[MS19-P07] Synthesis and Crystal Structure of Pb₄(V₃O₈)₂(SeO₃)₃
Vadim M. Kovrugin a,b, Marie Colmont a, Olivier Mentré b, Oleg I. Siidra b, Sergey V. Krivovichev b

aUnité de Catalyse et de Chimie du Solide, UMR 8181, Université Lille 1, 59652 Villeneuve d’Ascq Cédex, France.
bDepartment of Crystallography, Geological faculty, St. Petersburg State University, 199034, University emb. 7/9, St. Petersburg, Russia.
E-mail: kovrugin_vm@hotmail.com

Crystals of Pb₄(V₃O₈)₂(SeO₃)₃ (I) were obtained by hydrothermal method from aqueous solution and PbO, V₂O₅, SeO₂ in ratio 1:2:10. The reaction was performed in 23 mL Teflon-lined Parr reaction vessel heated in Thermo Scientific mechanical convection oven up to 210°C and hold over 96 hours. Afterward the vessel was cooled to room temperature at a rate of 5°C/h. Products consisted of orange platy crystals of I up to 300 μm in maximal dimension.

Crystals selected for data collection were mounted on a Bruker DUO four-circle diffractometer equipped with an APEX II CCD detector and monochromated MoKα radiation. The structure of I was solved by direct methods. The following twinning matrix was applied during the refinement [-100 0-10 -0.80-1]. I is triclinic, P1, a=7.1337(3)Å, b=7.1869(3)Å, c=21.5324(10)Å, α=90.138(2)°, β=98.139(2)°, γ=94.775(2)°, V=1088.92(8)Å³, R =0.0640 for 4721 unique reflections with |F₀|≥4σ₀.

There are six symmetrically inequivalent V sites in the structure of I. V−O distances vary in the range of 1.604−2.634Å and 1.606−2.080Å in VO₆ octahedra and VO₅ square pyramids, respectively. Se−O bonds are in the range of 1.676−1.739 Å. The structure of I contains four symmetrically distinct Pb²⁺ cations. All Pb−O bonds ≤3.5Å were taken into consideration. Coordination of Pb atoms is distorted and variable due to the stereochemical activity of lone electron pair.

The structure of I is based on ‘vanadium bronzes’ derivative chains formed by edge- and corner-sharing VO₆ octahedra, VO₅ square pyramids and SeO₃ trigonal pyramids. [(V₃O₈)₂(SeO₃)₃]₈⁻ chains are oriented along [100] and interconnected by Pb²⁺ cations into 3D framework. Comparison with the other similar compounds is given.

This work was supported by the French National Research Agency ANR (grant ANR 2011 JS08 003 01) and the Agency Campus France, the Russian Foundation for Basic Research RFBR (grant 12-05-31349) and Russian Federal Grant-in-Aid Program «Cadres» (agreement no. 8313).

Keywords: lead; vanadium; selenites; oxides; crystal structure; X-ray analysis