TINTINAITE,[1] THE ANTIMONY ANALOGUE OF KOBELLITE

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ABSTRACT

Re-examination of kobellite, a Pb-Bi-Sb sulphosalt, from the type locality, Hvena, Sweden, and from three new North American localities has led to a redefinition of the mineral and the discovery of its new antimony analogue. Electron probe analyses show that a solid solution series is present and extends from the high bismuth member to the pure antimony end-member. The ideal formula for the kobellite series is $5PbS \cdot 4(X)_2S_8$ where X is Bi and/or Sb.

The structural cell is orthorhombic with space group *Pnnm*. The unit-cell dimensions, as determined by Nuffield (1948) for kobellite with approximately 70% Bi, are $a = 22.62$, $b = 34.08$, $c = 4.02$ Å, while those of the pure antimony member from Tintina, Yukon, are $a = 22.30$, $b = 34.00$, $c = 4.04$ Å. Densities of the minerals in the series increase from 5.47 for the antimony end-member to 6.99 for the bismuth end-member.

It is proposed that the name kobellite be retained for minerals in the series having Bi:Sb = >1:1, and that antimony-rich members (Bi:Sb = <1:1) be assigned the new name tintinaite.

INTRODUCTION

Kobellite was first identified and named by Setterberg in 1839 from the cobalt mine of Hvena, Sweden. In his description, Setterberg proposed a formula of $Pb_2(Bi,Sb)_2S_5$ with Bi:Sb = ~2:1. However, Rammelsberg (1875) rejected Setterberg's analysis and, on the basis of analyses by himself and Genth, he deduced the formula $Pb_3(Bi,Sb)_2S_6$. Keller (1889) later reported a new occurrence of kobellite at the Silver Bell mine, Ouray, Colorado, and from the analysis of this material he confirmed the original formula proposed by Setterberg. On the basis of this early work, the mineral is given in Dana's System of Mineralogy as a doubtful species of unknown crystallography and composition, possibly $Pb_2(Bi,Sb)_2S_5$. The proposed formula for kobellite is thus equivalent to an antimonian cosalite ($Pb_2Bi_2S_5$) to which it is very similar in physical properties.

Beyond this original work, very little had been added to the description of kobellite until Nuffield (1948) showed by single-crystal and *x*-ray powder methods that the Hvena kobellite was not merely an antimonian cosalite but was an independent species. On the basis of the two earlier unsatisfactory analyses by Setterberg and Keller, and with a new specific gravity measurement, Nuffield suggested $6PbS.FeS.2Bi_2S_3.Sb_2S_3$ as a

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tentative formula until the question of cell contents could be settled by a new and reliable analysis.

MATERIALS STUDIED

In addition to the Hvena (Sweden) and Ouray (Colorado) occurrences, kobellite has been reported from five other localities (Table 1), but in none of these has an attempt been made to settle the question of cell contents.

The *x*-ray identification of kobellite from the dump of the Deer Park mine in the Rossland area of British Columbia (R.I.T.) and from the Tintina Silver Mines, Tintina, Yukon (Hunt, 1964) provided an opportunity to make new observations on this mineral. The following additional specimens labelled as kobellite were also examined: U.S.N.M. C-754, Leadville, Colorado; U.S.N.M. R-1025, Old Lout mine, Ouray, Colorado; H.M.M. 82436, Red Mountain, Colorado; National Mineral Collection (Ottawa) specimen 7101, Ibex mine, Leadville, Colorado; U.B.C. specimen from Dodger Tungsten mine, Salmo, British Columbia. *X*-ray powder diffraction and polished section studies of these specimens showed kobellite to be present only in the Salmo material. In addition, however, Paul Desautels of the U.S. National Museum kindly provided us with kobellite from another new locality, Raleigh, North Carolina.

DESCRIPTION OF NEW OCCURRENCES

Rossland, B.C.

Kobellite was identified in a specimen collected from the dump of the Deer Park mine by one of us (R. I. T.) during the course of a mineralogical investigation of the ores from the district. Identification was made by its *x*-ray powder pattern and was confirmed by determination of its optical rotation properties. At this locality kobellite occurs as blades enclosed in granular quartz. The blades in some cases form parallel aggregates which are up to 2 mm long by approximately 0.5 mm across. Less commonly, fine blades form somewhat radiating fibrous clusters. Subhedral to euhedral crystals of pyrite and arsenopyrite are associated with the kobellite. In some specimens the mineral is also closely associated with small amounts of pyrrhotite, chalcopyrite and magnetite.

Native bismuth, intergrown with galena, has been identified from a locality only a short distance northeast of the Deer Park deposit. Other bismuth and antimony-bearing minerals—bismuthinite, galenobismutite, and boulangerite, are found elsewhere in the Rossland camp.

Specimens of the Rossland material are catalogued in the Royal Ontario Museum Collection, Toronto, Ontario as M27173, M27174, M27175, M27176.

TABLE 1. LOCALITIES FOR KOBELLITE

Occurrence	*Reference*	*Comments*
Hvena, Sweden	Setterberg 1841; Nuffield 1948	Associated with pyrrhotite, chalcopyrite
Ouray, Colorado	Keller 1889	All analyses average 7–8% Ag + Cu + Fe + Zn
Boliden deposit, Sweden	Ödman 1941	Both Bi and Sb sulphosalts present. Analysis shows 5% Fe
Dodger Tungsten Mine, Salmo. B.C.	Thompson 1954	Associated with bismuthian robinsonite
Ustarasaisk deposit, Russia	Sakharova 1955	
Jilové area, Bohemia	Morávek 1956	*X*-ray powder data listed; Bi and Sb indicated by spectroscopy
Central Spanish Pyrenees	van der Lingen 1960	With arsenic-bearing minerals and native Bi. Identification tentative

Tintina Silver Mines, Yukon

During the course of a mineralogical investigation of ore specimens from Tintina Silver Mines by Hunt (1964), a mineral identified by *x*-ray powder photographs as kobellite was reported. The mineral occurs as small masses measuring up to 2 mm in diameter and as veinlets filling interstices and fractures in malachite-stained weathered quartz. Like kobellite from Sweden, its colour is lead-grey, it has a black streak and a metallic lustre. The (010) cleavage is well developed. Micro-hardness was determined to be *C*- (Talmage).

The principal associated mineral is jamesonite. Additional minerals identified in the area were argentian tetrahedrite, galena, sphalerite, pyrite, arsenopyrite, marcasite, bournonite, owyheeite, pyrrhotite and chalcopyrite, with the first three being the most abundant. It is interesting to note that none of the sulphosalts are bismuth-bearing.

The specimen of the Tintina "kobellite" kindly donated to the writers for this study by Miss Hunt is catalogued in the systematic collections of the Geological Survey of Canada while other specimens are catalogued in the research collections of the Department of Geology, University of Toronto.

Superior Stone Quarry, Raleigh, North Carolina

The specimen of kobellite (U.S.N.M. 118786) from this locality was identified originally by John S. White Jr., Division of Mineralogy, Smithsonian Institution.

The quarry is located in Wake County and is also known as the Crabtree or Nello Teer Quarry. Although Mr. White has not visited the locality (priv. comm.), he reports that the "kobellite" appears to occur in richly mineralized granite pegmatite veins associated with bismuthinite, jamesonite, and tetrahedrite. Other common minerals in the seams are chalcopyrite, pyrite, siderite, tourmaline, garnet and rutile. Small vuggy areas contain well-formed crystals of monazite, muscovite, rutile and barite.

The small unattached grains provided for this study contained only bismuthinite, tetrahedrite and kobellite. Partial electron probe analysis of the bismuthinite gave approximately 15% antimony. Weissenberg single-crystal photographs of this material gave cell dimensions $a = 11.12$, $b = 11.30$, $c = 3.96$ Å as compared to $a = 11.15$, $b = 11.29$, $c = 3.98$ Å for pure bismuthinite.

ELECTRON PROBE ANALYSIS

Kobellite occurs in such small quantities and intermixed with other physically similar minerals so that the only reliable method of determining its composition is with the electron probe. In this study, four specimens proved suitable for analysis, the results of which are given in Table 2.

The only specimen not analyzed at the Geological Survey of Canada was the one from Hvena, Sweden, which was analyzed by J. C. Rucklidge, Department of Geology, University of Toronto.

The results of the analyses show that a solid-solution series is present, extending from the high bismuth content of the Swedish material to the pure antimony end-member from Tintina. The Rossland and the North Carolina specimens occur in the middle of the series with the former being closer to the antimony member. The compositions of these four minerals clearly show that a continuous series is probably present.

The atomic proportions derived from the analyses (Table 2) show a variation in the Pb:(Bi,Sb) ratio extending from 1:1.24 to a high of 1:1.61. For this range of values, it is possible to deduce only two cell contents which approximately correspond with the unit-cell and specific-gravity measurements, namely $10PbS.7(X)_2S_3$ and $5PbS.4(X)_2S_3$ where X is Bi and/or Sb. For the pure antimony (Tintina) member, the calculated densities for $10PbS.7Sb_2S_3$ and $5PbS.4Sb_2S_3$ are, respectively, 5.111 with $Z = 2$, and 5.475 with $Z = 4$. The predicted density (Jambor, 1967) of $10PbS.7Sb_2S_3$ is about 5.60, whereas the predicted value for

TABLE 2. MICROPROBE ANALYSES AND DENSITIES OF MINERALS IN THE KOBELLITE SERIES

Weight %	Hvena, Sweden	Raleigh, N.C.	Rossland, B.C.	Tintina, Yukon
Pb	33.2	38.0	42.3	42.3
Bi	37.6	28.5	23.0	n.d.
Sb	9.6	15.0	17.6	34.5
Cu	1.0	—	n.d.*	n.d.
Ag	0.5	—	n.d.	n.d.
Fe	0.6	—	n.d.	n.d.
S	18.6	18.0	20.0	21.3
	101.1	99.5	102.9	98.1
Atomic Proportions:				
Pb	5.00	5.00	5.00	5.00
Bi	5.60 } 8.05	3.70 } 7.05	2.70 } 6.20	0.00 } 6.90
Sb	2.45	3.35	3.50	6.90
S	18.10	15.25	15.30	16.30
Atomic % Bismuth in the (Bi, Sb) position	69.6	52.4	43.5	0.00
Calculated Density†	6.51	6.27	6.16	5.48
Measured Density	6.48	6.31	—	5.51 (predicted)

*Not detected.

†Densities calculated from the analytical Bi:Sb ratios substituted in the formula $5PbS.4(X)_2S_3$.

$5PbS.4Sb_2S_3$ is about 5.51. The latter formula is therefore the obvious selection.

X-RAY CRYSTALLOGRAPHY

Rotation and Weissenberg *x*-ray diffraction studies by Nuffield (1948) showed that the Hvena, Sweden, kobellite was a distinct species having orthorhombic symmetry with cell dimensions of $a = 22.62$, $b = 34.08$, $c = 4.02$ Å, space group *Pnmm*. The results obtained in the present study from Weissenberg and precession examination of kobellites from four additional localities are listed in Table 3.

The films confirm the orthorhombic symmetry, but only the Salmo material gave the space group *Pnmm*. Photographs of material from the other three localities showed that the conditions for non-extinction were $0kl$ with $k + l = 2n$, $h0l$ with $h + l = 2n$, giving a space group of *Pnnm* or *Pnn*2. This difference in space group is due only to the systematic extinction of the $h0l$ reflections which, in the Weissenberg films, are very poor. Since the space group *Pnnm* is the most common in this series, the films obtained by Nuffield of the Sweden material kindly were lent to us for a closer study. Comparing these films with those obtained in this study indicated that *Pnnm* is more likely the space group for the mineral from this locality.

TABLE 3. UNIT-CELL DATA FOR THE KOBELLITE SERIES

	Hvena, Sweden (Nuffield)	Salmo, B.C. (This study)	Raleigh, N.C. (This study)	Rossland, B.C. (This study)	Tintina, Yukon (This study)
a	22.62 Å	22.56 Å	22.53 Å	22.50 Å	22.30 Å
b	34.08	34.00	33.97	34.00	34.00
c	4.02	4.04	4.04	4.03	4.04
Volume	3099.0 Å^3	3098.8 Å^3	3092.0 Å^3	3082.9 Å^3	3063.1 Å^3
Space Group	*Pnnm* or *Pnmm*	*Pnmm*	*Pnnm*	*Pnnm*	*Pnnm*
Z	4	4	4	4	4
Cleavage	010	010	010	010	010

The other important feature of the single-crystal study is the variation in unit-cell dimension *a* from the pure antimony end-member to the high bismuth member. This relationship is illustrated in Figure 1 which shows *a* versus the atomic percent bismuth in the (Bi,Sb) position.

As can be seen, the graph shows a linear relationship up to 75% Bi which could probably be extended to the pure end-member. The variation in the *a* cell dimension with change in composition may be distinct enough so that the *a* value can be used as a measure of the amount of Bi in the

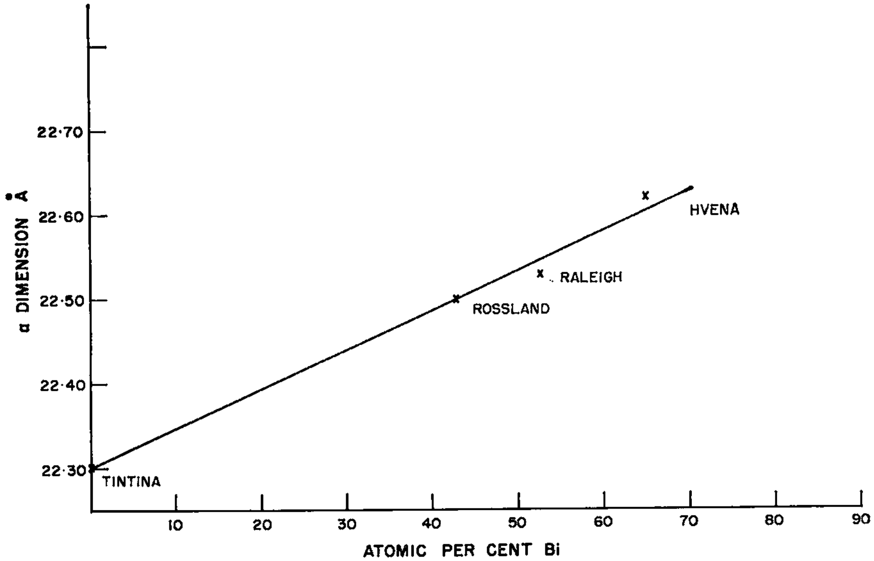

FIG. 1. Shows the variation in unit-cell dimensions *a* versus atomic percent bismuth in the (Bi,Sb) position.

(Bi,Sb) position. This correlation with the single crystal data may be fortuitous, but is amazingly good considering that the unit-cell values were not all determined in one laboratory. In an attempt to obtain more precise unit-cell dimensions, the powder data were run through a refinement programme giving values for the Tintina material of $a = 22.33$, $b = 34.07$, $c = 4.04$ Å and the Hvena material, $a = 22.56$, $b = 33.94$, $c = 4.02$ Å. In the refinement programme, it is possible to derive quite different unit-cell dimensions depending on which indices are included. For this reason, the *x*-ray powder data are not considered to be sufficiently precise to warrant further refinement. The refined unit-cell values were therefore only used for indexing the Tintina and Hvena powder patterns.

Although microprobe analyses of the Salmo mineral could not be obtained, the above graphical relationship indicates that its Bi content would be about 55–60 atomic percent, close enough to suspect the possible presence of an ordered compound with Bi:Sb = 1:1. This, however, is at variance with the reduction in space-group symmetry from *Pnnm* to *Pnmm*. As only one single-crystal fragment of poor quality was examined, further study is clearly needed before any meaningful conclusion can be reached.

X-RAY POWDER DIFFRACTION DATA

X-ray powder data for the minerals examined in this study are given in Table 4. All measurements were made on films obtained with 114.6 mm diameter cameras and nickel-filtered copper radiation. The only reasonably distinctive pattern in the series is that of the pure antimony end-member from Tintina. A characteristic feature is the appearance of a medium strong reflection at 2.022(6) which appears as a doublet (2.039 and 2.019) on the other patterns.

DENSITY

In the original description of kobellite from Sweden, Setterberg (1841) determined the density to be 6.29–6.33. Nuffield (1948) was able to measure with a Berman balance several clean fragments from this locality and obtained a value of 6.48 ± 0.05. In the present study, only the North Carolina specimen proved suitable for density measurements. Two fragments (total weight 5.5 mg) taken from a polished section were measured on a Berman balance by H. R. Steacy, of the Geological Survey of Canada, and a value of 6.31 was obtained.

As the microprobe analyses of each element (Table 2) are subject to error, only the analytical Bi:Sb ratios have been substituted in the

TABLE 4. KOBELLITE SERIES: X-RAY DIFFRACTION DATA
Camera Diameter 114.6 mm. Nickel-Filtered Copper Radiation

Tintina				Hvena				Rossland		Raleigh	
I(est)	*d*(obs)Å	*hkl*	*d*(calc)Å*	*I*(est)	*d*(obs)Å	*hkl*	*d*(calc)Å*	*I*(est)	*d*(obs)Å	*I*(est)	*d*(obs)Å
<½	10.63	210	10.61							<½	11.20
<½	10.08	130	10.12					1	10.19	<½	10.13
½	9.33	220	9.34								
<½	8.57	040	8.52					½	8.51		
<½	8.06	230	7.96	½	8.03	230	7.99	½	8.04	<½	8.05
<½	7.24	310	7.27								
½	6.83	320	6.82	½	6.95	320	6.88	½	6.83		
<½	6.53	150	6.52								
<½	6.22	330	6.23	½	6.26	330	6.26	½	6.27	<½	6.21
<½	5.70	060	5.68	1	5.70	060	5.66	1	5.70	½	5.74
				½	5.37	420	5.35	½	5.38	<½	5.33
				½	5.07	260	5.06				
				½	4.76	170	4.74	½	4.68	<½	4.70
½	4.50	360	4.51	½	4.54	360	4.52	½	4.50	½	4.52
1	4.32	450	4.32	½	4.34	450	4.34	½	4.35	½	4.36
1	4.26	080	4.26	½	4.27	080	4.24	1	4.25	½	4.26
½	4.14	530	4.16								
4	3.96	460	3.98	4	3.98	460	3.99	3	3.99	4	3.98
		540	3.96			540	3.98				
<½	3.87	121	3.87								
½	3.76	131	3.75	½	3.75	131	3.74	1	3.75	½	3.75
3	3.72	600	3.72								
		221	3.71								
2	3.60	231	3.60	1	3.57	630	3.58	1	3.59	½	3.59
8	3.51	560	3.51	10	3.54	560	3.53	9	3.53	10	3.54
10	3.40	640	3.41	1	3.44	640	3.44				
		0.10.0	3.41								
		331	3.39	9	3.41	0.10.0	3.39	10	3.40	9	3.41

TABLE 4—*Concluded*

Tintina				Hvena				Rossland		Raleigh	
I(est)	*d*(obs)Å	*hkl*	*d*(calc)Å*	*I*(est)	*d*(obs)Å	*hkl*	*d*(calc)Å*	*I*(est)	*d*(obs)Å	*I*(est)	*d*(obs)Å
4	3.27	341	3.28	4	3.27	341	3.27	4	3.28	3	3.29
3	3.11	660	3.11	1	3.12	660	3.13	2	3.14	1	3.14
		071	3.11								
1	3.06	1.11.0	3.07					½	3.08	½	3.07
2	3.00	361	3.01					2	3.00	1	3.00
½	2.96	670	2.96	1	2.98	670	2.97				
3	2.89	531	2.90	½	2.90	531	2.90	3	2.90	1	2.90
5	2.87	371	2.87	2	2.85	371	2.86	3	2.85	2	2.87
4	2.83	461	2.84							2	2.84
7	2.71	381	2.73	5	2.72	381	2.72	6	2.72	5	2.72
		471	2.72			471	2.71				
<½	2.65	840	2.65								
2	2.60	481	2.59	½	2.60	641	2.61	1	2.61	½	2.61
						481	2.59				
				½	2.57	780	2.57			<½	2.57
<½	2.526	2.10.1	2.536	1	2.523	2.10.1	2.527	½	2.523	½	2.528
<½	2.481	721	2.477								
1	2.341	591	2.349	½	2.353	591	2.348	½	2.348	1	2.325
		1.11.1	2.334								
1	2.294	811	2.291	1	2.305	811	2.303	1	2.305	1	2.277
½	2.231	10.0.0	2.233	½	2.253	10.0.0	2.256	½	2.252	½	2.255
				½	2.185	1.13.1	2.179			<½	2.189
1	2.156	2.13.1	2.157	2	2.150	2.13.1	2.149	3	2.156	1	2.151
3	2.114	901	2.114	2	2.129	901	2.127	3	2.129	2	2.126
<½	2.081			½	2.092			½	2.087	½	2.094
				1	2.039			2	2.039	1	2.038
6	2.022			1	2.011			2	2.019	2	2.015
½	1.976			½	1.976			½	1.981	½	1.981

*Calculated from refined cell dimensions (p. 377).

formula $5PbS.4(X)_2S_3$ for the purpose of calculating the densities of the minerals. For kobellite from Hvena, Sweden, the calculated density is 6.51, in excellent agreement with the measured value of 6.48. Likewise, the measured and calculated densities of the North Carolina kobellite (6.31 and 6.27 respectively) agree well.

The effect of bismuth content on density throughout the Bi–Sb series is graphically shown in Fig. 2. The predicted density of the end-member compound $5PbS.4Bi_2S_3$ is 6.99.

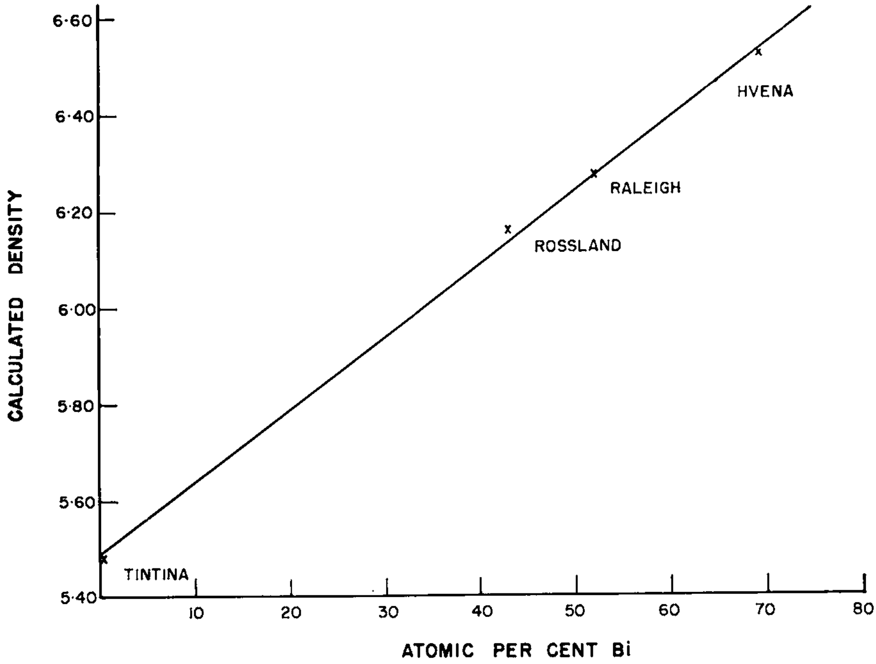

FIG. 2. Shows the relationship between calculated density and atomic percent bismuth in the (Bi,Sb) position.

ROTATION PROPERTIES AND REFLECTIVITIES

The only specimen on which rotation and reflectivity measurements were made was the Rossland material. The rotation angles and phase differences were measured at a series of wave-lengths using established techniques (Cameron, 1961), and the results were in essential agreement with the recorded values for selenium-bearing kobellite from Sweden (van Rensburg & Cameron 1965). At a wave-length of 546 mμ the rotation angle, Ar, and phase difference, 2θ, were determined to be 1.84° and 0.8°+, respectively.

The approximate uniradial reflectivities at a wave-length of 546 mμ are as follows:

Rp	36.6
Rm	39.0 to 41.1
Rg	45.4

They were measured using silicon and tungsten as standards, with reflectivities at this wave-length of 37.0% (NPL Report, Reference N2538, August 22, 1966) and 53.3%, respectively. Only seven grains suitable for measurements were present in the polished section prepared and the value of *Rm* could only be determined to lie between 39.0% and 41.1%. However, the mineral is almost certainly optically positive (Cameron, 1963).

Calculation of the reflectivity ratio R_2/R_1 from the measured rotation properties (Berek 1953) gives a value of 0.879. This gives a calculated value of 39.9% for *Rp*, which is much too great. There is thus a conflict between the measured uniradial reflectivities and rotation properties. Some errors in reflectivity are to be anticipated due to the great number of factors which influence the measurements. However, even if maximum and minimum values are rejected as erroneous (giving values of 37.5% and 44.4% for *Rp* and *Rg*, respectively) the ratio of *Rp* and *Rg* is only increased to 0.845, which indicates that the measured rotation angle should be as great as 2.42°. It seems probable that the rotation properties have not as yet been determined on a grain with the proper orientation to give maximum values.

NOMENCLATURE

The minerals described here are apparently members of a continuous solid-solution series with the general formula $5PbS.\ 4(X)_2S_3$ where X is Bi and/or Sb. The series may be arbitrarily divided at the Bi:Sb = 1:1, with minerals in the bismuth-rich portion being assigned the name kobellite. Minerals in the antimony-rich portion are assigned the new name tintinaite, the name being derived from the locality for the antimony end-member. It is also suggested that the solid-solution series be referred to as the kobellite series.

The formula proposed in this study for tintinaite, namely $5PbS.4Sb_2S_3$, is identical to another rare sulphosalt mineral, plagionite. However, the two minerals are structurally different. Plagionite has cell dimensions $a = 13.45$, $b = 11.81$, $c = 19.94$ Å, $\beta = 107°11$, space group $C2/c$ as compared to tintinaite of dimensions $a = 22.30$, $b = 34.00$, $c = 4.04$ Å, space group *Pnnm*. In this respect, the minerals are to be considered as distinct species.

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