (2)^\circ$, monoclinic (Harel, Knobler & McCullough, 1969)]. As a result of these observations the structure of the title compound was determined.

Experimental. The compound was prepared by the method used for the Zn compound (see above) and an orange prismatic crystal (~0·23 × 0·13 × 0·20 mm)

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with developed faces (100), ($\bar{1}$00), (130), ($\bar{1}\bar{3}$0), (0$\bar{1}$3) and (01$\bar{3}$) was selected and mounted for data collection. D_m by flotation in CCl_4 and $C_2H_2Br_4$.

Space group $P\bar{1}$ by successful refinement; cell parameters determined with a Philips PW 1100 automatic four-circle diffractometer equipped with a graphite monochromator as described previously (Gatehouse & Miskin, 1974), and are the mean of ten refinements of the orientation matrix during data collection. Three standard reflections measured at 3 h intervals. No decomposition occurred. Data collected using the $\omega/2\theta$ scan technique with a symmetric scan width of $\pm 1{\cdot}30°$ in ω from the calculated Bragg angle, with an allowance for dispersion, at a scan rate of $0{\cdot}05°\ s^{-1}$. No reflection was sufficiently intense to warrant the insertion of an attenuation filter. Data processed using a program written specifically for the PW 1100 diffractometer (Hornstra & Stubbe, 1972). Values of I and $\sigma(I)$ corrected for Lorentz and polarization effects. Absorption correction applied based on indexed crystal faces, max. and min. transmission factors 0·6226 and 0·4637 respectively. 2291 unique reflections measured to $2\theta = 70°$, 1896 $[I \geq 3\sigma(I)]$ used in analysis; index range $h \pm 11$, $k \pm 10$, l 0/9. As $Z = 1$, Cu placed on the origin, Cr located in subsequent difference Fourier synthesis followed by rest of non-H atoms in next Fourier calculation. Function minimized in full-matrix least-squares refinement $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma^2(F_o)]^{-1}$. Following several cycles of isotropic refinement and location of H atoms, non-H atoms were refined using anisotropic thermal parameters, H atoms were refined using a single isotropic thermal parameter (92 variable parameters), final $R = 0{\cdot}025$ and $wR = 0{\cdot}034$ (for observed reflections); max. Δ/σ in final cycle 0·2; final difference Fourier synthesis had $\Delta\rho$ within $-0{\cdot}89$ and $0{\cdot}42\ e\ Å^{-3}$. Scattering factors for neutral atoms and corrections for anomalous dispersion used (*International Tables for X-ray Crystallography*, 1974). All calculations were performed on the Monash University VAX 11/780 computer system. Major programs used: *SHELX*76 (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964). Fig. 1, showing the

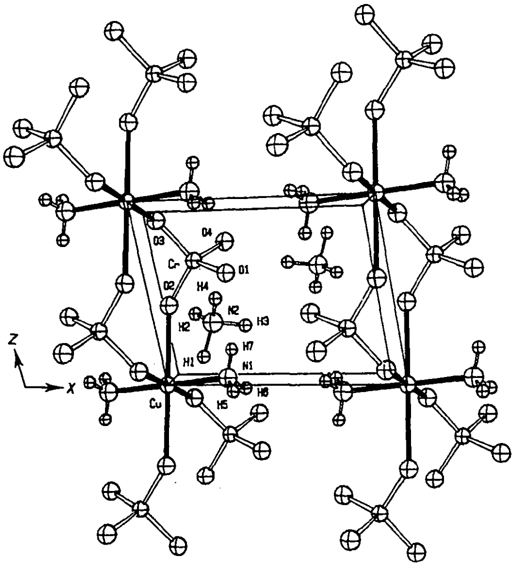

Fig. 1. *ORTEP* drawing of the structure and unit-cell packing of $(NH_4)_2[Cu(CrO_4)_2(NH_3)_2]$ together with the numbering scheme used. Thermal ellipsoids are shown at the 50% probability level; H atoms are represented by spheres of arbitrary size.

Table 1. *Fractional coordinates* (Cr $\times 10^5$; O, N $\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
Cu	0	0	0	166 (1)
Cr	21516 (3)	29813 (4)	68761 (4)	149 (1)
O(1)	3440 (2)	1316 (2)	6098 (2)	229 (5)
O(2)	667 (2)	2474 (2)	4389 (2)	242 (5)
O(3)	904 (2)	2617 (2)	9092 (2)	211 (4)
O(4)	3582 (2)	5562 (2)	7948 (3)	270 (5)
N(1)	2651 (2)	−74 (3)	389 (3)	211 (5)
N(2)	2625 (2)	6463 (2)	3535 (3)	231 (5)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Interatomic distances* (Å), *selected angles* (°) *and hydrogen-bond dimensions with e.s.d.'s in parentheses*

Cu–N(1)	1·972 (2) ×2	Cr–O(4)	1·636 (1)
Cu–O(3)	2·022 (2) ×2	Cr–O(2)	1·641 (1)
Cu–O(2)	2·425 (1) ×2	Cr–O(1)	1·650 (2)
		Cr–O(3)	1·686 (1)
N(1)–Cu–O(3)	88·99 (7)	O(4)–Cr–O(2)	108·05 (8)
N(1)–Cu–O(2)	91·65 (2)	O(4)–Cr–O(1)	109·74 (7)
O(3)–Cu–O(2)	90·61 (5)	O(4)–Cr–O(3)	109·06 (6)
		O(2)–Cr–O(1)	109·27 (7)
		O(2)–Cr–O(3)	110·29 (7)
		O(1)–Cr–O(3)	110·40 (8)

$X\cdots$H–Y	$X\cdots$H	H–Y	$X\cdots Y$	$X\cdots$H–Y
O(1^{i})$\cdots$H(3)–N(2)	1·92 (3)	0·89 (3)	2·807 (2)	174 (3)
O(1)$\cdots$H(7)–N(1)	2·29 (3)	0·84 (3)	3·099 (2)	163 (3)
O(1^{ii})$\cdots$H(2)–N(2)	2·36 (3)	0·90 (4)	2·969 (2)	125 (2)
O(2^{iii})$\cdots$H(2)–N(2)	2·22 (3)	0·90 (4)	2·926 (2)	135 (3)
O(3^{iv})$\cdots$H(1)–N(2)	1·94 (2)	0·92 (2)	2·819 (2)	160 (4)
O(1^{iv})$\cdots$H(6)–N(1)	2·39 (4)	0·78 (3)	3·055 (3)	144 (3)
O(4)$\cdots$H(4)–N(2)	2·15 (4)	0·85 (4)	2·995 (3)	176 (2)
O(4^{v})$\cdots$H(5)–N(1)	2·25 (4)	0·87 (4)	3·113 (2)	175 (2)

Symmetry code: $1-x$, $1-y$, $1-z$; (ii) x, $1+y$, z; (iii) $\bar{x}$, $1-y$, $1-z$; (iv) x, y, $z-1$; (v) x, $y-1$, $z-1$.

Table 3. *Unit-cell dimensions of* $(NH_4)_2[M(CrO_4)_2(NH_3)_2]$ *where* M = Ni, Cu, Zn *and* Cd

M	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
Ni	7·393 (2)	7·024 (2)	5·739 (2)	112·67 (3)	94·53 (3)	106·17 (3)
Cu	7·362 (2)	6·932 (1)	5·895 (1)	112·39 (3)	92·79 (2)	107·06 (3)
Zn	7·434 (2)	5·911 (2)	12·389 (3)	90	103·67 (3)	90*
Cd	7·604 (2)	6·037 (2)	12·479 (3)	90	104·65 (3)	90*

* As $A2/m$ unit cells.

structure and unit-cell packing, was drawn using *ORTEP* (Johnson, 1965).

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters and selected interatomic distances and angles are given in Tables 1 and 2 respectively.*

As in the analogous Zn compound (Harel *et al.*, 1969), the structure consists of octahedra linked by chromate tetrahedra into chains that are separated by ammonium ions. As set out in Table 3, the Zn compound is monoclinic ($C2/m$); the Zn atom is located on the origin and the Cr atom on a mirror plane.

In the Cu compound described here, the Cu atom is located on the origin of $P\bar{1}$ and coordinated by four O and two N atoms. The two pairs of O atoms are from chromate groups and have widely differing Cu—O distances; thus Cu—O(2) 2·425 (1) and Cu—O(3) 2·022 (2) Å, compared with Zn—O(1) 2·23 (2) Å in the Zn complex. The Cu—N(1)(ammonia) distance is 1·972 (2) Å [Zn—N(1) 2·00 (3) Å] and is similar to that of Cu—O(3), thus indicating a distortion of the octahedron of the type described by the Jahn–Teller theorem for Cu$^{\rm II}$.

The chromate tetrahedron is not constrained by symmetry and the Cr—O distances have a mean of 1·65 (2) Å compared with that in the Zn compound of 1·66 (1) Å.

The chains of octahedra and tetrahedra in the Cu compound are parallel to the shortest, *c*, axis; the axial length 5·895 Å corresponds to the *b* axis of the Zn compound (5·911 Å) to which the chain in that compound is parallel.

Interchain ammonium ions are involved in a number of possible hydrogen bonds as indicated in Table 2. Following the criteria set out by Hamilton & Ibers (1968), it is suggested that O(1) and O(4) each form single hydrogen bonds to N(1) and N(2), and that H(2) is involved in a bifurcated hydrogen bond to O(1) and O(2).

Preliminary experiments involving other $M^{\rm II}$ ions have produced crystals of the corresponding Ni and Cd compounds (Table 3) that are analogous to the Cu and Zn compounds respectively; further studies are intended.

The assistance of Miss J. Excell, a third-year student, is acknowledged.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43905 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Die Strukturen der Lithium-Vanadiumbronzen β-$Li_{1,5}V_{12}O_{29}$ (I) und β'-$Li_{2,1}V_{12}O_{29}$ (II)

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Abstract. (I): $M_r = 1085{\cdot}7$, monoclinic, $C2/m$, $a = 28{\cdot}2196\ (4)$, $b = 3{\cdot}6105\ (2)$, $c = 10{\cdot}1200\ (2)$ Å, $\beta = 102{\cdot}088\ (2)°$, $V = 1008{\cdot}23\ (6)$ Å^3, $Z = 2$, $D_x = 3{\cdot}576$ Mg m^{-3}, $\mu = 5{\cdot}19$ mm^{-1}, $F(000) = 1025{\cdot}0$, final $R = 0{\cdot}027$ for 3072 unique observed reflections. (II): $M_r = 1089{\cdot}9$, monoclinic, $C2/m$, $a = 28{\cdot}0428\ (9)$, $b = 3{\cdot}6206\ (3)$, $c = 10{\cdot}1286\ (4)$ Å, $\beta = 100{\cdot}070\ (3)°$, $V = 1012{\cdot}53\ (9)$ Å^3, $Z = 2$, $D_x = 3{\cdot}575$ Mg m^{-3}, $\mu = 5{\cdot}19$ mm^{-1}, $F(000) = 1028{\cdot}6$, final $R = 0{\cdot}040$ for 1777 unique observed reflections. (I) + (II): $T = 298$ K,

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