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Crystal chemistry and crystallography of some minerals in the tetradymite group

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ABSTRACT

Selen-tellurium is a mixture of the minerals selenium and tellurium. The atoms in stibarsen are ordered (completely or partially) so that the correct chemical formula is AsSb. Matildite $AgBiS_2$, bohdanowiczite $AgBiSe_2$, and volynskite $AgBiTe_2$, show atomic ordering in space group $P\bar{3}m1$ with unit-cell dimensions similar to those of tsumoite, BiTe. The S and Te atoms are apparently disordered in ingodite to yield the chemical formula Bi(S,Te) and in sulphotsumoite to yield the chemical formula Bi(Te,S). The unnamed mineral of Aksenov et al. (1968) seems to be tellurian tsumoite, $(Bi_{0.58}Te_{0.42})Te$. The synthetic composition of Godovnikov et al. (1966) is equivalent to bismuthian tsumoite, $Bi(Te_{0.75}Bi_{0.25})$. Platynite is possibly plumbian sulfurian nevskite, $(Bi_{0.650}Pb_{0.345})(Se_{0.657}S_{0.348})$.

Kawazulite, Bi_2SeTe_2, is isostructural with tetradymite. Csiklovaite is a mixture of tetradymite and bismuthinite. The chemical formula of skippenite is probably $Bi_2(Se,Te,S)_3$, like that of tellurian paraguanajuatite. The Se and S are disordered in laitakarite to yield the chemical formula $Bi_4(Se,S)_3$. As S and Te ordering was not observed in josëite or josëite-B, there is possibly a complete solid solution series from tellurian ikunolite, $Bi_4(S,Te)_3$, to sulfurian pilsenite, $Bi_4(Te,S)_3$. The unnamed mineral of Yingchen (1986) is apparently tellurian ikunolite. Rucklidgeite, Bi_3Te_4, has an antipilsenite, Bi_4Te_3, structure type. The unnamed mineral of Harañczyk (1978) is rucklidgeite.

INTRODUCTION

The narrow definition of the tetradymite group was given by Strunz (1970) to include only $Bi_2(S + Se + Te)_3$ minerals. These hexagonal or trigonal minerals have an approximately cubic closest-packed layer crystal structure (Pauling, 1975) in which the five planes within the tetradymite layer are Te-Bi-S-Bi-Te. The axis [0001] of the hexagonal cell corresponds to [111] of the approximately cubic closest-packed crystal structure, where the planes are stacked perpendicular to [111]. The relationship between different ratios of Bi:(S + Se + Te) and Te:S with respect to the *d*-value with the strongest corresponding intensity in X-ray diffraction patterns was discussed by Spiridonov (1981). The definition of the tetradymite mineral group was widened later by Bayliss et al. (1986) to include as essential those chemical elements from group Va (As, Sb, and Bi) and group VIa (S, Se, and Te) to produce eight subgroups each with a different number of planes in the basic layer repeat unit layer. Table 1 lists minerals within the tetradymite group, which may be subdivided on the basis of the number of approximately cubic closest-packed planes in the layered repeat unit, where each plane contains one chemical element or a random distribution of two or more chemical elements. Differences in atomic size and electronegativity allow ordered crystal structures at low temperature, but there are disordered crystal structures with extensive solid solution at high temperature.

The purpose of this paper is to compare and contrast the various mineral species within the tetradymite group in order to investigate some of the problems associated with minerals in this group. The principal objectives were as follows:

1. To obtain X-ray powder-diffraction data for minerals where no published data exist, or discredit the mineral (e.g., selen-tellurian, csiklovaite).
2. To use published X-ray powder-diffraction data to determine if the crystal structures are ordered, partially ordered, or disordered in order to ascertain the correct chemical formula (e.g., stibarsen, volynskite, ingodite, sulphotsumoite, kawazulite, skippenite, laitakarite, josëite, josëite-B).
3. To reindex the published X-ray powder-diffraction data and obtain refined unit-cell dimensions (e.g., matildite, bohdanowiczite).
4. To identify unnamed minerals, incorrectly named minerals, and synthetic phases within the tetradymite group.
5. To review the literature and ascertain the status of platynite.

METHODS

In the approximately cubic closest-packed layered metallic minerals, the number of possible crystal structure types is limited. All of the crystal structure types have atoms with symmetry-fixed values of coordinates *x* and *y*, whereas the coordinate *z* may or may not have a fixed value. Some atoms may have ordered, partially ordered, or disordered distributions. From these possible ordered, partially ordered, or disordered crystal structure types with

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TABLE 1. Subgroups within the tetradymite group

No. of atom planes	Space group	Subgroup name	Mineral	Chemical formula
1	$P3_121$	Te	selenium	Se
			tellurium	Te
2	$R\bar{3}m$	As	antimony	Sb
			arsenic	As
			bismuth	Bi
			stibarsen	AsSb
4	$P\bar{3}m1$	tsumoite	tsumoite	BiTe
			sulphotsumoite	Bi(Te,S)
			nevskite	BiSe
			platynite	(Bi,Pb)(Se,S) (?)
			ingodite	Bi(S,Te)
			bohdanowiczite	$AgBiSe_2$
			matildite	$AgBiS_2$
			volynskite	$AgBiTe_2$
5	$R\bar{3}m$	tetradymite	tellurantimony	Sb_2Te_3
			tellurobismuthite	Bi_2Te_3
			paraguanajuatite	Bi_2Se_3
			skippenite	$Bi_2(Se,Te)_3$
			tetradymite	Bi_2STe_2
			kawazulite	Bi_2SeTe_2
7	$R\bar{3}m$	josëite	ikunolite	Bi_4S_3
			josëite	$Bi_4(S,Te)_3$
			laitakarite	$Bi_4(Se,Te)_3$
			pilsenite	Bi_4Te_3
			josëite-B	$Bi_4(Te,S)_3$
			poubaite	$(Bi,Pb)_3(Se,Te)_4$
			rucklidgeite	Bi_3Te_4
9	$P\bar{3}m1$	protojosëite	protojosëite	$Bi_5(Te,S)_4$ (?)
14(?)	$P\bar{3}m1$	aleksite	aleksite	$Bi_2PbS_2Te_2$ (?)
20(?)	?	hedleyite	hedleyite	Bi_7Te_3 (?)

TABLE 2. Unit-cell dimensions obtained by least-squares refinement

Mineral	Chemical formula	Powder diffraction file or specimen no.	*a* (Å)	*c* (Å)
stibarsen	AsSb	31-80	4.0255(11)	10.837(9)
matildite	$AgBiS_2$	24-1031	4.0662(21)	18.958(17)
bohdanowiczite	$AgBiSe_2$	29-1441	4.2049(14)	19.650(11)
volynskite	$AgBiTe_2$	18-1173	4.468(7)	20.75(5)
ingodite	$Bi(S_{0.56}Te_{0.43}Se_{0.01})$	37	4.2477(24)	23.075(22)
ingodite	$Bi(Te_{0.55}S_{0.45})$	77	4.246(3)	23.26(3)
sulphotsumoite	$Bi(Te_{0.67}S_{0.33})$	38-442	4.3045(18)	23.475(14)
tsumoite	$(Bi_{0.58}Te_{0.42})Te$	22-117	4.364(3)	24.32(4)
tsumoite	$Bi(Te_{0.75}Bi_{0.25})$	19-176	4.453(3)	23.90(3)
laitakarite	$Bi_4(Se_{0.67}S_{0.33})_3$	14-220	4.2239(14)	39.94(3)
ikunolite	$Bi_{3.5}S_{2.0}Te_{1.5}$		4.258(3)	39.54(4)
josëite	$Bi_{3.9}S_2Se_{0.1}Te_{1.0}$	22-364	4.2402(12)	39.774(15)
josëite	$Bi_{4.1}S_{1.9}Te_{1.0}$	12-735	4.2415(10)	39.769(16)
josëite-B	$Bi_{4.3}S_{1.1}S_{0.2}Te_{1.4}$	9-435	4.3326(25)	40.92(5)
rucklidgeite	$(Bi,Pb)_3Te_4$	29-234	4.4180(13)	41.513(15)
rucklidgeite	Be_3Te_4		4.389(16)	42.0(35)
protojosëite	$Bi_{5.5}Te_{1.9}S_{1.6}$		4.3386(11)	57.804(18)
aleksite	$Bi_2PbS_2Te_2$	29-765	4.2423(25)	79.73(5)

values for z estimated from similar crystal structures, X-ray powder-diffraction patterns were calculated with the program of Smith (Pennsylvania State University). The number of crystal-structure models may vary from two for a two-plane structure type such as stibarsen, to ten for a seven-plane structure type such as josëite.

The calculated intensities for each possible crystal-structure model were compared to the observed intensities of the published X-ray powder-diffraction data. The observed X-ray powder-diffraction data were reindexed, and improved unit-cell dimensions were obtained by least-squares refinement of unit-cell parameters. If the calculated intensities are similar to the visually estimated observed intensities in the literature, then a qualitative conclusion may be made on the order-disorder to yield probable chemical formula, structure type, and unit-cell dimensions. The calculated X-ray powder-diffraction data of the crystal-structure models that give the best possible fit to the observed X-ray powder-diffraction data are given in the tables in order to show the goodness-of-fit.

X-ray powder-diffraction patterns of the very small specimens available were obtained with a 114.6 mm Gandolfi camera (Fe radiation; Mn filter). The intensities were measured with an automated densitometer.

TE SUBGROUP

Selen-tellurium, (Se,Te) with Se:Te = 2:3, was originally described from the El Plomo silver mine, Ojojoma District, Tegucigalpa, Honduras by Dana and Wells (1890). Only one phase consisting of both Se and Te has been found naturally, even though at least five Se-Te phases have been synthesized. The crystal structure of both minerals (i.e., selenium, tellurium) in space group $P3_121$ has Se or Te in 3(a) at $(x,0,\frac{1}{3})$ with x about 0.25. A complete solid-solution series occurs at moderate temperatures (200–450 °C) from selenium ($a = 4.37$, $c = 4.95$ Å, PDF 6-362) to tellurium ($a = 4.46$, $c = 5.93$ Å, PDF 4-554) without superstructure reflections (Lanyon and Hockings, 1966). Appreciable segregation occurs if a melt is not cooled instantaneously. A phase with both Se and Te in fixed proportions would require a crystal structure type different from that of selenium and tellurium. As yet such a phase has not been synthesized, and it is unlikely to occur because of the difference in atomic radii of Se and Te.

Enquiries for selen-tellurium at eight large mineralogical museums revealed only one specimen, NMNH R186 (Smithsonian Museum). This specimen, from the only known locality, Honduras, was initially examined with a Kevex solid-state detector on an SEM. Distinct grains were identified as quartz, barite, selenium, tellurium, and selen-tellurium. The mineral assemblage of the specimen is identical to that described by Dana and Wells (1890), so that this specimen probably comes from the type locality. The specimen itself has not been established conclusively to be a type specimen; however, none of the type specimen appears to exist.

The grains were too fine for mounting in a polished block for electron-probe analysis. An X-ray powder-diffraction photograph taken of the fine grains containing both Se and Te was identified as a mixture of selenium and tellurium. The unit-cell dimensions of selenium and tellurium are similar to those of pure Se and Te, so that negligible substitution of Te occurs in selenium and negligible substitution of Se occurs in tellurium.

Therefore, selen-tellurium may be discredited and is

TABLE 3. X-ray powder-diffraction data for stibarsen

hkl	I_{obs}	I_{calc} Order	I_{calc} Disorder
003	30	6	0
101	10	15	0
012	100	100	100
104	60	35	39
110	70	28	32
015	10	5	0
006	10	6	7
113	10	8	0
021	5	4	0
202	40	13	17
024	20	8	11
107	10	2	0
116	20	10	15
122	40	8	13
018	5	4	6
009	10	1	0
214	10	6	10
300	10	3	4
125	5	3	0
033	5	3	0
208	5	2	3
10.10	10	2	3
220	20	2	3
036	5	3	6
312	20	3	6
02.10	5	2	3
134	10	3	6
315	10	3	0
042	10	2	3
21.10	10	4	6
11.12	5	4	7
10.13	5	2	0
045	5	2	0

TABLE 4. X-ray powder-diffraction data for volynskite

Volynskite *d*	Volynskite *hkl*	Volynskite I_{obs}	Volynskite I_{calc}	Tellurobismuthite *d*	Tellurobismuthite I_{obs}
6.28		5			
4.95		10		5.078	8
2.78	101	10	2		
3.60	012	5	2		
3.36	013	5	2		
3.21		80		3.222	100
3.09	104	100	100		
2.84	105	10	1		
2.33	107	30	1	2.376	25
2.28		10			
2.21	110	50	40	2.192	25
2.15	018	30	36		
1.98	019	10	1		
1.82	024	30	21	1.812	8
1.73	00.12	10	6		
1.61	119	20	0.2	1.611	6
1.55	208	20	13		
				1.490	8
1.45	211	10	0.3		
1.42	213	5	1		
1.41	214	20	18		
1.37	11.12	10	16		
1.30	300	10	7		
1.27	128	10	13		
1.23	10.16	10	6		

shown to be a mixture of selenium and tellurium. A proposal to discredit selen-tellurium as a mixture has been approved by the International Mineralogical Association Commission on New Minerals and Mineral Names (IMA CNMMN).

AS SUBGROUP

Trzebiatowski and Bryjak (1938) show a continuous solid solution between As and Sb at 400 °C; however, a minimum melting point in a continuous solid solution is characteristic of a transformation in the solid state. As the unit-cell dimensions of stibarsen (PDF 31-80) are midway between those of As (PDF 5-632) and Sb (PDF 35-732), the chemical composition should be approximately midway between As and Sb. The specimen for PDF 31-80 contained arsenic intergrown with stibarsen, which suggests that stibarsen may exsolve from paradocrasite, $AsSb_3$ (Leonard et al., 1971). Stibarsen rather than allemontite is the correct mineral species name (Hey, 1982).

Stibarsen X-ray powder-diffraction data from PDF 31-80 were refined in space group $R\bar{3}m$ to produce the unit-cell dimensions given in Table 2. X-ray powder-diffraction patterns were calculated both for an ordered AsSb crystal structure model with As at 0,0,0 and Sb at 0,0,½, and for a disordered crystal-structure model (As,Sb). The calculated intensities are compared to the visually estimated observed intensities of PDF 31-80 in Table 3. As 12 observed reflections have $I = 0$ with a disordered crystal-structure model, but these reflections have $I > 0$ with a completely ordered crystal structure, stibarsen is concluded to be ordered and have the chemical formula AsSb. The visually estimated observed intensity data are not sufficiently accurate to exclude partial disorder of As and Sb.

TSUMOITE SUBGROUP

Matildite, $AgBiS_2$, as described by Geller and Wernick (1959), shows ordering in space group $P\bar{3}m1$ compared to tsumoite, BiTe, which has been described by Yamana et al. (1979). The refined unit-cell dimensions based on the matildite X-ray diffraction data (PDF 24-1031) from Harris and Thorpe (1969) are given in Table 2. The value of F_{29} (Smith and Snyder, 1979) increases from 2(0.060,242) to 5(0.070,84), where F_N = overall value of $F_N(|\Delta_{2\theta}|, N_{poss})$ with N as the number of observed reflections. The increase in F_{29} reflects a significant change in unit-cell dimensions during refinement.

Bohdanowiczite, $AgBiSe_2$, as described by Geller and Wernick (1959) shows ordering in space group $P\bar{3}m1$ compared to tsumoite, BiTe. The refined unit-cell dimensions based on the X-ray diffraction data for bohdanowiczite (PDF 29-1441) from Pringle and Thorpe (1980) are given in Table 2. The value of F_{30} increases from 4(0.045,162) to 10(0.051,59).

Volynskite, $AgBiTe_2$, was described by Bezsmertnaya and Soboleva (1965). The XRD data for volynskite (PDF 18-1173) were indexed using space group $P\bar{3}m1$; the unit-cell dimensions in Table 2 correspond to a value of F_{20} of 2(0.150,78). Reflections that could not be indexed were largely those of tellurobismuthite, Bi_2Te_3, as shown in Table 4 (PDF 15-863), in addition to weak reflections for

Table 5. Atomic coordinates of volynskite, space group $P\bar{3}m1$

Atom	Equipoint	x	y	z
Ag	1(b)	0	0	½
Ag	2(d)	⅓	⅔	⅔
Bi	1(a)	0	0	0
Bi	2(d)	⅓	⅔	0.163
Te	2(c)	0	0	¼
Te	2(d)	⅓	⅔	0.417
Te	2(d)	⅓	⅔	0.917

which $d = 6.28$ and 2.28 Å. An X-ray powder-diffraction pattern was calculated for an ordered crystal-structure of volynskite, as in the cases of matildite and bohdanowiczite; atomic coordinates are given in Table 5. The calculated intensities in Table 4 are similar to the visually estimated observed intensities of PDF 18-1173, indicating an ordered crystal structure.

Ingodite, Bi_2STe, was described by Zav'yalov and Begizov (1981) as having space group $P3{*}1$. The range of 12 chemical analyses given by Zav'yalov et al. (1984) varies from $Bi_{2.00}Pb_{0.02}S_{1.12}Se_{0.02}Te_{0.86}$ to $Bi_{1.38}Pb_{0.24}S_{0.95}Te_{1.42}$. The refined unit-cell dimensions of their specimen 37 ($Bi_{2.00}Pb_{0.02}S_{1.12}Se_{0.02}Te_{0.86}$) and specimen 77 ($Bi_{1.93}S_{0.94}Te_{1.14}$) are given in Table 2. An ordered crystal-structure model of Bi_2STe with atomic coordinates similar to those of matildite, $AgBiTe_2$, produced a calculated X-ray powder-diffraction pattern significantly different from the observed X-ray powder-diffraction pattern of ingodite. A crystal-structure model of Bi(S,Te), similar to that of tsumoite, with *z* atomic coordinate adjusted for the smaller S atom has atomic coordinates given in Table 6. The intensities of the calculated X-ray powder-diffraction pattern given in Table 7 are similar to the visually estimated observed X-ray powder-diffraction intensities of ingodite. Therefore, the chemical formula of ingodite (specimen 37) is inferred to be Bi(S,Te), whereas specimen 77 is inferred to be tsumoite, Bi(Te,S). The visually estimated observed intensity data are not sufficiently accurate to exclude partial ordering of S, Se, and Te.

Sulphotsumoite, Bi_3Te_2S, was originally described by Zav'yalov and Begizov (1982) as having space group $P\bar{3}m1$. The refined unit-cell dimensions of sulphotsumoite (PDF 38-442) are given in Table 2. Disordered Te and S in a crystal-structure model similar to that of tsumoite, with formula $Bi(Te_{0.67}S_{0.33})$ have atomic coordinates similar to Te + S of ingodite as given in Table 6. The intensities of the calculated X-ray powder-diffraction pattern given in Table 7 are similar to the visually estimated observed X-ray powder-diffraction intensities of sulphotsumoite. Therefore, the chemical formula of sulphotsumoite is inferred to be Bi(Te,S). The visually estimated observed intensity data are not sufficiently accurate to exclude partial ordering of S and Te.

Table 6. Atomic coordinates of ingodite, space group $P\bar{3}m1$

Atom	Equipoint	x	y	z
Bi	2(c)	0	0	0.124
Bi	2(d)	⅓	⅔	0.291
Bi	2(d)	⅔	⅓	0.459
S + Te	2(c)	0	0	0.362
S + Te	2(d)	⅓	⅔	0.056
S + Te	2(d)	⅔	⅓	0.211

Table 7. X-ray powder-diffraction data for ingodite, sulphotsumoite, and tsumoite

	Ingodite			Sulpho-tsumoite		$(Bi_{0.58}Te_{0.42})Te$		$Bi(Te_{0.75}Bi_{0.25})$	
	I_{obs}								
hkl	37	77	I_{calc}	I_{obs}	I_{calc}	I_{obs}	I_{calc}	I_{obs}	I_{calc}
005	20	30	12	30	3			20	4
101		20	12	20	2	12	0.3	10	2
102						25	1		
103		10	6	10	8				
007						25	0.1		
104	100	100	100	100	100	100	100	100	100
105		<10	1		3				
	<10	10							
106	<10	10	1	20	3	25	1		
108	50	60	39	60	37	38	43	70	38
00.11						25	1		
110	40	50	34	50	35	38	33	80	37
111				10	4				
10.10				10	2				
00.12, 115	30	40	14	30	8	75	9	50	6
10.11	<10	10	1	20	1				
117	<10	10	2						
204	20	30	18	40	18	25	18	40	19
206				10	1				
119	50	10	1						
10.13	<10	10	4	10	4				
		10							
11.10		10	0.1	10	1				
208	20	20	11	30	11	50	12	50	12
10.14				10	1				
209				10	2				
11.12	10	30	15	30	13	38	18	70	14
211	10	10	2	10	0.3				
213	10	10	1						
214	20	30	15	40	15	25	15	40	16
10.16								40	5
11.14	20	30	1	10	1				
20.13	10	10	2	10	1				
218	30	20	11	30	10	25	11	30	11
300	30	20	5	20	6			10	6

Note: The numbers 37 and 77 are specimen numbers from Zav'yalov et al. (1984).

The unnamed mineral of Aksenov et al. (1968) has Bi:Te = 2:5. The X-ray powder-diffraction data (PDF 22-117) were indexed assuming space group $P\bar{3}m1$, as in tsumoite, and the refined unit-cell dimensions are listed in Table 2. Crystal-structure models were constructed with both Bi and Te ordered and disordered in the tsumoite structure type. Many of the calculated X-ray powder-diffraction intensities are similar to the visually estimated observed intensities in Table 7; however, some significantly higher observed intensities are probably due to incorrect atomic coordinates. Although the exact nature of the Bi-Te order-disorder cannot be established, the mineral seems to be a tellurian tsumoite, $(Bi_{0.58}Te_{0.42})Te$, even though the chemical composition is far removed from BiTe.

A synthetic phase with Bi:Te = 5:3 was cooled from a molten solution at 700–270 °C at 5 °C/h by Godovnikov et al. (1966). The X-ray powder-diffraction data (PDF 19-176) were indexed by those authors on a unit cell of the hedleyite type; however, the large difference between the observed and calculated *d*-values indicates that the unit-cell dimensions are incorrect. The data were reindexed on a structure of the tsumoite type and the refined unit-cell dimensions are given in Table 2. A crystal-structure model for $Bi(Te_{0.75}Bi_{0.25})$ was inferred having coordinates similar to those of tsumoite, BiTe. The intensities of the calculated X-ray powder-diffraction pattern given in Table 7 are similar to the visually estimated observed X-ray powder-diffraction intensities of the synthetic phase (PDF 19-176). Therefore, the composition of the synthetic phase is equivalent to bismuthian tsumoite, $Bi(Te_{0.75}Bi_{0.25})$.

Platynite from Fahlun, Sweden, was originally described by Fink in 1910 (Palache et al., 1944). Although the specimen has been lost, Strunz (1963) quotes from a 1933 private communication from F. E. Wickman who states that the unit cell is hexagonal with dimensions $a = 8.49$ and $c = 20.80$ Å; however, the structure is not known. As this *c* dimension suggests a tsumoite-type structure, the original analysis may be recalculated without chalcopyrite and insolubles, and expressed as $(Bi_{0.650}Pb_{0.345})(Se_{0.657}S_{0.348})$. Therefore, platynite may be plumbian sulfurian nevskite.

Berry and Thompson (1962) stated that platynite is similar to selenjoseite. Ramdohr (1980) gave data for four X-ray powder-diffraction reflections for platynite, but he could not remember (Ramdohr, personal communication, 1983) the locality of his specimen and was unable to supply either the original X-ray film or a specimen. The composition of platynite by Nikitin et al. (1989) is similar to that of poubaite; however, the X-ray powder data could not be indexed on a cell for any mineral of the tetradymite group. Therefore, platynite remains a questionable species.

TETRADYMITE SUBGROUP

Tetradymite, Bi_2STe_2, has been synthesized by Glatz (1967), Evdokimenko and Tsypin (1971), and Abrikosov and Beglaryan (1973). The compositional limits of tetradymite have been shown to be Bi_2STe_2-$Bi_2S_{1.3}Te_{1.7}As$ determined by Kuznetsov and Kanishcheva (1970) with internally consistent X-ray powder-diffraction data. Pauling (1975) has explained why the substitution of Te by S increases the chemical stability.

Kawazulite, Bi_2SeTe_2, was originally described by Kato (1970) as the Se analogue of tetradymite. The crystal structure of synthetic Bi_2SeTe_2 was solved by Bland and Basinski (1961), and confirmed by Nakajima (1963), in space group $R\bar{3}m$ (166) with the atomic coordinates of Se 0,0,0, Te 0,0,0.211, and Bi 0,0,0.396. As Se does not have a significantly greater radius than Te, there is the possibility that the Se and Te may be disordered. The calculated intensities of an X-ray powder-diffraction pattern of kawazulite with the ordered tetradymite structure type are similar to the visually estimated observed X-ray powder-diffraction intensities of kawazulite (PDF 29-248) listed in Table 8. The visually estimated observed intensity data are not sufficiently accurate to exclude partial ordering of Se and Te.

TABLE 8. X-ray powder-diffraction data of kawazulite and skippenite

	Kawazulite		Skippenite		
				I_{calc}	
hkl	I_{obs}	I_{calc}	I_{obs}	Disordered	Ordered
003	10	3	50	16	33
006	40	10	60	11	7
101	30	9	60	10	8
009, 104	10	4	20	6	5
015	100	100	100	100	100
107	10	2		0	0
018	20	3	10	7	10
00.12	10	1		0	0
10.10	50	38	70	35	36
01.11	10	5	50	5	3
110	50	34	80	32	32
00.15	10	6	30	5	6
116, 10.13	10	9	30	11	10
01.14, 021	10	2	20B	2	2
205	20	18	40	16	16
10.16, 00.18, 208	10	5	30	5	5
11.12	6	1		0	0
02.10	10	11	40	9	9
01.17	10	1		0	0
11.15	10	13	50B	10	10
01.20		4	30	3	3
125	10	14	40B	11	11
02.16, 11.18	10	5	20	5	5
21.10		4	40	7	7

Csiklovaite was introduced by Koch (1948) for a mineral from Csiklova, Rumania, with the chemical formula Bi_2S_2Te. The validity of the mineral has been questioned by some compilers; e.g., Fleischer (1987). To obtain X-ray powder-diffraction data of csiklovaite, eight specimens consisting of several small grains and two small vials were obtained from G. Grasselly, who supplied the type specimens used by the late Sandor Koch to describe csiklovaite.

In reflected light, three areas with distinctly different colors can be seen as follows: (1) creamy yellow, (2) light blue-gray, and (3) darker blue-gray. This description matches that of Koch (1948). The eight specimens were analyzed for As, Bi, Pb, S, Se, and Te with an electron microprobe under operating conditions of 20 kV, 15 mA, 3 μm beam size, and 5 s count times. Grains from the vials were identified by X-ray powder-diffraction as a mixture of tetradymite, $Bi_2S_{1.1}Se_{0.1}Te_{1.8}$ (creamy yellow), galenobismutite (light blue-gray), and bismuthinite (dark blue-gray).

Bismuthinite and galenobismutite occur as lamellar and myrmekitic intergrowths within tetradymite. These fine-grained minerals are therefore difficult to separate. A mixture of 60 wt% tetradymite (Bi_2STe_2) and 40 wt% bismuthinite (Bi_2S_3) would approximately reproduce the chemical analysis of csiklovaite reported by Koch (1948).

No evidence has been found in studies of synthetic phases (Glatz, 1967; Kuznetsov and Kanishcheva, 1970; Evdokimenko and Tsypin, 1971) to suggest a phase with a composition near that of csiklovaite. A phase with the composition of csiklovaite does not exist among the synthetic phases or type specimens. A proposal to discredit csiklovaite as a mixture has been approved by the IMA CNMMN.

Skippenite, $Bi_2Se_2(Te_{0.9}S_{0.1})$, was described by Johan et al. (1987), and was inferred to be the Se analogue of csiklovaite. The crystal structure determination of Bi_2Se_2Te by Nakajima (1963) shows a partially disordered crystal structure with atomic coordinates for Se at 0,0,0, $(Se_{0.5},Te_{0.5})$ at 0,0,0.2115, and Bi at 0,0,0.3985 in space group $R\bar{3}m$, yielding a chemical formula similar to that of kawazulite, $Bi_2Se(SeTe)$. X-ray powder-diffraction patterns were calculated for a partially disordered crystal-structure model and an ordered crystal-structure model with the atomic coordinates Te 0,0,0, Se 0,0,0.212, and Bi 0,0,0.396 in space group $R\bar{3}m$. The calculated X-ray powder-diffraction intensities of both crystal-structure models as given in Table 8 are similar to the estimated observed X-ray powder-diffraction intensities of Johan et al. (1987). The chemical formula of skippenite is probably either $Bi_2Se(Se,Te,S)_2$ or $Bi_2(Se,Te,S)_3$, as is true for tellurian paraguanajuatite.

JOSËITE SUBGROUP

Laitakarite, Bi_4SSe_2, was described by Vorma (1960); however, Nakajima (1963) stated that the Se and S could be either ordered or disordered within space group $R\bar{3}m$. The refined unit-cell dimensions of laitakarite (PDF 14-220) are given in Table 2. An X-ray powder-diffraction pattern was calculated for a disordered crystal-structure having space group $R\bar{3}m$ with atomic coordinates similar to those of ikunolite, (Bi_4S_3; Kato, 1959) and pilsenite, (Bi_4Te_3; Yamana et al., 1979). The $S_{0.333}Se_{0.667}$ occupies sites with coordinates 0,0,0 and 0,0,0.426, whereas Bi occupies sites at 0,0,0.142 and 0,0,0.287. A second calculated X-ray powder-diffraction pattern was calculated for an ordered model with the S atom at 0,0,0, and the two Se atoms at 0,0,0.426. The calculated data for both the ordered and disordered crystal-structure models, which are given in Table 9, can be compared to the visually estimated observed intensities (PDF 14-220) of Vorma (1960). The calculated data of the disordered crystal-structure model are similar to the observed data (PDF 14-220), especially for 003 and 006, so that the correct formula of laitakarite is $Bi_4(Se,S)_3$.

Josëite was named after Sañ José, Minas Gerais, Brazil by Kenngott in 1853 (Palache et al., 1944). The chemical formula and refined unit-cell dimensions for space group $R\bar{3}m$ are given in Table 2. The data in PDF 9-435 (Li, 1957) are for a type-locality specimen. Calculated X-ray powder-diffraction patterns were produced for ordered crystal-structure models for Bi_4STe_2 and Bi_4S_2Te, and disordered crystal-structure models for $Bi_4(Te_2S)$ and $Bi_4(S_2Te)$. The atomic coordinates of $Bi_4(S_2Te)$ were taken as Bi at 0,0,0.1433 and 0,0,0.2856, and $S_{0.667}Te_{0.333}$ at 0,0,0 and 0,0,0.4260 for space group $R\bar{3}m$. The atomic coordinates of $Bi_4(STe_2)$ were taken as Bi at 0,0,0.1447 and 0,0,0.2842, and $S_{0.333}Te_{0.667}$ in 0,0,0 and 0,0,0.4260 for space group $R\bar{3}m$. Josëite with ordered S and Te has 006 strong, whereas josëite-B with ordered S and Te has 003 strong. The calculated intensities in Table 9 are similar to the visually estimated observed intensities of josëite (PDF 12-735) given by Peacock (1941) and josëite-B (PDF 9-435) as consistent with the disordered crystal structure types, $Bi_4(S,Te)_3$ and $Bi_4(Te,S)_3$.

The original X-ray powder-diffraction photographs (57.3 mm diameter Debye-Scherrer) of josëite by Berry and Thompson (1962) were borrowed from the Royal Ontario Museum, and new X-ray powder-diffraction films were taken of specimens ROM M19602a (josëite) and ROM M19602b (josëite-B). As neither 003 nor 006 was observed in any of these photographs or reported in the literature, the S and Te are inferred to be disordered. In addition, a wide solid-solution range between Bi_4Te_3 (pilsenite) and Bi_4S_3 (ikunolite) is suggested by the chemical formulae in Table 2 and by so-called josëite-C ($Bi_{4.1}S_{2.2}Te_{0.7}$) from Godovikov et al. (1970) and josëite from Minster et al. (1968). Therefore, josëite, $Bi_4(S,Te)_3$, is inferred to have a chemical formula similar to that of tellurian ikunolite, whereas josëite-B, $Bi_4(Te,S)_3$, is inferred to have a chemical formula similar to that of sulfurian pilsenite.

The unnamed mineral of Yingchen (1986) gave a chemical analysis with Bi 75.42, Te 19.2, and S 6.65 wt%. The X-ray powder-diffraction data were indexed in space group $R\bar{3}m$ as for ikunolite, and the refined unit-cell dimensions are given in Table 2. As 003 is not observed, a disordered crystal structure is suggested in which the chemical formula is $(Bi_{3.5}Te_{0.5})(S_{2.0}Te_{1.0})$; i.e., this phase is tellurian ikunolite.

Rucklidgeite [$(Bi,Pb)_3Te_4$] was originally described by Zav'yalov and Begizov (1977); however, the Pb does not appear to be essential so that the end-member chemical formula is taken as (Bi_3Te_4). The unit-cell dimensions (PDF 29-234) were refined and are given in Table 2. An X-ray powder-diffraction pattern was calculated using a crystal-structure model similar to that for Bi_4Se_3 of Stasova (1968). The model is for space group $R\bar{3}m$, with Bi at 0,0,0 and 0,0,0.429, and Te at 0,0,0.143 and 0,0,0.286. The intensities of the calculated X-ray powder-diffraction pattern given in Table 9 are similar to the visually observed intensities of PDF 29-234 (Zav'yalov and Begizov, 1977) and PDF 38-458 (Roberts and Harris, 1988).

The unnamed mineral (Bi_3Te_4) of Harañczyk (1978) is rucklidgeite. The unit-cell dimensions were refined assuming space group $R\bar{3}m$ and are given in Table 2.

Wehrlite was reexamined by Ozawa and Shimazaki (1982) and found to be a mixture of pilsenite, Bi_4Te_3, and hessite, Ag_2Te. The discreditation was approved by the IMA CNMMN.

DISCUSSION

The data of Brown and Lewis (1962) show a wide range of solid-solution of Bi and Te from 32 to 60% Te. The data of Godovnikov et al. (1966) for synthetic phases

TABLE 9. X-ray powder-diffraction data of laitakarite, josëite, josëite-B, and rucklidgeite

	Laitakarite			Josëite		Josëite-B		Rucklidgeite	
		I_{calc}							
hkl	I_{obs}	Ordered	Disordered	I_{obs}	I_{calc}	I_{obs}	I_{calc}	I_{obs}	I_{calc}
003		3	8		4		3		1
006	3	3	0		2		1	5	1
009	15	7	8	10	9	10	6	20	2
101		2	1						
012	15	14	15	10	14	10	8	20	3
104	1	1	2						
015, 00.12	10	12	17	5	10	5	4		
107	100	100	100	100	100	100	100	100	100
10.10								5	0.1
01.11	5	1	1	10	1	10	0.1		
10.13								3	0.1
01.14	40	41	41	30	41	40	41	60	41
110	40	34	34	30	34	50	35	60	35
10.16	5	3	4	5	4	10	4	10	1
01.17				5	0.1	5	0.2	5	0.1
119		5	6		6		4	10	1
00.21	10	8	8	10	8	20	8	25	8
10.19, 202	3	5	5	3	2				
11.12, 205	3	7	7	3	3				
027	30	18	18	10	18	30	19	40	18
11.15	1	0	0	5	1				
01.22				3	0.2				
20.14	15	12	12	10	12	20	12	45	12
11.21	15	17	17	10	17	10	17	35	18
02.16	1	1	1						
217	15	15	15	10	15	20	15	30	15
10.28	3	7	7	3	0.1		7	10	7
12.14	8	11	11	10	11	10	11	25	12
300	1	5	5	3	5	10	6		
21.16	5	1	1	10	2				
309	1	1	1	3	1				
11.33	3B	1	1	3	1				
30.21	5	6	7	5	7				

Note: The vvs, vs, s, ms, m, mw, w, vw, and vvw were taken as 100, 40, 30, 15, 10, 8, 5, 3, and 1, respectively.

indicate a chemical formula of $Bi_{1.25}Te_{0.75}$, which extends the solid-solution range far beyond the ideal composition of BiTe. In contrast, the data of Brebrick (1968) for the Bi-Te system show a series of individual phases with 50–60 at.% Te after annealing for several days at 525 °C, near the melting point. The identification of additional phases may be due to the use of more sophisticated X-ray techniques having better resolution.

The suggested disorder of S-Te in the natural series Bi_4S_3-Bi_4Te_3 (ikunolite–josëite-C–josëite–josëite-B–pilsenite) is difficult to explain. Most of the relatively few observed specimens have approximately integer ratios of S:Te, which would favor an ordered structure at low temperatures.

The unnamed mineral of Gamyanin et al. (1980) is Bi_2Te. The X-ray powder-diffraction data were indexed with a hexagonal unit cell with a = 4.476(5) and c = 5.997(12) Å; however, two reflections with d-values of 4.16 and 3.57 Å could not be indexed either with this unit cell or a supercell.

Imamov and Semiletov (1971) described Bi_4Se_5 (a = 4.21, c = 51.54 Å), Sb_4Te_5 (a = 4.27, c = 53.79 Å), and Bi_4Te_5 (a = 4.41, c = 54.9 Å) as having space group $P\bar{3}m1$. Protojosëite was redefined by Zav'yalov and Begizov (1983). Their X-ray powder-diffraction data were indexed using space group $P\bar{3}m1$, and the refined unit-cell dimensions, which are listed in Table 2, imply a structure with a repeat unit of nine closest-packed planes.

Aleksite was originally described by Lipovetskii et al. (1978). Their X-ray powder-diffraction data (PDF 29-765) were indexed using space group $P\bar{3}m1$, and the refined unit-cell dimensions are listed in Table 2. Hedleyite, which was described by Warren and Peacock (1945), was given the chemical formula Bi_7Te_3; however, both Bi_5Te_2 and Bi_8Te_3 are in better agreement with the chemical analyses. The hexagonal subcell has refined dimensions a = 4.475(4) and c = 5.367(8) Å.

Imamov and Semiletov (1971) consider that synthetic equivalents of the tetradymite-group minerals have an odd number of approximately cubic closest-packed planes, including the structures of Bi_2Se, Bi_2Se_3, Bi_4Se_3, Bi_4Se_5, Bi_6Se_5, Bi_8Se_7, and Bi_8Se_9, which have all been synthesized and characterized by Stasova (1968). The well-documented minerals listed in Tables 1 and 2 basically fit the relation involving an odd number of planes; however, the crystal structures of protojosëite (9 atom planes), aleksite (14 atom planes?), or hedleyite (20 atom planes?) have not been established (Table 1).

The published X-ray powder-diffraction data were obtained using a variety of kinds of equipment with different degrees of precision, and with intensities recorded on films with different exposures and estimated visually by different people. Published intensities tend to be very inaccurate, and are subject to preferred orientation in the phases under discussion because of the common {0001} perfect cleavage. Most investigators overestimated the intensity of weak reflections by using a scale of 5 to 100 instead of 2 to 100. The intensities at the lower 2θ angles often reflect the degree of order-disorder, whereas the intensities at the higher 2θ angles often appear to be independent of such effects. In contrast, the majority of well-refined crystal structures of the tetradymite group were obtained with a carefully measured series of 0001 reflections (nine to 36 of them).

The discrepancies between the calculated intensities and the published observed intensities may be accounted for by the poor accuracy of the visually estimated observed reflections, the errors in estimated atomic coordinates, the possibility of partial order rather than either complete order or disorder, or the possibility that further structural complexities exist because of the hierarchy of subcells present in each X-ray powder-diffraction pattern.

Members of the tetradymite group present complex problems, many of which remain unresolved due to incomplete data. To confirm the structure type and the degree of order-disorder in the mineral species within the tetradymite group, a series of crystal-structure determinations must be undertaken either by single-crystal or Rietveld methods.

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