

Natromelansonite, $\text{Na}_3\text{Zr}[\text{Si}_7\text{AlO}_{19}]\cdot 4\text{-}5\text{H}_2\text{O}$, a new member of the rhodesite mero- plesiotype series from Mont Saint-Hilaire, Quebec, Canada

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Short Title: Natromelansonite, a new mineral

Abstract

Natromelansonite, $\text{Na}_3\text{Zr}[\text{Si}_7\text{AlO}_{19}]\cdot 4\text{-}5\text{H}_2\text{O}$, was found at the Poudrette (Demix) quarry, Mont Saint-Hilaire, Quebec, Canada in a highly altered pegmatite together with a clay mineral, steacyite, polyolithionite, and rhodochrosite. It occurs as an outer zone of tabular crystals to 0.1 x 0.3 x 1 mm in size flattened on (001). The inner zone is made of melansonite. The mineral is grey with white powder colour and vitreous lustre. The cleavage is parallel to {010}, perfect. The Mohs hardness is 3.5. The mineral has green fluorescence under short-wave ultraviolet light. The D_{calc} is 2.31 g/cm³. The IR spectrum is reported. The composition (wt.%, average of 8 analyses) is Na₂O 10.08, K₂O 1.72, CaO 0.24, BaO 0.32, MnO 0.10, Al₂O₃ 6.86, Y₂O₃ 0.35, Yb₂O₃ 0.55, SiO₂ 56.23, ZrO₂ 14.78, H₂O 9.52, total 100.75. The empirical formula calculated on the basis of O = 23 *apfu* and H = 8 *apfu* is $\text{Na}(\square\text{Na}_{0.38}\text{Ca}_{0.02}\text{Mn}_{0.01})_2(\text{Na}_{0.70}\text{K}_{0.28}\text{Ba}_{0.02})_{\Sigma 1.00}(\text{Zr}_{0.91}\text{Y}_{0.02}\text{Yb}_{0.02})_{\Sigma 0.95}(\text{Si}_{7.08}\text{Al}_{1.02})_{\Sigma 8.10}\text{O}_{19}\cdot 4\text{H}_2\text{O}$. The mineral is monoclinic, $P2_1/m$, $a = 6.5156(3)$ Å, $b = 24.061(1)$ Å, $c = 6.9759(6)$ Å, $\beta = 90.453(5)^\circ$ and $V = 1093.61(9)$ Å³ and $Z = 2$. The strongest reflections of the powder X-ray



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diffraction pattern $[d, \text{\AA}(hkl)]$ are: 12.02(100)(020), 6.97(89)(001), 6.51(39)(100), 3.416(37)(160), 3.062(42)($\bar{1}61$, 102, 161, $\bar{1}12$), 3.018(38)(230, 042), 2.864(40)(240, 132). The crystal structure, solved and refined from single-crystal X-ray diffraction data ($R_1 = 0.042$), is based on a double sheet of tetrahedra (T) and a sheet of octahedra (O) that alternate along the [010] direction forming a TOT structure typical for members of the rhodesite mero-plesiotype series.

Keywords: natromelansonite, new mineral, crystal structure, mero-plesiotype series, Mont Saint-Hilaire,

Introduction

Minerals of rhodesite mero-plesiotype series (Ferraris and Gula, 2005; Cadoni and Ferraris, 2011), are often found in agpaitic and hyperagpaitic environments. One of the members of the series monteregianite-(Y), $\text{KNa}_2\text{Y}[\text{Si}_8\text{O}_{19}] \cdot 5\text{H}_2\text{O}$, has only been found at the famous alkaline igneous complex Mont Saint-Hilaire, Quebec, Canada (Chao, 1978; Ghose et al., 1987). It usually occurs in mirolitic cavities in igneous breccia and marble xenoliths (Horvath et al., 2019, and the references therein) as tabular-bladed crystals. Therefore, when we came across a specimen from Mont Saint-Hilaire with what appeared to be typical monteregianite-(Y) crystals but from a highly altered pegmatite it immediately attracted our attention. Its examination showed that the crystals are zoned and composed of melansonite, $\text{KNa}_2\text{Zr}[\text{Si}_7\text{AlO}_{19}] \cdot 4\text{-}5\text{H}_2\text{O}$, which previously was found in only one specimen from a contact between a marble xenolith and the host nepheline syenite at Mont Saint-Hilaire (Gore and McDonald, 2023) and the new species natromelansonite, ideally $\text{Na}_3\text{Zr}[\text{Si}_7\text{AlO}_{19}] \cdot 4\text{-}5\text{H}_2\text{O}$. Natromelansonite is named as the Na-analogue of melansonite.

Both the new mineral and the name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association, proposal IMA 2023-076. The holotype specimen of natromelansonite (catalogue number CMNMC 90813) is from the collection of the Canadian Museum of Nature, Canada, and was originally labelled as “monteregianite-(Y)”. A part of the holotype used for structure determination was deposited at the Natural History Museum in Oslo under catalogue number KNR 44447.

Occurrence and general appearance

Natromelansonite was found at the Poudrette quarry (Demix quarry), Mont Saint-Hilaire, Quebec, Canada in a highly altered pegmatite in nepheline syenite (Horvath et al., 2019, and the references therein). It occurs on rhodochrosite together with polyolithionite and beige aggregates of poorly diffracting clay mineral and steacyite microcrystals. The specimen was collected by Gilles Haineault in 2004.

Natromelansonite occurs as an outer zone of tabular crystals up to 0.1 x 0.3 x 1 mm in size (Fig. 1) flattened on (001) where the inner zone is melansonite. The boundary between the two species is sharp and marked by a thin zone of uranium-bearing melansonite (Fig. 2). The observed crystal forms are dominant pinacoid {001} with two other pinacoids {001} and {100}.

Physical and optical properties

Natromelansonite is grey with white powder colour and vitreous lustre (Fig. 1). The cleavage is parallel to {010}, perfect. The fracture is stepped. The Mohs hardness is 3.5. The mineral has green fluorescence under short-wave ultraviolet light. The density calculated using the empirical formula and unit-cell volume refined from the single-crystal XRD data is 2.31 g/cm³.

Natromelansonite is optically biaxial (+), $\alpha = 1.510(2)$, $\beta = 1.516(2)$, $\gamma = 1.524(3)$, $2V$ (meas.) = 86(3)^o (from a spindle-stage extinction curve), $2V$ (calc.) = 82^o.

Experimental methods

Chemical data were obtained using a JEOL 8230 SuperProbe electron microscope equipped with five WDS spectrometers (University of Ottawa – Canadian Museum of Nature MicroAnalysis Laboratory, Canada) with an accelerating voltage of 15 kV and a beam current of 10 nA. Both natromelansonite and melansonite are unstable under an electron beam, so a larger beam diameter of 20 μm was used to minimize the damage. The matrix correction method was ZAF or Phi-Rho-Z calculations (Armstrong, 1988). The following reference materials were used: albite (NaK α), sanidine (KK α , AlK α , and SiK α), diopside (CaK α), tephroite (MnK α), sanbornite (BaL α), YIG (YL α), YbPO₄ (YbL α), zircon (ZrL α), and UO₂ (UM α). Both the peak and the background count times were 20 seconds for all elements. The intensity data were corrected for Time Dependent Intensity loss (or gain) using a self-calibrated correction for KK α , SiK α , and NaK α . The time/counts analysis showed significant Na migration in natromelansonite (Fig. 3).

The Fourier transform infrared (FTIR) spectrum of natromelansonite was obtained at the Canadian Conservation Institute, Canada using a Bruker Hyperion 2000 microscope interfaced to a Tensor 27 spectrometer with a wide-band mercury cadmium telluride (MCT) detector. A small grain of natromelansonite was mounted and compressed on a low-pressure diamond anvil microsample cell and analysed in transmission mode. The spectrum was collected between 400–4000 cm^{-1} by averaging 256 scans at a 4 cm^{-1} operating resolution.

Powder X-ray diffraction data were collected at the Canadian Museum of Nature, Canada using a Bruker D8 Discover microdiffractometer equipped with a DECTRIS EIGER2 R 500K detector and I μ S microfocus X-ray source ($\text{CuK}\alpha_1$, $\lambda = 1.54060 \text{ \AA}$) with the $\text{K}\alpha_2$ contribution removed using the “Strip $\text{K}\alpha_2$ ” tool in Bruker Diffrac.EVA V4.3. The instrument was calibrated using a statistical calibration method (Rowe, 2009). A powder ball $\sim 200 \mu\text{m}$ in diameter, mounted on a fiber pin mount, was analysed with continuous Phi rotation and 10° rocking motion along the Psi axis of the Centric Eulerian Cradle stage.

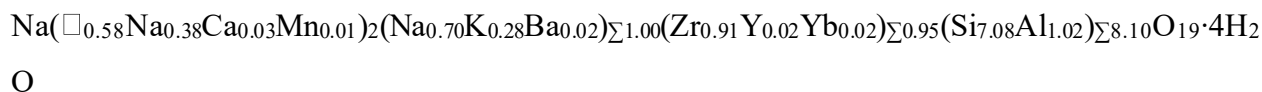
Single-crystal X-ray studies were carried out at the Natural History Museum, University of Oslo, Norway on a Rigaku XtaLAB Synergy-S diffractometer equipped with a HyPix 6000HE detector ($\text{MoK}\alpha$, $\lambda = 0.71073 \text{ \AA}$) operating at 50 kV and 1 mA at room temperature. The data were collected and processed using Rigaku's CrysAlis Pro software.

Results

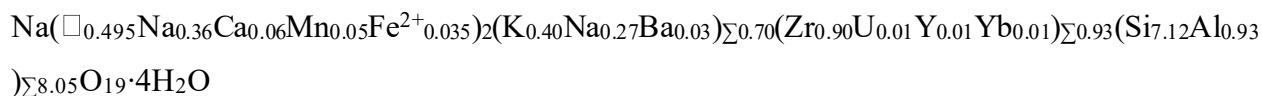
Chemical data

Chemical data on natromelansonite, melansonite and uranium-bearing melansonite are given in Table 1. All the formulae are calculated on the basis of O = 23 per formula unit (*pfu*) and H = 8 pfu. The H content is estimated to be 8 pfu for simplicity. The empirical formulae written taking into account the structural data are as follows:

Natromelansonite



Melansonite



Uranium-bearing melansonite

$\text{Na}(\square_{0.65}\text{Na}_{0.285}\text{Ca}_{0.05}\text{Mn}_{0.015})_2(\text{K}_{0.52}\text{Ba}_{0.02})_{\Sigma 0.54}(\text{Zr}_{0.65}\text{Y}_{0.12}\text{U}_{0.09}\text{Yb}_{0.05})_{\Sigma 0.91}(\text{Si}_{7.57}\text{Al}_{0.62})_{\Sigma 8.19}\text{O}_{19}\cdot 4\text{H}_2\text{O}$

The ideal end-member formula for natromelansonite is $\text{Na}_3\text{Zr}[\text{Si}_7\text{AlO}_{19}]\cdot 4\text{H}_2\text{O}$, which requires Na_2O 12.24, Al_2O_3 6.71, SiO_2 55.35, ZrO_2 16.22, H_2O 9.48, total 100 wt.%.

Natromelansonite does not react with an aqueous HCl solution at room temperature.

Infrared spectroscopy

The infrared (IR) spectrum of natromelansonite (Fig. 4) shows IR bands of O–H-stretching (in the range from 3275 to 3490 cm^{-1}) and O–H–O bending (at 1638 cm^{-1}) vibrations characteristic of H_2O molecules. The bands at 1023 and 1160 cm^{-1} corresponds to symmetric and asymmetric Si–O stretching vibrations. The band at 622 cm^{-1} can be assigned to Zr–O stretching vibrations. The band assignment was made in accordance with Chukanov and Chervonnyi (2016). The obtained spectrum is very similar to that of monteregianite-(Y) that was reported by Chukanov (2014).

X-ray diffraction data

The indexed powder X-ray diffraction data are given in Table 2. Parameters of the monoclinic unit cell refined from the powder data are as follows: $a = 6.5156(3)$ Å, $b = 24.061(1)$ Å, $c = 6.9759(6)$ Å, $\beta = 90.453(5)^\circ$ and $V = 1093.61(9)$ Å³.

The single-crystal X-ray diffraction data were indexed in the $P2_1/m$ space group with the following unit-cell parameters: $a = 6.51296(10)$ Å, $b = 24.0944(4)$ Å, $c = 6.97551(11)$ Å, $\beta = 90.7466(13)^\circ$ and $V = 1094.55(3)$ Å³. The structure was refined to $R1 = 0.042$ on the basis of 4430 independent reflections with $I > 2\sigma(I)$ using the SHELXL-2018/3 program package (Sheldrick, 2015). Crystal data, data collection and structure refinement details are given in Table 3, atom coordinates, equivalent displacement parameters, site occupancy factors and bond valence sums (BVS) in Table 4, selected interatomic distances in Table 5, and anisotropic displacement parameters in Table S1. The crystallographic information file (CIF) for natromelansonite is available as a Supplementary file. It was also deposited in the Inorganic Crystal Structure Database (ICSD; #CSD 2307107).

Description and discussion of the crystal structure

Natromelansonite is pseudorthorhombic. Its monoclinic unit cell ($P2_1/m$) is very close to the orthorhombic cell ($Pmma$) of melansonite (Gore and McDonald, 2023) and is not doubled as it was described in monteregianite-(Y) (Ghose et al., 1987; Table 6). Group $P2_1/m$ is a *translationengleiche* subgroup of $Pmma$ and such pseudosymmetry in monteregianite-related minerals was predicted by Ghose et al. (1987).

The natromelansonite crystal structure consists of two different types of sheets normal to the b axis: (a) a double sheet of tetrahedra (T) (Fig. 5) and (b) a sheet of octahedra (O) (Fig. 6). The T and O sheets alternate along the $[010]$ direction forming a TOT topology (Fig. 7) typical for members of the rhodesite mero-plesiotype series (Ferraris and Gula, 2005; Cadoni and Ferraris, 2011), including melansonite, $\text{KNa}_2\text{Zr}[\text{Si}_7\text{AlO}_{19}] \cdot 4\text{-}5\text{H}_2\text{O}$ (Gore and McDonald, 2023), monteregianite-(Y), $\text{KNa}_2\text{Y}[\text{Si}_8\text{O}_{19}] \cdot 5\text{H}_2\text{O}$ (Chao, 1978; Ghose et al., 1987), delhayelite, $\text{K}_4\text{Na}_2\text{Ca}_2[\text{AlSi}_7\text{O}_{19}]_2\text{F}_2\text{Cl}$ (Sahama and Hytönen, 1959; Pekov et al., 2009), hydrodelhayelite $\text{KCa}_2[\text{AlSi}_7\text{O}_{17}(\text{OH})_2] \cdot 3\text{H}_2\text{O}$ (Dorfman and Chigarov, 1979; Pekov et al., 2009), and fivegite, $\text{K}_4\text{Ca}_2[\text{AlSi}_7\text{O}_{17}(\text{O}_{2-x}\text{OH}_x)][(\text{H}_2\text{O})_{2-x}\text{OH}]\text{Cl}$ (Pekov et al., 2011). Members of the series are characterized by nearly identical T sheets which may show a different ratio between upwards and downwards pointing tetrahedra which is related to the number, charge and coordination number of the cations in the O sheet and to the Si/Al ratio within the silicate sheet. Most of the compounds have a T:O ratio of 8:19 (T = Si, Al). Monoclinic and orthorhombic members are known.

The O sheet in natromelansonite (Fig. 6) consists of alternating edge-sharing fully occupied $\text{Zr}(1)\text{O}_6$ and $\text{Na}(1)\text{O}_4(\text{H}_2\text{O})_2$ octahedra and a highly distorted partially occupied (40%) $\text{Na}(2)\text{O}_4(\text{H}_2\text{O})_2$ octahedron.

The T sheet is composed of two single silicate sheets of the apophyllite type with four- and eight-membered rings (Fig. 5a). They are formed by Si1, Si2, and Si3-centred tetrahedra with the $\langle\text{Si}-\text{O}\rangle$ distance in the range of 1.608-1.610 Å, and a larger Si4-centred tetrahedron with the $\langle\text{Si}-\text{O}\rangle$ distance of 1.664 Å, with a mixed Si/Al occupancy $\text{Si}_{0.5}\text{Al}_{0.5}$. The BVS at the Si4 site is 3.65 valence units (νu), which confirms mixed occupancy at the site. The T sheet can be also described using the structure hierarchy concept (Hawthorne et al., 2019) as based on the 3-connected plane net 4.82 plus an oikodoméic (up/down) operation $(u^3d)_1(u^4du^2d)_1$ with all d tetrahedra occupied by Al atoms.

The two single sheets are linked via two vertex-sharing Si4-centred tetrahedra (Si4-O10-Si4, Fig. 5b), which resulted in creation of large channels. Interstitial Na and K atoms occur in the

channels in a split site with occupancies of 0.54, 0.21, and 0.25 for the Na₃, Na₄ and K₅ sites, respectively. The Na₃–Na₄, Na₃–K₅ and Na₄–K₅ distances are 2.28 Å, 1.60 Å and 0.84 Å, respectively. Sodium is the dominant extra-framework cation.

The presence of H₂O⁰ molecules is confirmed by the BVS at the O₁₁, O₁₂, O₁₃, and O₁₄ sites (0.10, 0.04, 0.32, and 0.01 *vu*, respectively; Table 4) and by the presence of the bands of O–H stretching and H–O–H bending bands in the IR spectrum of natromelansonite (Fig. 4). Some of these sites (O₁₂, O₁₄) are partially occupied, as it was observed in the crystal structures of monteregianite-(Y) (Ghose et al., 1987) and melansonite (Gore and McDonald, 2023). In general, water content varies significantly in members of the rhodesite mero-pleisotype series (Ferraris and Gula, 2005; Cadoni and Ferraris, 2011). In addition, the variable partial occupancy of multiple sites means that the water content varies from grain to grain in one mineral. Furthermore, the anisotropic thermal vibrations of ligand water molecules are very pronounced which complicates proper refinement of these sites in the structure. Thus, the H content is estimated to be 8 pfu in the ideal end-member formula for simplicity, and that number is used in the formula calculations. But the actual water content seems to be variable in the range 4–5 H₂O *pfu* in both melansonite and natromelansonite.

The resulting structural formula of natromelansonite is



The idealised formulae of melansonite and natromelansonite

The idealized formula of melansonite was given as (Na, □)₂KZrSi₈O₁₉·5H₂O in the original publication (Gore and McDonald, 2023). However, detailed examination of the published data combined with our data shows that the formula should be reconsidered taking into account the behaviour of Al and Na in melansonite. One Si-centred tetrahedron (Si(2)O₄) in the structure of melansonite is significantly larger than the others with the <Si–O> distance of 1.66 Å, indicating that Al atoms are ordered at this site as they are in natromelansonite, and the idealized occupancy at the site should be Si_{0.5}Al_{0.5}. The published chemical data showing ~1.0 *apfu* of Al (6.32 wt. % Al₂O₃) corroborate this. The Si₂ site in melansonite is analogous to the Si₄ site in the structure of natromelansonite where Si(2)O₄ tetrahedra linking single silicate sheets together. Similar ordering was observed in delhayelite, hydrodelhayelite (Pekov et al., 2009), and fivegite (Pekov et al., 2011); three other members of the rhodesite mero-pleisotype series (Cadoni and Ferraris, 2011).

The Na content (0.71 *apfu*, 2.82 wt. % Na₂O) in melansonite from the original find from a marble xenolith at Mont Saint-Hilaire seems to be unexpectedly low which could be explained by migration of Na under electron beam as it was suggested by Gore and McDonald (2023) and the very high count time of 100 seconds. As we showed, Na migration in melansonite and natromelansonite is significant even with a 20 μm beam (Fig. 3). Additionally, the crystals are very thin, only ~2 μm, which further complicated obtaining realisable data on Na content in the mineral. Melansonite from our find is characterised by a significantly higher Na content (8.20 wt. % Na₂O, Table 1) both due to (1) a larger crystal size which allowed us to reduce Na migration by using a larger beam diameter (20 μm) as compared to a 10 μm beam used by Gore and McDonald (2023) and (2) lower contents of admixed cations Ca (0.92 wt. % CaO) and Ba (0.59 wt. % BaO) in contrast to 1.75 wt. % CaO and 1.35 wt. % BaO in melansonite from the original find (Gore and McDonald, 2023).

Furthermore, the occupancy at the Na site in the O sheet of melansonite was fixed at 28% and the refined site scattering value at the site is unknown (Gore and McDonald, 2023). The full dataset including the cif file was not provided by the authors of the description so the reasoning behind this value is unclear, but it is probable that it could be somewhat higher as it was observed in both natromelansonite and monteregianite-(Y) (Ghose et al., 1987).

Considering all the above, (□Na) instead of □₂ in the formula would much better represent the actual distribution of the cations at the Na₂ site. Furthermore, the water content in both minerals is variable in the range 4-5 H₂O *pfu*. Thus, the melansonite formula should be written as Na(□Na)KZr(Si₇Al)O₁₉·4-5H₂O or, in a simplified form, KNa₂Zr(Si₇Al)O₁₉·4-5H₂O and the formula of its Na-analogue natromelansonite as Na(□Na)NaZr(Si₇Al)O₁₉·4-5H₂O or Na₃Zr(Si₇Al)O₁₉·4-5H₂O.

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Competing interests: The authors declare none.

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Fig. 1. Grey tabular prismatic crystals composed of natromelansonite and melansonite on a beige aggregate of a poorly diffracting clay mineral and steacyite microcrystals. Specimen CMNMC 90813. Photo: François Génier.

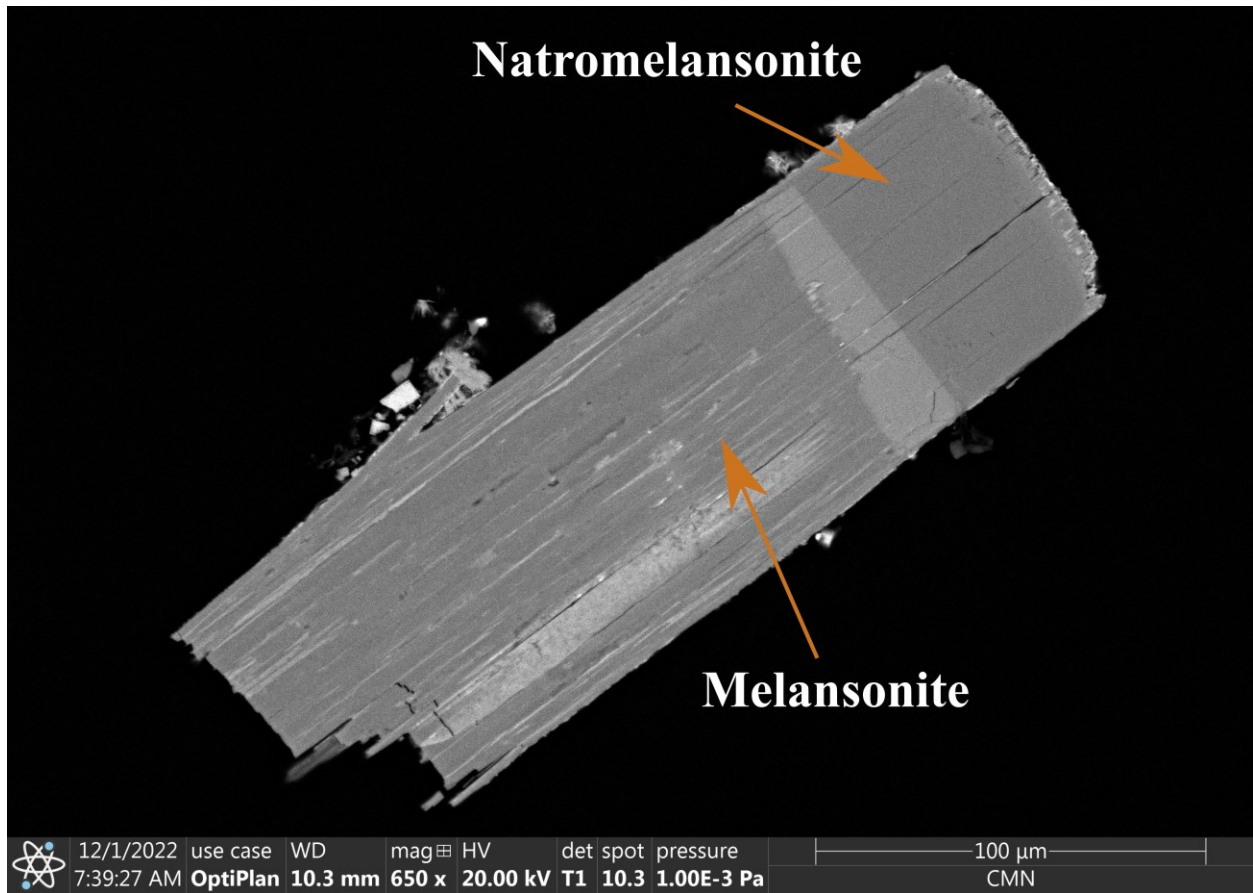


Fig. 2. A crystal composed of melansonite and natromelansonite zones. Bright zone – U-bearing melansonite. Polished section. SEM (BSE) image.

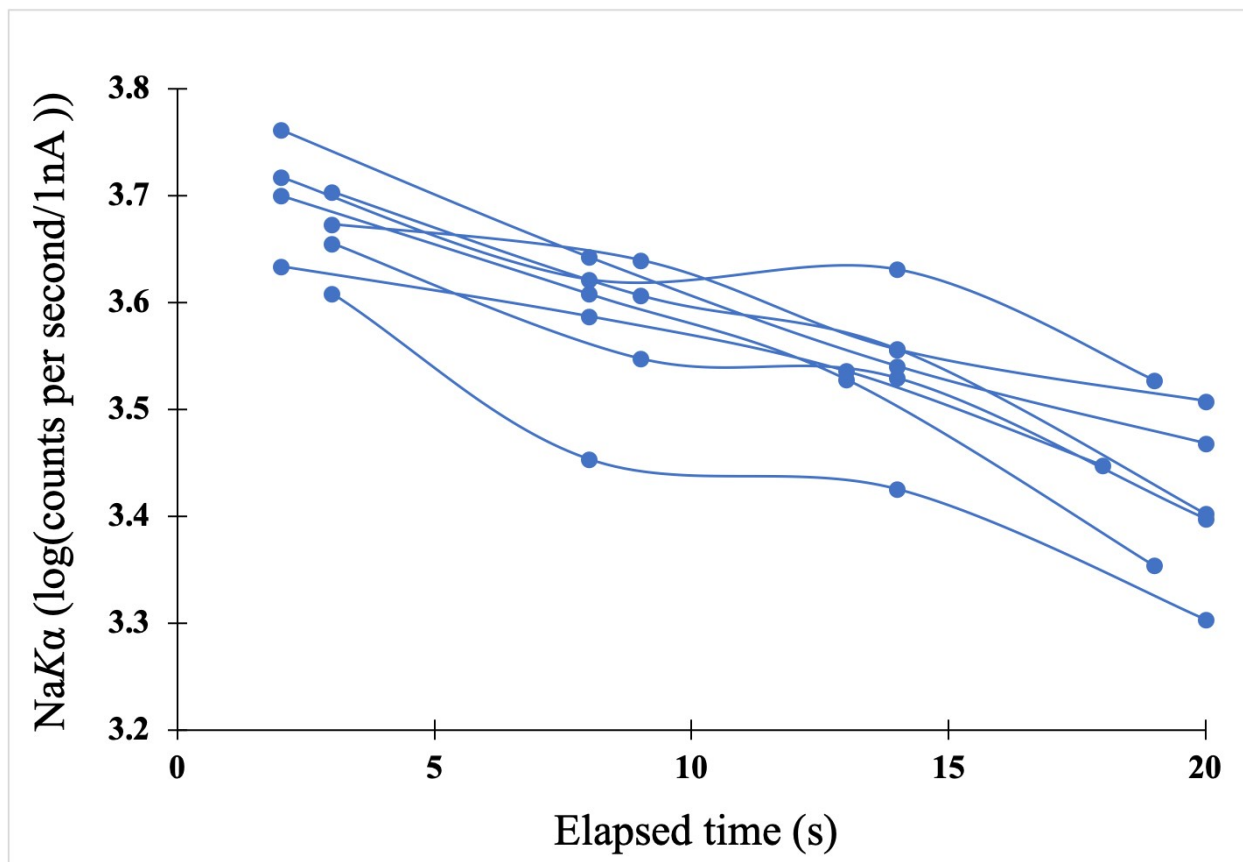


Fig. 3. Time dependent intensity loss for NaK α in natromelansonite (8 analyses).

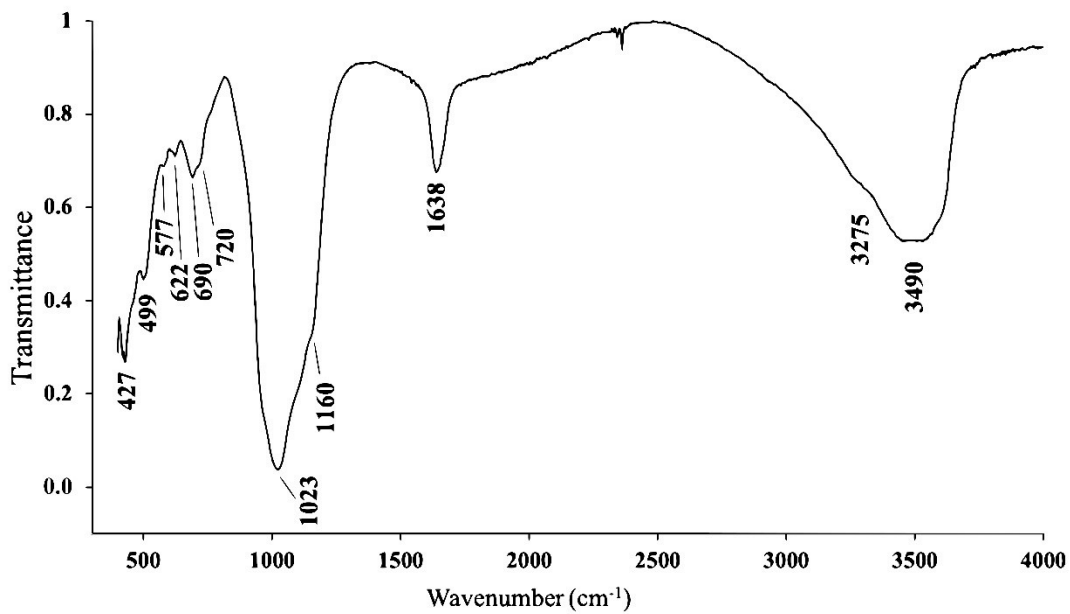
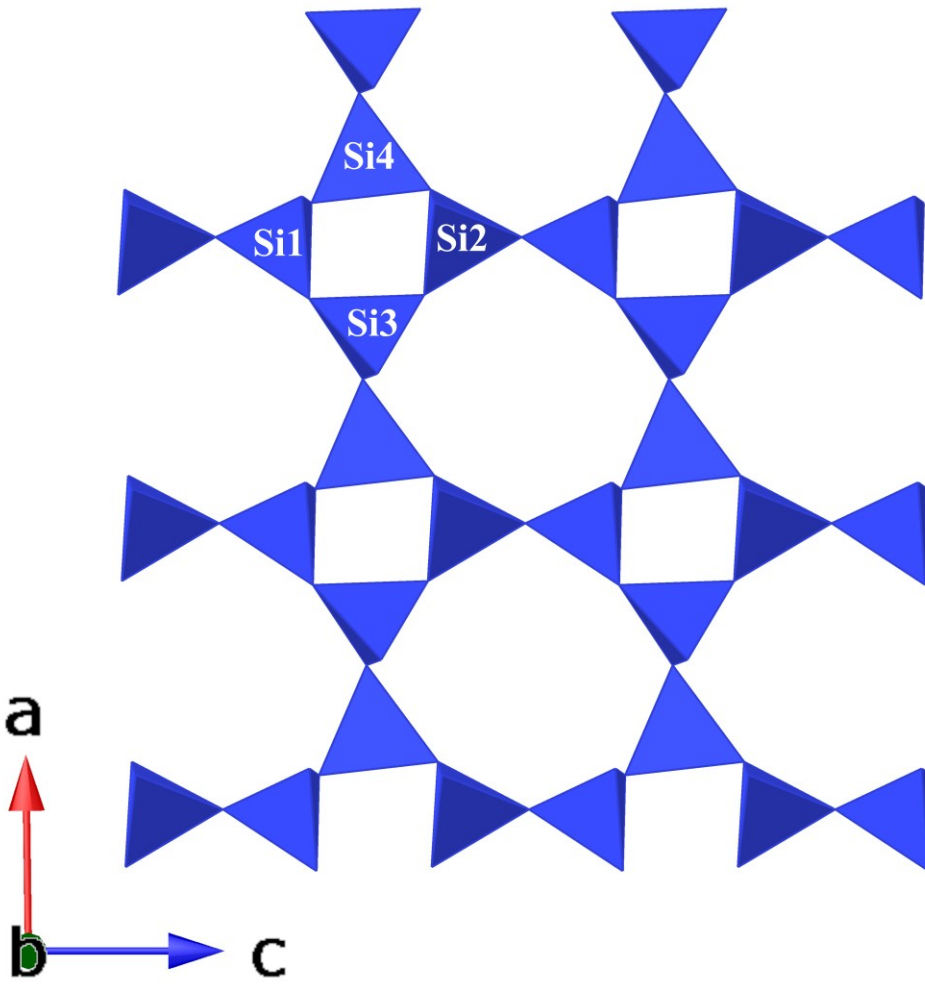


Fig. 4. Infrared spectrum of natromelansonite



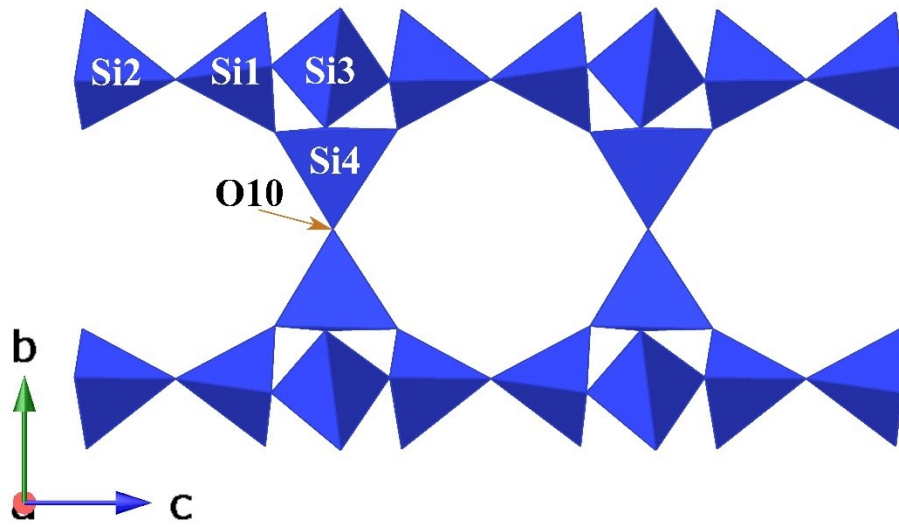
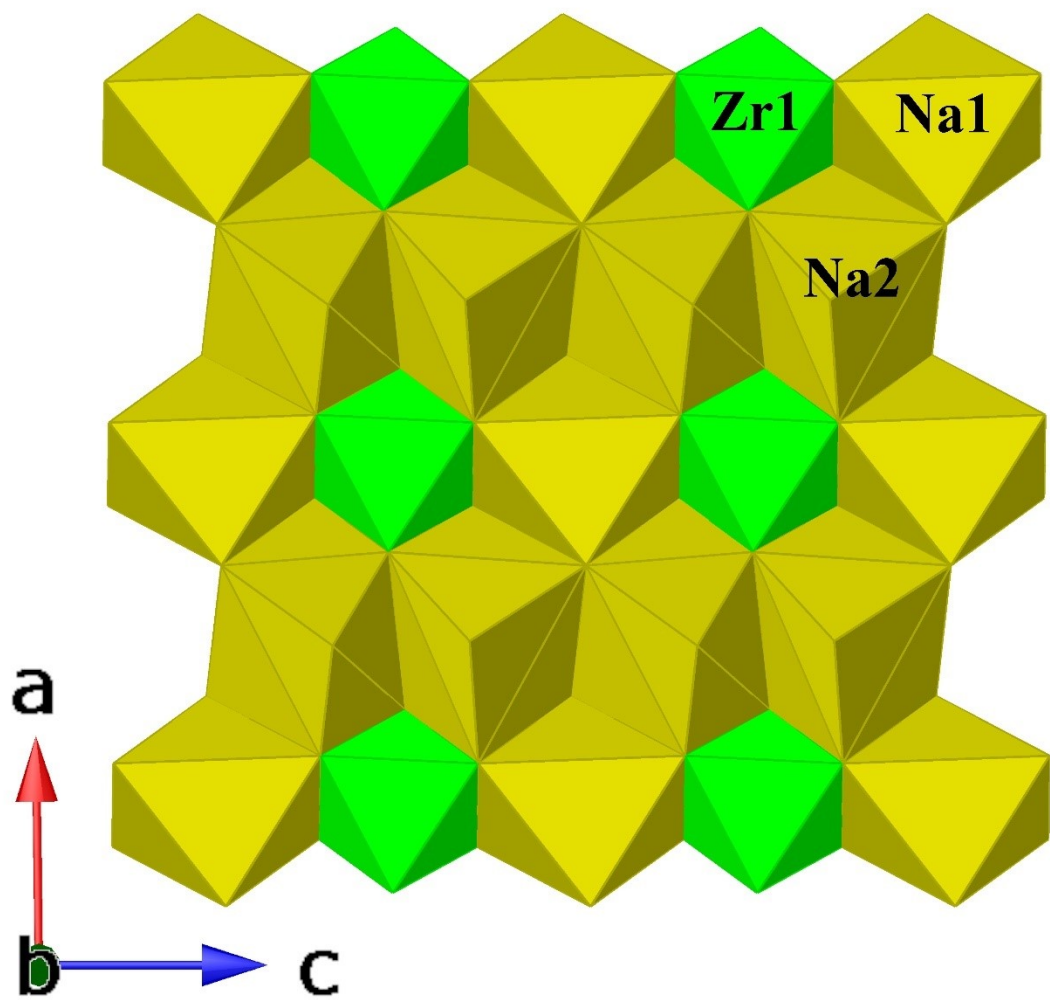


Fig. 5. Double sheet of tetrahedra (T) (a) in the structure of natromelansonite, where the single sheet consists of four- and eight-membered rings (b).

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Fig. 6. Sheet of octahedra (O) in the structure of natromelansonite.

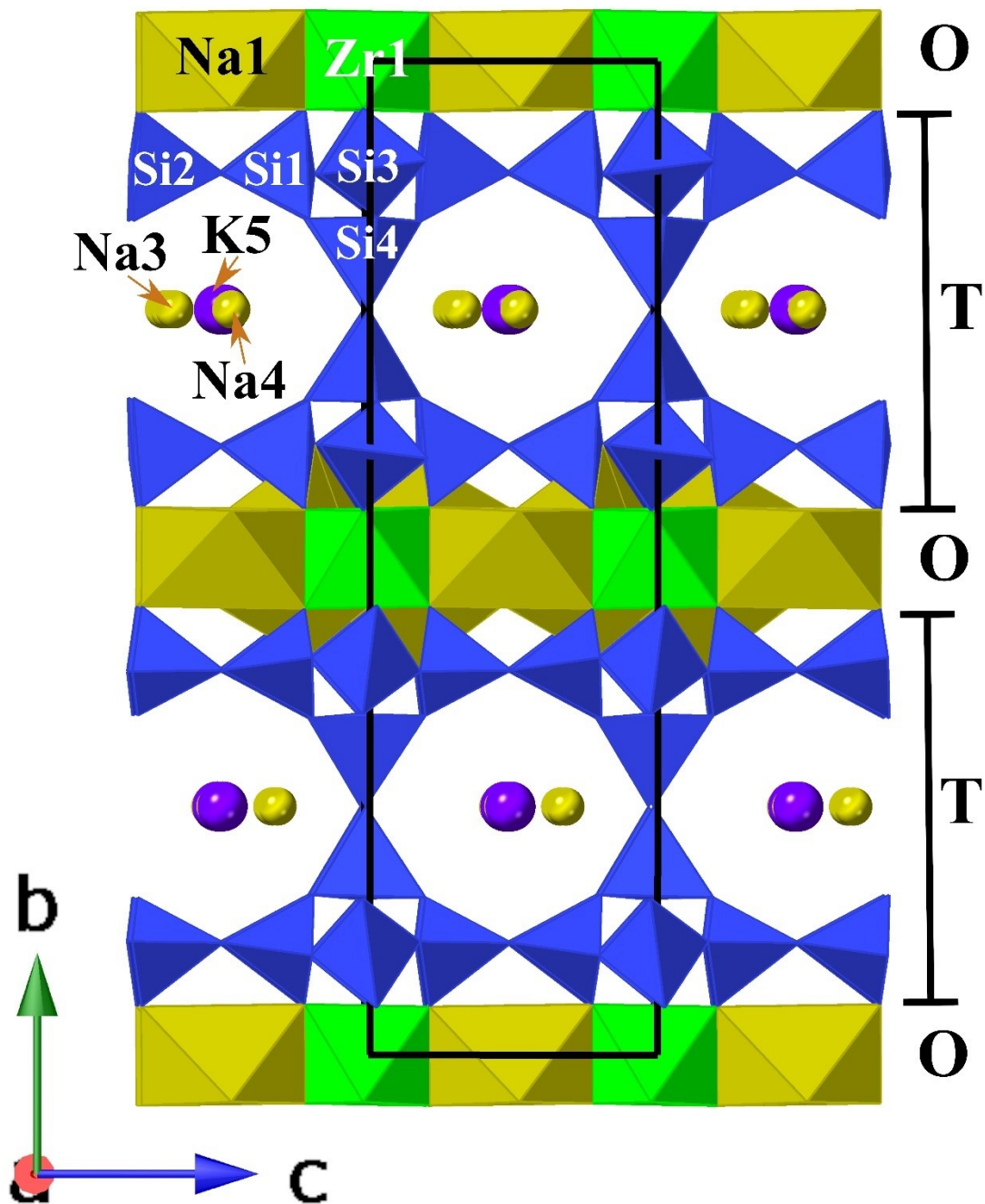


Fig. 7. General view of the crystal structure of natromelansonite. Projection on (100). The unit cell is outlined. T and O sheets are indicated. Yellow and purple spheres are extra framework Na and K atoms, respectively.