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The crystal structure of $Nb_3O_2Cl_5$, an original Nb_3 cluster oxyhalide

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Abstract

The new $Nb_3O_2Cl_5$ oxychloride was synthesized at 700°C in a sealed silica tube from a stoichiometric mixture of $NbCl_5$, Nb_2O_5, and Nb. The crystal structure of this new compound was determined by single crystal X-ray diffraction (*Pnnm*, $a = 8.060(2)$, $b = 14.496(3)$, $c = 6.695(2)$ Å, $V = 782.2(4)$ Å^3, $Z = 4$; $d_{calc} = 4.14$ g/cm^3, $R = 0.036$, $R_\omega = 0.047$). It consists of $[Nb_3(\mu_3\text{-}Cl^i)(\mu_2\text{-}Cl^i)(\mu_3\text{-}O^{i-a})_{2/2}(\mu_3\text{-}O^{a-i})_{2/2}(\mu_2\text{-}Cl^{a-a})_{4/2}(\mu_3\text{-}Cl^{a-a-a})_{3/3}]$ units, in which the Nb_3 triangle is face-capped by one chlorine atom and edge-capped by one chlorine and two oxygen atoms. In addition, each of the two oxygens is linked to an adjacent Nb_3 cluster, while Cl^{a-a} and Cl^{a-a-a} bridge two and three Nb_3 clusters, respectively. The new O^{i-a} ligand gives relatively short Nb–Nb intercluster distances (3.50 Å). The linkages between the clusters lead to zigzag chains along two directions of the space, building layers bridged together by chlorine atoms. In this compound, six valence electrons remain for the Nb–Nb bonding states, a good agreement with previous molecular orbital calculations performed on Nb_3 cluster compounds. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: A. Inorganic compounds; B. Chemical synthesis; C. X-ray diffraction; D. Crystal structure

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