

H., Zaban A., Borse P. H., Kulkarni S. K., Doran G. S., Whitfield H. J. (2002) Proceedings of National Academy of Sciences of the United States of America, Vol. 99.

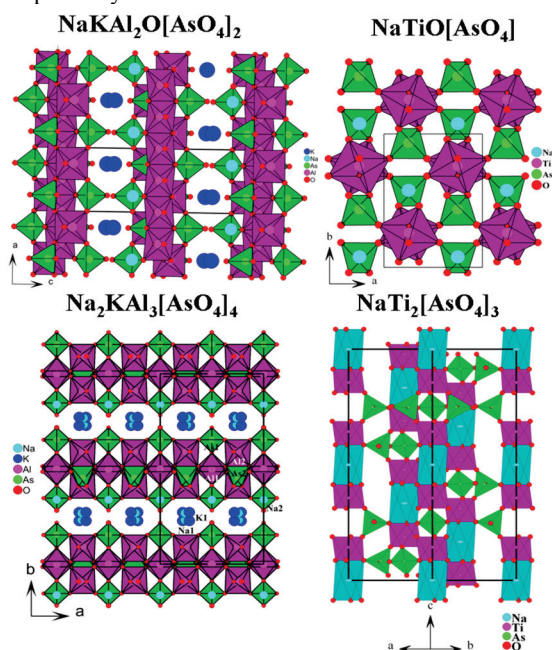
**Keywords:** ab-initio calculations, phonon properties, titanium oxide compounds

#### FA2-MS16-P58

**Crystal structures of NaTiO[AsO<sub>4</sub>], NaTi<sub>2</sub>[AsO<sub>4</sub>]<sub>3</sub>, NaKAl<sub>2</sub>O[AsO<sub>4</sub>]<sub>2</sub> and Na<sub>2</sub>KAl<sub>3</sub>[AsO<sub>4</sub>]<sub>4</sub>.** Hamdi Ben Yahia, Ute Ch. Rodewald, Rainer Pöttgen. *Institut für Anorganische und Analytische Chemie, Universität Münster, Corrensstrasse 30, D-48149 Münster, Germany.*

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The title compounds with structures isotopic to the  $\alpha$ -CaTiO[SiO<sub>4</sub>] [1], Nasicon [2], K<sub>2</sub>Fe<sub>2</sub>O[AsO<sub>4</sub>]<sub>2</sub> [3] and K<sub>3</sub>Fe<sub>3</sub>[AsO<sub>4</sub>]<sub>4</sub> [4] types have been synthesised by a solid state reaction route using a salt flux. The crystal structures were determined using single crystal data. The compound NaTiO[AsO<sub>4</sub>] crystallises in space group  $P2_1/c$ ,  $Z = 4$  with unit cell parameters:  $a = 6.7170(9)$ ,  $b = 8.7707(12)$ ,  $c = 7.2447(10)$  Å,  $\beta = 114.77(1)^\circ$ , whereas NaTi<sub>2</sub>[AsO<sub>4</sub>]<sub>3</sub> crystallises in space group  $R\bar{3}c$ ,  $Z = 6$  with unit cell parameters:  $a = 8.8057(5)$ ,  $c = 22.2406(15)$  Å. The NaKAl<sub>2</sub>O[AsO<sub>4</sub>]<sub>2</sub> and Na<sub>2</sub>KAl<sub>3</sub>[AsO<sub>4</sub>]<sub>4</sub> crystallise with the orthorhombic unit cell parameters  $a = 8.2368(6)$ ,  $b = 5.5228(3)$ ,  $c = 17.0160(13)$  Å, S.G.  $Pnma$ ,  $Z = 4$  and  $a = 10.5049(9)$ ,  $b = 20.4816(12)$ ,  $c = 6.3574(6)$  Å, S.G.  $Cmce$ ,  $Z = 4$ , respectively. NaKAl<sub>2</sub>O[AsO<sub>4</sub>]<sub>2</sub> and Na<sub>2</sub>KAl<sub>3</sub>[AsO<sub>4</sub>]<sub>4</sub> are build up of [Al<sub>2</sub>As<sub>2</sub>O<sub>9</sub>]<sup>2-</sup> and [Al<sub>3</sub>As<sub>4</sub>O<sub>16</sub>]<sup>3-</sup> layers, respectively. In-between these layers are located the seven or eight coordinated sodium and potassium cations. The structure of NaTiO[AsO<sub>4</sub>] and NaTi<sub>2</sub>[AsO<sub>4</sub>]<sub>3</sub> consist of a three dimensional framework of corner-sharing AsO<sub>4</sub> tetrahedra and TiO<sub>6</sub> octahedra. The negatively charged frameworks [TiAsO<sub>5</sub>] and [Ti<sub>2</sub>As<sub>3</sub>O<sub>12</sub>] give rise to different interstices in which are located the seven and six coordinated sodium atoms, respectively.



[1] Sper J.A., Gibbs G.V., *Am. Mineral.* 1976, 61, 238. [2] Sizova R.G., Voronkov A.A., Shumiatskaia N.G., Ilyukhin V.V., Belov N.V., *Sov. Phys. Dokl.* 1972, 17, 618. [3] Chang R.S., Wang S.L., Lii K.H., *Inorg. Chem.* 1997, 36, 3410. [4] Ouerfelli N., Zid M.F., Jouini T., *Acta Crystallogr.* 2005, E61, i67.

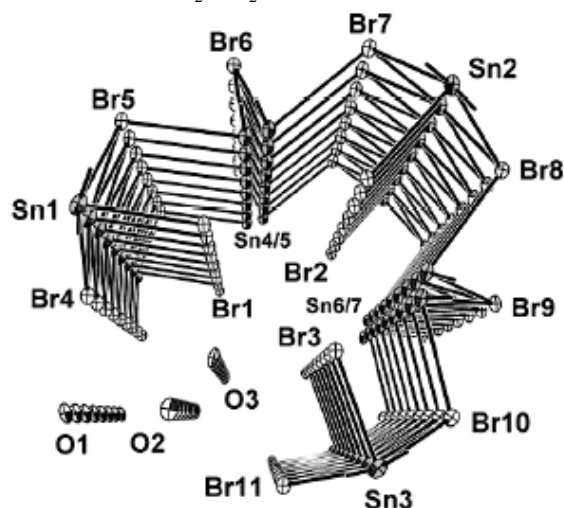
**Keywords:** Arsenate (V), Crystal structure determination, Single crystal diffraction, Salt flux synthesis

#### FA2-MS16-P59

**Intermediates in the course of the synthesis of tin(II) bromide, SnBr<sub>2</sub>.** Fei Ye<sup>a</sup>, Hans Reuter<sup>a</sup>. <sup>a</sup>*Institute of Chemistry, University of Osnabrück, Germany.*

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Since more than a century, tin(II) bromide, SnBr<sub>2</sub>, is prepared by heating metallic tin with hydrobromic acid at reflux [1]. It is also known since that time that this reaction proceeds via different solid intermediates which are described to be hydrates of tin(II) bromide or adducts of tin(II) bromide and hydrogen bromide or combinations of hydrates and adducts [2]. In contrast to the importance of this reaction and the long time this reaction is applied there is only little known about the nature and structures of these intermediates. Only Andersson published in the early 70<sup>th</sup> of the last century some preliminary structural results on compounds which he assigned sum formula like 2SnBr<sub>2</sub> · H<sub>2</sub>O [3], 3SnBr<sub>2</sub> · H<sub>2</sub>O [4] and 6SnBr<sub>2</sub> · 5H<sub>2</sub>O [5]. Because no atomic positions were given, we have started to reinvestigate this reaction and its intermediates to close this gap in the chemistry of bivalent tin. Here we present our results on the compound with composition 5SnBr<sub>2</sub> · 3H<sub>2</sub>O · HBr.



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**Keywords:** tin compounds, hydrates, adducts of hydrogen halides