The Canadian Mineralogist
Vol. 41, pp. 1167-1172 (2003)

VASILYEVITE, $(Hg_2)^{2+}{}_{10}O_6I_3Br_2Cl(CO_3)$, A NEW MINERAL SPECIES FROM THE CLEAR CREEK CLAIM, SAN BENITO COUNTY, CALIFORNIA

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ABSTRACT

Vasilyevite, a new mineral species of ideal composition (involving complete anion order) $(Hg_2)^{2+}{}_{10}O_6I_3Br_2Cl(CO_3)$, is triclinic, $P\bar{1}$, with unit-cell parameters measured on a single-crystal X-ray diffractometer: a 9.344(2), b 10.653(2), c 18.265(4) Å, α 93.262(5), β 90.548(4), γ 115.422(4)°, V 1638.3(9) Å^3, a:b:c 0.8771:1:1.7145, Z = 2. The strongest seven lines of the X-ray powder-diffraction pattern [d in Å(I)(hkl)] are: 7.645(60)($\bar{1}1\bar{1}$), 4.205(80)($01\bar{4}$), 3.296(50)($\bar{1}15$,105), 3.132(90)($12\bar{3}$,$\bar{1}3\bar{3}$), 2.894(100)($\bar{3}12$,$\bar{3}2\bar{2}$), 2.722(80)(124) and 2.629(50)(130, $\bar{1}40$). The mineral has been identified on five micromount specimens collected from a small prospect pit within the dumps surrounding the long-abandoned Clear Creek mercury mine, New Idria district, San Benito County, California. It is most closely associated with native mercury, eglestonite, montroydite, cinnabar and an undefined Hg oxyhalide in a host rock principally composed of quartz and ferroan magnesite. Vasilyevite occurs in small shallow quartz-lined vugs as anhedral cryptocrystalline masses, less than 0.5 mm in size, and as a somewhat elongate spheroidal anhedral mass, 0.3 mm in diameter, which is partly hollow and has a shell thickness of approximately 30 μm. The mineral is silvery grey to black to dark red-black with a red-brown streak. Physical properties include: adamantine to metallic luster, opaque to translucent (on very thin edges), nonfluorescent, no cleavage, very brittle, uneven fracture, estimated hardness approximately 3, density 9.57 g/cm^3 (calculated from chemical formula and unit-cell parameters derived from crystal structure). In polished section, vasilyevite is weakly bireflectant, nonpleochroic and moderately to strongly anisotropic in green, blue and grey tints. In reflected plane-polarized light, it is grey to white with abundant orange-red to blood-red internal reflections. Measured values of reflectance obtained in air and in oil for a single fragment are tabulated. Averaged and corrected results of electron-microprobe analyses yield Hg_2O 89.1, I 7.0, Br 2.5, Cl 0.6, CO_2 [0.8] (from crystal structure), S 0.1, sum 100.1, less O = I + Br + Cl + S 0.88, total 99.22 wt.%, corresponding to $Hg^{1+}{}_{20.82}O_{6.85}I_{2.69}(Br_{1.52}Cl_{0.82})_{\Sigma 2.34}[(CO_3)_{0.89}S^{2-}{}_{0.15}]_{\Sigma 1.04}$, based on O + I + Br + Cl + S = 14.7

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apfu (atoms per formula unit), as determined from the crystal structure. The original value for Hg, 85.7 wt.%, was converted to Hg_2O after the crystal structure was determined. The infrared-absorption spectrum confirms the presence of CO_3. The mineral name honors V.I. Vasilyev of the Institute of Geology of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia, for his contributions to the study of new and rare Hg-bearing minerals.

Keywords: vasilyevite, new mineral species, mercurous oxide iodide bromide chloride carbonate, electron-microprobe data, reflectance data, X-ray data, Clear Creek mine, San Benito County, California.

Sommaire

La vasilyevite, nouvelle espèce minérale dont la composition idéale (impliquant un degré d'ordre complet) serait $(Hg_2)^{2+}{}_{10}O_6I_3Br_2Cl(CO_3)$, est triclinique, $P\bar{1}$, et ses paramètres réticulaires, tels que mesurés sur monocristal, sont: *a* 9.344(2), *b* 10.653(2), *c* 18.265(4) Å, α 93.262(5), β 90.548(4), γ 115.422(4)°, *V* 1638.3(9) Å^3, *a*:*b*:*c* 0.8771:1:1.7145, *Z* = 2. Les sept raies les plus intenses du spectre de diffraction X [*d* en Å(*I*)(*hkl*)] sont: 7.645(60)($\bar{1}1\bar{1}$), 4.205(80)($01\bar{4}$), 3.296(50)($\bar{1}15$,105), 3.132(90)($12\bar{3}$,$\bar{1}3\bar{3}$), 2.894(100)($\bar{3}12$,$\bar{3}2\bar{2}$), 2.722(80)(124) et 2.629(50)(130,$\bar{1}40$). Nous l'avons identifié sur cinq échantillons micromontés, prélevés dans les haldes entourant l'ancienne mine de mercure de Clear Creek, district de New Idria, comté de San Benito, en Californie. La nouvelle espèce est le plus étroitement associée à mercure natif, eglestonite, montroydite, cinnabre, et un oxyhalogénure de Hg non identifié, dans une roche hôte principalement composée de quartz et de magnésite ferreuse. La vasilyevite se trouve dans des petites cavités peu profondes en masses xénomorphes cryptocristallines, moins de 0.5 mm de diamètre, et dans un cas, en masse sphéroïdale quelque peu allongée, 0.3 mm de diamètre, partiellement creuse, et ressemblant à une coquille dont l'épaisseur est environ 30 μm. Le minéral est gris argenté à noir à rouge foncé noirâtre, avec une rayure rouge-brun. Parmi ses propriétés physiques, on note: éclat adamantin à métallique, opaque à translucide (aux bordures très minces), nonfluorescent, sans clivage, très cassant, fracture inégale, dureté approximative 3, densité 9.57 g/cm^3 (calculée à partir de la formule chimique et des paramètres réticulaires dérivés de l'ébauche de la structure cristalline). En lames polies, la vasilyevite est légèrement biréflectante, non pléochroïque, et modérément à fortement anisotrope, en teintes de vert, bleu et gris. En lumière réfléchie polarisée en plan, elle est grise à blanche avec d'abondants reflets internes orange-rouge à rouge-sang. Nous fournissons les valeurs de réflectance pour un fragment, mesurées dans l'air et dans l'huile. Les résultats moyens et corrigés des analyses à la microsonde électronique sont: Hg_2O 89.1, I 7.0, Br 2.5, Cl 0.6, CO_2 [0.8] (pris de la structure cristalline), S 0.1, somme 100.1, moins O = I + Br + Cl + S 0.88, pour un total de 99.22% (poids), ce qui correspond à $Hg^{1+}{}_{20.82}O_{6.85}I_{2.69}(Br_{1.52}Cl_{0.82})_{\Sigma 2.34}[(CO_3)_{0.89}S^{2-}{}_{0.15}]_{\Sigma 1.04}$, sur une base de O + I + Br + Cl + S = 14.7 atomes par unité formulaire, tel que déterminée selon la structure cristalline. La valeur originale de la concentration du mercure, 85.7% (poids), a été transformée en termes de Hg_2O une fois la structure déterminée. Le spectre d'absorption infrarouge confirme la présence de CO_3. Le nom honore V.I. Vasilyev, de l'Institut de Géologie de la succursale sibérienne de l'Académie des Sciences de Russie, à Novosibirsk, et souligne ses contributions à l'étude des espèces nouvelles et rares des minéraux de mercure.

(Traduit par la Rédaction)

Mots-clés: vasilyevite, nouvelle espèce minérale, oxyde mercureux à iodure, bromure, chlorure et carbonate, données à la microsonde électronique, données de réflectance, données de diffraction X, mine Clear Creek, comté de San Benito, Californie.

Introduction

The new mineral species described here, vasilyevite, was first identified in the spring of 1994 by the senior author during routine X-ray powder-diffraction characterization of a suite of mercury-bearing oxy-halide phases that have iodine as the dominant anion. These minerals were originally collected by one of us (GED) from a small prospect pit within the dumps surrounding the long-abandoned Clear Creek mercury mine, New Idria district, San Benito County, California (lat. 36°22'59"N, long. 120°43'58"W). Subsequent scanning electron microscopy (SEM) and energy-dispersion analysis indicated major Hg with subordinate I, Br and Cl, strongly suggestive of a potentially new mercury halide; results of our subsequent mineralogical study support this contention. This mineral is the third of six new mercury oxyhalides which we hope to fully characterize over the next few years. All must be considered very rare; despite assiduous searches and numerous X-ray powder determinations, only five small vasilyevite-bearing specimens have so far been found.

The mineral is named *vasilyevite* in honor of Vladimir Ivanovich Vasilyev (b. 1929), of the Institute of Geology of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia, for his numerous contributions to the study of new and rare Hg-bearing minerals, particularly those discovered in the former Soviet Union. He is the senior author of the formal descriptions of chursinite $[(Hg_2)^{2+}]_3(AsO_4)_2$, grechishchevite $Hg_3S_2(Br,Cl,I)_2$, kadyrelite $Hg^{1+}{}_4(Br,Cl)_2O$, kelyanite $Hg_{36}Sb_3(Cl,Br)_9O_{28}$, kuzminite $Hg^{1+}{}_2(Br,Cl)_2$, kuznetsovite $[(Hg_3)^{4+}]Cl(AsO_4)$, lavrentievite $Hg_3S_2(Cl,Br)_2$, poyarkovite $Hg^{1+}{}_3ClO$, and shakhovite $Hg^{1+}{}_4SbO_3(OH)_3$. Surprisingly, none of these minerals has, as yet, been identified at Clear Creek. The mineral and mineral name have been approved by the Commission on New Minerals and Mineral Names,

IMA (2003–016). Holotype material, consisting of four micromount specimens, two gelatin capsules with micrometric specks of pure material, one SEM stub and two single-crystal mounts, is housed in the Systematic Reference Series of the National Mineral Collection at the Geological Survey of Canada, Ottawa, Ontario, under catalogue number NMC68094. The polished sections used for both the quantitative reflectance and electron-microprobe studies are preserved at The Natural History Museum, London, U.K., under catalogue number BM2003,5.

Occurrence and Associated Minerals

Vasilyevite is an extremely rare constituent at the Clear Creek claim. We estimate less than 20 μg of material is available for study. It has been identified on five micromount specimens that were collected in the mid-1990s and occurs within quartz-lined vugs less than 0.5 mm across in centimeter-sized quartz veins. The host rock is a brecciated silicate–carbonate rock composed principally of quartz and ferroan magnesite. The vugs containing the new mineral are typically monomineralic. Associated minerals found in adjacent vugs are globules of native mercury, eglestonite, montroydite, cinnabar and an undefined Hg oxyhalide [informally called CCUK–10 in Dunning *et al.* (2004)] that occurs as red-orange fibrous-to-acicular aggregates. In four of the micromounts, vasilyevite occurs as anhedral, almost cryptocrystalline masses, less than 0.5 mm in size, within shallow vugs. In the fifth specimen containing vasilyevite, the mode of occurrence and habit are identical to that of tedhadleyite (Roberts *et al.* 2002), which leads us to believe that the mineral formed *in situ* as a replacement of native mercury during a period of high activity of I (with lower Br and Cl) in the fluid phase. This somewhat elongate spheroidal mass, 0.3 mm in longest dimension, is partly hollow and has no obvious crystal form. The outermost "shell" is approximately 30 μm thick; portions of this were used for quantitative electron-microprobe analyses, quantitative reflectance study and crystal-structure analysis. Other Hg-bearing minerals identified from the Clear Creek claim are listed in Roberts *et al.* (2001) and references therein. To that listing, we can add tedhadleyite $Hg^{2+}Hg^{1+}{}_{10}O_4I_2(Cl,Br)_2$ (Roberts *et al.* 2002) and aurivilliusite $Hg^{2+}Hg^{1+}OI$ (Roberts *et al.* 2004). Dunning *et al.* (2003) provided a complete description of the history, geology, mineralogy (vasilyevite is given the designation CCUK–11) and geochemistry of the Clear Creek claim.

Physical Properties

Vasilyevite is silvery grey to black to dark red-black (similar in color to both wattersite and tedhadleyite), with a red-brown streak. The main mass is opaque, although very thin edges of fragments are translucent. It is very brittle, has an uneven fracture and an adamantine to metallic luster, and is nonfluorescent under both short- and longwave ultraviolet light. There is no cleavage, and the Mohs hardness is approximately 3 (the mineral is easily scratched by a needle), but a more precise value could not be determined. The density could not be measured owing to the dearth of material; the calculated density, obtained on the basis of the chemical formula and unit-cell parameters derived from an investigation of the crystal structure (Cooper & Hawthorne 2003), is 9.57 g/cm^3. Twinning was not observed megascopically, nor was it observed in the crystal-structure or reflectance study.

X-Ray Crystallography

An anhedral fragment of vasilyevite, 0.03 × 0.08 × 0.10 mm in size, was investigated with a Bruker *P*4 four-circle diffractometer equipped with a CCD detector. Its symmetry is triclinic. The crystal-structure determination (Cooper & Hawthorne 2003) indicates that $P\bar{1}$ is the correct space-group. The measured unit-cell parameters from the structure analysis are: *a* 9.344(2), *b* 10.653(2), *c* 18.265(4) Å, α 93.262(5), β 90.548(4), γ 115.422(4)°, *V* 1638.3(9) Å^3, *a*:*b*:*c* 0.8771:1:1.7145.

The unit-cell parameters, *a* 9.250(5), *b* 10.629(4), *c* 18.182(6) Å, α 93.06(4), β 90.35(5), γ 115.16(4)°, *V* 1615(1) Å^3, *a*:*b*:*c* 0.8703:1:1.7106, and Z = 2, were refined from 27 powder reflections representing *d* values between 5.293 and 1.481 Å for which unambiguous indexing was possible on the basis of the calculated intensities derived from the crystal structure. A fully indexed powder-diffraction pattern is presented in Table 1. The powder data are unique and bear no resemblance to any other inorganic phase listed in the Powder Diffraction File, and no synthetic equivalent is known in the chemical literature. This is the first reported mercury oxy-halide carbonate in either natural or synthetic form.

Chemical Composition

A small anhedral fragment of vasilyevite was analyzed with a Cameca SX–50 electron microprobe, with an operating voltage of 20 kV, a beam current of 10 nA, a 10-s count time for both peak and background, and a beam spot 4 μm across. The following standards were used: cinnabar (Hg,S), iodargyrite (I), halite (Cl) and synthetic KBr (Br). An energy-dispersion spectrum indicated the absence of elements with an atomic number greater than 9 other than those reported here. The mineral is very unstable under the electron beam; the longer it is subjected to electrons, the higher the Hg value tends to be above the ideal value. The average of four determinations (and ranges) gave Hg 85.7 (85.3 – 86.4), I 7.0 (6.0 – 7.9), Br 2.5 (2.0 – 2.9), Cl 0.6 (0.6 – 0.7) and S 0.1 (0.0 – 0.1) wt.%. The precision of these values is probably average at best, owing to the aforementioned instability under the electron beam. The

valence state for Hg, as well as the number of O and C atoms, were determined by crystal-structure analysis prior to final interpretation of the electron-microprobe results. The paucity of material prevented quantitative determination of the amount of CO_2. However, the presence of C as CO_3 was confirmed both by crystal-structure analysis and by powder infrared-absorption study; the amount of CO_2 was therefore calculated by stoichiometry. After the crystal structure was successfully determined, the Hg value, given above, was converted to Hg_2O. This gives Hg_2O 89.1, I 7.0, Br 2.5, Cl 0.6, CO_2 [0.8], S 0.1, sum 100.1, less O = I + Br + Cl + S 0.88, total 99.22 wt.%. With O + I + Br + Cl + S = 14.7 *apfu* (atoms per formula unit), as determined from structure, the empirical formula for vasilyevite is $Hg^{1+}_{20.82}O_{6.85}I_{2.69}(Br_{1.52}Cl_{0.82})_{\Sigma 2.34}[(CO_3)_{0.89}S^{2-}_{0.15}]_{\Sigma 1.04}$. The chemical formula, determined from the structure refinement, $(Hg_2)^{2+}_{10}O_6I_3(Br_{1.6}Cl_{1.4})_{\Sigma 3}[(CO_3)_{0.8}S^{2-}_{0.2}]_{\Sigma 1}$, requires Hg_2O 88.38, I 8.06, Br 2.71, Cl 1.05, CO_2 0.75, S 0.14, sum 101.09, less O = I + Br +Cl + S 1.09, total 100.00

TABLE 1. X-RAY POWDER-DIFFRACTION DATA FOR VASILYEVITE

	$I_{est.}$	dÅ$_{(meas.)}$	dÅ$_{(calc.)}$	*hkl*		$I_{est.}$	dÅ$_{(meas.)}$	dÅ$_{(calc.)}$	*hkl*
	3	9.771	9.602	010				2.325	$\bar{3}25$
	5	8.365	8.368	100		40	2.318	2.317	$\bar{3}1\bar{5}$
	60	7.645	7.610	$\bar{1}1\bar{1}$				2.312	$\bar{4}20$
	10	6.797	6.808	$01\bar{2}$		3	2.281	2.279	$31\bar{3}$
	3	6.452	6.400	012	*	5	2.250	2.251	$\bar{3}4\bar{3}$
	3	6.088	6.056	102	*	15	2.200	2.199	$\bar{3}43$
*	30	5.293	5.290	110		15	2.176	2.178	$04\bar{4}$
	5	5.089	5.097	$\bar{1}2\bar{1}$				2.173	$\bar{2}4\bar{5}$
*	20	4.983	4.981	013	*	5	2.139	2.139	$\bar{3}26$
*	5	4.835	4.838	$\bar{1}13$				2.102	$02\bar{8}$
	15	4.640	4.625	$\bar{2}10$		20b	2.091	2.096	$\bar{4}33$
	40	4.489	4.487	$\bar{2}11$				2.096	314
			4.476	$\bar{2}1\bar{1}$				2.086	$\bar{4}01$
*	80	4.205	4.204	$01\bar{4}$		15	2.052	2.055	$23\bar{3}$
	10	4.147	4.129	$\bar{2}12$				2.053	$\bar{4}02$
	3	3.934	3.936	104	*	15	2.038	2.035	$\bar{2}5\bar{3}$
*	5	3.883	3.879	113	*	10	2.001	2.002	$\bar{4}1\bar{4}$
			3.561	120		5	1.972	numerous	numerous
	10b	3.524	3.534	$\bar{1}30$	*	5	1.946	1.947	045
			3.494	$\bar{2}03$		10	1.929	1.930	$\bar{3}07$
*	30	3.348	3.349	210				1.928	$\bar{1}53$
	50	3.296	3.299	$\bar{1}15$	*	20	1.898	1.898	051
			3.292	105		15	1.849	1.852	$\bar{1}29$
	90	3.132	3.151	$12\bar{3}$				1.851	119
			3.127	$\bar{1}3\bar{3}$	*	20	1.831	1.830	412
*	30	3.030	3.029	$\bar{2}24$	*	20	1.816	1.816	$\bar{4}52$
*	20	2.957	2.961	032				1.773	$\bar{3}3\bar{8}$
	100	2.894	2.904	$\bar{3}12$		15	1.764	1.764	$21\bar{9}$
			2.886	$\bar{3}2\bar{2}$				1.762	241
	3	2.769	2.766	$\bar{3}30$	*	20	1.735	1.735	$\bar{5}42$
*	80	2.722	2.723	124	*	25	1.689	1.689	1110
*	5	2.675	2.675	$\bar{1}2\bar{6}$	*	10	1.661	1.660	039
	50	2.629	2.638	130				1.624	$\bar{4}28$
			2.623	$\bar{1}40$		10	1.611	1.615	$\bar{4}2\bar{8}$
*	10	2.590	2.592	007				1.614	334
			2.542	034	*	30	1.572	1.571	$06\bar{3}$
	5	2.537	2.538	$\bar{2}16$		5b	1.525	1.528	$34\bar{1}$
			2.530	$\bar{3}24$				1.522	$\bar{2}211$
	30	2.495	2.503	222	*	10	1.513	1.513	$37\bar{1}$
			2.485	$\bar{2}42$		3	1.497	1.499	$\bar{2}6\bar{7}$
	3	2.404	2.405	215				1.498	$\bar{6}42$
*	40	2.375	2.379	$11\bar{7}$	*	15	1.481	1.481	064

114.6 mm Debye-Scherrer powder camera employing Ni-filtered Cu radiation (λ Cu$K\alpha$ = 1.54178 Å). Intensities estimated visually. Not corrected for shrinkage and no internal standard used; * = lines used for unit-cell refinement. Indexed on a 9.250(5), b 10.629(4), c 18.182(6) Å, α 93.06(4), β 90.35(5), γ 115.16(4)°.

wt.%. The ideal end-member composition involving complete anion order is $(Hg_2)^{2+}{}_{10}O_6I_3Br_2Cl(CO_3)$ and requires Hg_2O 87.94, I 8.03, Br 3.37, Cl 0.75, CO_2 0.93, sum 101.01, less O = I + Br + Cl 1.01, total 100.00 wt.%. Clearly, the crystal-chemical formula of vasilyevite would not have been successfully unraveled on the basis of chemical composition alone; we would never have expected to find carbonate in a mercury oxy-halide that megascopically resembles a metallic phase. Thus vasilyevite is yet another example of the use of crystal-structure analysis to determine the exact chemical formula of a complex mineral species (*cf.* Hawthorne & Grice 1990).

TABLE 2. REFLECTANCE DATA FOR VASILYEVITE

λnm	R_1	R_2	$^{im}R_1$	$^{im}R_2$
400	30.2	30.8	14.6	15.3
420	29.9	30.6	14.0	14.8
440	29.5	30.4	13.5	14.4
460	28.9	29.8	13.2	14.1
470	28.6	29.5	13.0	13.9
480	28.2	29.2	12.7	13.6
500	27.6	28.7	12.3	13.1
520	27.0	27.8	11.8	12.5
540	26.4	27.3	11.3	12.1
546	26.2	27.1	11.1	11.8
560	25.7	26.6	10.7	11.5
580	24.9	25.9	10.4	11.1
589	24.6	25.7	10.2	10.9
600	24.3	25.3	10.0	10.7
620	23.6	24.8	9.7	10.4
640	23.0	24.1	9.2	10.0
650	22.8	24.0	9.1	9.8
660	22.6	23.6	9.0	9.6
680	22.2	23.1	8.7	9.3
700	21.9	22.9	8.6	9.2

Optical Properties

In plane-polarized reflected light (~3200 K), vasilyevite is grey with low reflectance, white against quartz (in which it is embedded), weakly bireflectant, but not pleochroic. It is distinctly anisotropic, with green, blue and grey tints. These optical properties are generally masked by orange-red to blood-red internal reflections, which are common in air and in oil. Reflectance measurements were made in the visible region

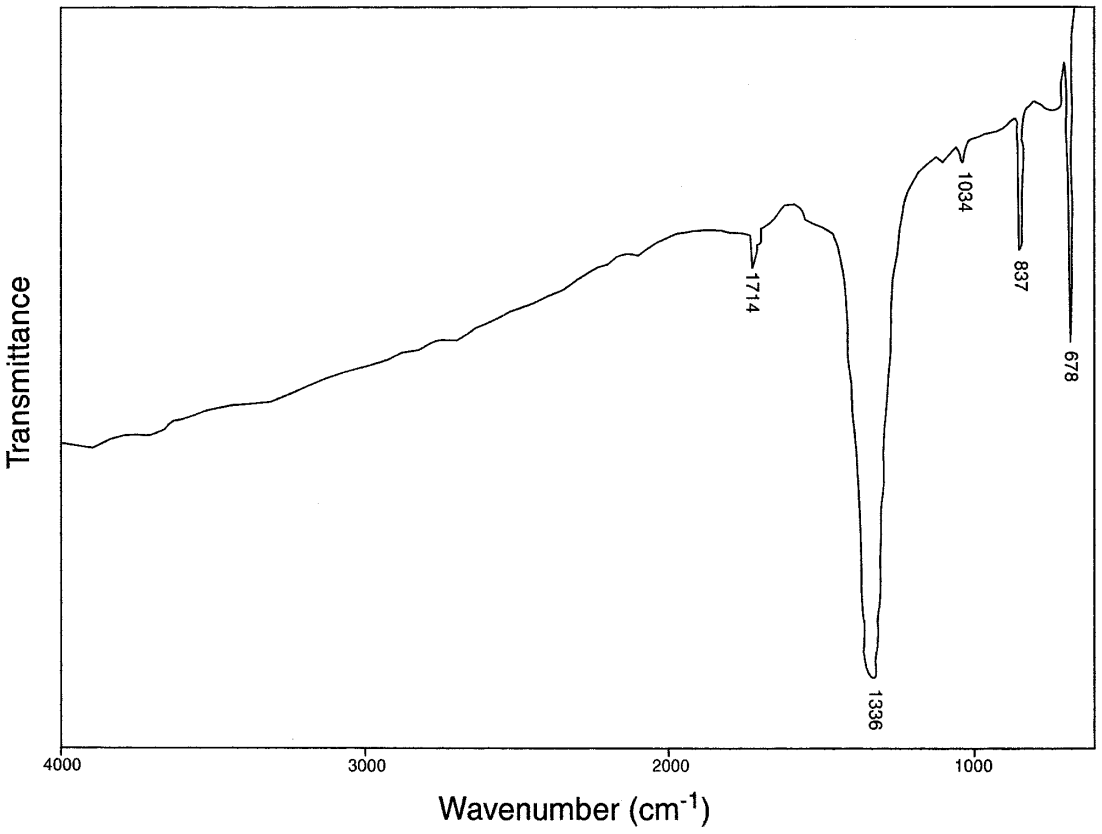

FIG. 1. Infrared-absorption spectrum for vasilyevite.

(400–700 nm) at intervals of 20 μm using a Leitz MPV–SP microscope-spectrophotometer. A WTiC reflectance standard (Zeiss 314) was used as a reference for air and oil (Leica N_D = 1.518) measurements. These were done with 20× objectives, the numerical apertures of which were confined to 0.40, and the diameter of the measured discs was 9 μm. The reflectance curves (R_1, R_2) are more or less parallel and descend continuously from short to long wavelength. In Table 2, we summarize the data collected from the only grain available in polished section. The absolute bireflectance in air ranges between 0.6 and 1.2%, and in oil, it ranges between 0.6 and 0.9%.

Infrared Spectroscopy

The procedures for acquiring the infrared-absorption spectrum of vasilyevite are identical to those reported by Roberts *et al.* (1994). The sample was analyzed using a Bomen Michelson MB–100 FTIR spectrometer equipped with a wide-band mercury–cadmium telluride detector. The transmittance spectrum (Fig. 1) clearly shows diagnostic bands for the carbonate group: ν_3 at 1336 cm^{-1}, very strong, ν_1 at 1034 cm^{-1}, very weak, ν_2 at 837 cm^{-1}, medium, and ν_4 at 678 cm^{-1}, medium strong.

Acknowledgements

The authors thank George Zagler (Institut für Mineralogie, Universität Salzburg) for the reflectance measurements, E. Moffatt (Canadian Conservation Institute) for the infrared spectrum of vasilyevite, M. Clarke (GSC) for redrafting Figure 1, K. Mooney (GSC) for typing the manuscript, and Drs. N.V. Pervukhina, J.K. McCormack, J.A. Mandarino and R.F. Martin for comments and corrections, which greatly improved the final version of this manuscript. FCH was supported by a Canada Research Chair in Crystallography and Mineralogy, and by Discovery, Major Equipment and Major Facilities Access grants from the Natural Sciences and Engineering Research Council of Canada.

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Received July 18, 2003, revised manuscript accepted September 22, 2003.