

Non-Stoichiometric Monoclinic Cr₅Se₈ Prepared at High-Pressure and High-Temperature and the Crystal Structure Refined from Rietveld Data

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Dedicated to Dr. Hj. Mattausch on the occasion of his 60th birthday

The non-stoichiometric chromium selenide Cr_{5.095(5)}Se₈ was prepared under high-pressure high-temperature conditions. The structure was refined from X-ray powder data with the Rietveld method in the non-conventional monoclinic space group $F2/m$, $a = 12.2992(2)$, $b = 7.12753(12)$, $c = 11.4486(2)$ Å, $\beta = 90.927(1)^\circ$ and $V = 1003.49(3)$ Å³. Three of the four unique Cr sites are fully occupied, and one site is only partially occupied. The structure may be viewed as being composed of alternating full and metal deficient layers which are oriented perpendicular to the crystallographic c axis of the pseudo-hexagonal unit cell. All Cr atoms are in an octahedral environment of six Se atoms. The CrSe₆ octahedra of neighbouring layers share common faces whereas the octahedra with layers are joined by common edges. As a result short Cr-Cr distances of 2.867(3) and 2.951(6) Å are found across common faces while Cr-Cr separations between CrSe₆ octahedra sharing edges are significantly longer. From a formal point of view charge balance requires a formulation as Cr⁴⁺_{0.715}Cr³⁺_{4.38}Se₈. On the basis of the distortion of the CrSe₆ octahedra the Cr⁴⁺ ions are mostly located on two different sites.

Key words: Chromium Chalcogenides, High Pressure Synthesis, Rietveld Refinement