Canadian Mineralogist
Vol. 28, pp. 351-356 (1990)

REDEFINITION OF VOLKOVSKITE AND ITS DESCRIPTION FROM SUSSEX, NEW BRUNSWICK

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ABSTRACT

Volkovskite has been identified from the potash deposits in New Brunswick. A chemical analysis gave: K_2O 3.8, CaO 17.5, SrO 0.4, B_2O_3 60.5, Cl 2.8, H_2O 13.5, Sum 98.5, less O ≡ Cl 0.6, Total 97.9 wt.%. The empirical formula (based on 47 O + Cl) is: $K_{1.01}(Ca_{3.91}Sr_{0.05})_{\Sigma 3.96}B_{21.77}O_{46.01}H_{18.77}Cl_{0.99}$. These data and the results of a complete crystal-structure determination ($R = 1.9\%$) gave the following structural formula: $KCa_4[B_5O_8(OH)]_4[B(OH)_3]_2Cl \cdot 4H_2O$. The structural study also showed that volkovskite is triclinic, *P*1 with *a* 6.575(2), *b* 23.921(8), *c* 6.522(2) Å, α 90.58(3)°, β 119.10(2)°, γ 95.56(3)°, *V* 890.15 Å³, *Z* = 1. The cell parameters, refined from the X-ray powder-diffraction data, are: *a* 6.580(4), *b* 23.937(8), *c* 6.521(3) Å, α 90.40(6)°, β 119.09(5)°, γ 95.67(5)°, *V* 891.3(5) Å³, *Z* = 1. The strongest six lines in the X-ray powder-diffraction pattern [*d* in Å (*I*) (*hkl*)] are: 11.89(7b)(020), 7.91(9)(030), 5.40(7)(110), 3.39(6)(070), 3.26(10) ($\bar{1}02$) and 2.641(6)(090). It is optically biaxial (+), α 1.523, β 1.530, γ 1.596, 2*V*(calc.) 37°. The measured and calculated densities are, respectively, 2.27(3) and 2.28 g/cm³. The Gladstone–Dale compatibility is –0.034, *i.e.*, excellent. A redefinition of volkovskite, using data obtained from the New Brunswick material, has been approved by the Commission on New Minerals and Mineral Names, I. M. A. A specimen of this material is designated as the neotype.

Keywords: volkovskite, redefinition, neotype, borate, New Brunswick, U.S.S.R., potash.

SOMMAIRE

Nous avons identifié la volkovskite dans les dépôts de potasse du Nouveau Brunswick. Une analyse chimique a donné: K_2O 3.8, CaO 17.5, SrO 0.4, B_2O_3 60.5, Cl 2.8, H_2O 13.5, somme 98.5, moins O ≡ Cl 0.6, total 97.9% en poids. La formule empirique, fondée sur 47 (O + Cl), est $K_{1.01}(Ca_{3.91}Sr_{0.05})_{\Sigma 3.96}B_{21.77}O_{46.01}H_{18.77}Cl_{0.99}$. Ces données et les résultats d'une détermination complète de la structure cristalline, jusqu'à un résidu *R* de 1.9%, ont mené à la formule structurale $KCa_4[B_5O_8(OH)]_4[B(OH)_3]_2Cl \cdot 4H_2O$. L'étude structurale a aussi montré que la volkovskite est triclinique, *P*1, avec *a* 6.575(2), *b* 23.921(8), *c* 6.522(2) Å, α 90.58(3)°, β 119.10(2)°, γ 95.56(3)°, *V* 890.15 Å³, *Z* = 1. Les paramètres réticulaires suivants ont été affinés à partir du cliché de poudre: *a* 6.580(4), *b* 23.937(8), *c* 6.521(3) Å, α 90.40(6)°, β 119.09(5)°, γ 95.67(5)°, *V* 891.3(5) Å³, *Z* = 1. Les six raies les plus intenses du cliché de poudre [*d* in Å(*I*) (*hkl*)] sont: 11.89(7b)(020), 7.91(9)(030), 5.40(7)(110), 3.39(6)(070), 3.26(10) ($\bar{1}02$) et 2.641(6)(090). C'est un minéral biaxe positif, α 1.523, β 1.530, γ 1.596, 2*V* calculé 37°. Les densités mesurée et calculée sont 2.27(3) et 2.28, respectivement. La compatibilité en termes de l'indice de Gladstone–Dale est de –0.034, et donc excellente. Une redéfinition de la volkovskite à la lumière de ces données nouvelles a été approuvée par la Commission des nouveaux minéraux et des noms de minéraux de l'IMA. Nous avons désigné un échantillon de ce matériau comme néotype.

(Traduit par la Rédaction)

Mots-clés: volkovskite, redéfinition, néotype, borate, Nouveau Brunswick, URSS, dépôts de potasse.

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INTRODUCTION

A mineral recovered from water-insoluble residues from salt drill cores from an unspecified locality in the U.S.S.R. was described as a new mineral by Kondrat'eva *et al.* (1966). It was named *volkovskite* in honor of A. I. Volkovskaya, the petrographer who found the mineral. Although a proposal for this new mineral was not submitted to the Commission on New Minerals and Mineral Names, I. M. A. for approval, a fairly complete description was published and included physical, chemical, optical, crystallographic and X-ray powder-diffraction data.

Sutherland (1976) carried out mineralogical examinations of drill cores recovered in the early stages of the exploration and development of some New Brunswick potash deposits. Among the minerals he studied was a hydrated potassium calcium borate. Although an X-ray powder-diffraction study and a chemical analysis were carried out, it could not be identified.

Simmons & Berger (1980) reported the presence of volkovskite in a borate mineral assemblage from the upper Louann Salt in Clarke County, Alabama. The volkovskite was recovered in the water-insoluble residue from the salt as transparent plates and rare elongate prisms. Further details on volkovskite from this occurrence are given by Simmons & Webber (1989).

In 1981, a further study of the borate assemblage from the New Brunswick potash deposits was undertaken. We found an unknown mineral apparently related to volkovskite, because the X-ray powder-diffraction data are practically identical to those of the type material from the U.S.S.R. However, we were unable to obtain samples of the Soviet material for direct comparison from any North American museum. Eventually, through the generosity of the staff of the Fersman Mineralogical Museum of the Academy of Sciences of the U.S.S.R., we were able to obtain a small amount of the type material and to confirm the identity of the New Brunswick mineral. Our identification of volkovskite was reported by Roulston & Waugh (1981); we began a detailed study to describe this rare mineral completely. The data from this study, when compared with the original data for the Soviet material, made it apparent that a redefinition of the mineral was necessary.

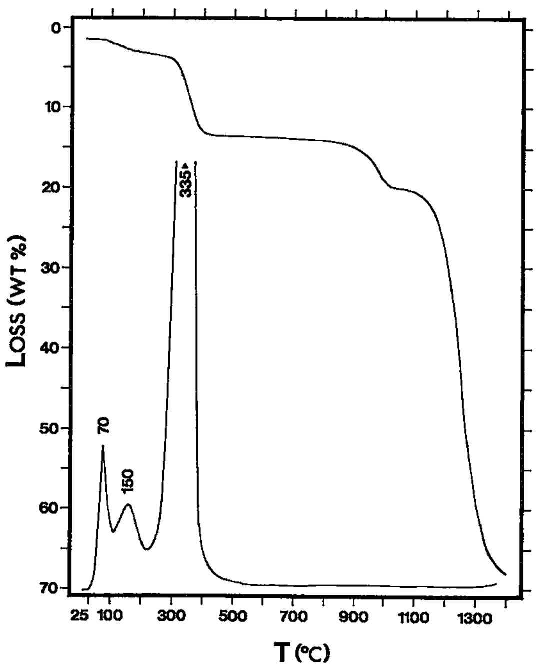

FIG. 1. Thermogravimetric curve (upper) and evolved water curve (lower) for volkovskite from New Brunswick. The latter curve represents ion peak height plotted on a linear scale.

OCCURRENCE OF THE NEW BRUNSWICK MINERAL

The general geology of the New Brunswick potash and salt deposits was described by Roulston & Waugh (1981), who also listed the minerals found in each of them. The following description is a brief summary of the information given by Roulston & Waugh (1981). The southern New Brunswick potash and salt deposits are part of a thick sequence of Mississippian evaporites known as the Windsor Group, which occurs in the Moncton sub-basin in the southwestern part of the northeast-trending Fundy Basin. The Penobsquis and the Salt Springs evaporite deposits consist of a basal anhydrite, a lower halite member, a "sylvinite" ore zone, a middle halite member, an upper anhydrite unit and an upper halite member. Most of the borate minerals are found in the middle halite member and are readily separated from the halite matrix by solution in water. The borate assemblage includes: colemanite, priceite, veatchite, boracite, hilgardite-4M, hydroboracite, szaibelyite and volkovskite. A detailed study of the hilgardite-4M was published by Rachlin *et al.* (1986). Non-borate minerals present in the deposits are: gypsum, anhydrite, sylvite, a chlorite-group mineral, "illite", halite, danburite and howlite.

The volkovskite described in this paper is from the Denison-Potacan Potash Company's mine at Sussex, New Brunswick.

DESCRIPTION OF VOLKOVSKITE

We had hoped that a complete comparative study of type volkovskite from the U.S.S.R. and the New Brunswick material could be made. However, the type material is very rare, and only enough could be spared for a Gandolfi X-ray powder-diffraction pattern.

General appearance and physical properties

The New Brunswick volkovskite occurs as thin, almost micaceous plates up to one centimeter in diameter. The mineral is transparent, colorless to pink, and has a white streak and a vitreous luster. It is nonfluorescent in short-wave and long-wave ultraviolet radiation. The mineral is brittle and has a Mohs hardness of about 2 ½. The cleavage is perfect on {010}. The density measured by Berman balance in toluene is 2.27(3) g/cm^3. The density calculated from the unit-cell parameters and the formula derived from the chemical analytical data is 2.28 g/cm^3; this value also was calculated from the cell parameters and the theoretical composition.

Optical properties

An optical study, using the method described by Hurlbut (1984), gave the following results: biaxial positive, α 1.523, β 1.530, γ 1.596, $2V$ (calc.) 37°. Measurements were made using a filter that transmits light with a wavelength of approximately 589 nm.

Chemical Composition and Thermal Data

A chemical analysis of the New Brunswick mineral was carried out with an ARL–SEMQ electron microprobe using an operating voltage of 15 kV and a sample current of 0.025 μA, measured on brass. Homogeneity was confirmed with a small beam-spot, and the analyses were done with a 60-μm beam-spot. The analytical data were obtained using the following standards: celestine (Sr), microcline (K), wollastonite (Ca) and NaCl (Cl). Wavelength-dispersion scans showed no other elements with atomic numbers greater than 11. The data were corrected using a modified version of the MAGIC–4 program. The B_2O_3 content was determined by wet chemical analysis, and H_2O was determined by thermogravimetric analysis and evolved gas analysis (TGA–EGA).

Thermogravimetric and evolved gas data were obtained using a Mettler TA–1 Thermoanalyzer coupled to an Inficon IQ200 quadrupole mass spectrometer. The 6.5 mg hand-picked sample contained very minor unidentified visible inclusions. It was weighed at 22°C at a relative humidity of approximately 40%. The sample was then subjected to a vacuum for 22 hours at room temperature, during which it lost 1.5 ± 1 wt.%. Upon heating at 10°C/minute to nearly 1400°C, it lost a further 68 wt.% in four distinct steps. The first two losses were primarily of water: 2.0 wt.% between 40 and 215°C (with peaks at roughly 70° and 150°C) and 10.0 wt.% between 215 and 470°C (with a peak at 355°C). Minor peaks of O_2, CO_2 and mass 27 (presumably BO^+) were noted during the second loss. The last two losses were primarily of unidentified volatiles that condensed before reaching the spectrometer: 6.4 wt.% between 470 and 1045°C, and 49 wt.% between 1045 and 1397°C. However, a minor broad peak of HCl was noted at 1300°C. The thermogravimetric curve and evolved water curve are given in Figure 1.

Table 1 shows the chemical analytical data reported by Kondrat'eva *et al.* (1966), Sutherland (1976), Simmons & Webber (1989) and in the present study. Kondrat'eva *et al.* (1966) attributed the presence of K and Cl to physical impurities in their mineral and did not consider them in the calculation of formulae. Their empirical formula is

TABLE 1. CHEMICAL ANALYTICAL DATA FOR VOLKOVSKITE

	1	2	3	4	5
K_2O	2.42	3.76	2.55	3.8	3.84
Na_2O	0.14				
CaO	14.12	18.75	19.76	17.5	18.28
SrO	4.06	minor		0.4	
B_2O_3	59.80	65.36	61.00	60.5	62.42
Cl	1.98		2.83	2.8	2.89
H_2O	16.30	12.42	(14.50)	13.5	13.22
Sum	98.82		(100.64)	98.5	100.65
less O=Cl	-0.45		-0.64	-0.6	-0.65
Total	98.37	100.29	(100.00)	97.9	100.00

Notes: 1. Kondrat'eva *et al.* (1966). 2. Sutherland (1976). 3. Simmons & Webber (1989). H_2O (by difference) originally given as 13.86 wt.%, is corrected here to reflect the O=Cl of -0.64 required by the Cl-content. 4. This study. 5. Ideal composition for $KCa_4[B_5O_8(OH)]_4[B(OH)_3]_2Cl{\cdot}4H_2O$.

TABLE 2. UNIT-CELL DATA FOR VOLKOVSKITE

Crystal system	monoclinic		triclinic		
Space group	$P2_1$		P1		
	1	2	3	4	5
Parameters					
a	6.57(1)Å	6.571(3)Å	6.51(4)Å	6.580(4)Å	6.575(2)Å
b	48.30(8)	48.30(2)	23.80(6)	23.937(8)	23.921(8)
c	6.51(2)	6.505(3)	6.52(2)	6.521(3)	6.522(2)
α	-----	-----	91.5(4)°	90.40(6)°	90.58(3)°
β	119° 05(6)'	119.05(4)°	118.4(4)°	119.09(5)°	119.10(2)°
γ	-----	-----	94.8(3)°	95.67(5)°	95.56(3)°
V	1805.4Å^3	1805(1)Å^3	884(4)Å^3	891.3(5)Å^3	890.15Å^3
Z	8	8	1	1	1

Notes: 1. Kondrat'eva *et al.* (1966). Original data for their monoclinic cell. Their erroneous unit cell volume (1805.27Å^3) has been corrected here.
2. This study. The monoclinic cell parameters refined from the X-ray powder diffraction data given by Kondrat'eva *et al.* (1966) using their cell parameters.
3. This study. The triclinic cell refined from the X-ray powder diffraction data given by Kondrat'eva *et al.* (1966) using the cell parameters derived from the crystal structure determination by Le Page & Lee (1985).
4. This study. Cell parameters refined from the X-ray powder diffraction data using the cell parameters derived from the crystal structure determination by Le Page & Lee (1985).
5. Cell parameters derived from the crystal structure determination by Le Page & Lee (1985).

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

(1)				(2)			
I	$d_{meas.}$	$d_{calc.}$	hkl	I	$d_{meas.}$	$d_{calc.}$	hkl
7b	11.89	11.886	020	30	12.2	12.075	040
9	7.91	7.924	030	100	8.1	8.050	060
5	5.93	5.943	040	60	6.03	6.038	080
1	5.69	5.706	1$\bar{1}$0				
7	5.40	5.410	110	60	5.42	5.411	130
				10b	4.94	4.938	150
2	4.40	4.395	130	20b	4.41	4.415	$\bar{1}$70
1	3.98	3.985	041				
1	3.87	3.884	1$\bar{5}$0	20b	3.87	3.886	$\bar{1}$91
				40	3.65	3.667	$\bar{1}$.10.1
				40	3.47	3.476	0.11.1
6	3.39	3.396	070	50	3.33	3.316	101
10	3.26	3.260	$\bar{1}$02	90	3.28	3.277	$\bar{2}$11
3	3.15	3.149	$\bar{1}$22	10	3.15	3.141	$\bar{1}$42
3b	3.006	3.009	$\bar{1}$32	10	2.98	2.989	171
2	2.928	2.926	23$\bar{1}$				
4b	2.875	2.875	2$\bar{1}$0	60	2.86	2.863	$\bar{1}$82
5b	2.794	2.796	210	70	2.81	2.809	1.15.1
3	2.705	2.705	220	10	2.69	2.683	0.18.0
6	2.641	2.641	090	60	2.63	2.630	$\bar{2}$.11.2
3	2.590	2.591	230				
2	2.512	2.510	$\bar{1}$62	20	2.49	2.494	$\bar{2}$92
1	2.459	2.457	$\bar{2}$60				
1	2.416	2.416	161				
1	2.378	2.377	0.10.0	30	2.36	2.367	$\bar{1}$.14.2
1	2.325	2.333	1$\bar{8}$1	30	2.31	2.310	1.15.1
2b	2.255	2.253	171	10	2.25	2.245	$\bar{2}$.13.2
1	2.198	2.198	260	20	2.19	2.194	0.14.2
1	2.172	2.171	$\bar{2}$2$\bar{1}$				
4	2.148	2.148	$\bar{2}$3$\bar{1}$	70	2.15	2.151	$\bar{3}$21
3b	2.112	2.113	$\bar{3}$12	60	2.10	2.099	261
3	2.028	2.029	231	20	2.04	2.040	$\bar{1}$73
3b	1.992	1.992	082				
3b	1.982	1.981	$\bar{3}$61	70	1.98	1.983	2.10.1
1b	1.905	1.903	300				
2b	1.809	1.809	$\bar{3}$61	50	1.821	1.820	$\bar{1}$.22.2
				50	1.775	1.774	2.15.3
1	1.700	1.700	350	10	1.698	1.695	$\bar{2}$.23.0
1	1.675	1.675	182				
1	1.656	1.656	0.$\bar{1}\bar{4}$.1	20	1.661	1.661	$\bar{1}$.25.2
1	1.630	1.629	$\bar{2}$.13.0	30	1.639	1.639	$\bar{4}$22
				20	1.572	1.572	143
				10	1.557	1.556	$\bar{1}$44
				20	1.490	1.490	$\bar{2}$.13.4
				20	1.463	1.463	0.21.3
				30	1.428	1.428	4.14.3
				20	1.377	1.378	3.27.1
				10	1.354	1.354	4.18.3
				10	1.341	1.341	0.35.1

Notes:
1. This study. Indexed on the refined triclinic cell: a 6.580, b 23.937, c 6.521Å, α 90.40°, β 119.09°, γ 95.67°.
2. Data of Kondrat'eva *et al.* (1966). Indexed on the refined monoclinic cell: a 6.571, b 48.30, c 6.505Å, β 119.05°.

$(Ca_{0.87}Sr_{0.13})_{\Sigma 1.00}B_{5.92}O_{13}$ (based on O = 13). They presented the simplified formula as: $(Ca,Sr)O{\cdot}3B_2O_3{\cdot}3H_2O$. If K and Cl are included in the calculation of the empirical formula (based on O + Cl = 47), it becomes $(K_{0.63}Na_{0.06})_{\Sigma 0.69}(Ca_{3.09}Sr_{0.48})_{\Sigma 3.57}B_{21.09}O_{46.31}H_{22.21}Cl_{0.69}$, and the simplified formula is $KCa_{3.5}B_{21}O_{46}H_{22}Cl$.

Sutherland (1976) reported no information on Cl content. He presented the simplified formula as $KCa_4B_{23}O_{40}{\cdot}8H_2O$. The formula has a net charge of −2. If Sutherland's data are recalculated to give 46 oxygen ions and sufficient chlorine is added for one Cl ion, the empirical formula becomes: $K_{0.96}Ca_{4.01}B_{22.50}O_{46.00}H_{16.52}Cl_{1.00}$, and the simplified formula is $KCa_4B_{22.5}O_{46}H_{16.5}Cl$. The chemical data given by Simmons & Webber (1989) yield the empirical formula $K_{0.66}Ca_{4.29}B_{21.39}O_{46.03}H_{19.64}Cl_{0.97}$ (based on O + Cl = 47) and $KCa_4B_{21.5}O_{46}H_{19.5}Cl$ in a simplified form.

The data from the present study give the empirical formula $K_{1.01}(Ca_{3.91}Sr_{0.05})_{\Sigma 3.96}B_{21.77}O_{46.01}H_{18.77}Cl_{0.99}$ (based on O + Cl = 47), and the simplified formula, $KCa_4B_{22}O_{46}H_{18}Cl$. The structural formula is $KCa_4[B_5O_8(OH)]_4[B(OH)_3]_2Cl{\cdot}4H_2O$. Although we had insufficient material from the type specimen to do a complete analysis, subsequent electron-microprobe scans of a small flake of type volkovskite from the U.S.S.R. and a crystal from New Brunswick gave the same relative peak heights for Ca, K and Cl.

Crystallography

The original data given by Kondrat'eva *et al.* (1966) are for a monoclinic cell with space group $P2_1$, *a* 6.57(1), *b* 48.30(8), *c* 6.51(2) Å, β 119°05(6)′, *V* 1805.4 Å³, *Z* = 8 (the unit-cell volume is given as 1805.27 Å³ in the original description.). Note that this is a markedly pseudo-hexagonal cell. No single-crystal data were reported by Sutherland (1976), Simmons & Berger (1980) or Simmons & Webber (1989), although Sutherland (1976) presented sixteen lines from a diffractometer trace, and Simmons & Webber (1989) noted that their X-ray powder-diffraction data correspond closely with those given by Kondrat'eva *et al.* (1966).

Using precession methods, we were not able to

TABLE 4. COMPARISON OF DATA FOR VOLKOVSKITE FROM THE U.S.S.R. AND NEW BRUNSWICK

	U.S.S.R. Kondrat'eva *et al.* (1966)	NEW BRUNSWICK This study
Formula	$(Ca, Sr)B_6O_{10}{\cdot}3H_2O$	$KCa_4[B_5O_8(OH)]_4[B(OH)_3]_2Cl{\cdot}4H_2O$
Crystallography	Monoclinic	Triclinic
Space group	$P2_1$	P1
Parameters		
a	6.57Å	6.580(4)Å
b	48.30	23.937(8)
c	6.51	6.521(3)
α	——	90.40(6)°
β	119°05′	119.09(5)°
γ	——	95.67(5)°
V	1805.4Å³	891.3(5)Å³
Z	8	1
Habit	platy on {010}	platy on {010}
Phys. Properties		
Colour	colourless	colourless to pink
Lustre	vitreous	vitreous
Cleavage	perfect on {010}	perfect on {010}
$D_{meas.}$	2.29-2.34 g/cm³	2.27(3) g/cm³
$D_{calc.}$	2.39 g/cm³	2.29 g/cm³
Optical data		
Biaxial	+	+
α	1.536	1.523
β	1.539	1.530
γ	1.603	1.596
$2V_{calc.}$	25°	37°

obtain usable photographs because of the small repeat along b^*. To solve this problem, a crystal-structure determination was performed (Le Page & Lee 1985). The results of this 4-circle study showed that the mineral is triclinic, $P1$, a 6.575(2), b 23.921(8), c 6.522(2) Å, α 90.58(3)°, β 119.10(2)°, γ 95.56(3)°, V 890.15 Å^3. Details of this structural study are summarized here. The structure was refined to R_F = 1.9% (R_W = 2.0%) on 9915 unique reflections observed up to 60° 2θ with Mo$K\alpha$ radiation. All the atomic positions were refined, including those for the hydrogen atoms. The structure is made up of four identical (010) layers of $[B_5O_8(OH)]_8$, only slightly different from those in the nasinite structure, described by Corazza *et al.* (1975), and two isolated $B(OH)_3$ groups. The Ca ions bond pairs of layers, and one of those two pairs of layers also contains K and Cl. The paired layers are joined together by H-bonding. The layers are pseudohexagonal in projection, but not in three dimensions, which explains the near identity of a and c and the interaxial angle of about 120°. The structure determination confirmed the empirical formula derived from the chemical analytical data.

The unit-cell parameters obtained by refining the X-ray powder-diffraction data are compared in Table 2 with those derived from the 4-circle data and with the data given by Kondrat'eva *et al.* (1966). The X-ray powder-diffraction data given by Kondrat'eva *et al.* (1966) were used to obtain refined unit-cell parameters for the monoclinic and triclinic unit cells; the results also are given in Table 2. Table 3 gives complete X-ray powder-diffraction data for volkovskite from New Brunswick and the U.S.S.R.

Volkovskite from New Brunswick occurs as pseudohexagonal, platy crystals tabular on {010}. The following forms were identified: {010}, $\{0\bar{1}0\}$, {001}, $\{00\bar{1}\}$, $\{2\bar{8}1\}$, $\{2\bar{8}\bar{1}\}$ and $\{1\bar{8}\bar{1}\}$.

Compatibility

The Gladstone–Dale compatibility, as defined by Mandarino (1979, 1981a), is -0.034 (excellent) for the New Brunswick material and -0.051 (good) for the Soviet material. In each case, the density was calculated as shown by Mandarino (1981b) from the chemical analytical data and the parameters of the triclinic unit cell.

Conclusions

All the data for the mineral described as volkovskite by Kondrat'eva *et al.* (1966) from the U.S.S.R. and the mineral from New Brunswick studied here are compared in Table 4. From this comparison, it is clear that the two minerals are the same. Because the new data from the New Brunswick mineral necessitate changes in the chemical formula and unit cell given by Kondrat'eva *et al.* (1966), a proposal was submitted to the Commission on New Minerals and Mineral Names, I. M. A., to redefine volkovskite. This follows the procedures given by Nickel & Mandarino (1987). The proposal was approved.

The data given in the preceding pages for New Brunswick material are considered the definitive data for volkovskite. A sample of volkovskite from New Brunswick has been designated as the neotype of this species according to the procedures outlined by Dunn & Mandarino (1987) and is deposited in the Mineral Collection of the Royal Ontario Museum under the registration number M44196.

Acknowledgements

The authors express their appreciation to the late Mr. R. C. Staveley for generating the initial interest in this study. They also thank the scientific staff of the Fersman Mineralogical Museum, Academy of Sciences, U. S. S. R., particularly Dr. L. V. Bulgak, for generously providing some of the type volkovskite. The referees, Drs. G. Y. Chao and L. A. Groat, are thanked for their careful review of the manuscript.

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Received November 16, 1989, revised manuscript accepted February 28, 1990.