

Table 2. Selected bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

Cl(1)—Os	2.326 (4)	Cl(2)—Os—Cl(1)	90.7 (2)
Cl(2)—Os	2.338 (5)	Cl(3)—Os—Cl(1)	90.5 (2)
Cl(3)—Os	2.346 (4)	Cl(3)—Os—Cl(2)	90.3 (2)
C(2)—N(1)	1.49 (3)	C(6)—N(1)—C(2)	112 (2)
C(6)—N(1)	1.50 (3)	C(3)—C(2)—N(1)	110 (2)
C(3)—C(2)	1.51 (3)	C(7)—C(2)—N(1)	112 (2)
C(7)—C(2)	1.50 (3)	C(7)—C(2)—C(3)	109 (2)
N(4)—C(3)	1.54 (3)	N(4)—C(3)—C(2)	109 (2)
C(5)—N(4)	1.50 (3)	C(5)—N(4)—C(3)	112 (2)
C(6)—C(5)	1.52 (3)	C(6)—C(5)—N(4)	109 (2)
C(8)—C(7)	1.51 (3)	C(5)—C(6)—N(1)	113 (2)
O(9)—C(8)	1.42 (3)	C(8)—C(7)—C(2)	114 (2)
		O(9)—C(8)—C(7)	115 (2)

Craciunescu & Ghirvu, 1980; Anadon, Craciunescu, Larrañaga, Sanz & Doadrio, 1977; Ruiz Perez, 1983, and references therein).

The bond lengths and angles given in Table 2 show that the $(\text{Os}^{\text{IV}}\text{Cl}_6)^{2-}$ anion is nearly octahedral. The distances Os^{IV}—Cl in the range 2.326–2.346 (5) Å are shorter than previously reported for Os^{III}—Cl (Blake, Heath, Smith, Yellowlees & Sharp, 1988) and Os^{II}—Cl bonds (Chakravarty, Cotton & Tocher, 1985; Mukhopadhyay & Ray, 1987), but almost identical to those reported by Robinson, Hinckley, Matusz & Kibala (1988) for the same anion. The shortest Os—Os ($\frac{1}{2} + x, \frac{1}{2} - y, z$) distance is 7.654 (1) Å.

The deposited distances, angles and torsion angles of the piperazinium cation are similar to those reported by Sangin & Brisse (1984). Intermolecular distances and the deposited IR data support the existence of a weak intermolecular hydrogen bond between the cation and the chloride ions (Pimentel & McClellan, 1960; Harlow, Wells, Watt & Simonsen, 1974; Willet, Haugen, Lebsack & Morrey, 1974).

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Redetermination of the Structure of (Acetonitrile)chlorocopper(I) $[(\text{H}_3\text{CCN})\text{CuCl}]$ at 178 K

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Abstract. $[\text{Cu}(\text{C}_2\text{H}_3\text{N})\text{Cl}]$, $M_r = 140.0$, orthorhombic, $Pnma$, $a = 8.545 (3)$, $b = 3.8691 (10)$, $c = 12.692 (4)$ Å, $V = 419.6 (3)$ Å³, $Z = 4$, $D_x = 2.217 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 5.6 \text{ mm}^{-1}$, $F(000) = 272$, $T = 178$ K, $R = 0.022$ for 491 reflections. All non-H atoms lie in the mirror

planes at $y = 0.25$. Although the Cu atom displays slightly larger displacement parameters than the Cl atom [$U_{eq} 0.0317 (2)$ and $0.0225 (2)$ Å² for Cu and Cl], there seems no cogent reason to prefer the non-centrosymmetric space group $Pna2_1$ with the Cu atom displaced from the mirror plane. Zigzag chains

—Cu(NCCH₃)—Cl— are connected in pairs parallel to the short x axis, and joined by Cu—Cl cross-links to form infinite pleated sheets. The Cu atoms thus attain (somewhat distorted) tetrahedral coordination geometry, with Cu—N 1.935 (3), Cu—Cl 2.399 (1) ($\times 2$), 2.406 (1) Å.

Experimental. A colourless octahedron 0.55 × 0.5 × 0.45 mm was mounted in inert oil and rapidly transferred to the cold gas stream of the diffractometer (Siemens *R*3 with LT-2 low-temperature attachment). Intensities were registered to $2\theta_{\max}$ 55° using monochromated Mo $K\alpha$ radiation; ω scan technique. Of 1833 measured reflections (hemisphere with $-l$, expect for inaccessible reflections), 546 were unique ($R_{\text{int}} = 0.031$, index ranges $h -11$ to 11, $k -5$ to 5, $l -16$ to 0) and $491 > 4\sigma(F)$ considered observed. The orientation matrix was refined from setting angles of 49 reflections in the 2θ range 20–25°. Three check reflections showed no significant intensity variation. An absorption correction based on ψ scans was applied, with transmission factors 0.53–0.75.

The structure was solved by direct methods and subjected to anisotropic full-matrix least-squares refinement on F . H atoms were tentatively identified in difference Fourier syntheses and held fixed. An extinction correction of the form $F_{\text{corr}} = F[1 + 0.002xF^2/\sin 2\theta]^{-0.25}$ was carried out; the refined value of x was 0.0049 (4). The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0002F^2$; final $R = 0.022$, with $wR = 0.029$. 32 parameters; $S = 1.4$; maximum $\Delta/\sigma = 0.001$, maximum, minimum $\Delta\rho = 0.36$, -0.46 e Å⁻³. Program system: *SHELXTL-Plus* (Sheldrick, 1987). Atomic scattering factors and f' , f'' values from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atom coordinates are given in Table 1, with derived bond lengths and angles in Table 2.* Fig. 1. shows a stereoview of the packing of the compound.

Related literature. The structure was determined at room temperature from photographic data by Massaux, Bernard & Le Bihan (1969). In the redetermination, standard deviations of bond lengths are reduced by almost an order of magnitude. Healy, Kildea, Skelton & White (1989) redetermined the structures of (H₃CCN)Cu X ($X = \text{Cl}, \text{Br}, \text{I}$) at room temperature and discuss the possibility that the Cu atom is displaced from the mirror plane, giving space group *Pna2*₁. The thermal parameters of the Cu atom are indeed rather larger than those of the Cl

Table 1. *Atomic coordinates (× 10⁵) and equivalent isotropic displacement coefficients (Å² × 10⁴)*

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cu	89052 (4)	25000	44202 (3)	317 (2)
Cl	97440 (8)	25000	62297 (5)	225 (2)
N	66958 (33)	25000	40854 (20)	294 (7)
C(1)	37954 (33)	25000	35056 (24)	270 (8)
C(2)	54214 (35)	25000	38249 (20)	235 (8)

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

Cu—Cl	2.406 (1)	Cu—N	1.935 (3)
Cu—Cl ⁱ	2.399 (1)	N—C(2)	1.138 (4)
C(1)—C(2)	1.448 (4)		
Cl—Cu—N	120.0 (1)	Cl—Cu—Cl ⁱ	100.7 (1)
N—Cu—Cl ⁱ	113.2 (1)	Cl ⁱ —Cu—Cl ⁱⁱ	107.5 (1)
Cu—Cl—Cu ⁱ	79.3 (1)	Cu ⁱ —Cl—Cu ⁱⁱ	107.5 (1)
Cu—N—C(2)	175.8 (2)	N—C(2)—C(1)	179.5 (3)

Symmetry code: (i) $2 - x$, $0.5 + y$, $1 - z$; (ii) $2 - x$, $-0.5 + y$, $1 - z$.

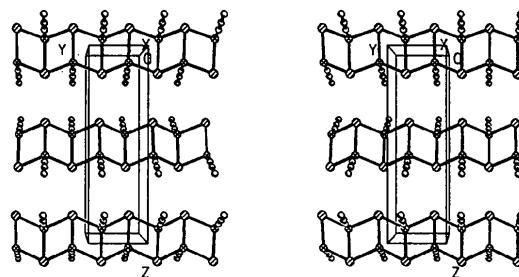


Fig. 1. Stereographic packing diagram of the title compound. Radii are arbitrary.

atom, but the U_{22} component [0.0382 (2) Å²] is not unusually large. It is, however, unlikely that X-ray data, even at low temperature, can absolutely rule out the lower symmetry, particularly if only a small deviation from centrosymmetry is postulated. The structures of Cu¹ polymers are discussed by Wells (1984); pleated sheets, as in the current structure, are a common motif.

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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 54884 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0086]