to recrystallize the pigments from other solvents tended to produce multiply twinned crystals. Densities of the crystals were measured by flotation in mixtures of either carbon tetrachloride and bromoform or carbon tetrachloride and benzene. Estimated accuracy was ±0·003 g.cm^{-3}. The calculated densities are given for comparison. The crystal classes were deduced from Laue photographs. Oscillation, zero, first, second and third layer Weissenberg photographs were taken about two crystallographic axes for each crystal with filtered Cu $K\alpha$ radiation. From these photographs the systematic absences and cell dimensions were obtained. As no corrections were applied for film shrinkage, the cell dimensions, with one exception, are probably accurate to ±1% and the angles to ±2°.

In the case of C. I. Pigment Yellow 4, which crystallizes in the triclinic class, the cell dimensions were obtained using Buerger's (1942) method of level offsets.

In view of the small offsets measured, there was some doubt concerning the accuracy of some of the results.

As a consequence low-angle diffraction lines from the powder pattern of the pigment, which could be indexed unambiguously, were used in conjunction with the program *CEDI* (by courtesy of D. Williams, Dept. of Chemistry, Imperial College, London), whereby the cell dimensions and an estimate of their errors were obtained. These are given for comparison.

During the course of the work the structure of C. I. Pigment Yellow 1 (Hansa *G*) was determined independently by Mez (1968). The unit cell chosen by him is different from the one obtained in this present investigation, but the two unit cells are equivalent. In this investigation the *a* axis chosen was that giving the most prominent row of pinacoidal spots on the first layer Weissenberg photograph and for this reason the present cell is preferred.

Further work is proceeding on the crystal structure determinations of Hansa 10*G*, its mono-bromo analogue and C. I. Pigment Red 2.

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The structure of the potassium salt of μ_4-oxo-hexa-μ-chloro-tetra[chlorocuprate(II)], $K_4Cu_4OCl_{10}$.

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$K_4Cu_4OCl_{10}$ crystallizes in space group $C2/c$ with two molecules in the unit cell having dimensions a = 14·70 (1), b = 14·88 (1), c = 8·95 (1) Å, β = 104·74 (5)°. The structure has been refined by full-matrix least-squares techniques to a final R value of 6·8%.

Crystals of potassium μ_4-oxo-hexa-μ-chloro-tetra[chlorocuprate(II)], $K_4Cu_4OCl_{10}$, can be prepared by slowly cooling a melt of K_2CuCl_4 and CuCl under oxygen. The anion $[Cu_4OCl_{10}]^{4-}$ of this potassium salt occurs also in the tetramethylammonium salt (Bertrand & Kelley, 1969) and is analogous to the central unit in the complexes $Cu_4OCl_6L_4$ with L = triphenylphosphine (Bertrand, 1967), pyridine (Kilbourn & Dunitz, 1967) and methylpyridine (Gill & Sterns, 1970).

The crystals are irregularly shaped and orange-red. Precession photographs showed the extinction rules hkl: $h+k=2n$, $h0l$: $l=2n$; the true space group, $C2/c$, followed from the refinement. A least-squares fit for several θ, $-\theta$ values measured on a Nonius three-circle diffractometer resulted in the following cell parameters: a = 14·70 (1), b = 14·88 (1), c = 8·95 (1) Å, β = 104·74 (5)°. On the diffractometer we measured 544 unique reflexions with intensities significantly above background and with $\sin\theta/\lambda \le 0{\cdot}48$. We used a θ, 2θ scan and Zr-filtered Mo K radiation.

The structure was solved from the three-dimensional Patterson function. Scattering factors and the corrections for anomalous scattering were taken from *International Tables for X-ray Crystallography* (1962). Full-matrix least-squares refinement with isotropic vibration parameters resulted in a final conventional R value (based on F) of 6·8% the weighted R value $\{\sum w(|F_o|-|F_c|)^2/\sum|wF_o|^2\}^{1/2}$ was 8·9%.

The final atomic parameters with their estimated standard deviations are given in Table 1. A listing of $|F_o|$ and $|F_c|$ may be obtained on request. The atomic numbering scheme and the bond distances are given in Fig. 1. The other important structural parameters, together with their e.s.d.'s, are listed in Table 2. The bond distances and angles are all comparable with those in the analogous compounds mentioned above. The asymmetric unit consists of half a molecule, the second half is generated by the crystallographic twofold rotation axis which passes through Cl(3), O, Cl(4).

Table 1. $K_4Cu_4OCl_{10}$, *final least-squares parameters*

	Fractional coordinates			
	x	y	z	B
O	0	0·2168 (14)*	$-\frac{1}{4}$	1·3 (4)
Cu(1)	0·0614 (2)	0·2914 (2)	−0·3680 (3)	1·82 (7)
Cu(2)	0·0921 (2)	0·1413 (2)	−0·1149 (3)	2·08 (8)
Cl(1)	0·1956 (4)	0·2042 (5)	−0·2742 (7)	2·5 (1)
Cl(2)	−0·0521 (4)	0·2249 (5)	−0·5848 (7)	2·5 (1)
Cl(3)	0	0·4152 (7)	$-\frac{1}{4}$	2·7 (2)
Cl(4)	0	0·0198 (8)	$-\frac{1}{4}$	4·3 (3)
Cl(5)	0·1214 (4)	0·3820 (5)	−0·5196 (8)	2·5 (1)
Cl(6)	0·2061 (4)	0·0598 (5)	0·0423 (7)	2·3 (1)
K(1)	0·1590 (4)	0·1295 (4)	0·3758 (7)	2·8 (1)
K(2)	0·1315 (4)	0·4271 (4)	0·1401 (7)	3·0 (1)

* Numbers in parentheses here and elsewhere are the estimated standard deviations in the least significant digit. None are given for parameters fixed by symmetry.

Table 2. $K_4Cu_4OCl_{10}$ *bond lengths* (Å) *and angles* (°)

Copper environment

Cu(1)		Cu(2)	
Cu(1)-O	1·909 (12)	Cu(2)-O	1·930 (12)
Cu(1)-Cl(1)	2·334 (7)	Cu(2)-Cl(1)	2·517 (7)
Cu(1)-Cl(2)	2·424 (7)	Cu(2)-Cl(2)	2·370 (8)
Cu(1)-Cl(3)	2·409 (8)	Cu(2)-Cl(4)	2·395 (9)
Cu(1)-Cl(5)	2·247 (7)	Cu(2)-Cl(6)	2·249 (7)
O——Cu(1)-Cl(1)	87·4 (4)	O——Cu(2)-Cl(1)	81·9 (3)
O——Cu(1)-Cl(2)	83·1 (3)	O——Cu(2)-Cl(2)	84·1 (3)
O——Cu(1)-Cl(3)	85·4 (5)	O——Cu(2)-Cl(4)	84·6 (6)
O——Cu(1)-Cl(5)	175·1 (3)	O——Cu(2)-Cl(6)	176·5 (5)
Cl(5) -Cu(1)-Cl(1)	97·1 (3)	Cl(6) -Cu(2)-Cl(1)	95·3 (2)
Cl(5) -Cu(1)-Cl(2)	93·3 (3)	Cl(6) -Cu(2)-Cl(2)	95·7 (3)
Cl(5) -Cu(1)-Cl(3)	93·1 (3)	Cl(6) -Cu(2)-Cl(4)	98·3 (3)
Cl(1) -Cu(1)-Cl(2)	114·1 (3)	Cl(1) -Cu(2)-Cl(2)	122·3 (3)
Cl(1) -Cu(1)-Cl(3)	130·1 (2)	Cl(1) -Cu(2)-Cl(4)	109·9 (2)
Cl(2) -Cu(1)-Cl(3)	113·9 (2)	Cl(2) -Cu(2)-Cl(4)	124·0 (2)

Oxygen environment		Non-bonded Cu···Cu distances	
Cu(1)-O-Cu(1′)	108·9 (1)	Cu(1)···Cu(1′)	3·107 (5)
Cu(1)-O-Cu(2)	109·2 (1)	Cu(1)···Cu(2)	3·130 (5)
Cu(1)-O-Cu(2′)	110·3 (1)	Cu(1)···Cu(2′)	3·150 (5)
Cu(2)-O-Cu(2′)	108·9 (1)	Cu(2)···Cu(2′)	3·139 (5)

Chlorine (bridge) environment		Non-bonded Cl···Cl distances	
Cu(1)-Cl(1)-Cu(2)	80·2 (2)	Cl(1)···Cl(2)	4·00 (1)
Cu(1)-Cl(2)-Cu(2′)	82·1 (2)	Cl(1)···Cl(3)	4·30 (1)
Cu(1)-Cl(3)-Cu(1′)	80·3 (2)	Cl(1)···Cl(4)	4·02 (1)
Cu(2)-Cl(4)-Cu(2′)	81·9 (2)	Cl(2)···Cl(1′)	4·28 (1)
		Cl(2)···Cl(3)	4·05 (1)
		Cl(2)···Cl(4)	4·21 (1)
		Cl(1)···Cl(5)	3·43 (1)
		Cl(1)···Cl(6)	3·53 (1)
		Cl(2)···Cl(5)	3·40 (1)
		Cl(2)···Cl(6)	3·43 (1)

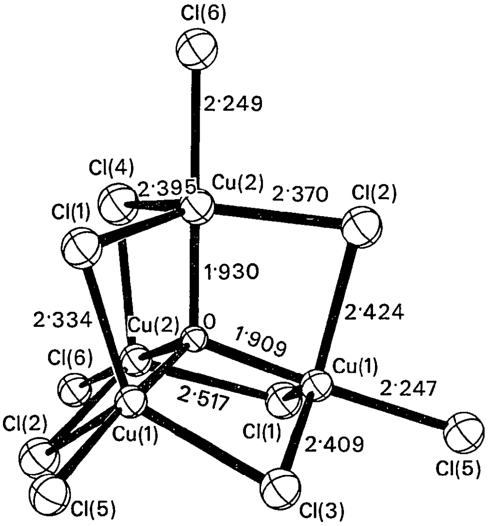

Fig. 1. The $[Cu_4OCl_{10}]^{4-}$ ion.

The tetrahedron formed by the Cu^{2+} ions and the oxygen in the centre is somewhat irregular [Cu–O–Cu: 108·9 (1)°–110·3 (1)°], as is the octahedron formed by the bridging chlorines [Cu–Cl–Cu: 80·2 (2)°–82·1 (2)°]. The displacements of Cu(1) and Cu(2) out of the plane of the nearest bridging Cl ions are 0·19 and 0·23 Å, respectively. The deviation from trigonal symmetry around the O–Cu axis is shown by the Cl(bridge)–Cu–Cl′(bridge) angles, which range from 109·9 (2) to 130·1 (2)°.

The potassium ions are in the open spaces between the anions. The shortest K–Cl distance of 3·1 Å compares well with the distance in solid KCl (3·138 Å).

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