

Suseinargiuite, $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$, the Na-Bi analogue of wulfenite, from Su Seinaregiu, Sardinia, Italy

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Abstract: The new mineral species suseinargiuite, $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$, has been discovered in the Mo-Bi occurrence of Su Seinaregiu, Sarroch, Cagliari, Sardinia, Italy. It occurs as hemispherical aggregates of very small acicular crystals, up to some micrometres in length. It is colourless, with a pearly to adamantine lustre. In the studied specimens, suseinargiuite is associated with wulfenite. Micro-Raman spectra were collected in the region between 100 and 2000 cm^{-1} . The following bands have been observed (in cm^{-1}): 131, 188, 319, ~ 376 , ~ 772 , and 876. Electron microprobe data collected on a chemically zoned grain gave (outer and inner zone, respectively – in wt%): MoO_3 49.03, Bi_2O_3 42.97, PbO 2.89, Na_2O 3.69, total 98.58, and MoO_3 45.59, Bi_2O_3 34.47, PbO 12.04, Na_2O 3.03, total 95.13. On the basis of 4 O atoms per formula unit, the chemical formulae are $(\text{Na}_{0.35}\text{Bi}_{0.54}\text{Pb}_{0.04})_{\Sigma=0.93}\text{Mo}_{0.99}\text{O}_4$ and $(\text{Na}_{0.31}\text{Bi}_{0.46}\text{Pb}_{0.17})_{\Sigma=0.94}\text{Mo}_{0.99}\text{O}_4$, respectively. Main diffraction lines are [d in Å (relative intensity) hkl]: 3.146 (100) 112, 2.912 (13) 004, 2.652 (18) 200, 1.964 (34) 204, 1.875 (15) 220, 1.728 (19) 116, and 1.616 (28) 312 and 132. Unit-cell parameters, refined from the powder X-ray diffraction data in a tetragonal setting, space group $I4_1/a$, are $a = 5.296(1)$, $c = 11.673(2)$ Å, $V = 327.4(1)$ Å³, $Z = 4$. Owing to the lack of suitable crystals and the very small amount of available material, the crystal structure of suseinargiuite was not solved. However, X-ray powder diffraction data, micro-Raman spectra, as well as chemical analysis, show the close correspondence between suseinargiuite with synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$, which displays a scheelite-type structure.

Key-words: suseinargiuite; new mineral; sodium; bismuth; molybdate; scheelite-type structure; Su Seinaregiu; Sardinia; Italy.

1. Introduction

In the last few years, the small Su Seinaregiu prospect has provided a significant number of molybdenum secondary minerals, represented by bismuth-molybdenum oxides (sardignaite, gelosaite, and mambertiite; Orlandi *et al.*, 2010, 2011, 2015b) and molybdates (tancaite-(Ce), ichnusaite, and nuragheite; Orlandi & Bonaccorsi, 2010; Orlandi *et al.*, 2014, 2015a). It is noteworthy that, among the twelve valid mineral species characterized by the oxoanion $(\text{MoO}_4)^{2-}$, three (*i.e.* one quarter) have their type locality at Su Seinaregiu, which consequently represents a reference locality for the study of these minerals.

During the routine check of specimens from this Sardinian locality through qualitative energy-dispersive spectrometry (EDS) analysis, two samples, provided to us by the mineral collector Fernando Caboni, were found to contain Na, Bi, Mo, and minor Pb as the only elements with $Z > 9$. X-ray powder diffraction patterns collected on a very small grain by using a Gandolfi camera indicated the

identity between this unknown mineral and the synthetic compound $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$, having a scheelite-type structure (Teller, 1992; Hanuza *et al.*, 1997; Wařkowska *et al.*, 2005) and actively studied for its optical and luminescent properties (*e.g.*, Mazurak *et al.*, 1987; Rico *et al.*, 2002; Volkov *et al.*, 2002). Owing to the lack of suitable crystals for single-crystal X-ray diffraction and the very small amount of available material that did not allow a Rietveld refinement, a micro-Raman spectrum was also collected, in order to confirm the identity with its synthetic analogue. Additionally, other grains have chemistry and X-ray powder diffraction patterns intermediate between those of the new compound and wulfenite, PbMoO_4 , suggesting the possible existence of a solid solution between $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$ and PbMoO_4 .

The new mineral species was named suseinargiuite, from its type locality, Punta de Su Seinaregiu (or more simply Su Seinaregiu), Sarroch, Cagliari, Sardinia, Italy. The mineral and its name have been approved by the IMA CNMNC (IMA 2014–089). The holotype specimen of suseinargiuite is deposited in the mineralogical collection of the Museo di

Storia Naturale, Università di Pisa, via Roma 79, Calci (PI), Italy, with catalogue number 19692.

This paper presents the definition of suseinargiuite, describing its occurrence and its relationships with wulfenite and synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$.

2. Occurrence and mineral description

The Su Seinargiu prospect is located on the southern coast of Sardinia, northwest of the town of Sarroch. The Mo-Bi mineralization occurs as vein systems embedded in leucogranite porphyry, hosted within shales of Ordovician-Silurian age metamorphosed up to the greenschist facies. The vein mineralogy is very simple, being composed by quartz and molybdenite, with minor amounts of other sulphides (chalcopyrite, galena, sphalerite, and bismuthinite) and rare native bismuth (Orlandi *et al.*, 2013). A pervasive hydrothermal alteration is common throughout the magmatic rocks, resulting in the appearance of clay minerals replacing plagioclase and K-feldspar.

Molybdenite is also frequently altered into secondary minerals, initially indicated as “molybdite” by Caboi *et al.* (1978). The recent mineralogical studies carried out by Orlandi *et al.* (2013) showed actually the occurrence of a wide series of minerals formed by hydrothermal and/or supergene alteration of the primary Mo-Bi mineral assemblage.

Suseinargiuite occurs as hemispherical aggregates (up to 0.2–0.3 mm in diameter) of very small acicular crystals, up to few micrometres in length. Crystals are colourless, transparent, with a pearly to adamantine lustre. The mineral is brittle. Owing to the very small crystal size, micro-hardness was not measured. Density was not measured owing to the very low amount of available material; calculated density, based on the ideal formula, is 5.597 g/cm^3 . In plane-polarized transmitted light, suseinargiuite is transparent, colourless, with a high relief. With crossed polars, extinction is parallel and birefringence is high. The mean refractive index is 2.11 according to the Gladstone-Dale relationship (Mandarino, 1979, 1981) using the ideal formula and the calculated density.

In the studied specimens, suseinargiuite is associated with wulfenite in small vugs of quartz veins.

2.1. Chemical data

Preliminary qualitative chemical analyses were performed using a Philips XL30 scanning electron microscope equipped with an EDAX DX4 system. The only elements with $Z > 9$ detected in suseinargiuite are Na, Bi, Mo, and minor Pb. Quantitative chemical analyses were carried out with a Cameca SX100 electron-microprobe in wavelength-dispersive mode using the following analytical conditions: accelerating voltage 20 kV, beam current 15 nA, beam size 1 μm . Standards (element, emission line) were: albite (Na $K\alpha$), metallic Mo (Mo $L\alpha$), metallic Bi (Bi $M\alpha$), and galena (Pb $M\alpha$). Tungsten was sought but was not detected. The studied grain turned out to be chemically zoned (Fig. 1), with an outer zone enriched in Na and Bi and a Pb-enriched core. Analytical data for the outer and inner zones are given in Table 1. Low analytical totals are related to the

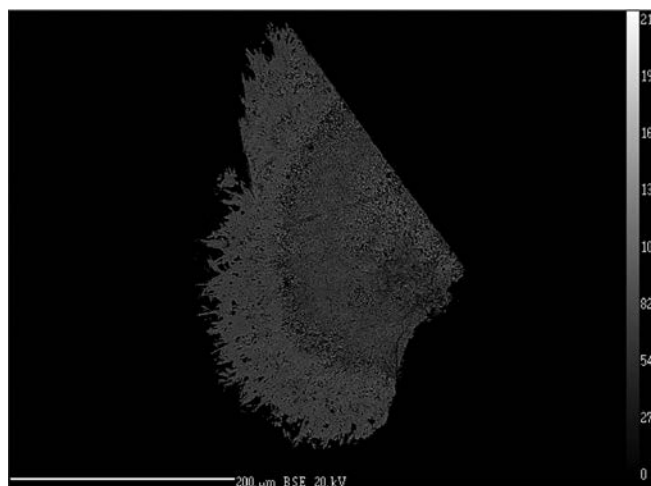


Fig. 1. Back-scattered electron image of an aggregate of acicular crystals of suseinargiuite. The outer zone (light grey) is depleted in Pb with respect to the inner zone (dark grey).

Table 1. Electron-microprobe analyses of suseinargiuite.

Oxide	Outer zone ($n = 12$)			Inner zone ($n = 14$)		
	wt%	range	e.s.d.	wt%	range	e.s.d.
MoO_3	49.03	47.54–51.14	1.11	45.59	43.49–48.01	1.25
Bi_2O_3	42.97	41.55–44.14	0.95	34.47	32.77–37.49	1.21
PbO	2.89	1.36–4.87	1.10	12.04	8.81–13.82	1.34
Na_2O	3.69	3.28–4.39	0.39	3.03	2.76–3.40	0.20
Total	98.58	96.92–100.47	1.00	95.13	93.20–97.71	1.38
apfu ($\text{O} = 4$ apfu)						
Mo	0.994	0.988–0.999	0.004	0.993	0.980–0.999	0.005
Bi	0.538	0.522–0.558	0.013	0.464	0.448–0.482	0.008
Pb	0.038	0.017–0.065	0.015	0.170	0.118–0.197	0.022
Na	0.346	0.315–0.399	0.030	0.307	0.284–0.343	0.018
Na + Pb + Bi	0.922	0.909–0.943	0.012	0.941	0.917–0.998	0.022

porous micro-crystalline texture of the sample, in particular for the inner zone, where the small size of intergrown crystals precluded spot analysis on a pore-free area.

The empirical formulae, based on 4 oxygen atoms per formula unit (apfu), are $(\text{Na}_{0.35(3)}\text{Bi}_{0.54(1)}\text{Pb}_{0.04(2)})_{\Sigma=0.93(1)}\text{Mo}_{0.99(1)}\text{O}_4$ and $(\text{Na}_{0.31(2)}\text{Bi}_{0.46(1)}\text{Pb}_{0.17(2)})_{\Sigma=0.94(2)}\text{Mo}_{0.99(1)}\text{O}_8$ for the outer and inner zones, respectively.

Without taking into account the Na deficit relatively to Bi (see § 3), the ideal formula of suseinargiuite is $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$, corresponding to (in wt%) MoO_3 52.17, Bi_2O_3 42.21, Na_2O 5.62, total 100.00.

2.2. X-ray crystallography

The X-ray powder diffraction pattern of suseinargiuite was collected using a 114.6 mm Gandolfi camera with Ni-filtered $\text{Cu K}\alpha$ radiation and is given in Table 2. Figure 2 shows the X-ray powder diffraction pattern compared with that calculated on the basis of the structural model of synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$ using the atomic coordinates and displacement parameters given by Wařkowska *et al.* (2005). The shift towards lower 2θ values is due to Pb incorporation (see below).

The unit-cell parameters of suseinargiuite, refined from the powder data using the method of Holland & Redfern (1997) on the basis of ten unequivocally indexed reflections, are: $a = 5.296(1)$, $c = 11.673(2)$ Å, $V = 327.4(1)$ Å³, $Z = 4$, assuming the space group $I4_1/a$ in agreement with Wařkowska *et al.* (2005). The $c:a$ ratio calculated from the unit-cell parameters is 2.204.

2.3. Micro-Raman spectroscopy

Raman analyses were carried out employing a confocal Raman microprobe (Horiba Jobin-Yvon T64000) coupled with an optical microscope and equipped with an Ar^+ laser

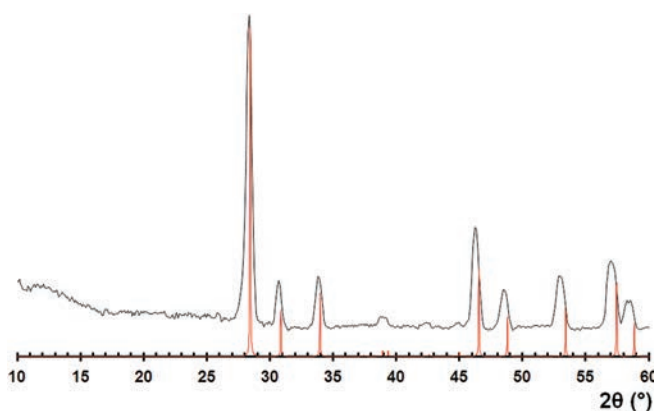


Fig. 2. X-ray powder diffraction pattern for suseinargiuite: comparison between observed (in black) and calculated pattern (in red – based on the atomic coordinates and displacement parameters given by Wařkowska *et al.*, 2005). (online version in colour)

source ($\lambda_0 = 514.5$ nm), a double subtractive stage plus spectrograph and diffraction gratings of 1800 gr/mm, and a liquid-nitrogen-cooled CCD Symphony detector (1024×256 pixels). Raman spectra were collected on the same grain used for electron-microprobe analysis. Spectra were collected in the region between 100 and 2000 cm^{-1} , with a slit and hole aperture of $100\text{ }\mu\text{m}$, laser beam power on the sample of 1.5 mW, and an integration time of 10 s, for two accumulation cycles. The diameter of the laser spot on the sample surface was $\sim 2\text{ }\mu\text{m}$ for the fully focused laser beam at $50\times$ objective magnification. The spectral resolution was 1 cm^{-1} and the instrument was calibrated against the Stokes Raman signal of pure Si at 520 cm^{-1} using a silicon wafer. Instrument control and data acquisition, as well as the processing of Raman spectra, were performed with the software LabSpec 5 (Horiba Jobin-Yvon).

The Raman spectrum in the range $100\text{--}2000\text{ cm}^{-1}$ is shown in Fig. 3 together with the position of the main bands. No Raman bands were detected above 2000 cm^{-1} .

Table 2. X-ray powder diffraction data for suseinargiuite. Calculated intensity and d_{hkl} were obtained using the software Powdercell 2.3 (Kraus & Nolze, 1996) on the basis of the structural model reported by Wařkowska *et al.*, 2005. The five strongest reflections are given in bold. The asterisk * indicates the reflections used for the refinement of the unit-cell parameters. Only calculated reflections with $I_{\text{calc}} \geq 1$ are reported.

I_{obs}	d_{obs}	I_{calc}	d_{calc}	$h\ k\ l$
100	3.146*	100	3.135	1 1 2
13	2.912*	14	2.894	0 0 4
18	2.652*	19	2.637	2 0 0
4	2.313*	2	2.311	2 1 1
		2	2.287	1 1 4
2	2.135*	1	2.120	1 0 5
2	2.014*	1	2.013	1 2 3
34	1.964*	28	1.949	2 0 4
15	1.875*	12	1.865	2 2 0
19	1.728*	15	1.714	1 1 6
28	1.616	10,13	1.603	3 1 2, 1 3 2
11	1.580*	10	1.568	2 2 4

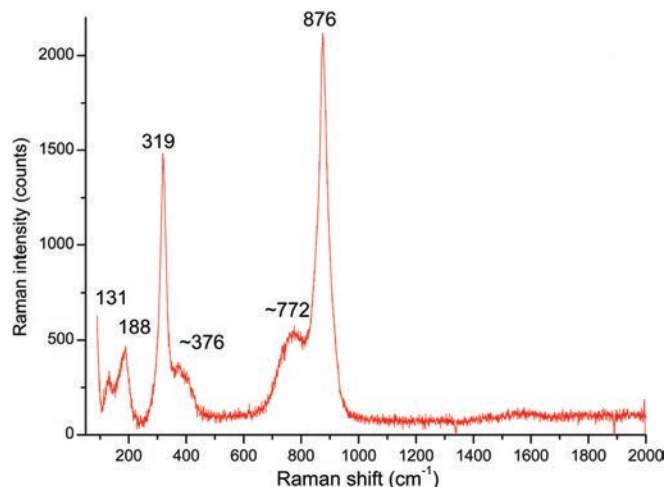


Fig. 3. Micro-Raman spectrum of suseinargiuite. (online version in colour)

The spectrum of suseinargiuite compares very well with those reported by Hanuza *et al.* (1997) for synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$. The following bands have been observed (in cm^{-1} ; in parentheses the wavenumber given by Hanuza *et al.*, 1997): 131 (130), 188 (192), 319 (322), ~ 376 (377), ~ 772 (768), and 876 (876). The observed bands correspond to vibrational modes of (MoO_4) groups.

3. Crystal-chemistry of suseinargiuite

Owing to the lack of suitable crystals, the crystal structure of suseinargiuite was not solved. However, the mineral is the analogue of synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$ whose crystal structure has been reported by several authors (*e.g.*, Teller, 1992; Hanuza *et al.*, 1997; Wařkowska *et al.*, 2005).

Suseinargiuite has a scheelite-type structure, commonly adopted by ABO_4 compositions. In these compounds, A^+ and A^{3+} ions are randomly distributed at the eight-fold coordinated Ca site (using the site notation of the scheelite structure), whereas Mo is tetrahedrally coordinated at the W site. According to Teller (1992), $\text{Na}_{0.5}\text{Bi}_{0.5}\text{MoO}_4$ and $\text{Na}_{0.32}\text{Bi}_{0.56}\square_{0.18}\text{MoO}_4$ adopt the $I4_1/a$ scheelite structure, whereas Hanuza *et al.* (1997) proposed a slightly distorted scheelite structure with space group $I\bar{4}$. Finally, Wařkowska *et al.* (2005) assumed the $I4_1/a$ space group as the correct one for this compound.

As stated above, suseinargiuite has been observed in association with wulfenite. Qualitative chemical data and X-ray powder diffraction patterns suggest a solid solution between suseinargiuite and wulfenite through the substitution mechanism $\text{Na}^+ + \text{Bi}^{3+} = 2\text{Pb}^{2+}$. Chemical analyses showed different $(\text{Na} + \text{Bi})/\text{Pb}$ ratios, whereas the X-ray powder diffraction patterns collected on samples from Su Seinargiu showed a shift in the peak positions, *e.g.*, the d_{112} spacing moves from 3.146 Å in suseinargiuite to 3.24 Å in wulfenite. This is in accordance with the increase of unit-cell parameters from Pb-free synthetic suseinargiuite up to wulfenite (Table 3). Particularly, suseinargiuite has a unit-cell volume (327.4 Å^3) between those of its stoichiometric Pb-free end-member and of wulfenite (~ 322 and 357.5 Å^3 , respectively).

Table 3. Unit-cell parameters of suseinargiuite compared with synthetic compounds of the $(\text{Na}_x\text{Bi}_{1-x})\text{MoO}_4$ series and natural wulfenite.

Compound	<i>a</i> (Å)	<i>c</i> (Å)	<i>V</i> (Å ³)	<i>c/a</i>	(Bi/Na) _{at}
Suseinargiuite	5.296	11.673	327.4	2.2041	variable
Teller (1992) S	5.271	11.5801	321.82	2.1967	0.5/0.5
Teller (1992) NS	5.2785	11.6410	324.35	2.2054	0.56/0.32
Hanuza <i>et al.</i> (1997)	5.267	11.565	320.83	2.1957	0.5/0.5
Wařkowska <i>et al.</i> (2005)	5.2744	11.578	322.09	2.1951	0.5/0.5
Wulfenite – Lugli <i>et al.</i> (1999)	5.434	12.107	357.5	2.2280	

Note: S = stoichiometric; NS = non-stoichiometric

Moreover, it is noteworthy that chemical data show a consistent deficit of Na relatively to Bi. Previous authors (*e.g.*, Sleight *et al.*, 1975; Teller, 1992) observed the occurrence of vacancies in synthetic $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$. Consequently, assuming that no Na volatilization occurs during the electron-microprobe analysis, two possible substitution mechanisms could be invoked to explain the observed chemistry of suseinargiuite:

- i) $\text{Na}^+ + \text{Bi}^{3+} = 2\text{Pb}^{2+}$, with a strong increase of the unit-cell volume (Table 3);
- ii) $3\text{Na}^+ = \text{Bi}^{3+} + 2\square$.

According to Teller (1992), this second substitution also increases the unit-cell volume, principally by an increase of the *c* parameter (Table 3).

Consequently, the formula of suseinargiuite could be written $(\text{Na}_{0.5-x-y}\text{Bi}_{0.5+x/3-y}\square_{2x/3}\text{Pb}_{2y})\text{MoO}_4$ (*Z* = 4). The outer and the inner zones of the analyzed grains correspond to $x \sim 0.13$, $y \sim 0.02$ and $x \sim 0.10$, $y \sim 0.09$.

4. Conclusion

Suseinargiuite is the Na-Bi analogue of wulfenite and fits the 07.GA group of Strunz & Nickel (2001) classification, *i.e.* molybdates without additional anions or H_2O . The qualitative chemical data suggest the possible existence of a wide solid solution between suseinargiuite and wulfenite; unfortunately, owing to the extremely low amount of available material, data are still incomplete and further studies are mandatory to accurately describe the chemical variability in the series $(\text{Na}_{0.5}\text{Bi}_{0.5})\text{MoO}_4$ – PbMoO_4 .

Suseinargiuite is the seventh new mineral species containing Mo found at Su Seinargiu, which therefore confirms its role as a world-class locality for the study of secondary molybdenum minerals.

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