The Crystal Structure of $CuSe_2Cl$

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Copper Chalcogen Halides, Crystal Structure of $CuSe_2Cl$, X-Ray

The crystal structure of $CuSe_2Cl$ has been determined by single-crystal X-ray diffraction. $CuSe_2Cl$ crystallizes monoclinically in space group $P2_1/c$ with the lattice parameters a = 768.3(9) pm, b = 462.5(5) pm and c = 1455.2(3) pm, β = 135.2(4)°, Z = 4. The structure could be shown to be isotypic with $CuTe_2Cl$.

$CuSe_2Cl$ was synthesized under hydrothermal conditions from hydrogen chloride solution [1]. The crystals are black needles of 5–10 mm length with metallic lustre. The reflection intensities up to 2θ = 60° were measured on a Philips fourcircle diffractometer PW 1100 (graphite-monochromator, MoK_α-radiation) in the ω-scan mode. The ratio of measuring times for background to peak intensity was 0.5. The intensity of two reference reflections remained constant during the data collection. The structure was solved by applying direct methods (SHELXS 86 [2]). An E-Map showed two outstanding peaks which were interpreted as the atoms Se1 and Se2. A subsequent calculated Fourier synthesis yielded the Cu and the Cl atom. On the basis of the best isotropic model the reflection data were corrected for absorption by the program DIFABS [3]. The final refinement with anisotropic temperature factors and 668 unique reflections converged at R_w = 0.067 (Table I).

The crystal structure of $CuSe_2Cl$ is built up of pseudo-fourfold screws of Se atoms along b. The screws are connected by Cu–Cl–Cu links. Each copper atom is tetra-coordinated, with the four ligands Se1, Se2 and two Cl forming a slightly distorted tetrahedron (Fig. 1).

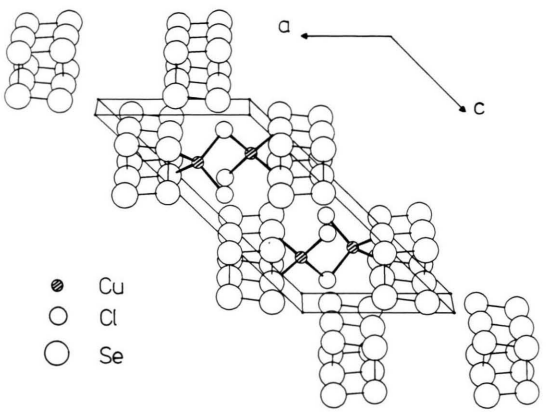

Fig. 1. Crystal structure of $CuSe_2Cl$, view down b.

Table I. Data pertaining to the structure determination of $CuSe_2Cl$.

Lattice parameters	a = 768.3(9) pm	b = 462.5(5) pm	c = 1455.2(3) pm	β = 135.2(4)°
Space group		$P2_1/c$		
Z		4		
Atomic positions	x	y	z	U_{eq}
Cu at 4(e)	.6769(4)	.1400(6)	.2526(3)	512(22)
Cl at 4(e)	.6898(8)	.8880(12)	.3932(5)	477(35)
Se1 at 4(e)	.9980(3)	.4965(5)	.3494(2)	399(14)
Se2 at 4(e)	.2644(3)	.2846(5)	.3444(2)	420(14)
Theta range		$2° \leq \theta \leq 30°$		
Measured sections of reciprocal space		whole sphere		
Number of measured reflections		3039		
Number of unique reflections		668		
R_w-Value		0.067		
μ(Mo)		257.7 cm^{-1}		
Calculated density ϱ		4.70 $g \cdot cm^{-3}$		

U_{eq} is given in pm^2.

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$CuSe_2Cl$ forms a connecting link between the crystal chemistry of Te and that of Se in the known copper tellurium and copper selenium halides [4–8]. While these compounds contain either screws of tellurium or six-membered rings of selenium, $CuSe_2Cl$ is the first selenium compound built up of screws of Se. It is isotypic with $CuTe_2Cl$ [4]. The Se–Se distances in the screw alternate at 231.6(4) pm and 239.3(3) pm and are comparable to those in elemental Se (237 pm) [9]. The Se–Se–Se angles alternate at 102.9(1)° and at 104.4(1)°.

Additional crystal structure data have been deposited at the Fachinformationszentrum Energie, Physik, Mathematik GmbH, D-7514 Eggenstein-Leopoldshafen 2, FRG. Inquieries should be accompanied by the depository number CSD-52669, the names of the authors, and the literature citation.

[1] A. Rabenau, H. Rau, and G. Rosenstein, Z. Anorg. Allg. Chem. **374,** 43 (1970).
[2] G. M. Sheldrick, SHELXS 86, an integrated system for solving, refining and displaying crystal structures from diffraction data, University of Göttingen (1986).
[3] N. Walker and D. Stuart, Acta Crystallogr. **A 39,** 158 (1983).
[4] J. Fenner, Acta Crystallogr. **B 32,** 3084 (1976).
[5] H. M. Haendler and P. M. Carkner, J. Solid State Chem. **29,** 35 (1979).
[6] J. Fenner and A. Rabenau, Z. Anorg. Allg. Chem. **426,** 7 (1976).
[7] P. M. Carkner and H. M. Haendler, J. Solid State Chem. **18,** 183 (1976).
[8] W. Milius and A. Rabenau, Mater. Res. Bull. **22** (11), 1493 (1987).
[9] P. Cherin and P. Unger, Inorg. Chem. **6,** 1589 (1967).