Preparation and Crystal Structure of Cs$_4$Nb$_2$S$_{11}$

Kurt O. Klepp*, Gerald Gabl

Department of Inorganic Chemistry, Linz University, Altenbergerstr. 69, A-4040 Linz, Austria

Z. Naturforsch. 53b, 1236–1238 (1998); received April 14, 1998

Chalcogenide, Thioniobate, Polysulfide, Niobium, Cesium

The new polychalcogenide Cs$_4$Nb$_2$S$_{11}$ was prepared from the melt. Cs$_4$Nb$_2$S$_{11}$ is orthorhombic, $oP6_8$, s.g. Pca$_2$$_1$ (No.29), $Z = 4$ with $a = 13.775(9)$ Å, $b = 8.043(9)$ Å, $c = 18.306(5)$ Å. The crystal structure was determined from diffractometer data and refined to a conventional $R$ of 0.052 (1104 Fo’s, 154 variables). It is characterized by asymmetric discrete binuclear moieties [Nb$_2$S$_{11}$]$^{4-}$ which are separated by the alkali cations. Each Nb atom is side-on coordinated by two S$_2^{2-}$ groups, one bridging and one terminal sulfide ligand. Nb-S bond lengths are 2.15(1) Å to 2.22(1) Å (terminal) and 2.44(1)Å to 2.51(1) Å (others). A further longer Nb-S bond (2.86(1)Å and 2.90(1)Å, resp.) expands the coordination of the two crystallographically independent transition metal atoms to distorted pentagonal bipyramidal configurations. The Nb-Nb-distance is 3.517(3) Å. The anionic groups are arranged in hexagonal close packed slabs running parallel to (001). The atomic arrangement corresponds to that of K$_4$Ta$_2$S$_{11}$.

* Reprint requests to Prof. Dr. K. O. Klepp.