

The crystal structure of caratiite

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ABSTRACT. The crystal structure of caratiite, $K_4Cu_4O_2(SO_4)_4MeCl$ (a 13.60(2), c 4.98(1) Å, $Z = 2$) was solved in space group $I4$ from single crystal data, and refined to $R = 0.035$ for $654F_o > 6\sigma(F_o)$. The atomic arrangement is characterized by $Cu_2O(SO_4)_2$ chains parallel to $[001]$, with Cu in $(4+1)$ co-ordination and O surrounded tetrahedrally by four Cu atoms. These chains are interconnected by K atoms to a framework, the channels of which house the *Me* and Cl atoms. The nature of the *Me* atoms is not completely clear; most probably the position is half occupied by Cu, fully occupied by Na, or occupied by a mixture of the two.

CARATIITE is a naturally occurring sulphate-chloride of copper and potassium described elsewhere (Clark *et al.*, 1984) on lava from Mount Vesuvius, Naples, Italy. Clark *et al.* found a tetragonal body-centred cell with a 13.60(2) and c 4.98(1) Å. This structure determination has been carried out by single crystal X-ray methods on part of the type material, kindly provided by the authors of the original description.

Experimental. Preliminary work confirmed the crystal system, lattice constants and centring of the cell published by Clark *et al.* (1984). The determination of the Laue symmetry was, however, unexpectedly difficult. Most of the apparently single crystals showed Laue symmetry $4/mmm$, with only $(h+k+l = \text{odd})$ systematic absences characteristic of a body-centred cell. Trial solutions from a three-dimensional data set collected from one such grain failed in all five characteristic space groups $I4/mmm$, $I422$, $I4mm$, $I\bar{4}2m$, and $I\bar{4}m2$. The subsequent investigation of approximately 20 'single crystals' by Weissenberg photographs revealed that the apparent Laue symmetry was caused by coaxial twinning; several grains showed deviations from this Laue symmetry towards $4/m$. Finally a single crystal was found, evidence of its untwinned nature being obtained from the lack of observable $\bar{h}kl$ counterparts to several strong hkl reflections. As the only systematic extinction rule was again that of a body-centred cell, the possible space groups were $I4/m$, $I\bar{4}$, and $I4$. The dimensions of this grain were $0.08 \times 0.08 \times 0.27$ mm³. Unit cell data are given in Table I.

The crystal was mounted parallel to $[001]$ on an automatic Stoe-Stadi-2 two-circle diffractometer. A total of 1234 reflections was recorded up to $\sin \theta/\lambda = 0.70 \text{ \AA}^{-1}$ (Mo- $K\alpha$ radiation, graphite monochromator, ω -scan). The data were corrected for absorption resulting from the crystal shape, as well as for Lorentz and polarization effects (program SHELX-76; Sheldrick, 1976). Symmetry-related reflections were averaged, resulting in a set of 706 unique reflections, of which 654 with $F_o > 6\sigma(F_o)$ were used in the structure refinement.

TABLE I. Crystal data of caratiite

Type material: Mt. Vesuvius, Naples (Italy)	
$a = 13.60(2) \text{ \AA}$	$c/a = 0.366$
$c = 4.98(1) \text{ \AA}$	$V = 921.1 \text{ \AA}^3$
Space group $I4-C_2^2$ (no. 79)	
$Z = 2\{K_4[Cu_4O_2(SO_4)_4]MeCl\}$ (for <i>Me</i> cf. text)	
$D_x = 2.76 \text{ g cm}^{-3}$	
$\mu(\text{Mo-}K\alpha) = 67.3 \text{ cm}^{-1}$ *	

* Based on $Me = Cu_{0.5}$.

For the structure determination a three-dimensional Patterson synthesis was carried out. A solution was obtained in space group $I4$ for the atoms Cu, K, and S. A subsequent Fourier summation revealed the positions of all the oxygen atoms and of both the atoms *Me* and Cl. The peak height of *Me* corresponded to an atom with atomic number near 14; in the refinement the position was treated as half-occupied by Cu (for a more detailed discussion see below). During the final stages of the least-squares refinement (program SFLS-5; Prewitt, 1966) the atomic coordinates and the anisotropic temperature factors were included in the list of variables, together with the isotropic extinction factor of Zachariasen (1967). The largest correction factor for F_c was 0.84 (110 reflection). Complex scattering factors were taken from the

TABLE II. Structural parameters of caratiite

Atom	Wyckoff position	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B_{equiv}
K	8(c)	0.1905(1)	0.1279(1)	-0.0379(6)	19(1)	30(1)	222(9)	2(1)	5(3)	15(3)	1.92
Cu	8(c)	0.05627(6)	0.40789(6)	0.00000†	15(1)	15(1)	116(4)	2(1)	-1(2)	3(2)	1.12
O	4(b)	0.0	0.5	0.7572(23)	17(6)	16(6)	93(27)	-1(6)	0	0	1.11
S	8(c)	0.4426(1)	0.2347(1)	-0.0179(6)	19(1)	12(1)	118(8)	2(1)	-3(3)	8(3)	1.14
O _s (1)	8(c)	0.4230(6)	0.2103(5)	0.2705(15)	44(4)	19(3)	133(28)	5(3)	5(9)	3(8)	1.99
O _s (2)	8(c)	0.3807(5)	0.1706(5)	-0.1884(18)	18(3)	36(4)	246(29)	-1(3)	5(9)	-40(10)	2.12
O _s (3)	8(c)	0.5456(4)	0.2157(4)	-0.0778(17)	18(3)	18(3)	354(40)	1(2)	4(9)	8(8)	2.03
O _s (4)	8(c)	0.4158(5)	0.3369(4)	-0.0531(20)	41(4)	15(3)	346(44)	11(3)	4(11)	25(10)	2.52
Me	2(a)	0.0	0.0	0.4482(12)	45(2)	β_{11}	188(26)	0	0	0	2.85
Cl	2(a)	0.0	0.0	-0.0513(18)	26(1)	β_{11}	579(43)	0	0	0	3.15

ATF = $\exp \left[- \sum_{i=1}^3 \sum_{j=1}^3 h_i h_j \beta_{ij} \right]$. $B_{\text{equiv.}} = 4/3 \left[\sum_{i=1}^3 \beta_{11}/a_i^{*2} \right]$. The β_{ij} were multiplied by 10^4 . E.S.D.s are given in parentheses.

† Invariant, fixes the origin.

'International Tables for X-ray Crystallography' (1974). For the 654 reflections with $F_o > 6\sigma(F_o)$ the refinement converged to $R = 0.035$, and for all 706 reflections to $R = 0.039$. Structure factor lists have been deposited at the Institut für Mineralogie und Kristallographie, Universität Wien, and with the Mineralogy Library, British Museum (Natural History), and are available on request.

Table II gives the atomic coordinates, anisotropic thermal vibration factors, and the equivalent isotropic B values. Interatomic distances, bond angles and selected bond valence sums are compiled in Tables III and IV. Table V gives the root-mean-square amplitudes of the thermal ellipsoids.

Discussion. The most novel features of the atomic arrangement are prominent $\text{Cu}_2\text{O}(\text{SO}_4)_2$ chains with symmetry 4_2 , the central parts of which consist of strings of edge-sharing OCu_4 tetrahedra with Cu-O c. 1.92 Å and Cu-O-Cu angles ranging from 98 to 115° (fig. 1). The dimensions of the OCu_4 tetrahedron are similar to those found in dolerophane, $\text{Cu}_2\text{O}(\text{SO}_4)$, in which, however, only two such tetrahedra share a common edge (Flügel-Kahler, 1963). Tetrahedral co-ordination of oxygen to four Cu atoms would not agree with the results obtained from other chemical compounds, if the oxygens were also linked to hydrogen as in hydroxyl groups or water molecules (e.g. Zemann, 1961 and 1972). The bond valence sum (Brown and Wu, 1976) of 2.03 for the oxygen under discussion also indicates that it is indeed an oxo-oxygen (Table IV).

The Cu atoms are surrounded at distances of 1.90–2.07 Å by two O and by two O_s atoms of different sulphate groups in the same chain (average O-Cu-O angle = 87.3°). The co-ordination is

approximately square planar, characteristic of divalent copper (cf. Wells, 1949 and 1962; Zemann, 1961 and 1972). A fifth oxygen atom, from a third sulphate group (again in the same chain), with Cu-O_s = 2.29 Å, completes a (4 + 1) co-ordination,

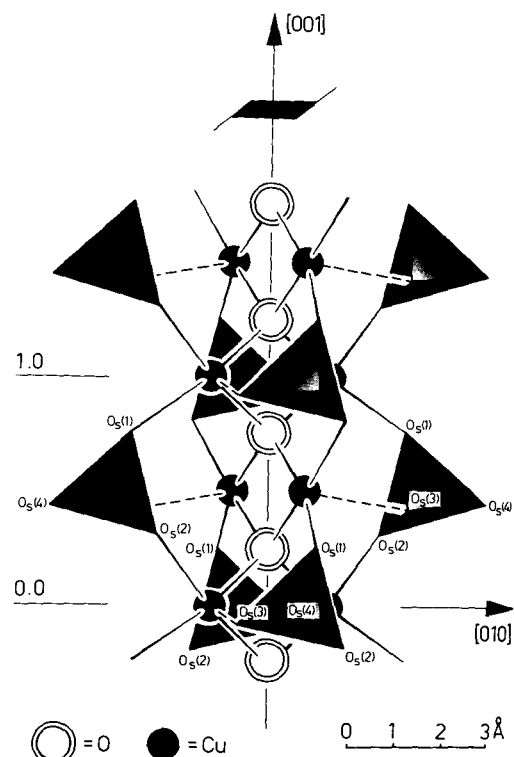


FIG. 1. Caratiite: diagram of the $\text{Cu}_2\text{O}(\text{SO}_4)_2$ chain.

TABLE III. *Interatomic distances (in Å) and angles (in °) with E.S.D.s in parentheses for caratiite*

Cation-anion distance		Ligand A	Ligand B	Distance A-B	Angle cation A-B
K ¹ -O _s (1) ⁸	= 2.852(7)	O _s (1) ⁸	O _s (4) ⁷	3.784(12)	83.1(2)
K ¹ -O _s (1) ¹⁹	= 3.240(8)	O _s (1) ⁸	O _s (4) ⁸	2.360(11) ^c	47.7(3)
K ¹ -O _s (2) ¹	= 2.755(7)	O _s (1) ¹⁹	O _s (2) ¹	3.593(11)	73.2(2)
K ¹ -O _s (3) ¹⁸	= 2.851(7)	O _s (1) ¹⁹	O _s (3) ¹⁸	3.649(11)	73.3(2)
K ¹ -O _s (3) ¹⁹	= 3.180(8)	O _s (1) ¹⁹	O _s (3) ¹⁹	2.407(11) ^c	44.0(3)
K ¹ -O _s (4) ⁷	= 2.854(9)	O _s (2) ¹	O _s (3) ¹⁸	3.722(11)	83.2(2)
K ¹ -O _s (4) ⁸	= 2.984(9)	O _s (2) ¹	O _s (3) ¹⁹	2.893(10) ^b	57.8(3)
K ¹ -Cl ¹	= 3.121(1)	O _s (3) ¹⁸	O _s (4) ⁷	3.158(8) ^a	67.2(3)
		O _s (3) ¹⁹	O _s (4) ⁸	3.158(8) ^a	61.6(3)
		O _s (4) ⁷	Cl ¹	3.520(10) ^d	72.0(2)
		O _s (4) ⁸	Cl ¹	3.532(10) ^d	70.7(2)
Cu ¹ -O ³	= 1.902(7)	O ³	O ¹⁷	2.490(23) ^e	80.6(5)
Cu ¹ -O ¹⁷	= 1.948(7)	O ³	O _s (1) ⁸	3.046(7)	102.9(3)
Cu ¹ -O _s (1) ⁸	= 1.992(7)	O ³	O _s (2) ⁷	3.955(11)	169.5(4)
Cu ¹ -O _s (2) ⁷	= 2.069(8)	O ³	O _s (3) ¹⁰	3.109(6)	95.3(2)
Cu ¹ -O _s (3) ¹⁰	= 2.292(5)	O ¹⁷	O _s (1) ⁸	3.892(10)	162.1(4)
		O ¹⁷	O _s (2) ⁷	2.844(7)	90.1(3)
		O ¹⁷	O _s (3) ¹⁰	3.431(8)	107.8(2)
		O _s (1) ⁸	O _s (2) ⁷	2.808(12)	87.4(3)
		O _s (1) ⁸	O _s (3) ¹⁰	3.024(9)	89.5(3)
		O _s (2) ⁷	O _s (3) ¹⁰	2.893(10) ^b	83.0(3)
S ¹ -O _s (1) ¹	= 1.498(8)	O _s (1) ¹	O _s (2) ¹	2.418(12)	108.6(5)
S ¹ -O _s (2) ¹	= 1.480(7)	O _s (1) ¹	O _s (3) ¹	2.407(11) ^c	109.2(5)
S ¹ -O _s (3) ¹	= 1.455(5)	O _s (1) ¹	O _s (4) ¹	2.360(11) ^c	106.5(5)
S ¹ -O _s (4) ¹	= 1.448(5)	O _s (2) ¹	O _s (3) ¹	2.389(9)	109.0(4)
		O _s (2) ¹	O _s (4) ¹	2.408(9)	110.7(4)
		O _s (3) ¹	O _s (4) ¹	2.418(8)	112.8(4)
Me ¹ -O _s (4) ^{4, 7, 13, 18}	= 2.496(5)	O _s (4) ^{4, 7}	O _s (4) ^{13, 18}	3.530(12)	90.0(3)
Me ¹ -Cl ¹	= 2.488(10)	O _s (4) ^{4, 7, 13, 18}	Cl ¹	3.520(10) ^d	89.9(3)
Me ¹ -Cl ²	= 2.492(10)	O _s (4) ^{4, 7, 13, 18}	Cl ²	3.532(10) ^d	90.2(3)

Common edges of co-ordination polyhedra are indicated: ^a two K atoms; ^b one K and one Cu atom; ^c one K and one S atom; ^d one K and one Me atom; and ^e two Cu atoms.

Edges of co-ordination polyhedra are only given up to 4 Å. The transformation of the atomic parameters given in Table II is as follows: 1 x, y, z ; 2 $x, y, 1-z$; 3 $x, y, -1+z$; 4 $-1/2+x, -1/2+y, 1/2+z$; 5 $-x, -y, z$; 6 $-x, 1-y, 1+z$; 7 $1/2-x, 1/2-y, 1/2+z$; 8 $1/2-x, 1/2-y, -1/2+z$; 9 $y, -x, z$; 10 $y, 1-x, z$; 11 $1/2+y, 1/2-x, 1/2+z$; 12 $1/2+y, 1/2-x, -1/2+z$; 13 $-1/2+y, 1/2-x, 1/2+z$; 14 $-y, x, z$; 15 $1-y, x, z$; 16 $1/2-y, 1/2+x, 1/2+z$; 17 $1/2-y, 1/2+x, -1/2+z$; 18 $1/2-y, -1/2+x, 1/2+z$; 19 $1/2-y, -1/2+x, -1/2+z$.

also a well-known feature in the stereochemistry of copper (cf. Zemann, 1961 and 1972; Wells, 1962). The five oxygens form a somewhat distorted tetragonal pyramid, the Cu atom being slightly shifted from the centre of the basal square towards the interior of the pyramid. The sixth neighbour, a Cu atom, occurs at a distance of 2.94 Å; this distance represents the common Cu-Cu edges in the central Cu-O string.

The interatomic distances in the sulphate group are the typical values found by other workers (cf. Baur, 1964; Wuensch, 1972).

The Cu₂O(SO₄)₂ chains are interconnected by potassium atoms to form an electrostatically neutral three-dimensional arrangement of formula K₂[Cu₂O(SO₄)₂] which contains channels parallel to [001] (fig. 2). Each K atom connects two copper oxysulphate chains only. As is often the case in

TABLE IV. The co-ordination of the oxygen and chlorine atoms in caratiite with E.S.D.s in parentheses

Co-ordination of the oxygen and chlorine atoms	s	Angles at the oxygen and chlorine atoms
O ¹ -Cu ^{2,6} = 1.902(7)	0.54	Cu ² ,Cu ⁶ 101.0(6)
O ¹ -Cu ^{13,16} = 1.948(7)	0.47	Cu ^{2,6} ,Cu ^{13,16} 114.7(1)
		Cu ¹³ ,Cu ¹⁶ 97.8(6)
	$\Sigma = 2.03$	
O _s (1) ¹ -K ⁷ = 2.852(7)	0.13	K ⁷ ,K ¹¹ 92.6(3)
O _s (1) ¹ -K ¹¹ = 3.240(8)	0.04	K ⁷ ,Cu ⁷ 120.5(2)
O _s (1) ¹ -Cu ⁷ = 1.992(7)	0.41	K ⁷ ,S ¹ 104.3(3)
O _s (1) ¹ -S ¹ = 1.498(8)	1.47	K ¹¹ ,Cu ⁷ 92.6(3)
		K ¹¹ ,S ¹ 92.1(3)
	$\Sigma = 2.05$	Cu ⁷ ,S ¹ 134.7(3)
O _s (2) ¹ -K ¹ = 2.755(7)	0.18	K ¹ ,Cu ⁸ 119.0(2)
O _s (2) ¹ -Cu ⁸ = 2.069(8)	0.33	K ¹ ,S ¹ 120.2(3)
O _s (2) ¹ -S ¹ = 1.480(7)	1.56	Cu ⁸ ,S ¹ 119.9(3)
	$\Sigma = 2.07$	
O _s (3) ¹ -K ¹¹ = 3.180(8)	0.05	K ¹¹ ,K ¹² 111.2(2)
O _s (3) ¹ -K ¹² = 2.851(7)	0.13	K ¹¹ ,Cu ¹⁵ 98.0(2)
O _s (3) ¹ -Cu ¹⁵ = 2.292(5)	0.18	K ¹¹ ,S ¹ 95.4(4)
O _s (3) ¹ -S ¹ = 1.455(5)	1.68	K ¹² ,Cu ¹⁵ 116.8(2)
		K ¹² ,S ¹ 117.6(3)
	$\Sigma = 2.04$	Cu ¹⁵ ,S ¹ 113.5(3)
O _s (4) ¹ -K ⁷ = 2.984(9)	0.09	K ⁷ ,K ⁸ 117.1(2)
O _s (4) ¹ -K ⁸ = 2.854(5)	0.13	K ⁷ ,S ¹ 99.9(4)
O _s (4) ¹ -S ¹ = 1.448(5)	1.72	K ⁷ ,Me ⁸ 94.4(3)
O _s (4) ¹ -Me ⁸ = 2.496(5)	0.11	K ⁸ ,S ¹ 113.0(4)
		K ⁸ ,Me ⁸ 94.9(3)
	$\Sigma = 2.05$	S ¹ ,Me ⁸ 137.5(3)
Cl ¹ -K ^{1,5,9,14} = 3.121(1)		K ^{1,5} ,K ^{9,14} 90.0(1)
Cl ¹ -Me ¹ = 2.488(10)		K ^{1,5,9,14} ,Me ¹ 88.8(2)
Cl ¹ -Me ³ = 2.492(10)		K ^{1,5,9,14} ,Me ³ 91.2(2)

The bond valences *s* for the oxygen atoms were calculated by the method of Brown and Wu (1976). The transformation of the atomic parameters is according to Table III.

crystal structure determinations, the co-ordination around the potassium is not clear cut (cf. Cocco *et al.*, 1972). Table III gives the seven shortest K-O_s distances, up to K-O_s = 3.24 Å. This limit is arbitrary—an eighth neighbour with K-O_s = 3.38 Å suggests a larger limit, whereas the K-Cl bond of 3.12 Å, as well as the ionic radii of K⁺ and O²⁻, indicate a lower limit, e.g. *c.* 2.9 Å.

The channels in this atomic arrangement are not empty but house one Cl and one Me atom per K₄[Cu₄O₂(SO₄)₄]. Although the least-squares refinement gave a somewhat high overall temperature parameter for Cl (Table II), and although the

anisotropy of the thermal vibration ellipsoid of Cl is somewhat large (Tables II and V), there seems to be no doubt that any deviation from a full occupancy of this position by Cl can be, at the most, minor.

The Me atom is more problematic. Information from the X-ray analysis indicates that the site is occupied by an atom with (possibly average) atomic number 14 ± 3, and a not-too-rapid decrease in scattering power with increasing Bragg angle. Again, the overall temperature parameter and the ellipsoid of thermal vibration are somewhat large (Tables II and V). Combining the X-ray data with the electron probe analyses of Clark *et al.* (1984),

TABLE V. The root-mean-square amplitudes of the anisotropic thermal ellipsoids (in Å) for caratiite and their orientation with respect to the reciprocal lattice (in °)

Atom	r.m.s.	Angles with			Atom	r.m.s.	Angles with		
		a_1^*	a_2^*	c^*			a_1^*	a_2^*	c^*
K	0.13	9.2	97.2	95.8	S	0.14	14.0	77.9	96.9
	0.18	80.8	45.1	46.4		0.10	104.0	31.5	117.6
	0.15	90.9	134.2	44.2		0.13	89.7	61.5	28.6
Cu	0.11	49.2	136.7	102.1	O _s (1)	0.21	11.8	79.0	85.8
	0.13	54.2	48.7	118.1		0.13	101.2	40.3	51.9
	0.12	61.2	79.4	31.0		0.13	86.5	128.2	38.4
O	0.13	31.7	121.7	90.0	O _s (2)	0.13	18.4	99.4	105.6
	0.12	58.3	31.7	90.0		0.22	93.6	42.3	132.0
	0.11	90.0	90.0	0.0		0.14	72.0	49.3	46.2
Me	0.21	0.0	90.0	90.0	O _s (3)	0.13	42.2	47.8	88.2
	0.21	90.0	0.0	90.0		0.13	132.1	42.7	84.2
	0.15	90.0	90.0	0.0		0.21	87.4	95.5	6.1
Cl	0.16	0.0	90.0	90.0	O _s (4)	0.20	35.3	81.8	124.1
	0.16	90.0	0.0	90.0		0.09	108.1	22.2	102.5
	0.27	90.0	90.0	0.0		0.22	60.8	69.5	36.9

in good agreement with values obtained in this department (analyst F. Kluger), suggests that we are dealing with a half-occupied Cu position. However, the octahedral co-ordination of Me by four O_s and two Cl atoms, all at a distance of c. 2.50 Å, cannot be easily reconciled with the accepted stereochemical behaviour of Cu(II): one would rather expect a square planar co-ordination either by two Cl at c. 2.3 Å and two O_s at c. 2.0 Å or by four O_s at c. 2.0 Å (in both cases with two further neighbours to complete the (4+2) co-ordination).

In view of this difficulty, the role of Na should be considered. One interpretation is that Na substitutes in minor amounts on the K position. Another possibility is that there is a relative error of c. 10% in the Cu determination and that the Me position is fully occupied by Na. This interpretation agrees with the X-ray diffraction results but, unfortunately, again leads to discrepancies with observed behaviour in solid-state chemistry, because the Na atoms would have two Cl neighbours at a distance of 2.50 Å only. We have to admit, therefore, that a fully satisfactory interpretation of the Me position cannot be given at present. Such difficulties are, however, very common with atoms in crystal structure channels. Possibly the atoms of Cl and Me are not located on exactly the positions given here, but show statistical deviations with shifts of a

few tenths of an ångström. Further understanding of the atomic arrangement of caratiite must await the availability of more ample single crystal material.

Acknowledgements. We thank the authors of the original description of caratiite for kindly providing the working material, as well as for information prior to publication; also M. F. Kluger (Institut für Analytische Chemie der Universität Wien) for the microprobe analysis. Computing time was provided by 'Interuniversitäres Rechenzentrum Wien'. Dr A. M. Clark, London, has revised the manuscript.

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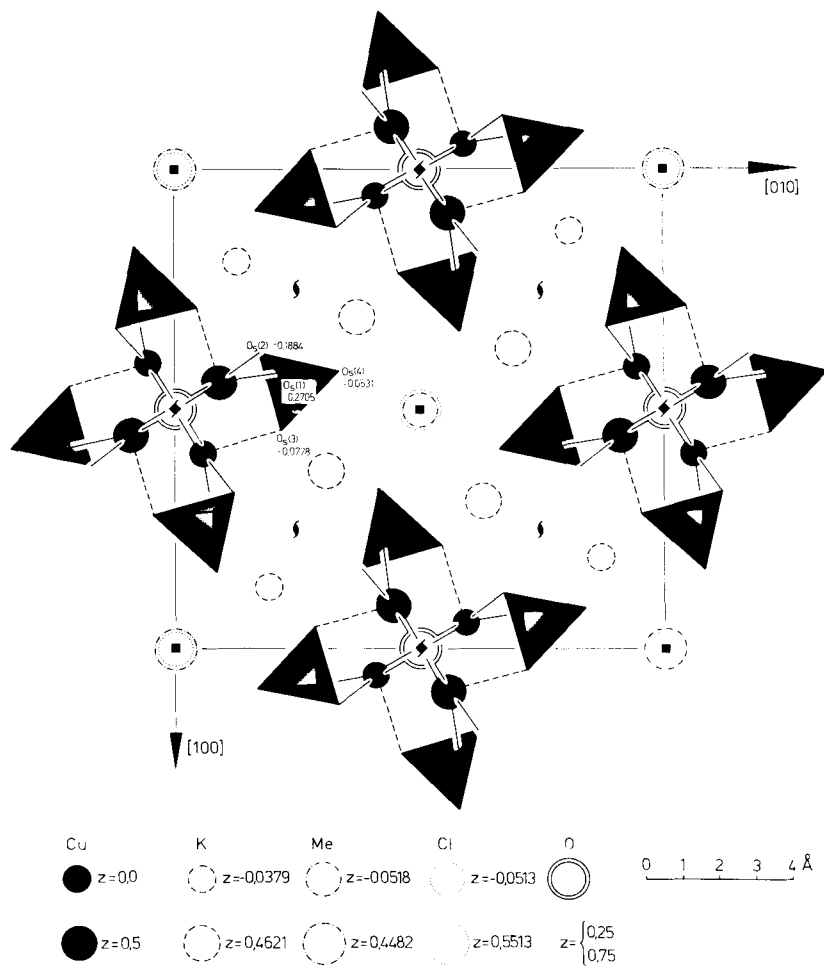


FIG. 2. Caratiite: projection of the structure parallel to [001].

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[Manuscript received 20 April 1984;
revised 29 June 1984]