

Vicanite-(Ce): A new Ca-Th-REE borosilicate from the Vico volcanic district (Latium, Italy)

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Abstract: Vicanite-(Ce), ideally $(\text{Ca, REE, Th})_{15}\text{As}^{5+}(\text{As}^{3+}_{0.5}\text{Na}_{0.5})\text{Fe}^{3+}\text{Si}_6\text{B}_4\text{O}_{40}\text{F}_7$, was found inside a volcanic ejectum collected from a pyroclastic formation of the Vico volcanic complex at Tre Croci, Vetralla (north of Rome), Viterbo province, Italy. The host rock is composed of K-feldspar (high sanidine), plagioclase, clinopyroxene plus minor quantities of magnetite and clinoamphibole. Vicanite-(Ce) occurs in small myrolitic cavities in association with zircon, thorite, thorian uraninite, betafite, thorian hellandite, titanite, antimonian asbecasite, apatite, stillwellite-(Ce), and other unidentified phases. It is found as rare euhedral crystals up to 0.3 mm across, yellowish green in colour; uniaxial (-), ϵ 1.722(2), ω 1.757(2), non pleochroic. The streak is white, and the mineral shows no fluorescence under UV radiation. The crystal chemical formula calculated from electron microprobe analyses, on the basis of 47(O+F) and B fixed to 4 atoms, is: $(\text{Ca}_{8.03}\text{Ce}_{2.32}\text{La}_{1.95}\text{Th}_{1.82}\text{Pr}_{0.44}\text{Nd}_{0.28}\text{U}_{0.19})_{\Sigma 15.06}(\text{As}^{5+}_{0.86}\text{P}_{0.14})_{\Sigma 1.00}(\text{As}^{3+}_{0.34}\text{Na}_{0.12})_{\Sigma 0.46}(\text{Fe}^{3+}_{0.57}\text{Ti}_{0.05}\text{Al}_{0.08})_{\Sigma 0.67}\text{Si}_{6.07}\text{B}_4(\text{O}_{36.57}\text{F}_{10.43})_{\Sigma 47}$. Density is higher than 4.2. The mineral is trigonal and crystallizes in the non-centrosymmetric $R\bar{3}m$ space group. The refined unit-cell parameters are: $a = 10.795(1)$, and $c = 27.336(4)$ Å, $Z = 3$ (hexagonal cell). The eight strongest X-ray diffraction lines are [d in Å (I) (hkl)]: 7.70 (50) (012), 4.42 (50) (202), 3.13 (50) (214), 2.993 (100) (027), 2.950 (70) (303), 2.698 (50) (220), 1.839 (50) (3,0,12), 1.802 (50) (2,0,14). The main feature of the structure is the presence of a new polyanion $(\text{Si}_3\text{B}_3\text{O}_{18})^{15-}$. The cell dimensions of vicanite-(Ce) are very close to those of okanoganite-(Y), but the chemical composition is rather different as no Y, Gd, Dy, Sm and Er are present in vicanite-(Ce). The name is for the Vican volcanic complex where the mineral was found. Holotype is deposited in Museo di Mineralogia, Università di Roma "La Sapienza" (MMUR).

Key-words: new mineral, vicanite-(Ce), borosilicate, X-ray data, infrared absorption spectrum, okanoganite-(Y), Vico volcanic district, Italy.

Introduction

The thermometamorphic ejecta which are abundant throughout the Quaternary pyroclastic formations of Latium, Italy, have been a source of rare and new mineral species since the last century. The work of researchers and mineral

collectors in the last few decades has resulted in the discovery of some unusual new minerals, such as latiumite (Tilley & Henry, 1953), tuscanite (Orlandi *et al.*, 1977), cancrinite-like phases (liottite, franzinite, Merlino & Orlandi, 1977a and b; sacrofanite, Burrigato *et al.*, 1980; giuseppetite, Mazzi & Tadini, 1981; pitiglianoite, Merlino

et al., 1991), zeolites (merlinoite, Passaglia *et al.*, 1977, vertumnite, Passaglia & Galli, 1977), and sulphates (cesanite, Cavarretta *et al.*, 1981). However, Th-U-REE-bearing minerals were only known to occur in the sand deposits along the Tyrrhenian coast where thorite (Bonatti & Gallitelli, 1950), perrierite-(Ce) (Bonatti & Gottardi, 1950), and monazite-(Ce) (Gottardi, 1958) were found. Recently, we have focused our attention on the identification of the accessory phases found in the syenitic ejecta, locally called "sanidiniti". This systematic research has led to the discovery of several Th-U-REE-bearing minerals (thorite, Maras, 1982; betafite, De Casa *et al.*, 1986; ekanite, Della Ventura *et al.*, 1986a; baddeleyite, Della Ventura *et al.*, 1986b; stillwellite-(Ce), Burns *et al.*, 1993) and the new mineral peprosiite-(Ce) (Della Ventura *et al.*, 1993). We presume that much work is still left to be done, both for the characterisation of these Th-U-REE-bearing phases and for the petrological and geochemical significance of this association inside this particular type of ejecta.

In June 1985, we received from Francesco S. Stoppani, a prominent mineral collector from Rome, a group of ejecta specimens which contained small bright yellowish green crystals inside myarolitic cavities which were megascopically unidentifiable. Both a preliminary SEM-EDS analysis and a Gandolfi X-ray diffraction pattern indicated that this mineral was a new species with unit-cell parameters similar to those of okanoganite-(Y) (Boggs, 1980) but with an unusual mineral chemistry. A single-crystal structure determination was undertaken in order to fully resolve the composition and the stoichiometry of this mineral (Callegari *et al.*, 1991).

Vicanite-(Ce) has been named after the Vican volcanic district (the Vico lake) where the mineral was found. Both the description and the mineral name were approved by the Commission on New Minerals and Mineral Names, I.M.A. (vote 91-050). Type specimen is deposited in the Museo di Mineralogia, Università di Roma "La Sapienza" (MMUR), Roma, Italy, under catalogue number 24509/1.

Occurrence

Vicanite-(Ce) occurs as rare grains in small myarolitic cavities inside an ejectum of syenitic composition. This ejectum was collected near Tre Croci, Vetralla, Viterbo province, Latium, Italy, in

a pyroclastic level belonging to the Vican volcanic sequence whose stratigraphic position is still rather uncertain. The host rock shows a hornfels-like texture in thin section, and the main constituents are: K-feldspar (high sanidine), plagioclase, clinopyroxene, clinoamphibole and magnetite. Small crystals of biotite are often present as inclusions in the clinoamphiboles. Accessory phases are: apatite, titanite, antimonian asbecasite (Della Ventura *et al.*, 1991; Sacerdoti *et al.*, 1993), thorian hellandite, betafite, thorite, zircon, thorian uraninite, stillwellite-(Ce) and other unidentified phases. Most of the accessory phases appears to have crystallised later than the host rock, except for vicanite-(Ce) and thorian hellandite which are occasionally found enclosed in the K-feldspar mass.

Physical and optical properties

Vicanite-(Ce) occurs as transparent yellowish green anhedral grains or rare rice-grain crystals consisting of a vertical prism terminated by a pyramid, up to 0.3 mm in size (Fig. 1). The streak is white and the lustre is vitreous. Fracture is conchoidal and there is no evidence of any cleavage. Twinning is sometimes present giving a pseudocubic habit.

Neither the hardness nor the density could be accurately measured because of the small crystal size. The hardness is estimated at 5–6 on the Mohs' scale and the density is greater than 4.2 g/cm³ as vicanite-(Ce) sinks in the Clerici solution; the calculated density is 4.73 g/cm³



Fig. 1. Euhedral "rice-grain" crystals of vicanite-(Ce). The mineral aggregate is about 0.8 mm in maximum length.

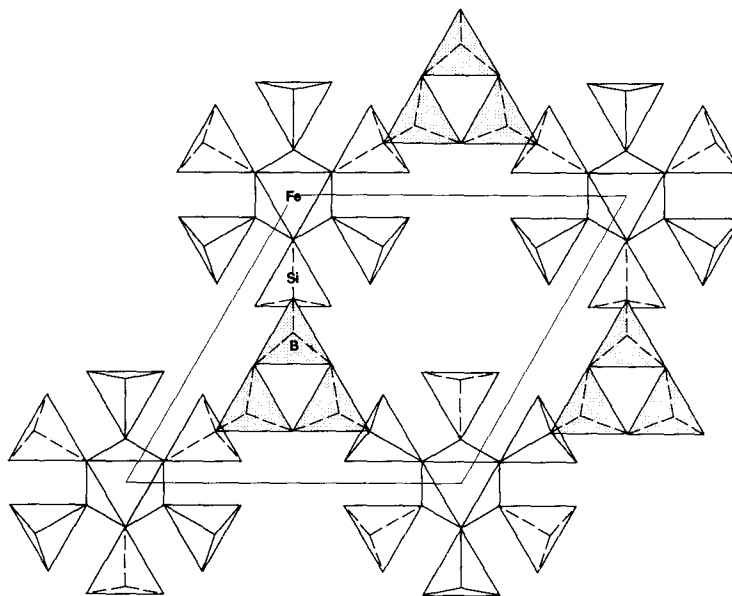


Fig. 2. The vicanite-(Ce) polyanion ($\text{Si}_3 \text{B}_3 \text{O}_{18}$)¹⁵⁻ made up of a ring of three BO_4 tetrahedra, linked via an apex to three SiO_4 tetrahedra.

based on the empirical formula and the measured unit-cell dimensions, with $Z = 3$.

A spindle stage was used to orientate one grain for refractive-index measurements. Vicanite-(Ce) is non-pleochroic, uniaxial negative, with ω 1.757(2), ϵ 1.722(2), measured with gel-filtered Na light ($\lambda = 589.9$ nm). It is non fluorescent in long- or short-wave ultraviolet radiation.

X-ray crystallography

The X-ray diffraction pattern of vicanite-(Ce) was obtained by means of a 114.6-mm diameter Gandolfi camera, Cu $K\alpha$ radiation, Ni filter. The least-squares refinement of the unit-cell parameters was obtained with the computer program LSQ82 (Hubbard, 1982) starting from the preliminary cell parameters obtained with a single-crystal diffractometer. Table 1 gives the X-ray diffraction pattern and refined unit-cell parameters for vicanite-(Ce), compared to those of okanoganite-(Y)

According to the structural refinement (Callegari *et al.*, 1991), the mineral has a rhombohedral symmetry with space group $R\bar{3}m$. The main feature of the structure is the presence of a new polyanion ($\text{Si}_3 \text{B}_3 \text{O}_{18}$)¹⁵⁻, not previously recognized in other borosilicate minerals. It is made up of a ring of three BO_4 tetrahedra, linked via an apex to three SiO_4 tetrahedra. These polyanions are linked to each other by octahedrally coordi-

nated Fe^{3+} , which also links isolated SiO_4 tetrahedra to form a complex layer of polyhedra perpendicular to the c direction. The thickness of this layer is about $1/3 c$ (Fig. 2). The linking of these layers along c is achieved by Ca, REE and Th atoms, whose coordination sphere is completed by additional O and F atoms. These O and F ions do not belong to the Fe-Si-B polyhedral layer, but to additional interlayer cations, namely: B1 (in triangular coordination); As1 (in tetrahedral coordination); As2 (in pyramidal coordination) and Na (in distorted ten-fold coordination). Both anionic and cationic interlayer positions may be vacant due to local charge balance. In particular the (As2) site is only half occupied by As^{3+} .

Chemical composition

Microchemical analyses of vicanite-(Ce) were obtained with Cameca Camebax Micro (Orléans) and SX50 (Rome) electron microprobes. Experimental conditions for both were 15 kV operating voltage, 10 nA sample current and a 10 μm beam diameter. Data were collected by a WDS system and corrected by a ZAF program. About forty chemical analyses were carried out over three different grains using the following standards: diopside (Si), rutile (Ti), albite (Na, Al), ThO_2 (Th); uraninite (U), fluorapatite (Ca, P, F), arsenic (As), Fe_2O_3 (Fe), zircon (Zr) and Drake & Weill's (1972) glasses for La, Ce, Pr, Nd. For

Table 1. Comparison between X-ray powder diffraction data of vicanite-(Ce) from Tre Croci and okanoganite-(Y).

I	vicanite-(Ce)			okanoganite-(Y)			
	d(Å) _{meas.}	d(Å) _{calc.}	h	k	l	d(Å) _{meas.}	I
50	7.70	7.72	0	0	3	9.01	4
			0	1	2	7.67	12
			1	0	4	5.48	7
			1	1	0	5.35	7
			0	1	5	4.67	13
40	4.61	4.61	0	2	1	4.57	5
50	4.42	4.42	2	0	2	4.35	41
30	3.60	3.60	0	2	4	3.82	4
			1	0	7	3.57	20
			2	0	5	3.51	18
10	3.48	3.48	1	2	2	3.39	16
			1	1	6		
			2	1	4	3.11	48
50	3.13	3.14	0	0	9	3.01	15
100	2.993	2.997	0	2	7	2.970	100
70	2.950	2.949	1	2	5	2.939	95
			3	0	3	2.926	50
30	2.757	2.759	2	0	8	2.734	15
50	2.698	2.699	2	2	0	2.676	32
10	2.622	2.620	2	1	7, 1 0 10	2.598	20
10	2.572	2.572	1	3	1	2.564	9
			3	0	6	2.554	9
			3	1	2	2.525	7
			1	3	4	2.404	4
			0	1	11	2.377	4
			0	2	10	2.338	4
			3	1	5	2.318	4
			0	4	2	2.285	4
			4	0	4	2.193	6
			3	0	9	2.154	26
			1	3	7	2.141	29
			2	3	2	2.101	13
			3	2	4	2.030	24
			2	2	9	1.996	19
			4	1	3	1.978	35
0	2	13	1.897	4			
40	1.881	1.881	3	2	7, 1 3 10	1.866	12
50	1.839	1.839	3	0	12	1.822	32
50	1.802	1.802	0	0	15, 2 3 8	1.800	7
			2	0	14	1.784	43
			5	0	5	1.755	18
10	1.770	1.769	1	2	14	1.695	4
50	1.686	1.686	0	5	7	1.672	16
50	1.572	1.573	1	5	5, 0 2 16	1.590	6
			3	0	15	1.558	6
			3	3	9	1.535	12
10	1.544	1.543	0	5	10	1.532	13
10	1.521	1.521	6	0	3	1.527	10
			2	0	17		
			4	1	12	1.507	9
10	1.501	1.501	3	2	13		
			5	0	2	1.487	7

Table 1. Continued.

I	vicanite-(Ce)		h	k	l	okanoganite-(Y)				
	d(Å) _{meas.}	d(Å) _{calc.}				d(Å) _{meas.}	I			
10	1.479	1.480	3	4	5	1.469	10			
			5	2	3	1.466	12			
			2	3	14	1.432	4			
			4	2	11	1.428	3			
			5	2	6, 6 1 2	1.409	3			
			3	5	1, 3 2 16	1.325	8			
			5	3	2, 2 1 19	1.318	10			
			0	4	17	1.313	3			
			10	1.286	1.286	6	0	12, 2 3 17	1.274	9
						1	2	20	1.262	4
10	1.265	1.265	1		21					
10	1.251	1.251	5	2	12	1.241	6			
			0	7	8, 5 3 8	1.238	6			
			4	3	13, 6 1 11	1.229	3			
			6	2	7, 1 7 3	1.221	3			
						1.118	8			
						1.184	8			
						1.180	3			
						1.143	9			
			1.135	7						

Space group
R3m *R3, R3̄, R32, R3m, R3̄m, R3c or R3̄c*
a(Å) = 10.795(1) *a*(Å) = 10.72(1)
c(Å) = 27.336(1) *c*(Å) = 27.05(8)
c/a = 2.532 *c/a* = 2.523
Z = 3 *Z* = 3

Table 2. Composition (wt%) of vicanite-(Ce) and okanoganite-(Y).

	vicanite-(Ce)		okanoganite-(Y)	vicanite-(Ce)		okanoganite-(Y)
SiO ₂	13.82	(12.84–14.81)	14.35	Y ₂ O ₃	bd	20.46
Al ₂ O ₃	0.16	(0.10– 0.22)	–	La ₂ O ₃	12.01 (11.83–12.11)	5.88
TiO ₂	0.14	(0.06– 0.19)	0.50	Ce ₂ O ₃	14.41 (14.13–14.56)	15.42
P ₂ O ₅	0.38	(0.26– 0.46)	–	Pr ₂ O ₃	2.77 (2.61– 3.08)	1.83
As ₂ O ₃	4.49	(3.46– 5.52)*	–	Nd ₂ O ₃	1.79 (1.67– 1.86)	7.70
Fe ₂ O ₃	1.66	(1.60– 1.75)	–	Sm ₂ O ₃	bd	1.64
FeO	–		1.60	Gd ₂ O ₃	bd	5.28
CaO	17.07	(16.90–17.32)	3.24	Tb ₂ O ₃	bd	0.44
Na ₂ O	0.14	(0.12– 0.15)	2.74	Dy ₂ O ₃	bd	2.08
ThO ₂	18.24	(17.91–18.57)	–	Ho ₂ O ₃	bd	0.96
UO ₂	1.96	(1.85– 2.05)	–	Er ₂ O ₃	bd	1.26
PbO	bd		0.63	Tm ₂ O ₃	bd	0.20
RE ₂ O ₃	30.98	(30.24–31.61)	64.75	Yb ₂ O ₃	bd	0.79
B ₂ O ₃	5.27**		3.1	Lu ₂ O ₃	bd	0.81
F	7.50	(7.06– 8.41)	11.15	total	30.98	64.75
	101.81		102.06			
–O=F	3.16		4.69			
total	98.65		97.37			

* see text for partition as As³⁺ and As⁵⁺
** calculated to give 4 B in the formula
bd = below detection

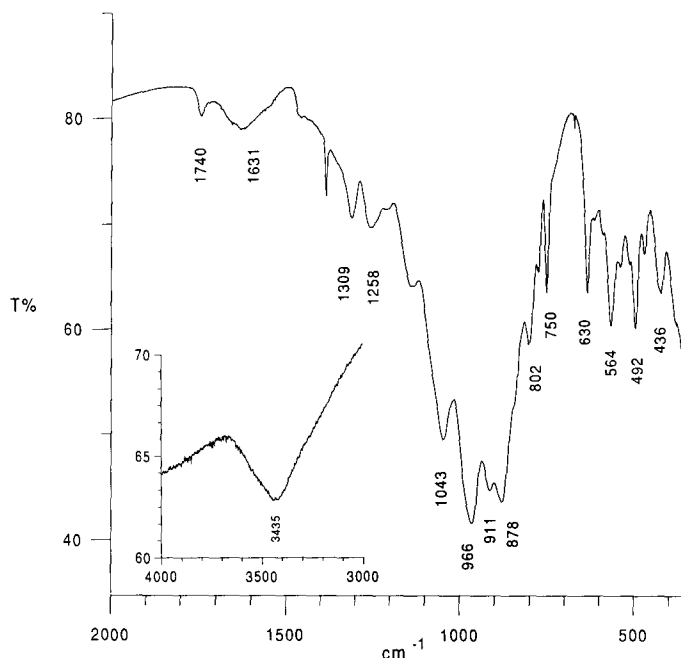


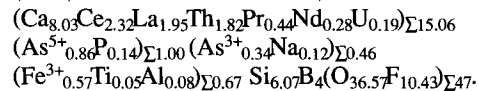
Fig. 3. Infrared absorption spectrum of vicanite-(Ce).

REE peak overlap the precautions suggested by Roeder (1985) were used. The analysis was duplicated because the first results at Orléans produced low analytical totals suggesting the presence of several wt% of unanalysed element(s) other than oxygen. After repolishing and recoating the sample, two further analytical runs were made in Rome which confirmed the low totals of 93 to 94 wt% oxide, with no other element observed on either energy-dispersive or wavelength-dispersive spectral scans. A comparison of the results showed them to be in excellent agreement for Si, Al, Ti, P, Fe and Ca, whereas significant differences were found in both actinide and REE concentrations. Although $\Sigma(\text{REE}_2\text{O}_3 + \text{ThO}_2 + \text{UO}_2)$ values are relatively constant, there are significant differences in the actinide concentration and in some REE, in particular La and Pr.

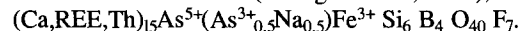
Backscattered-electron images showed a patchy zoning with brighter regions richer in the actinide elements. The smallest grain showed only one bright and well delimited region, slightly richer in the actinide elements. The analyses of this grain obtained in Orléans and Rome are in good agreement; they are presented in Table 2 as the mean value of 4 point analyses. Iron was analysed as total iron and considered as Fe^{3+} on the basis of the structure determination. Boron was detected

by ion microprobe. The measured value, 5.77 wt% B_2O_3 , must be high since the 4 B atoms indicated by the structure refinement would amount to 5.27 wt% B_2O_3 . This slight discrepancy is probably due to a matrix effect and/or to a higher B concentration in vicanite-(Ce) than in the synthetic and natural glasses (containing 15 wt% Al_2O_3 and between 0.03 and 0.62 wt% B_2O_3) used as B standards. Furthermore, as the structural refinement indicates, As is present as both As^{3+} and As^{5+} ; in the calculation of the crystal-chemical formula it was partitioned into As^{5+} so as to fill the cation site, and the remainder into As^{3+} . Values obtained (0.86 As^{5+} and 0.34 As^{3+}) correspond to 3.76 wt% As_2O_5 and 1.26 wt% As_2O_3 , respectively. Adding these values to the chemical analysis, the total becomes 99.18 %, closer to the theoretical composition.

The empirical formula, calculated on 47 (O+F) and B fixed to 4 atoms, is:



The simplified formula, as derived by the structure determination (Callegari *et al.*, 1991), is:



Due to the scarcity of material H_2O was not analysed. Only an infra-red spectrum was obtained,

which indicates that the mineral is anhydrous. The chemical compositions of vicanite-(Ce) and okanoganite-(Y) are compared in Table 2.

Infra-red spectroscopy

The infra-red spectrum of vicanite-(Ce) (Fig. 3) was recorded on a Perkin Elmer 1760 Fourier transform spectrophotometer equipped with a 7700 data-processing terminal. The sample was prepared by adding 1 mg of finely ground pure material to 200 mg of KBr and pressing the mixture into a pellet for 10 min under a load of 9000 kg. Eighty scans were averaged in the wave-number range 4000–250 cm^{-1} with a nominal resolution of 2 cm^{-1} .

The infra-red spectrum in the region 4000 to 2000 cm^{-1} shows no absorption bands attributable to OH or H₂O. The numerous absorption bands in the region 1200–250 cm^{-1} are very difficult to assign because of the chemical and structural complexity of the mineral.

Discussion

The cell parameters of vicanite-(Ce) are very close to those of okanoganite-(Y) and the physical properties too (Table 3), but the structure of okanoganite-(Y) is not known although the range of the possible space groups for the mineral does include that found for vicanite-(Ce); however, the chemical composition is rather different (see Table 2). No Y, Gd, Dy, Sm and Er are present in vicanite-(Ce), whereas no As, Th and U are present in okanoganite-(Y). Thus this mineral is interesting not only from a structural and mineralogical point of view, but also for the unusual crystal chemical distribution of the elements: as a matter of fact, it represents the first mineral with the polyanion $(\text{Si}_3 \text{B}_3 \text{O}_{18})^{15-}$ and is, besides dixenite, kraisslite and mcgovernite, yet another example of a silicate (as far as we know the first borosilicate) where As has been found both as As^{3+} and As^{5+} within the structure.

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Table 3. Physical properties for vicanite-(Ce) and okanoganite-(Y).

vicanite-(Ce)	$D_{\text{meas.}}$	okanoganite-(Y)
>4.28(2)		4.35(4)
Optical properties		
	Na light	White light
ω	1.757(2)	1.753(2)
ϵ	1.722(2)	1.740(2)

indebted to A.C. Roberts (Ottawa) for improving the English, M. Chaussidon (CNRS Nancy) for the B determination, the technicians of the Cartography Laboratory of the D.S.T. (Rome) for the drawing, and S. Fiori for supplying the picture of the vicanite-(Ce).

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