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I. Jahnsite, Segelerite, and Robertsite, Three New Transition Metal Phosphate Species

II. Redefinition of Overite, an Isotype of Segelerite

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III. Isotypy of Robertsite, Mitridatite, and Arseniosiderite

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With Two Chemical Analyses by

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Abstract

Three new species,—jahnsite, segelerite, and robertsite,—occur in moderate abundance as late stage products in corroded triphylite-heterosite-ferrisicklerite-rockbridgeite masses, associated with leucophosphite, hureaulite, collinsite, laueite, *etc*. Type specimens are from the Tip Top pegmatite, near Custer, South Dakota.

Jahnsite, $CaMn^{2+}Mg_2(H_2O)_8Fe^{3+}{}_2(OH)_2[PO_4]_4$, *a* 14.94(2), *b* 7.14(1), *c* 9.93(1)Å, β 110.16(8)°, *P*2/*a*, *Z* = 2, specific gravity 2.71, biaxial (−), 2*V* large, α 1.640, β 1.658, γ 1.670, occurs abundantly as striated short to long prismatic crystals, nut brown, yellow, yellow-orange to greenish-yellow in color. Forms are *c*{001}, *a*{100}, *i*{201}, *j*{$\bar{2}01$}, *k*{$\bar{1}01$}, *l*{011}, *m*{110}, and *n*{$\bar{1}11$}.

Segelerite, $CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$, *a* 14.826(5), *b* 18.751(4), *c* 7.307(1)Å, *Pcca*, *Z* = 8, specific gravity 2.67, biaxial (−), 2*V* large, α 1.618, β 1.635, γ 1.650, occurs sparingly as striated yellow-green prismatic crystals, with *a*{100}, *b*{010}, *m*{110} and *q*{121} with perfect {010} cleavage. It is the Fe^{3+}-analogue of overite; a restudy on type overite reveals the space group *Pcca* and the ideal formula $CaMg(H_2O)_4Al(OH)[PO_4]_2$.

Robertsite, $Ca_3Mn^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$, *a* 17.36, *b* 19.53, *c* 11.30Å, β 96.0°, *A*2/*a*, *Z* = 8, specific gravity 3.17, cleavage {100} good, biaxial (−) α 1.775, $\beta \approx \gamma = 1.82$, $2V \sim 8°$, pleochroism extreme (*Y*, *Z* = deep reddish brown; *X* = pale reddish-pink), occurs as fibrous masses and small wedge-shaped crystals showing *c*{001}, *a*{100}, *q*{031}. It is tabular parallel to {100}. The color is shiny black, blood-red in thin splinters, streak chocolate brown.

Mitridatite, $Ca_3Fe^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$, *a* 17.52(2), *b* 19.35(4), *c* 11.25(2)Å, β 95°55′, *A*2/*a*, *Z* = 8, specific gravity 3.24, α 1.785, $\beta \approx \gamma = 1.85$, 2*V* 5-10°, pleochroism extreme (*Y*, *Z* = deep greenish-brown, *X* = pale greenish yellow), shows *a*{100}, *c*{001} and *v*{$\bar{4}23$}. Arseniosiderite, $Ca_3Fe^{3+}{}_4(OH)_6(H_2O)_3[AsO_4]_4$ has *a* 17.76, *b* 19.53, *c* 11.30Å, β 96.0° based on the powder data. These species are isotypes of the robertsite structure.

Introduction

Rarely does an investigator in descriptive mineralogy have an opportunity to announce three species new to scientific intelligence which not only occur intimately associated but also appear as well-developed crystals. It is appropriate, for this reason, to commence with a detailed description of the paragenesis of these minerals since their compositions leave little doubt that their formation was due to the presence of Ca^{2+}, Fe^{3+}, Mn^{2+}, Mn^{3+}, Mg^{2+}, $[PO_4]^{3-}$, and $(OH)^-$ ions in solution at low temperature.

Following this paragenetic discussion, the crystal chemistry of each species shall be discussed separately.

The type specimens originate from the Tip Top pegmatite, near the village of Custer, in Custer County, South Dakota, although other localities have also come to light and one species (robertsite) has a Fe^{3+}-analogue (mitridatite) so widespread in its occurrence to suggest that the phase should elicit considerable interest in its crystal chemistry. The Tip Top pegmatite (NE ¼, SW ¼, sec. 8, T.4S, R.4E) is one of several in the region of the central Black Hills where a great abundance of triphylite-lithiophilite appeared as an accesory core mineral and was exposed during the course of mining activity. These crude giant crystals of triphylite suffered extensive retrograde oxidative and metasomatic reactions which resulted in the ferrisicklerite-sicklerite series $Li_{1-x}Fe^{3+}{}_x(Fe^{2+},Mn^{2+})_{1-x}[PO_4]$; the heterosite-purpurite series $(Fe^{3+},Mn^{3+})[PO_4]$; and the rockbridgeite-frondelite series $(Fe,Mn)^{2+}Fe^{3+}{}_4(OH)_5[PO_4]_3$. Vuggy cavities in these phases contain a host of late stage hydrated transition metal phosphates. Since casual inspection of such associations can be unreliable in identification of species, great care was taken in this study to examine all distinct phases by X-ray single crystal and powder diffraction techniques and, whenever necessary, to establish identity by comparison with descriptions of type materials. Phases discovered in these cavities include leucophosphite, $KFe^{3+}{}_2(OH)(H_2O)[PO_4]_2 \cdot H_2O$, as lovely greenish-grey, purplish-pink to deep amethystine prismatic crystals; hureaulite, $(Mn,Fe)^{2+}{}_5(H_2O)_4[PO_3(OH)]_2[PO_4]_2$, as pale yellow, to pale pink to red-brown prismatic crystals; collinsite, $Ca_2(Mg,Fe^{2+})(H_2O)_2[PO_4]_2$, as colorless to white radiating sprays, lath-like crystals, and flattened chisel-shaped rosettes; hydroxylapatite, $Ca_5(OH)[PO_4]_3$, as rounded colorless hexagonal prisms; whitlockite, *ca* $Ca_8Mg[PO_4]_6$, as colorless simple rhombohedra; bermanite, $Mn^{2+}Mn^{3+}{}_2(OH)_2(H_2O)_4[PO_4]_2$, as deep reddish-brown druses of thin tabular crystals; laueite, $Mn^{2+}Fe^{3+}{}_2(OH)_2(H_2O)_6[PO_4]_2 \cdot 2H_2O$, as striated orange laths; mitridatite, $Ca_3Fe^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$, as thin brittle greenish-brown plates and dull olive-green stains; and the new species jahnsite, $CaMn^{2+}Mg_2(H_2O)_8Fe^{3+}{}_2(OH)_2[PO_4]_4$; segelerite, $CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$; and robertsite, $Ca_3Mn^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$. For a general review of the myriad pegmatite phosphates, the reader is referred to Moore (1973).

a. The aquated oxidation products of triphylite-lithiophilite

The secondary phosphate paragenesis of the Tip Top pegmatite appears to fall into two closely related but distinct categories: the products of direct solution, oxidation, and recrystallization of the parent triphylite-lithiophilite and the products involving the addition of Ca^{2+} and Mg^{2+} cations. We have examined by electron microprobe analysis remnant primary triphylite occurring in the interior of the zone of oxidation and aquation and have obtained the composition $Li(Fe_{0.78}Mn_{0.22}Mg_{<0.10})[PO_4]$. Since the late stage Ca-Mg rich phosphates occur locally but occasionally in sufficient abundance, it is difficult to imagine a contribution of the Ca^{2+} and Mg^{2+} cations from the parent phosphates; it is proposed that these cations were introduced at a late stage from a separate source and were added to the pool of cations provided by the the parent phosphates.

The oxidation and aquation of triphylite leads directly to the formation of rockbridgeite or ferrisicklerite, where ferrisicklerite may in turn be oxidized further to heterosite or aquated to form rockbridgeite. These phases are observed as among the most persistent products of oxidation-aquation of triphylites in granitic pegmatites throughout the world. Since the isoelectronic point of Fe^{3+} cation occurs at lower pH values and earlier than Mn^{2+} and the Fe^{2+} remaining, leucophosphite crystallizes at low temperatures where water ligands are stable. The solution becomes progressively enriched in Mn^{2+}, which crystallizes later in hureaulite. At the final stages, laueite and mitridatite appear. Here, oxidation may produce Mn^{3+}, so that bermanite forms. With the depletion of $[PO_4]^{3-}$, todorokite, *ca* $Mn_{<8}(O,OH)_{16} \cdot 2H_2O$, crystallizes. Thus, the sequence triphylite-ferrisicklerite - rockbridgeite - leucophosphite, hureaulite + laueite-mitridatite-bermanite-todorokite appears repeatedly at the Tip Top, White Elephant and Linwood pegmatites in the Black Hills; at Hagendorf Süd, Bavaria; at Palermo No. 1, New Hampshire; and at the Sapucaia pegmatite, Brazil.

b. The addition of Ca^{2+} *and* Mg^{2+}

Collinsite, hydroxylapatite, whitlockite, and the three new species are not rare at the Tip Top pegmatite. In addition, they have been observed from the White Elephant pegmatite, having formed under similar conditions. Mitridatite is a particularly persistent phase at all stages of late crystallization and occurs widely as stains coating the silicates surrounding triphylite crystals at practically every pegmatite where triphylite occurs, suggesting that mitridatite is an important low temperature phase. Paragentically, the Ca^{2+}- and Mg^{2+}-bearing minerals occur at the sequence where leucophosphite and hureaulite crystallized. Robertsite appears in several generations: as shiny black botryoidal aggregates coating the cavity walls upon which are implanted jahnsite and collinsite, and as reddish platy rosettes implanted upon these minerals. In several specimens, it occurs as the sole mineral with stubby shiny black wedge-shaped crystals lining the cavities in the same. Collinsite, hydroxylapatite, and whitlockite usually appear as the last minerals to crystallize, encrusting the rather abundant jahnsite. The author suggests that the Fe^{3+} remaining in solution crystallized as jahnsite with the essentially pure Ca–Mg phosphates crystallizing last.

JAHNSITE

$CaMn^{2+}Mg_2(H_2O)_8Fe^{3+}_2(OH)_2[PO_4]_4$

Descriptive mineralogy

The new species jahnsite occurs as tabular short to long prismatic crystals with vitreous to subadamantine luster of a nut-brown, purplish-brown, yellow, yellow-orange, or greenish-yellow color. It can be easily confounded with laueite, pseudolaueite, stewartite, and childrenite. Experience has shown that almost certain distinction from these other phosphates can be achieved by analysis of crystal morphology. Persistent striations parallel to the prism (=[010]) distinguish it from hureaulite and leucophosphite. Laueite and stewartite usually reveal their triclinic character. Metastrengite usually possesses a delicate pink color not observed in jahnsite. Childrenite in well-developed crystals almost invariably shows good orthorhombic development with persistent $p\{111\}$ and $s\{121\}$.

The specific gravity of jahnsite is 2.706 (pycnometer), 2.718 (sink-float in methylene iodide-toluene), computed density 2.715 gm cm^{-3}, hardness 4, cleavage {001} good. The mineral is soluble in dilute HCl solution.

Crystal morphology

Crystals are commonly well-developed and euhedral, monoclinic holosymmetric (2/*m*), striated parallel to [010], short to long prismatic parallel to [010], often tabular parallel to $a\{100\}$. They frequently occur as parallel aggregates, twinned by reflection on $c\{001\}$. Large crystals may be up to 5 mm in length, though 0.1 to 0.5 mm are most typical. The forms include $c\{001\}$, $a\{100\}$, $i\{201\}$, $j\{\bar{2}01\}$, $k\{\bar{1}01\}$, $l\{011\}$, $m\{110\}$ and $n\{\bar{1}11\}$ with striae on $j\{\bar{2}01\}$ and $a\{100\}$. A typical development is presented in Figure 1.

Analysis of the reticular density (based on the X-ray cell) reveals that the form frequencies approximately follow the order of descending interplanar spacings (Table 1a). The form $b\{010\}$ is consistently absent; examination of single crystal data reveals that this plane is of zero intensity. Table 1a suggests that some crystals of jahnsite should reveal $\{\bar{2}11\}$ and $\{111\}$, but none of the crystals submitted to morphological examination presented these facets.

X-ray data

A partly indexed powder pattern is offered in Table 2a. Caution was taken to roll the ground sample into a small sphere with rubber cement. Throughout this study, the powder data were indexed on the basis of the strong single crystal intensities. Computations of the interplanar spacings were made from the single crystal studies.

Table 3a provides the structure cell data. In addition, we have solved its crystal structure and have refined the results to $R(hkl) = 0.09$. This allows

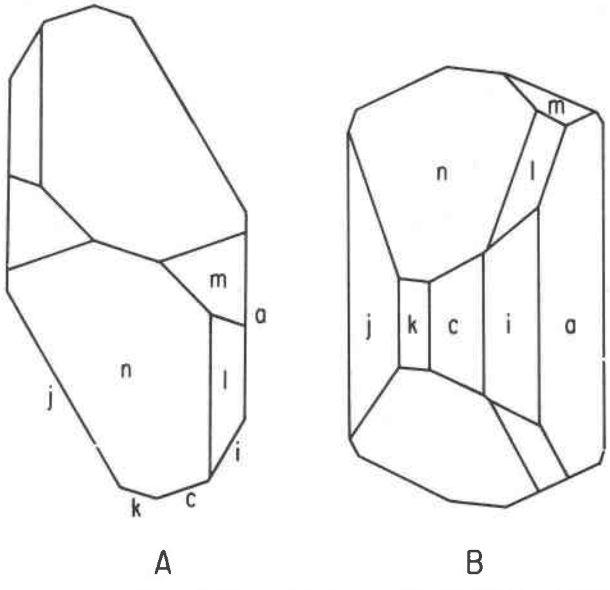

FIG. 1. Crystal of jahnsite showing the forms $c\{001\}$, $a\{100\}$, $i\{201\}$, $j\{\bar{2}01\}$, $k\{\bar{1}01\}$, $l\{011\}$, $m\{110\}$, and $n\{\bar{1}11\}$. Tip Top pegmatite. A. Plan. B. Clinographic projection.

Table 1. Jahnsite and Segelerite. Descending Interplanar Spacings, Intensities, and Appearance of Forms

JAHNSITE				SEGELERITE			
hkl	d(hkl)	I/Io	Appearance*	hkl	d(hkl)	I/Io	Appearance*
001	9.32	10	x	010	18.75	<1	x ?
010	7.14	< 1	a	020	9.38	9	x
200	7.01	1	x	200	7.41	<1	x
$\bar{2}01$	6.85	< 1	x	210	6.89	<1	a
110	6.36	2	x	030	6.25	<1	x ?
$\bar{1}11$	5.68	< 4	x	111	6.19	<1	a
011	5.67	< 4	x	220	5.82	1	x
210	5.01	< 1	a	121	5.37	6	x
$\bar{2}11$	4.94	< 6	a				
111	4.91	< 6	a				
201	4.86	< 1	x				
$\bar{2}02$	4.70	< 1	x				

*(*x* = *present*, *a* = *absent*)

for a precise description of the jahnsite crystal chemistry and the establishment of a correct formula, which otherwise would be a difficult and uncertain problem. Chains of Fe^{3+}–OH–Fe^{3+} corner-sharing octahedra run parallel to the *b*-axis and link to $[PO_4]^{3-}$ tetrahedra; alternating Ca–O and Mn^{2+}–O distorted octahedral edge-sharing chains also run in the *b*-direction, forming dense slabs parallel to {001}. These slabs are bridged by the $[PO_4]^{3-}$ tetrahedra which link by corners to the aquated Mg–O octahedra. The structure reveals a novel ligand stereoisomerism about the octahedral corner-chains which was predicted on combinatorial grounds by Moore (1970). It is allied to the childrenite-eosphorite and laueite-pseudolaueite structures.

Chemical analysis

Several electron probe analyses of jahnsite were made by Mr. T. Solberg utilizing apatite, wollastonite, wyllieite, and MgO standards. Owing to the brittle character of the crystals, the jahnsite samples were not polished and consequently the quantitative results in Table 4a are consistently low. Thus, computations of the cation composition were based on an integral number of phosphorus atoms. Valence states were inferred by the crystal structure analysis and the water content was determined by the Penfield tube technique conducted by Dr. A. L. Lingard of South Dakota School of Mines and Technology. Setting $P = 8.0$, the analysis computes to

$$Ca_{2.0}Mn^{2+}_{2.3}Mg_{3.5}Fe^{3+}_{3.3}Al_{0.8}(OH)_{4.1}[PO_4]_{8.0}\cdot 15.8\ H_2O.$$

Noting that all water molecules are bound to the Mg–O octahedra in the structure, we propose the ideal formula $CaMn^{2+}Mg_2(H_2O)_8Fe^{3+}_2(OH)_2[PO_4]_4$, of which there are two formula units in the cell.

Optical properties

The type sample is biaxial (−), 2*V* large, α 1.640, β 1.658, γ 1.670, all ± 0.003. For the brown crystals, absorption is $Y \gg Z > X$, with *X* pale purple, *Y* deep purplish brown, and *Z* yellow with a tinge of green. *Z* is $\|b$ and $X \wedge c$ *ca* 18°. Based on the Gladstone-Dale relationship, the tables of Larsen and Berman (1934), the ideal formula, and the average observed specific gravity, $\langle n \rangle$ calc = 1.668.

Table 2. Jahnsite and Segelerite. Powder Data

a. JAHNSITE				b. SEGELERITE			
I/I_o	d(obs)	d(calc)	hkl	I/I_o	d(obs)	d(calc)	hkl
10	9.27	9.320	001	9	9.31	9.376	020
1	7.08	7.012	200	1	5.74	5.815	220
2	6.32	6.360	110	6	5.34	5.372	121
4	5.66	5.683	$\bar{1}11$	4	4.97	5.014	211
		5.667	011	4	4.65	4.688	040
6 brd	4.91	4.942	$\bar{2}11$	2	3.936	3.962	240
		4.908	111	5	3.729	3.706	400
4	4.63	4.660	002	5	3.421	3.446	420
3	4.05	4.075	$\bar{1}12$	2	3.253	3.255	411
3	3.90	3.910	310			3.228	212
1	3.723	3.733	$\bar{4}01$	1	3.163	3.188	430
5	3.522	3.540	$\bar{3}12$	3	3.059	3.083	341
		3.506	400	10	2.868	2.907	440
3	3.416	3.426	$\bar{4}02$			2.882	042
3	3.268	3.284	311	2	2.813	2.821	161
2	3.165	3.165	$\bar{2}21$	4	2.583	2.577	412
5	2.950	2.962	401	3 brd	2.368	2.389	460
8 brd	2.825	2.867	$\bar{4}03$			2.375	062
		2.833	022	3	2.307	2.296	213
4	2.575	2.579	$\bar{4}21$	1	2.253	2.275	442
2	2.417	2.428	402	3	2.191		
3	2.341	2.349	$\bar{4}04$	2	2.123		
		2.340	$\bar{6}11$	1	2.060		
2	2.295	2.322	$\bar{6}12$	1	2.026		
3	2.002	2.007	422	4	1.988		
		2.007	403	4	1.963		
3	1.958	1.962	$\bar{4}24$	1	1.925		
4	1.945	1.951	024	1	1.893		
		1.947	$\bar{4}05$	3	1.844		
4	1.870	1.866	$\bar{8}02$	3	1.819		
2	1.787	1.784	040	3	1.733		
1	1.777			2	1.697		
2	1.746			1	1.667		
2	1.712			2	1.608		
1	1.669			1	1.593		
1	1.642			3	1.553		
4	1.573			2	1.540		
4	1.548			3	1.515		
3	1.524			1	1.449		
3	1.495			2	1.435		
+ 20 lines, less than 3				3	1.346		
				2	1.335		
				2	1.305		
				3	1.296		
				3	1.254		
				+ 15 lines, less than 3.			

(Fe/Mn radiation, 114.6mm camera diameter; film corrected for shrinkage)

TABLE 3. Jahnsite, Segelerite, and Overite. Structure Cell Data

	a. JAHNSITE	b. SEGELERITE	c. OVERITE
$\underline{a}$(Å)	14.94(2)	14.826(5)	14.78
$\underline{b}$(Å)	7.14(1)	18.751(4)	18.78
$\underline{c}$(Å)	9.93(1)	7.307(1)	7.14
β	110.16(8)°	-	-
space group	P2/a	Pcca	Pcca
specific gravity	2.706, 2.718	2.67	2.53
density (gm cm^{-3})	2.715	2.610	2.48
formula	$CaMg_2(H_2O)_8Mn^{2+}Fe_2^{3+}(OH)_2[PO_4]_4$	$CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$	$CaMg(H_2O)_4Al(OH)[PO_4]_2$
Z	2	8	8

a. Tip Top pegmatite. Refined coordinates from PAILRED diffractometer.

b. Tip Top pegmatite. Refined coordinates from PICKER diffractometer.

c. Fairfield Utah. Axial data and specific gravity of Larsen (1940).

Occurrences

Jahnsite is a relatively abundant mineral in the low temperature phosphate paragenesis at the Tip Top pegmatite. It usually occurs with leucophosphite, hureaulite, and collinsite, but granular masses up to 5 cm across consisting solely of the new mineral fill cavities in ferrisicklerite. These masses are actually densely interlocking turbid dull yellow-orange crystals invariably twinned on {001} resulting in a pseudo-orthorhombic appearance. It also occurs in variable amount at the White Elephant, Bull Moose, Big Chief, and Linwood pegmatites in the Custer region of South Dakota, invariably with hureaulite and leucophosphite. At the Linwood pegmatite, much of the bright yellow friable material filling cavities in heterosite and associated with collinsite and robertsite provides a powder pattern identical with jahnsite. Nut-brown striated crystals occur as warty aggregates with laueite and strunzite from the Palermo No. 1 pegmatite, North Groton, New Hampshire.

A related compound occurs in moderate abundance as orange splinters intergrown with (= replacing?) coarse fibrous black rockbridgeite from the Fletcher pegmatite, North Groton, New Hampshire. It corresponds to the "golden rockbridgeite" of New England collectors. The unit cell and space group are similar to those of type jahnsite, but the optical and physical properties indicate a different composition. This may be related to the "xanthoxenite-like phase" incompletely described by Mrose (1955). Yet another related phase occurs as tan, tabular, twinned crystals coating rose quartz from the Taquaral pegmatite, Minas Geraes, Brazil. It, too, provides a structure cell akin to jahnsite, and ARL electron probe analysis shows that it is the aluminum analogue of jahnsite. Both compounds are presently under further investigation.

TABLE 4. Jahnsite, Segelerite, and Overite. Chemical Analyses

	a. JAHNSITE		b. SEGELERITE		c. OVERITE	
	1	2	3	4	5	6
CaO	6.6	6.9	13.6	14.0	16.1	15.1
MgO	9.4	9.9	9.5	10.1	11.2	10.9
MnO	8.0	8.7	0.0	-		-
Fe_2O_3	15.1	19.6	16.4	20.0		-
Al_2O_3	2.1	-	0.4	-	13.7	13.7
P_2O_5	32.2	35.0	33.1	35.6	38.9	38.3
H_2O	18.8	19.9	19.1	20.3	22.04	22.0
	92.2	100.0	92.1	100.0	101.9	100.0

[1] ARL electron probe analysis computed as oxides. Unpolished crystals: T. Solberg, analyst. Water determined by Penfield tube: A. Lingard, analyst.

[2] For $CaMg_2(H_2O)_8Mn^{2+}Fe_2^{3+}(OH)_2[PO_4]_4$.

[3] See (1). Water determined by weight loss upon fusion.

[4] For $CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$.

[5] ARL electron probe analysis computed as oxides. Water from Larsen (1940). Polished crystal: A. J. Irving, analyst.

[6] For $CaMg(H_2O)_4Al(OH)[PO_4]_2$.

Name

The Commission on New Minerals and Mineral Names (IMA) has approved the name jahnsite prior to publication. It is a pleasure to christen this magnificent species **jahnsite** after Professor Richard

H. Jahns, outstanding scholar of pegmatites, and Dean of Earth Sciences, Stanford University.

SEGELERITE

$CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$

Descriptive mineralogy

This new species closely resembles jahnsite, to which it is chemically related, but is easily distinguished by its relatively simple orthorhombic morphology, pale green color, and long prismatic development with nearly square cross-section. Segelerite appears to be much less frequent than jahnsite; it occurs as striated prisms along joint fractures in dense ferrisicklerite-rockbridgeite, usually in close association with collinsite, minor hydroxylapatite, and globules of red-brown robertsite. The color is pale yellow-green, lively chartreuse, inclining to colorless. The cleavage is {010} perfect, hardness 4^-, specific gravity by sink-float in methylene iodide-toluene 2.67. The computed density is 2.610 gm cm^{-3} based on the structure cell and ideal formula. It is soluble in dilute HCl solution.

Crystal morphology

Crystals are up to 0.5 mm in length, orthorhombic ($2/m$ $2/m$ $2/m$), and striated parallel to the prism which is [001]. The cross-section shows a nearly square outline, with lustrous $a\{100\}$, $b\{010\}$, $m\{110\}$ and $q\{121\}$. A typical development is shown in Figure 2. The analysis of reticular density in Table 1b shows that the development conforms to the space group and structure cell shape.

X-ray data

A partly indexed powder pattern is offered in Table 2b, and the single crystal study is summarized in Table 3b. We note that the structure cell is related to that of jahnsite, but it is not possible to transform the jahnsite structure into that of segelerite continuously without changes in order of ligand groupings around the Mg–(O,H_2O) octahedra, and we conclude that a stereoisomeric relationship exists between the two species. Toward this end, we are investigating the structure of segelerite.

Overite: an isotype

We mention the close similarity between the structure cell and crystal morphology of segelerite with that of overite. Overite was described as a new species by Larsen (1940) who proposed the formula $Ca_3Al_8(PO_4)_8(OH)_6 \cdot 15\,H_2O$, $Z = 2$, and the cell data a 14.75, b 18.74, c 7.12 Å, space group *Bmam*. Through the assistance of Mr. John S. White, Jr., we obtained some single crystals from the type material, bearing U.S.N.M. No. R7898. X-ray, Weissenberg, and precession photographs about the c-axis conclusively show that the space group is *Pcca*, identical with that of segelerite. Recently, we examined the crystal chemistry of overite by electron probe analysis, following the procedures used for jahnsite and segelerite. The original formula is in error: overite is the aluminum analogue of segelerite, with the formula $CaMg(H_2O)_4Al(OH)[PO_4]_2$.

The overite analysis was performed by Dr. A. J. Irving who used diopside, anorthite, and wyllieite as standards. Since the crystal of overite was polished, it provided excellent results as shown in Table 4c.

Chemical analysis

ARL electron probe analyses run simultaneously with the jahnsite on several small single segelerite crystals gave the results in Table 4b. The water content was determined by weight loss upon combustion since there was no evidence for oxidizable ionic species.

Based on $P = 16.0$, the cation ratios establish $Ca_{8.3}Mg_{8.0}Fe^{3+}_{7.1}Al_{0.4}[PO_4]_{16.0}(OH)_{7.1} \cdot 30.7\,H_2O$ suggesting the ideal formula $CaMg(H_2O)_4Fe^{3+}(OH)[PO_4]_2$, with eight formula units in the cell.

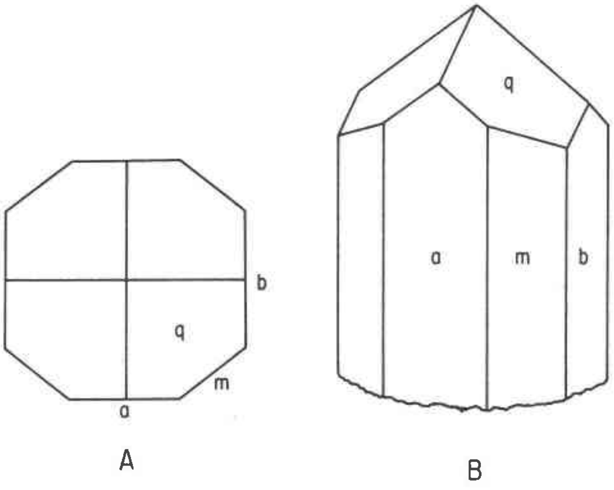

FIG. 2. Crystal of segelerite showing the forms $b\{010\}$, $a\{100\}$, $m\{110\}$, and $q\{121\}$. Tip Top pegmatite. A. Plan. B. Clinographic projection.

Optical properties

The type segelerite is biaxial (−), α 1.618, β 1.635, γ 1.650 ± 0.003, $2V$ large. The absorption is Z (yellow) > X, Y (colorless), with $X = b$, $Y = a$, $Z = c$.

Occurrence

So far, the Tip Top pegmatite is the only locality for segelerite. It usually occurs in intimate association with collinsite and hydroxylapatite. According to the crystal structure analysis of jahnsite, the ordered Mn^{2+} cations evidently stabilize that mineral. Since jahnsite is evidently rather abundant, sufficient Mn^{2+} was apparently available in solution to promote its formation. As Mn^{2+} is depleted, segelerite forms, providing there still remain some Fe^{3+} cations in solution. Segelerite appears later than jahnsite and for this reason is found in close association with the Ca–Mg phosphates rather than leucophosphite and hureaulite which have crystallized earlier with the jahnsite.

Name

The Commission on New Minerals and Mineral Names (IMA) has approved the name prior to publication. The lustrous well-developed crystals of this new phosphate **segelerite** make it fitting to honor Mr. Curt G. Segeler of Brooklyn, New York, amateur mineralogist, whose superb collection of micromounts, especially of pegmatite phosphates, reveals the care and meticulousness of his mineral identifications through optical and microchemical techniques.

ROBERTSITE

$Ca_3Mn^{3+}_4(OH)_6(H_2O)_3[PO_4]_4$

Descriptive mineralogy

Evidence provided here supports the statement that robertsite, mitridatite, and arseniosiderite belong to the same structure type. This is of considerable interest since mitridatite is a stable and persistent low temperature phase and occurs frequently as dull-green stains coating wall rocks in pegmatites which carry transition metal phosphate minerals.

Work on the entire mitridatite problem has been hindered through lack of suitable single crystal material. All three phases characteristically occur as fine fibrous masses of thin curved plates, dense nodules, and stains. We shall elaborate on the crystal chemistry of the robertsite-mitridatite-arseniosiderite group as a conclusion of this study.

Robertsite is moderately abundant, at least locally, and is probably rather widespread, especially in pegmatites where triphylite occurs in abundance. It is easily confused with the rockbridgeite-frondelite group of minerals, which commonly occur as shiny black to deep reddish-brown fibrous masses. The key differences include color and specific gravity. Rockbridgeite slivers are bluish to greenish in tint; robertsite is reddish-brown to deep blood-red. In methylene iodide, fragments of rockbridgeite-frondelite sink while those of robertsite (and mitridatite) float. Robertsite occurs as fine feathery aggregates of deep red to bronzy plates, botryoidal aggregates of shiny black fibers, shiny black plates, and lustrous small wedge-shaped crystals. A spectacular specimen, measuring 6 × 7 cm in area, shows the mineral as broad plates up to 5 mm across which are grouped as bunched radial aggregates along a fracture in a dense rockbridgeite-ferrisicklerite mass; its lustrous black color is reminiscent of hematite and manganite though it lacks their steel-grey tint. Robertsite lines cavities in rockbridgeite and ferrisicklerite upon which are placed crystals of leucophosphite, jahnsite, and hureaulite. A late generation of this platy robertsite often is deposited upon these minerals. Another specimen shows a dense mass of deep red-brown massive robertsite in which cavities are lined with lustrous wedge-shaped crystals of the same. It is this specimen from which crystals suitable for X-ray and morphological study were obtained. All specimens originated from the Tip Top pegmatite. The mineral also occurs at the Linwood pegmatite in a paragenesis similar to the Tip Top material, and in small amounts at the White Elephant and Gap Lode pegmatites.

The color is shiny black, blood-red to reddish-brown in thin slivers, streak chocolate brown, hardness 3.5, cleavage {100} good. The specific gravity of the crystals is 3.17 (sink-float in methylene iodide-toluene), 3.13 (Berman microbalance, on botryoidal fibers). Finely fibrous material is silky, deep reddish-brown, resembling goethite. Thin plates are reminiscent of lepidocrocite.

Crystal morphology

Single crystals of robertsite suitable for detailed morphological and X-ray study are rather rare, and only one specimen provided adequate material for the crystal structure analysis now in progress. Measurable crystals are thick tabular and wedge-shaped; very thick crystals are pseudo-rhombohedral in appearance. Crystals range from 0.05 to 0.5 mm in maximum dimension. They are frequently twinned by rotation normal to $\{100\}$ and often involve rotations by multiples of $\pi/3$ radians owing to the pseudo-hexagonal character of the crystal subcell. Untwinned crystals show prominent $a\{100\}$, $c\{001\}$ and $p\{031\}$, and establish beyond doubt the monoclinic symmetry of the species. In addition several doubly terminated crystals established the monoclinic $2/m$ holosymmetry. A typical development is shown in Figure 3.

X-ray crystallography

Determination of the correct space group and unit cell parameters proved difficult owing to the rarity of suitable single crystals. Despite the rather lustrous and even appearance of many crystals, goniometric examination revealed twinning by rotation of $\pm\ \pi/3$ radians on $\{100\}$. X-ray photographs of the same were almost impossible to decipher until a small untwinned fragment was secured. Rotation about [010], [001] and [100], and Weissenberg photographs of the $h = 0$, $l = 0$, $k = 0$ to 4 levels, established the monoclinic end-centered cell in Table 5. It exhibits a profound and persistent substructure which obeys the same extinction criteria and requires b (sub) $= b/3$. After the b-axis rotation photograph was prepared, successive rotation photographs were obtained by regluing the crystal $\pm\ \pi/3$ radians away from b. The three photographs, whose axes are perpendicular to a^*, reveal a pronounced pseudo-hexagonal cell. However, only the b-direction afforded evidence of a mirror plane although the axial translations are roughly of the same magnitude for all three photographs. This conclusively established the monoclinic character of the species. We note that $\sqrt{3}\,b(\text{sub}) = 11.27$ Å $\approx c$.

A partly indexed powder pattern appears in Table 5. Since the chemical analysis and optical properties shall be correlated with those of mitridatite and arseniosiderite—as are the X-ray data—we provide further discussion of the new species in the next section.

Name

The name **robertsite** was approved by the Commission of New Minerals and Mineral Names, International Mineralogical Association. It is a privilege to name this interesting species after Mr. Willard L. Roberts, Mineralogist, South Dakota School of Mines and Technology, in whose private cabinet are some of the most remarkable of Nature's mineralogical legacy. In addition, Mr. Roberts maintains a continued interest in the minerals of the Black Hills pegmatites, particularly the rarer species, and his compendious knowledge of minerals in general has consisently provided many fine specimens for research.

The Mitridatite and Arseniosiderite Problems: Isotypy with Robertsite

a. Mitridatite

Mitridatite is a name applied to many colloidal and fine-grained substances which occur as nodules and gumlike masses at numerous localities in the Kertch and Taman peninsulas in the Soviet Union. The most recent study on the problem, an investigation on mitridatites by Chukhrov, Moleva, and Ermílova (1958), provides powder and optical data. A firm knowledge of the mitridatite chemistry was

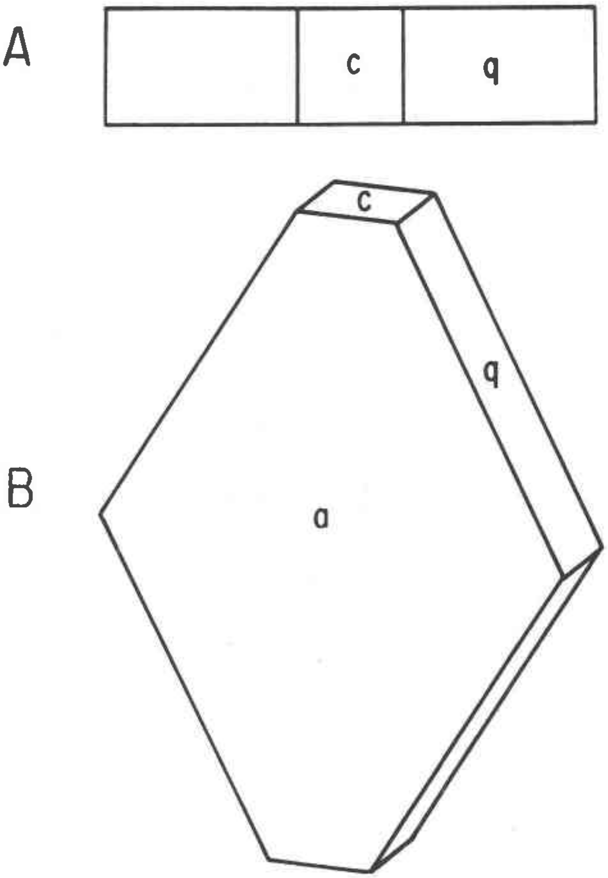

FIG. 3. Crystal of robertsite showing the forms $c\{001\}$, $a\{100\}$, and $q\{031\}$. Tip Top pegmatite. A. Plan. B. Clinographic projection.

TABLE 5. Robertsite, Mitridatite, and Arseniosiderite. Structure Cell Data

	1	2	3
a(Å)	17.36(2)	17.52(2)	17.76(4)
b(Å)	19.53(5)	19.35(4)	19.53
c(Å)	11.30(3)	11.25(2)	11.30
β	96.0°	95.92°(8)	[96.0]
space group	A2/a	A2/a	[A2/a]
specific gravity	3.13, 3.17	3.24	3.58, 3.60
density (gm cm-3)*	3.05	3.08	3.59
formula	$Ca_3Mn_4^{3+}(OH)_6(H_2O)_3[PO_4]_4$	$Ca_3Fe_4^{3+}(OH)_6(H_2O)_3[PO_4]_4$	$Ca_3Fe_4^{3+}(OH)_6(H_2O)_3[AsO_4]_4$
Z	8	8	8

*The density is computed from ideal formula and the cell data.

[1] Robertsite. Tip Top pegmatite. From rotation and Weissenberg data.

[2] Mitridatite. White Elephant pegmatite. From rotation, Weissenberg and precession data.

[3] Arseniosiderite. Mapimi, Mexico. From partly indexed powder data, matched with (1) and (2). Errors in b and c are probably ± 0.1 Å.

hindered by the lack of coarse-grained material, in particular of single crystals. In this study, we offer evidence of specific status for mitridatite and conclude on the basis of powder diffraction patterns that the material of Chukhrov *et al* is the fine-grained equivalent of our specimens and that the materials are in fact the same species.

Recently, Mr. W. L. Roberts provided the senior author with some excellent crystals which afforded powder patterns (Table 6) similar to robertsite and practically identical to the powder data of Chukhrov *et al.* In addition, minerals D, E, and F from the Borborema pegmatite, described by Murdoch (1955), appear to belong to the robertsite-mitridatite series. The prominent powder lines are 8.65/10; 5.60/5; and 2.74/6. The crystals, which line cavities in massive material and which are associated with jahnsite, collinsite, hydroxylapatite, and hureaulite, were collected on the dumps of the White Elephant pegmatite, whose paragenesis closely resembles that of the Tip Top pegmatite suite. Single crystals are thin tabular up to 0.2 mm and afford $a\{100\}$, $c\{001\}$ (small), and rather commonly, $v\{\bar{4}23\}$. This last form was firmly established on the basis of careful X-ray alignment. Precession photographs with a^* horizontal on the films afforded the opportunity to explore *b*- and *c*-axis projections in detail. The single crystal data in Table 5 thus derived clearly establish isotypy with robertsite, including similar sub- and super-structure reflections. Like robertsite, the strong substructure reflections predominate, resulting in similar and relatively simple X-ray powder patterns. Combinations of $a\{100\}$ and $v\{\bar{4}23\}$ result in a polyhedron (Fig. 4) with profound pseudo-rhombohedral appearance.

Mitridatite crystals are deep red to bronzy-red, pleochroic pale yellow-green to red. Crushed crystals and fine-grained material are dull olive-green, as is the streak, the latter aiding in distinction from robertsite whose streak is brown. The specific gravity is 3.24 ± 0.02, determined by sink-float on single crystals.

Mitridatite commonly occurs at the final stages of corrosion of primary transition metal phosphates in pegmatites. The dull green stains on silicates in phosphate-bearing pegmatites are in part mitridatite. Scrapings of such stains afford powder patterns similar to coarsely crystalline mitridatite but with considerable line broadening. These stains form at low temperature and coat large surfaces of pegmatite exposures after they have been abandoned and exposed to weathering. The occurrences at the Kertch and Taman peninsulas are in sedimentary oölitic iron ores. Vivianites, which occur as sedimentary nodules derived from the decomposition of organic remains as well as vivianites from pegmatites, develop in time a film of mitridatite, suggesting that ground waters provide Ca^{2+} cations for its formation. Mitridatite may prove to be a potentially important phase in the chemistry of soils. Despite the complexity of its crystal cell, we are approaching the problem of the crystal structure with enthusiasm.

b. Arseniosiderite

This species, long of uncertain status, occurs at many localities as a low temperature oxidation product of löllingite and arsenopyrite. In addition, it replaces earlier arsenates such as scorodite. Palache, Berman, and Frondel (1951) list it as hexagonal or tetragonal, based on optical data. The color is golden yellow in fine masses to reddish-brown in fibers to deep reddish or brownish black in granular material. It is extremely dichroic reddish-brown to practically colorless, according to Larsen and Berman (1934). The "mazapilite" of Koenig (1889) was shown by Foshag (1937) to be arseniosiderite.

Through the help of Mr. John S. White, Jr., we secured a specimen of the "mazapilite" and part of the material described by Foshag (1937). The powder data are identical for both and are offered in Table 6 for Foshag's material. The obvious relationship with robertsite and mitridatite provided opportunity to derive approximate crystal cell data (Table 5) by analogy with those minerals. Koenig (1889) obtained a specific gravity of 3.58 for "mazapilite," and Mrose in Palache *et al* (1951) obtained 3.60.

c. Optical data for robertsite, mitridatite, and arseniosiderite

New optical data for robertsite and mitridatite plus the data in Larsen and Berman (1934) for arsenio-

TABLE 6. Robertsite, Mitridatite, and Arseniosiderite. X-ray Powder Data

ROBERTSITE				MITRIDATITE		MITRIDATITE (Sample 2) (Chukhrov et al., 1958)		MITRIDATITE (Sample 3) (Chukhrov et al., 1958)		ARSENIOSIDERITE (Mapimi)			
I/I_0	d(obs)	d(calc)	hkl	I/I_0	d(obs)	I/I_0	d(obs)	I/I_0	d(obs)	I/I_0	d(obs)	d(calc)	hkl
10	8.63	8.63	200	10	8.64	7	8.81	6	8.60	10	8.84	8.83	200
5	5.61	5.63	031	6	5.55	8	5.57	8	5.60	5	5.62	5.63	031
2	4.32	4.32	500	3	4.30					3	4.58	4.54	202
1	4.14	4.13	$\bar{3}31$							4	3.28	3.31	402
2	3.48	3.52	$\bar{4}31$	1	3.49	2	3.49	1	3.48			3.26	060
4	3.27	3.25	033	4	3.20	6	3.19	5	3.18	4	3.22	3.24	$\bar{1}33$
		3.24	$\bar{1}33$							5	2.945	2.945	600
2	3.17	3.20	160							8	2.772	2.760	$\bar{2}04$
1	3.03	3.04	260	1	3.01	3	3.02	2	3.02	3	2.622		
1	2.937	2.952	233							1	2.602		
3	2.876	2.877	600	4	2.881	3	2.90	2	2.88	2	2.522		
1	2.807	2.809	004	1	2.765					2	2.491		
		2.803	$\bar{1}62$							2	2.245		
6	2.749	2.758	$\bar{2}04$	7	2.721	10	2.73	10	2.73	4	2.213		
		2.757	162	1	2.617					1	2.137		
4	2.590	2.584	$\bar{3}62$	4	2.562	6	2.56	6	2.57	2	1.891		
2	2.465	2.459	602	2	2.460	1	2.46	2	2.46	1	1.857		
2	2.223	2.249	404	3	2.207					1	1.797		
3	2.160	2.158	800	4	2.169	4	2.18	5	2.17	2	1.770		
2	2.112			2	2.106	3	2.10	3	2.10	4 brd	1.643		
2	2.048			1	2.068					+ 20 lines, <2.			
3	1.899			3	1.901	3	1.911	4	1.919				
2	1.745			2	1.741	2	1.750	2	1.745				
5	1.623			4	1.612	7	1.616	7	1.614				
1	1.585			1	1.588								
3	1.542			3	1.548	3	1.553	3	1.555				
1	1.464			2	1.474			2	1.483				
2	1.412			2	1.408	2	1.415	1	1.415				
2	1.375							1	1.396				
								1	1.372				

(114.6mm camera diameter, FeK_α radiation, corrected for shrinkage.)

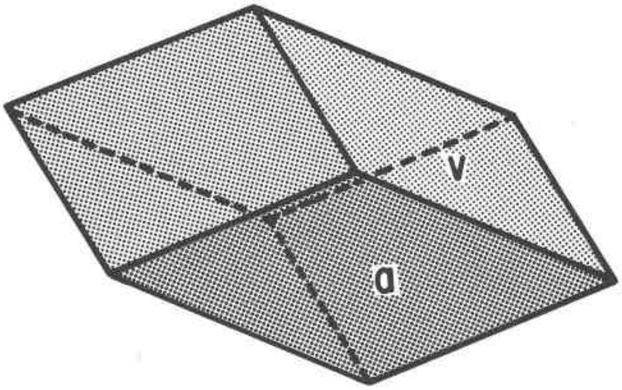

FIG. 4. Pseudo-rhombohedral crystal of mitridatite resulting from the forms $a\{100\}$ and $v\{\bar{4}23\}$. White Elephant pegmatite. Clinographic projection.

siderite and those of Chukhrov *et al* for mitridatite are listed in Table 7. We also provide Gladstone-Dale calculations for all three compounds and note the excellent agreement with the observed mean indices of refraction.

All three compounds are optically very similar; they are strongly birefringent, strongly absorbing in the Y and Z directions, biaxial negative, and with small $2V$. The acute bisectrix is roughly perpendicular to the plane of the good {100} cleavage. The data of Chukhrov *et al* (1958) show significantly lower indices, doubtless arising from the highly hydrated nature of their fine-grained material.

d. Chemistry of robertsite, mitridatite, and arseniosiderite

We submit two new chemical analyses, for robertsite fibers in association with single crystals from the Tip Top pegmatite and for massive dull green mitridatite closely associated with crystals from the White Elephant pegmatite. Both afforded powder patterns which are identical with those from crushed single crystals. The massive green mitridatite contained a substantial admixture of quartz which evidently crystallized at low temperature and which occurs as very small grains in the mass. These analyses, along with analyses for two rather coarse-grained arseniosiderites reported by Koenig (1889) (= "mazapilite") and Foshag (1937) appear in Table 8. Ten analyses for mitridatite from the Soviet Union appear as a compilation from several sources in Palache *et al* (1951). Since all specimens were of a fine-grained and colloidal nature, they contain varying amounts of water, a deficiency of CaO, and an excess of Fe_2O_3, suggesting mitridatite-goethite-hematite admixtures. Chukhrov *et al* (1958) offer five new analyses, all rather similar. We provide a list of the analyses of samples 2 and 3 in Table 8. They propose the formula $Ca_2Fe_3(PO_4)_{3-x}(OH)_{4+3x}\cdot nH_2O$. Their study leaves little doubt that our coarsely crystalline material is the same species and that the deviations in chemical analysis, specific grav-

TABLE 7. Robertsite, Mitridatite, and Arseniosiderite. Optical Data

	1	2	3	4	5
α	1.775(5)	1.785(5)	1.762	1.80	1.815
β, γ	1.82 (1)	1.85 (1)	1.770	1.88	1.898
<n> obs	1.805	1.828			1.870
<n> calc*	1.803	1.836			1.853
2V	∿8°	5-10°		0°±	uniaxial
sign	--	--		--	--
absorption	Y,Z >> X	Y,Z >> X		Y,Z >> X	Y,Z >> X
pleochroism	Y,Z deep reddish brown X pale reddish-pink	Y,Z deep greenish brown X pale greenish yellow		Y,Z dark reddish-brown X colorless	
orientation	X nearly $\perp$ {100}	X nearly $\perp$ {100}		X $\perp$ {100}	

[1] Robertsite. Tip Top pegmatite.
[2] Mitridatite. White Elephant pegmatite.
[3] Mitridatite. Fine-grained material (Sample 2). Chukhrov *et al* (1958).
[4] Arseniosiderite. Larsen and Berman (1934). They note X = *c* and perfect {001} cleavage.
[5] Arseniosiderite. Larsen and Berman (1934) for the variety "mazapilite".

*From Gladstone-Dale relationship for ideal formulae.

ity, and optical properties result from the fine-grained nature of their material. In addition, three analyses on fine-grained arseniosiderite are compiled in Palache *et al* (1951), but their status in the absence of X-ray patterns is uncertain. Thus, discussions of them would be of questionable value.

Our two analyses were computed into cations per unit cell utilizing the specific gravity averages and the cell volumes from the X-ray studies. The best agreement, which also agrees remarkably well with the arseniosiderite analyses, suggests ideal $Ca_3Mn^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$, with $Z = 8$ for robertsite and obvious isotypy among all three species. We note that the massive mitridatites reveal an excess of water which is repeated consistently in the other analyses for colloform materials, and submit that this excess is adsorbed and not a part of the crystal structure. In all analyses, the Ca^{2+} content is low by about 5 to 10 percent. This may result from slight excesses of trivalent cations and/or alkalies as substituents for Ca^{2+}, but more likely arises from partial vacancies over the large cation sites. Based on the ideal formulae, the computed densities are low by about 5 percent and suggest that the slight excesses of trivalent cations are real. A better fit can be obtained with the formula $Ca_6Fe^{3+}{}_9(OH)_{15}(H_2O)_9[PO_4]_8$, $Z = 4$, but we doubt whether the precise formula can be unambiguously stated without knowledge of the crystal structure. Accordingly, the simpler formulae are proposed in this study.

The agreement between the chemical analyses, the X-ray powder data, and the optical data leaves little doubt that the three species robertsite, mitridatite, and arseniosiderite belong to the same structure type. In addition, it is very likely that $Ca_3Mn^{3+}{}_4(OH)_6(H_2O)_3[AsO_4]_4$ will prove to be a stable phase and may exist in Nature.

We conclude by proposing the following formulae:

robertsite	$Ca_3Mn^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$
mitridatite	$Ca_3Fe^{3+}{}_4(OH)_6(H_2O)_3[PO_4]_4$
arseniosiderite	$Ca_3Fe^{3+}{}_4(OH)_6(H_2O)_3[AsO_4]_4$.

Specimens of the type materials for all three new species will be deposited in the U.S. National Museum.

Acknowledgments

The electron probe analyses on jahnsite and segelerite, requiring careful preparation and choice of standards, were conducted by Mr. T. Solberg. Dr. A. J. Irving kindly contributed to the study when the crystal-chemical relationship between overite and segelerite was recognized. Contributions of fragments of type materials from the U.S. National Museum were invaluable in providing results on species which could easily be confounded. Messrs. W. L. Roberts and C. Segeler provided samples of many fine specimens for research.

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