



Synthesis, crystal structure and infrared and Raman spectra of a sodium barium orthoarsenate (V) nonahydrate $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$



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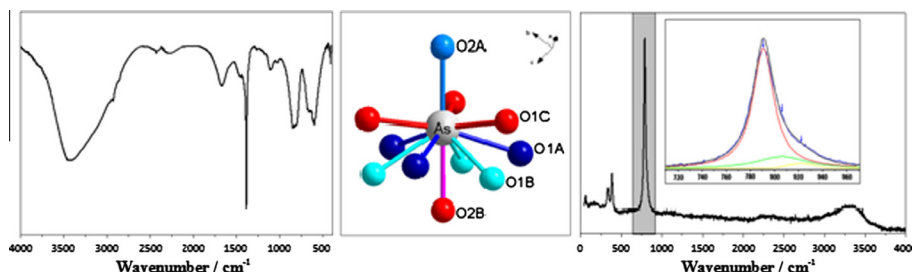
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HIGHLIGHTS

- A new orthoarsenate $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$, has been synthesized, structurally studied and characterized by vibrational spectroscopy.
- The structure is characterized by the presence of discrete AsO_4 tetrahedra and $\text{NaBa}(\text{H}_2\text{O})_9$ framework.
- The infrared bands in the region between 4000 and 1000 cm^{-1} show the existence of stronger, medium and low hydrogen bonds.
- The short hydrogen bond length $\text{O4}-\text{H42}\cdots\text{O2B}$ of 2.496 \AA was confirmed by infrared spectroscopy.
- The Raman spectrum at room temperature confirms the presence of AsO_4 tetrahedra.

GRAPHICAL ABSTRACT



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ABSTRACT

Crystals of a sodium barium orthoarsenate (V) nonahydrate were synthesized by slow evaporation from aqueous solutions. The crystal structure was solved and refined by single-crystal X-ray diffraction. The crystal is cubic, space group $P2_13$, $a = 10.838(2)\text{ \AA}$; $Z = 4$ and $V = 1273.1(2)\text{ \AA}^3$. Its structure is composed of BaO_9 and NaO_6 polyhedra and AsO_4 tetrahedral units. The short hydrogen bond length $\text{O4}-\text{H42}\cdots\text{O2B}$ of 2.496 \AA was confirmed by infrared spectroscopy. In addition, Raman spectrum was recorded in order to study the arsenate group and the cation motion (lattice modes) in the title compound.

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1. Introduction

Alkaline and alkaline earth arsenates are of interest in different research areas and have been the subject of several previous studies [1]. A great challenge in materials sciences is the design

of compounds with potential practical applications in fields such as the ion exchange, surface absorption chemistry, conductivity. In this way, the alkaline and alkaline earth metals arsenates with three-dimensional condensed framework can give rise to original physical properties, due to the great number of different cation arrangements that they exhibit [2,3]. These compounds attract interest in materials science because of the possibility to dope

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them with divalent or trivalent rare earth ions to produce efficient fluorescent materials [4,5].

The sodium barium orthoarsenate (V) nonahydrate $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$ has as related compounds $\text{NaSrAsO}_4(\text{H}_2\text{O})_9$ [6], $\text{NaSrPO}_4(\text{H}_2\text{O})_9$ [7], $\text{NaCaPO}_3\text{S}(\text{H}_2\text{O})_9$ [8], $\text{NaSrPO}_3\text{S}(\text{H}_2\text{O})_9$ [9], $\text{NaBaPO}_3\text{S}(\text{H}_2\text{O})_9$ [8], and the minerals $\text{Na}(\text{Sr},\text{Ba})\text{PO}_4(\text{H}_2\text{O})_9$ (nastrophite) [10] and $\text{NaBaPO}_4(\text{H}_2\text{O})_9$ (nabaphite) [11], which exhibit the same cubic symmetry $P 2_13$.

The $\text{NaSrAsO}_4(\text{H}_2\text{O})_9$ was the first example of orthoarsenate (V) nonahydrate structurally studied by X-ray methods using single crystal, but H-atom positions of the water molecules were not located [6]. The title compound provides the second example of orthoarsenates (V) nonahydrate, the first example being $\text{NaSrAsO}_4(\text{H}_2\text{O})_9$ [6]. The synthesis, structural and vibrational study of the $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$ compound are reported in the present work.

2. Experimental

2.1. Synthesis

The crystals of the title compound were prepared in aqueous solution of $\text{As}_2\text{O}_3/\text{Ba}(\text{NO}_3)_2$ in a ratio of 3/44. The solution was stirred while the pH was monitored, and 1 M NaOH solution was added dropwise till the pH reached 7. The resulting solution is kept under ambient conditions. After two weeks, colorless, transparent and platelet crystals were obtained.

2.2. Structure determination

A single crystal with dimensions $0.30 \times 0.26 \times 0.18 \text{ mm}^3$ was chosen for the structure determination and refinement. The data was collected on an Entraf-Nonuis CAD-4 diffractometer using the $\text{Mo K}\alpha$ radiation at room temperature [12]. The H atoms were located through the difference Fourier map and refined by imposing constraints at the distance and agitation. The reflections were corrected for Lorentz and polarization effects; absorption correction was obtained via a psi-scan [13] and secondary extinction correction was applied [13]. The crystal structure was solved and

Table 1

Single-crystal X-ray data, data collection and details of structure refinements for $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$.

<i>Crystal data</i>	
Chemical formula	$\text{NaBaAsO}_4(\text{H}_2\text{O})_9$
Formula weight	549.01
Crystal system	Cubic
Space group	$P 2_13$
Cell dimensions	$a = 10.838(2) \text{ \AA}$
Cell volume	$1273.1(2) \text{ \AA}^3$
Z	4
<i>Data collection</i>	
Diffractometer	Entraf-Nonuis CAD-4
Temperature	293(2) K
Shape	Platelet
Radiation, graphite monochromator:	$\text{Mo K}\alpha$ ($\lambda = 0.71073 \text{ \AA}$)
Scan mode	$w - 2\theta$
Theta range for data collection	$2.7\text{--}26.9^\circ$
Range of h, k, l	$-5 \leq h \leq 13, -5 \leq k \leq 13, -13 \leq l \leq 4$
Measured reflections	2649
Independent reflections	849 [$R_{\text{int}} = 0.080$]
<i>Refinement</i>	
Refinement method	Full-matrix least-squares on F^2
Parameters	78
$S(F^2)$	1.06
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.043; wR = 0.099$
Final R indices (all)	$R_1 = 0.0438; wR = 0.098$
$(\Delta\rho)_{\text{max}}/(\Delta\rho)_{\text{min}}$	$2.53/-1.90$ ($e \text{ \AA}^{-3}$)

Table 2

Analysis of the coordination polyhedra for $\text{NaBaAsO}_4(\text{H}_2\text{O})_9$; main interatomic distances (\AA) and bond angles ($^\circ$).

<i>AsO₄ tetrahedra</i>			
As—O2B	1.68(6)	O1C ^{vii} —As—O1C	117.3(5)
As—O1C ^{vii}	1.65(2)	O1C ^{vii} —As—O1C ^{viii}	117.3(5)
As—O1C	1.65(2)	O1C—As—O1C ^{viii}	117.3(5)
As—O1C ^{viii}	1.65(2)	O1C ^{vii} —As—O1C	117.3(5)
		O1C ^{vii} —As—O2B	99.5(9)
		O1C ^{viii} —As—O2B	99.5(9)
As—O2A	1.70(2)	O1A ^{vii} —As—O1A ^{viii}	109.3(5)
As—O1A ^{viii}	1.687(1)	O1A ^{vii} —As—O1A	109.3(5)
As—O1A	1.687(1)	O1A ^{viii} —As—O1A	99.5(9)
As—O1A ^{vii}	1.687(1)	O2A—As—O1A ^{viii}	109.7(5)
		O2A—As—O1	109.7(5)
		O2A—As—O1A ^{vii}	109.7(5)
As—O2A	1.70(2)	O1B ^{vii} —As—O1B	94.6(1)
As—O1B ^{vii}	1.65(2)	O1B—As—O1B ^{viii}	94.6(1)
As—O1B	1.70(2)	O1B ^{vii} —As—O1B ^{viii}	94.6(1)
As—O1B ^{viii}	1.70(2)	O2A—As—O1B ^{vii}	122.0(8)
		O2A—As—O1B	122.0(8)
		O2A—As—O1B ^{viii}	122.0(8)
<i>NaO₆ octahedron</i>			
Na—O4 ⁱⁱⁱ	2.413(8)	O4 ^{ix} —Na—O5 ^{ix}	107.9(2)
Na—O4 ^{ix}	2.413(8)	O4 ⁱⁱⁱ —Na—O5 ⁱⁱⁱ	107.9(2)
Na—O4	2.413(8)	O4—Na—O5 ^{ix}	79.0(2)
Na—O5 ^{ix}	2.418(8)	O4 ^{ix} —Na—O5 ⁱⁱⁱ	79.0(2)
Na—O5	2.419(8)	O5 ^{ix} —Na—O5 ⁱⁱⁱ	89.4(3)
Na—O5 ⁱⁱⁱ	2.419(8)	O4—Na—O5 ⁱⁱⁱ	158.8(2)
		O5—Na—O5 ⁱⁱⁱ	89.4(3)
		O4 ⁱⁱⁱ —Na—O4 ^{ix}	87.8(3)
		O4 ⁱⁱⁱ —Na—O5	79.0(2)
		O4 ⁱⁱⁱ —Na—O4	87.8(3)
		O4 ^{ix} —Na—O5	158.8(2)
		O4 ^{ix} —Na—O4	87.8(3)
		O4—Na—O5	107.9(2)
		O4 ⁱⁱⁱ —Na—O5 ^{ix}	158.8(2)
		O5 ^{ix} —Na—O5	89.4(3)
<i>BaO₉ polyhedron</i>			
Ba—O3 ⁱ	2.796(7)	Ba—O4 ^v	2.840(7)
Ba—O3	2.796(7)	Ba—O5 ⁱ	2.845(6)
Ba—O3 ⁱⁱ	2.796(7)	Ba—O5	2.845(7)
Ba—O4 ⁱⁱⁱ	2.840(7)	Ba—O5 ⁱ	2.845(6)
Ba—O4 ^{iv}	2.840(7)		

Symmetry codes: (i) $-z + 1, x - 1/2, -y + 1/2$; (ii) $y + 1/2, -z + 1/2, -x + 1$; (iii) $-y + 1/2, -z, x - 1/2$; (iv) $-x + 3/2, -y, z + 1/2$; (v) $-z + 1/2, -x + 1, y + 1/2$; (vi) $-x + 1, y + 1/2, -z + 1/2$; (vii) $z + 1, x - 1, y$; (viii) $y + 1, z, x - 1$; and (ix) $z + 1/2, -x + 1/2, -y$.

refined against F^2 in the space-group $P2_13$ using the SHELX-97 computer programs [14,15] included in the WingX software package [16]. The disordered model was refined by using the tools available in SHELX97 [16]. Structural figures have been obtained with the Diamand 2.1 [17]. The details of the structure refinement are given in Table 1. For geometrical parameters see Table 2. Further details of the crystal structure may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany, on quoting the depository number CSD – 426703.

2.3. Infrared and Raman spectra

The infrared spectrum in the spectral range between 4000 and 400 cm^{-1} was recorded as KBr pellets with a Perkin Elmer FTIR instrument 1760X at a resolution of 4 cm^{-1} .

Laser-Raman spectrum of crystal was recorded at room temperature in the range from 4000 to 50 cm^{-1} with a Renishaw M1000 Micro-Raman Imaging System using a laser wavelength of 633 nm and excitation through a Leica DMLM optical microscope (unpolarised laser light, 180 backscatter mode, spectral resolution

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