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KRIVOVICHEVITE, $Pb_3[Al(OH)_6](SO_4)(OH)$, A NEW MINERAL SPECIES FROM THE LOVOZERO ALKALINE MASSIF, KOLA PENINSULA, RUSSIA

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Abstract

Krivovichevite, $Pb_3[Al(OH)_6](SO_4)(OH)$, is a new sulfate found in a natrolite – aegirine – orthoclase vein within lujavrite at Mt. Lepkhe–Nelm, Lovozero alkaline massif, Kola Peninsula, Russia. It occurs as large (up to 1 cm diameter) equant grains within galena aggregates in natrolite, in intimate association with anglesite, cerussite, hydrocerussite, leadhillite and lanarkite. It is a late hydrothermal mineral produced by alteration of galena. Other associated minerals are anatase, arsenopyrite, fluorapatite, fluorite, halloysite, heulandite-Ca, heulandite-Sr, kupletskite, lamprophyllite, leucophanite, magnesio-arfvedsonite, manganneptunite, microcline, monazite-(La), polylithionite, quartz, opal, rhabdophane-(Ce) and titanite. The mineral is transparent and colorless, with a vitreous luster and a white streak. A cleavage was not observed, and the fracture is conchoidal. The Mohs hardness is about 3. The mineral is brittle. Krivovichevite is uniaxial (–): n(meas) ≈ 1.9 (589 nm), n(calc.) = 1.85. It is non-pleochroic. The mean chemical composition, determined with an electron microprobe is: PbO 76.49, CaO 0.02, Al_2O_3 5.38, SO_3 9.27, H_2O (Penfield method) 7.20, total 98.36 wt.%. The empirical formula, calculated on the basis of Pb + Ca + Al + S = 5 *apfu* is $Pb_{3.04}Al_{0.94}(S_{1.03}O_{3.98})(OH)_{7.08}$. An ideal formula taking into account the single-crystal study of its structure is: $Pb_3[Al(OH)_6](SO_4)(OH)$. Krivovichevite is trigonal, $R3c$, a 7.693(8), c 31.57(9) Å, V 1618(6) Å^3, Z = 6. The strongest six X-ray powder-diffraction lines [d in Å(I)(hkl)] are: 3.58(100)(201), 3.10(60)(116), 2.591(90)(119), 2.216(50)(030), 2.048(70)(036), 1.704(80)(317). The structure of krivovichevite has been refined to R_1 = 0.034 for 681 reflections. It contains isolated $Al(OH)_6^{3-}$ octahedra and SO_4^{2-} tetrahedra, Pb^{2+} cations and OH^- anions. The infrared spectrum indicates the presence of structural $(OH)^-$ and sulfate absorption bands. The mineral is named in honor of Sergey Vladimirovich Krivovichev, Russian mineralogist and crystallographer, Professor at St. Petersburg State University. Krivovichevite belongs to a new structure-type of minerals and inorganic compounds. Chemically related to krivovichevite are minerals of the beudantite and alunite groups.

Keywords: krivovichevite, new mineral species, sulfate, Lovozero alkaline massif, Kola Peninsula, Russia.

Sommaire

La krivovichevite, $Pb_3[Al(OH)_6](SO_4)(OH)$, nouvelle espèce minérale sulfatée, a été découverte dans une veine à natrolite – aegyrine – orthoclase recoupant la lujavrite au mont Lepkhe–Nelm, complexe alcalin de Lovozero, péninsule de Kola, en Russie. On la trouve en cristaux équidimensionnels atteignant 1 cm dans des aggrégats de galène dans la natrolite, en association intime avec anglésite, cérussite, hydrocérussite, leadhillite et lanarkite. Il s'agit d'un minéral tardif résultant de l'altération de la galène. Lui sont associés anatase, arsénopyrite, fluorapatite, fluorite, halloysite, heulandite-Ca, heulandite-Sr, kupletskite, lamprophyllite, leucophanite, magnésio-arfvedsonite, mangan-neptunite, microcline, monazite-(La), polylithionite, quartz, opale, rhabdophane-(Ce) et titanite. Le minéral est transparent et incolore, avec un éclat vitreux et une rayure blanche. Aucun clivage n'a été décelé, et la fracture est conchoïdale. La dureté de Mohs est d'environ 3. Le minéral est cassant. La krivovichevite est uniaxe (–): n (mesuré) ≈ 1.9 (589 nm), n (calculé) = 1.85. Elle est non pléochroïque. La composition chimique moyenne, déterminée avec une microsonde électronique, est: PbO 76.49, CaO 0.02, Al_2O_3 5.38, SO_3 9.27, H_2O (méthode de Penfield) 7.20, pour un total de 98.36% (poids). La formule empirique, calculée en fixant Pb + Ca + Al + S à 5 atomes par formule unitaire, est $Pb_{3.04}Al_{0.94}(S_{1.03}O_{3.98})(OH)_{7.08}$. La formule idéale proposée, fondée en partie sur les résultats de l'ébauche de la structure sur monocristal, est: $Pb_3[Al(OH)_6](SO_4)(OH)$. La krivovichevite est trigonale, $R3c$, a 7.693(8), c 31.57(9) Å, V 1618(6) Å^3, Z = 6.

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Les six raies les plus intenses du spectre de diffraction, méthode des poudres [d en Å(I)(hkl)] sont: 3.58(100)(201), 3.10(60)(116), 2.591(90)(119), 2.216(50)(030), 2.048(70)(036) et 1.704(80)(317). La structure de la krivovichevite a été affinée jusqu'à un résidu R_1 de 0.034 en utilisant 681 réflexions. Elle contient des octaèdres $Al(OH)_6^{3-}$ isolés, des tétraèdres SO_4^{2-}, des cations Pb^{2+} et des anions OH^-. Le spectre d'abosorption infrarouge indique la présence de groupes $(OH)^-$ structuraux et des bandes d'absorption attribuées au groupe sulfate. Le nom choisi honore Sergey Vladimirovich Krivovichev, minéralogiste et cristallographe russe, professeur à l'université de Saint Petersbourg. La krivovichevite représenterait un nouveau type structural parmi les minéraux et les composés inorganiques. Lui sont apparentées chimiquement les minéraux des groupes de la beudantite et de l'alunite.

(Traduit par la Rédaction)

Mots-clés: krivovichevite, nouvelle espèce minérale, sulfate, complexe alcalin de Lovozero, péninsule de Kola, Russie.

Introduction

The Lovozero alkaline massif is one of the largest (about 650 km^2) intrusive bodies of nepheline syenite of the foyaite–lujavrite series and foidolite (Fig. 1). It intrudes Archean granitic gneisses (2.8 Ga) and has a Devonian age, 360 Ma (Kogarko *et al.* 1983). The massif has stratified structure; a subhorizontal differentiated complex of rhythmically alternating lujavrite, foyaite and foidolite (about 800 m thick) is covered by a more uniform complex of eudialyte lujavrite (about 200 m thick). Both complexes contain numerous conformal lens-shaped bodies of poikilitic nepheline, sodalite and nosean syenites, xenoliths of Devonian volcanic-sedimentary rocks, pegmatites and hydrothermally affected rocks.

The Lovozero alkaline massif is characterized by a unique variety and diversity of mineral species (Semenov 1972, Khomyakov 1995, Iwanjuk *et al.* 1997, Pekov 2000). In this respect, it can be compared only with the Khibiny (Kola Peninsula, Russia) and the Mont Saint-Hilaire (Canada) agpaitic nepheline syenite complexes. Krivovichevite was discovered in one of the frequently sampled and well-studied veins found by

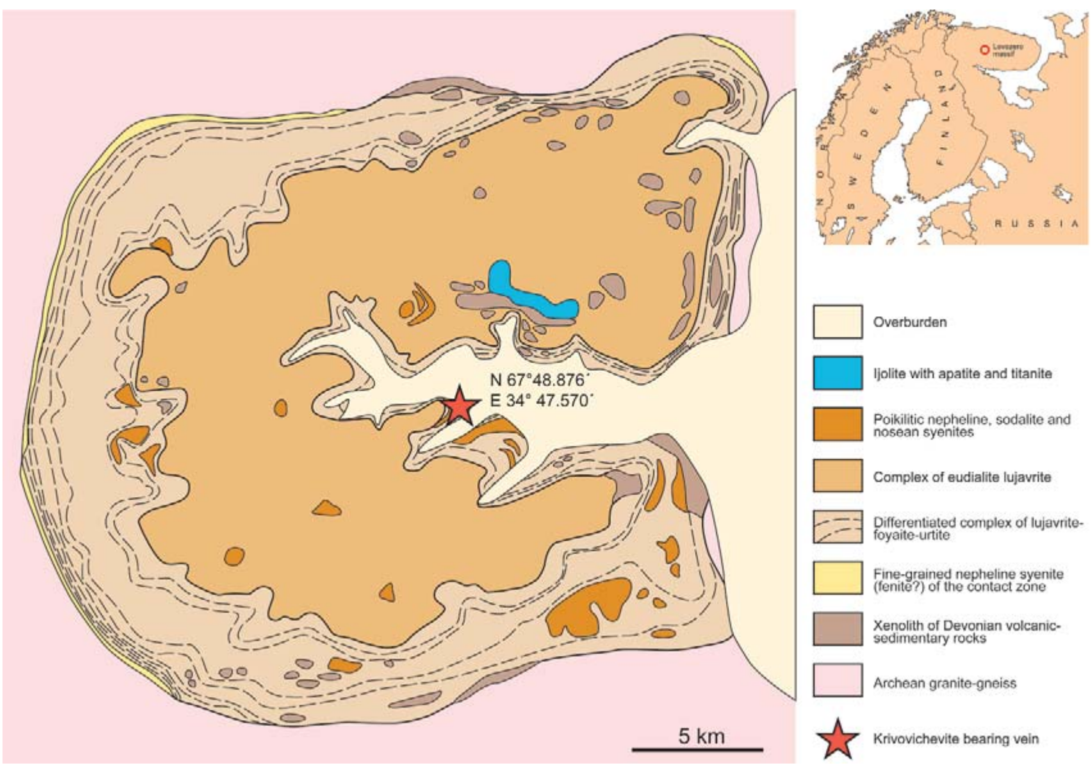

Fig. 1. Simplified geology of the Lovozero massif according to Bussen & Sakharov (1972), with additions.

Semenov in 1948 within lujavrite at Mt. Lepkhe–Nelm (Fig. 1). The vein is the type locality of kupletskite (Semenov 1956) and krivovichevite (this article).

Krivovichevite-bearing specimens were sampled in 2003 in the dumps of the natrolite core of the vein. X-ray powder-diffraction patterns and the chemical composition of all collected minerals were checked, and the study revealed several unknown mineral phases, including a new Pb–Al sulfate.

The mineral has been named krivovichevite (кривовичевит in Cyrillic) in honor of Sergey Vladimirovich Krivovichev (b. 1972), Russian mineralogist and crystallographer, Professor at the St. Petersburg State University, for his contributions to mineralogy of alkaline massifs as well as structural mineralogy of sulfate and Pb minerals (Hawthorne *et al.* 2000, Krivovichev & Burns 2000a, 2000b, 2002, 2003).

Both mineral and mineral name have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association (no. 2004–053). The holotype specimen of krivovichevite is deposited at the Mineralogical Museum of St. Petersburg State University, and a cotype specimen is deposited at the Geological and Mineralogical Museum of the Geological Institute of the Kola Science Centre of Russian Academy of Sciences, Apatity, Russia (no. 6281/1.07.2005).

Occurrence

Krivovichevite was found in a large (about 30 × 15 m) isometric concentrically zoned vein (Fig. 2), the marginal zone of which (up to 3 m wide) is composed by extremely coarse-grained aggregate of orthoclase, nepheline, sodalite, aegirine, magnesio-arfvedsonite, eudialyte and lamprophyllite. The intermediate zone (up to 4 m wide) consists of giant (up to 1 m long) orthoclase crystals, interstices of which contain aegirine, lamprophyllite and magnesio-arfvedsonite. The core zone (up to 6 m diameter) is made of columnar milky white natrolite with inclusions of tabular microcline crystals, elongate-prismatic magnesio-arfvedsonite crystals, Sr-rich fluorapatite, as well as numerous late-formed hydrothermal minerals: aegirine, anatase, anglesite, arsenopyrite, galena, fluorite, goethite, halloysite, heulandite-Ca, heulandite-Sr, kupletskite, leucophanite, mangan-neptunite, monazite-(La), mont-

FIG. 2. Natrolite – aegirine – orthoclase vein in lujavrite, Mt. Lepkhe–Nelm.

FIG. 3. Krivovichevite grain, which look like ice, with another sulfate of lead, covering a galena crystal, from a natrolite – aegirine – orthoclase vein in lujavrite, Mt. Lepkhe–Nelm. Field of view: 25 × 14 mm.

morillonite, polylithionite, rhabdophane-(Ce), sphalerite and titanite (Semenov 1972, Pekov 2000, our data). Contacts between all zones are gradational. Around the vein, the host-rock lujavrite is metasomatically altered to poikilitic nepheline syenite and microclinite (up to 6 m wide).

Krivovichevite forms large (up to 1 cm diameter), colorless, isometric grains (Fig. 3) within compact to porous polymineralic pseudomorphs (up to 2 cm diameter) after galena grains within columnar milky white natrolite. Other components of these pseudomorphs are anglesite, cerussite, hydrocerussite, lanarkite, leadhillite and several unknown Pb-bearing phases. Anglesite, $Pb(SO_4)$, occurs as colorless elongate-prismatic crystals (up to 3 mm long) on the walls of cavities within leached galena and natrolite. Lanarkite, $Pb_2(SO_4)O$, forms white flattened-prismatic crystals (up to 2 mm long) in voids in leached natrolite. Porous fine-grained leadhillite, $Pb_4(SO_4)(CO_3)_2(OH)_2$, fills voids (up to 3 mm diameter) within natrolite around leached grains of galena. Cerussite, $Pb(CO_3)$, forms thin (up to 1 mm wide) rinds consisting of colorless tabular crystals around galena inclusions in natrolite. Druses of small (up to 0.5 mm across) colorless plates of hydrocerussite, $Pb_3(CO_3)_2(OH)_2$, incrust walls of cavities in leached natrolite. X-ray powder data of all these minerals are in agreement with their standard powder X-ray-diffraction patterns (PDF–2 database).

Physical and Optical Properties

Macroscopically, krivovichevite is pale gray to colorless with a vitreous luster. The mineral is transparent and non-fluorescent. The streak is white. No cleavage is observed, and the fracture is conchoidal. The mineral is brittle, and has the Mohs hardness of about 3.

The mineral was found to be easily soluble in water and heavy liquids as well as alcohol, benzine, and acetone, which makes the determination of its density and optical properties a difficult task. In transmitted light, the mineral is colorless and nonpleochroic. Krivovichevite is uniaxial (–): n(meas.) ≈ 1.9; n(calc.) = 1.85. The calculated density is 5.37 gcm^{-3}. These incomplete data result in a poor Gladstone–Dale index of compatibility, 0.084 (Mandarino 1981).

Chemical Composition

The chemical composition of krivovichevite has been studied using a Cameca MS–46 electron microprobe (wavelength-dispersion mode) at the Geological Institute, Kola Science Centre of the Russian Academy of Sciences, Apatity, operated at 20 kV (25 kV for Pb) and 30 nA. The beam diameter was chosen to be 30 μm because of the instability of krivovichevite under a more focused beam. The following standards were used: synthetic PbSe (Pb), pyrope (Al), diopside (Ca) and barite (S). The H_2O content was determined by the Penfield method in purified material. Table 1 provides an average result for five analyzed grains (every analysis is average of 6–10 points for each grain).

The empirical formula of krivovichevite (based on Pb + Ca + Al + S = 5 *apfu*) is: $Pb_{3.04}\,Al_{0.94}\,(S_{1.03}\,O_{3.98})(OH)_{7.08}$. The simplified formula is $Pb_3Al(SO_4)(OH)_7$, which requires: PbO 77.53, Al_2O_3 5.90, SO_3 9.27, H_2O 7.30, total 100.00 wt.%.

The chemical compositions of Pb-bearing minerals intimately associated with krivovichevite are given in Table 2.

Crystal Structure

The crystal structure of krivovichevite has been solved using data collected by means of a CCD-type area detector in the precession method (Krivovichev *et al.*, in prep.). The structure belongs to a new structure-type of minerals and inorganic compounds. The structure contains isolated $Al(OH)_6^{3-}$ octahedra and $(SO_4)^{2-}$ tetrahedra, Pb^{2+} cations and OH^- anions. The structure is polar one because all $(SO_4)^{2-}$ tetrahedra have the same orientation along the c axis. Krivovichevite belongs to a family of Pb minerals and inorganic compounds based upon a pseudohexagonal arrangement of Pb^{2+} cations (Krivovichev & Burns 2000b). We consider it chemically related to minerals of the beudantite–alunite family (Hawthorne *et al.* 2000).

The structural formula for krivovichevite calculated on the basis of a single-crystal structure study can be written as $Pb_{3.04}[Al_{0.94}(OH)_{6.00}](S_{1.03}O_{3.98})(OH)_{1.08}$, and the simplified formula, as $Pb_3[Al(OH)_6](SO_4)(OH)$.

According to the single-crystal structure study, krivovichevite is trigonal, $R3c$. The powder X-ray-diffraction pattern of krivovichevite was obtained by means of the URS–1 instrument operated at 40 kV and 30 mA with a 114.7 mm Debye–Scherrer camera and Fe$K\alpha$ radiation (Table 3). The unit-cell parameters refined from the powder data are a 7.693(8), c 31.57(9) Å, V 1618(6) Å^3, Z = 6.

TABLE 1. CHEMICAL COMPOSITION OF KRIVOVICHEVITE

	Mean	Range	Standard deviation
PbO wt.%	76.49	75.67 – 77.05	0.60
CaO	0.02	0.00 – 0.06	0.03
Al_2O_3	5.38	4.88 – 5.44	0.25
SO_3	9.27	7.15 – 9.35	0.90
H_2O*	7.20		
Total	98.36		

Average result for five analyzed grains. * Determined by the Penfield method.

Infrared Spectroscopy

The infrared-absorption spectrum of purified material of krivovichevite was recorded using a UR–20 spectrometer. The spectrum (Fig. 4) is similar to spectra of minerals of the alunite–jarosite series (Bishop & Murad 2005) and shows 13 absorption bands caused by vibrations of metal–oxygen bonds, sulfate and $(OH)^-$ groups (Table 4), which is in a good agreement with theoretical C3v sulfate spectrum. Medium to strong peaks at 447, 610, 720 and 940 cm^{-1}, as well as the strongest doublet at 1084 and 1114 cm^{-1}, are due to sulfate-group vibrations. Bands at 595, 1023, 3410 and 3486 cm^{-1} can be assigned to both the stretching and bending vibrations of $(OH)^-$ groups. Medium to strong peaks at 458, 505 and 518 cm^{-1} are probably due to Al–O vibrations (Bishop & Murad 2005).

Discussion

Polymineralic pseudomorphs formed by the alteration of galena within the vein under consideration are usually porous and do not contain krivovichevite and other soluble minerals; we thus assume that krivovichevite could be leached from these pseudomorphs by meteoric water during supergene alteration. Therefore, we believe krivovichevite to be a late-stage hydrothermal mineral produced by low-temperature (<100°C) alteration of galena and natrolite by oxidizing aqueous

TABLE 2. CHEMICAL COMPOSITION OF Pb-BEARING MINERALS INTIMATELY ASSOCIATED WITH KRIVOVICHEVITE

	Anglesite	Lanarkite	Leadhillite	Cerussite	Hydrocerussite
PbO wt.%	73.73	84.56	76.83	84.10	86.38
SiO_2	0.06			0.29	0.67
SO_3	26.90	14.60	8.02		
CO_2*			7.15	16.18	10.38
H_2O*			1.47		2.33
Total	100.69	99.16	93.47	100.57	99.76

* Calculated on the basis of 4 *apfu* Pb for leadbillite, 1 *apfu* Pb for cerussite and 3 *apfu* Pb for hydrocerussite.

TABLE 3. X-RAY POWDER-DIFFRACTION DATA FOR KRIVOVICHEVITE

I	d_{obs}	d_{calc}	*h k l*	*I*	d_{obs}	d_{calc}	*h k l*
3	6.06	6.138	1 0 2	1	1.678	1.680	2 1 14
3	5.24	5.262	0 0 6			1.673	1 3 8
2	5.07	5.091	0 1 4	3	1.658	1.656	4 0 2
2	3.82	3.846	1 1 0	2	1.632	1.630	0 4 4
10	**3.58**	**3.613**	**1 1 3**	3	1.610	1.611	4 0 5
2	3.24	3.313	2 0 1	3	1.600	1.596	1 1 18
6	3.10	3.105	1 1 6	4	1.559	1.554	1 3 11
1	3.05	3.069	2 0 4			1.553	1 2 16, 2 2 12
2	2.643	2.636	1 0 11	3	1.523	1.527	2 3 1
9	**2.591**	**2.592**	**1 1 9**			1.521	3 2 2
2	2.544	2.545	0 2 8	3	1.505	1.501	2 3 4
3	2.502	2.510	1 2 1	4	1.477	1.473	0 4 10
1	2.439	2.399	1 2 4			1.471	3 1 13
3	2.338	2.339	2 1 5	3	1.444	1.448	2 3 7
5	**2.216**	**2.221**	**0 3 0**	2	1.439	1.440	1 4 3
2	2.202	2.199	1 2 7	4	1.428	1.429	1 3 14
2	2.177	2.173	3 0 3			1.427	0 2 20
		2.172	1 1 12			1.425	3 2 8
7	2.048	2.046	0 3 6	3	1.414	1.420	2 2 15
1	1.900	1.923	2 2 0	2	1.406	1.403	0 1 22
5	**1.893**	**1.893**	**2 1 11**			1.401	1 4 6
		1.892	2 2 3, 0 1 16	3	1.388	1.387	1 2 19
4	1.859	1.867	0 2 14	1	1.381	1.376	3 0 18
2	1.843	1.845	3 1 1	3	1.348	1.349	3 2 11
2	1.830	1.835	1 3 2			1.349	3 1 16
2	1.808	1.806	2 2 6			1.344	1 0 23
2	1.799	1.799	3 1 4			1.343	1 4 9
4	1.776	1.773	1 3 5	3	1.331	1.331	5 0 1
1	1.756	1.754	0 0 18			1.328	5 0 2
8	1.704	1.710	3 1 7	**5**	**1.318**	**1.318**	**2 0 22**
2	1.691	1.697	0 3 12				
		1.686	2 2 9				

Values of d_{obs} and d_{calc} are expressed in Å.

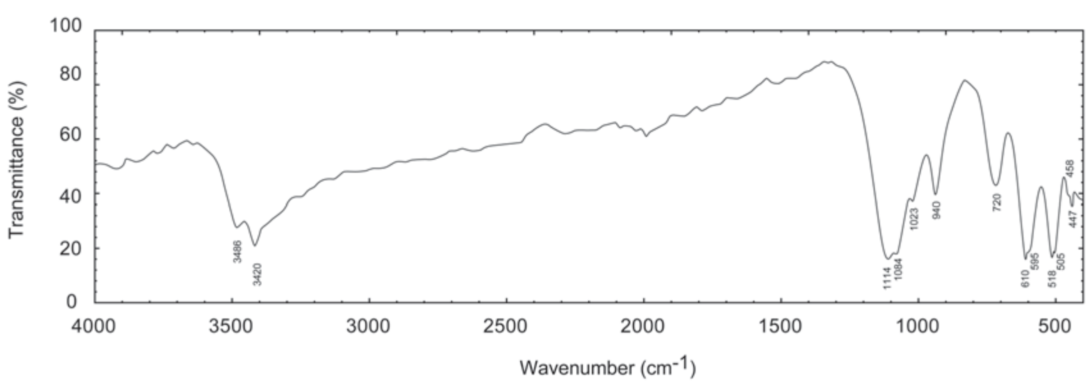

FIG. 4. Infrared-absorption spectrum of powdered krivovichevite.

TABLE 4. ABSORPTION BANDS IN INFRARED ABSORPTION SPECTRA OF KRIVOVICHEVITE, JAROSITE AND SYNTHETIC ALUNITE

Krivovichevite		Jarosite *		Synthetic alunite *		Assignment *
447	*m*	446	*w*	422	*m*	$\nu_2\ (SO_4)^{2-}$
458	*sh*					
505	*sh*	475	*s*	500	*w*	(Al, Fe^{3+})–O
518	*s*	511	*s*	518	*w*	
594	*sh*	580	*w*	594	*s*	$\gamma\ (OH)^-$
610	*s*	630	*m*	622	*s*	$\nu_4\ (SO_4)^{2-}$
720	*m*	661	*m*	668	*s*	
940	*m*	995	*sh*	989	*w*	$\nu_1\ (SO_4)^{2-}$
1023	*sh*	1005	*s*	1028	*m*	$\delta\ (OH)^-$
				1070	*sh*	
1084	*sh*	1085	*s*	1083	*s*	$\nu_3\ (SO_4)^{2-}$
1114	*s*	1185	*m*	1225	*m*	
3420	*sh*	3383	*s*	3485	*s*	$\nu\ (OH)^-$
3486	*s*	3405	*sh*	3515	*sh*	

* After Bishop & Murad (2005). The band strength is indicated as follows: *s* for strong, *m* for medium, *w* for weak, and *sh* for shoulder.

solutions; Na-rich opal and quartz are also coproduced by this reaction.

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