

## NEW DATA ON GIESSENITE FROM THE BJØRKÅSEN SULFIDE DEPOSIT AT OTOFTEN, NORTHERN NORWAY

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### ABSTRACT

New microprobe and crystallographic data are presented for giesSENite from Bjørkåsen, Otoften, Norway, which was originally described by Karup-Møller (1973). The chemical formula is  $\text{Cu}_{1.7}\text{Ag}_{0.1}\text{Fe}_{0.2}\text{Pb}_{26.4}\text{Bi}_{16.7}\text{Sb}_{2.9}\text{S}_{57}$ , or  $(\text{Cu,Fe})_2\text{Pb}_{26.4}(\text{Bi,Sb})_{19.6}\text{S}_{57}$  ( $Z = 2$ ). The symmetry is monoclinic, space group  $P2_1/n$ , with cell parameters  $a$  34.34(1),  $b$  38.05(1),  $c$  4.06(1) Å,  $\beta$  90.33(5)°. Deviations of symmetry from orthorhombic  $Pnmm$  are definite but not conspicuous. The same findings on symmetry apply to giesSENite from the type locality, Binnatal, Switzerland. GiesSENite represents the fourth member of the kobellite homologous series; it is a monoclinic, Bi-rich analogue of the orthorhombic, Sb-rich izoklakeite.

**Keywords:** giesSENite, chemical and crystallographic data, Bjørkåsen, Norway, izoklakeite, kobellite homologous series.

### SOMMAIRE

De nouvelles données, chimiques (microsonde) et cristallographiques, sont présentées sur la giesSENite de Bjørkåsen, Otoften, Norvège, originellement décrite par Karup-Møller (1973). L'analyse donne  $\text{Cu}_{1.7}\text{Ag}_{0.1}\text{Fe}_{0.2}\text{Pb}_{26.4}\text{Bi}_{16.7}\text{Sb}_{2.9}\text{S}_{57}$ , d'où la formule  $(\text{Cu,Fe})_2\text{Pb}_{26.4}(\text{Bi,Sb})_{19.6}\text{S}_{57}$  ( $Z = 2$ ). La structure est monoclinique, de groupe spatial  $P2_1/n$ , et les paramètres de la maille sont  $a$  34.34(1),  $b$  38.05(1),  $c$  4.06(1) Å,  $\beta$  90.33(5)°. La déviation de la symétrie  $Pnmm$ , quoique visible, n'est pas évidente. Les mêmes observations sur la symétrie sont valides pour la giesSENite du gisement type (Binnatal, Suisse). La giesSENite représente le quatrième membre de la série homologue de la kobellite; elle est l'analogue, monoclinique et riche en Bi, de l'izoklakeite, orthorhombique et riche en Sb.

(Traduit par la Rédaction)

**Mots-clés:** giesSENite, données chimiques et cristallographiques, Bjørkåsen, Norvège, izoklakeite, série homologue de la kobellite.

### INTRODUCTION

GiesSENite was described by Graeser (1963) as an orthorhombic mineral from Binnatal in Switzerland,

with the composition  $\text{CuPb}_9\text{Bi}_6\text{Sb}_{1.5}\text{S}_{30}$ . In 1973, Karup-Møller described a new occurrence of giesSENite from the Bjørkåsen sulfide deposit at Otoften, northern Norway. He also determined the symmetry to be orthorhombic and suggested the empirical formula  $\text{Cu}_{0.5}\text{Pb}_9\text{Bi}_6\text{Sb}_{1.5}\text{S}_{20}$ .

In 1981, Makovicky proposed a potential kobellite homologous series and found giesSENite to be the likely candidate for a higher homologue of kobellite. Re-examination of the Norwegian material preserved at the Geological Museum at Copenhagen using Weissenberg photographs indicated that the true symmetry of giesSENite is monoclinic (Makovicky 1981) and that it indeed represents a higher homologue of kobellite. The crystals examined are intimately twinned, unfit for crystal-structure determination. Thus the matter was put to rest until the discovery of orthorhombic izoklakeite (Harris *et al.* 1986, Zakrzewski & Makovicky 1986), which enabled us to carry out the structure determination (Makovicky & Mumme 1985) as well as to verify the proposed structure and composition of the kobellite homologues with  $N = 4$  (Zakrzewski & Makovicky 1986). A re-examination of tintinaite and of some kobellite specimens (Moëlo *et al.* 1984), as well as the description of orthorhombic "Sb-giesSENite" (in fact, the most Bi-rich izoklakeite known to date) by Armbruster *et al.* (1984), contributed further data to the detailed study of the kobellite homologous series. The chemical and structural implications of the entire study are to be found in Makovicky & Mumme (1985) and Zakrzewski & Makovicky (1986). The purpose of this contribution is to present new chemical and crystallographic data on giesSENite from Bjørkåsen and to interpret these in the light of the above studies.

### CHEMICAL COMPOSITION

GiesSENite from Bjørkåsen has been analyzed using a JEOL Superprobe 733 apparatus at the Institute of Mineralogy, University of Copenhagen. The ZAF corrections were performed using the JEOL PACM on-line set of programs. The following analytical wavelengths were used:  $\text{AgL}\alpha$ ,  $\text{CuK}\alpha$ ,  $\text{BiL}\alpha$ ,

TABLE 1. MICROPROBE DATA FOR GIESSENITE FROM BJØRKÅSEN

Analysis	Meas. points	Ag*	Cu*	Fe*	Pb*	Bi*	Sb*	S*	Total	Ag <sup>+</sup>	Cu <sup>+</sup>	Fe <sup>+</sup>	Pb <sup>+</sup>	Bi <sup>+</sup>	Sb <sup>+</sup>	S <sup>+</sup>
1	8	0.05(11)	0.89(4)	-	49.1(4)	30.8(7)	3.3(2)	15.9(4)	100.0(12)	0.1(2)	3.3(2)	-	55.7(3)	34.6(6)	6.3(4)	116.4(22)
2	8	0.14(8)	0.88(2)	0.09(2)	48.9(5)	31.2(5)	3.1(3)	16.4(1)	100.8(8)	0.3(2)	3.2(1)	0.4(1)	55.2(6)	34.9(5)	6.0(6)	119.8(6)
3	3	-	0.7(1)	-	48.3(10)	31.7(5)	3.5(2)	[16.0]	[100.2]	-	2.7(3)	-	54.9(11)	35.7(6)	6.8(2)	[117.4]

Standard deviations are indicated in parentheses in terms of the last digit. \* Data expressed in weight%.

<sup>+</sup> Data expressed in atomic proportions,  $\Sigma Me = 100$ . Analysis 1: mixed metal and sulfide standards;

analysis 2: sulfide standards; analysis 3: results of Karup-Møller (1973), mixed metal and sulfide standards;

S not analyzed, assumed to be 16 wt.%. Ranges for analyses 1 & 2: Bi 29.8 - 32.0 wt.%, Sb 2.4 - 3.4 wt.%.

PbL $\alpha$ , FeK $\alpha$  and SK $\alpha$ . For the composition designated (1) in Table 1, pure metals and galena (for Pb and S) were used, whereas for composition (2) in the same table, the following sulfide standards were used: Cu<sub>3</sub>SbS<sub>4</sub>, FeS<sub>2</sub>, Ag<sub>2</sub>S, PbS and Bi<sub>2</sub>S<sub>3</sub>. Fe was not determined in the first case.

In the first instance, the analytical results were used to check the compatibility of the phase studied with the general crystal-chemical formula of the kobellite homologous series,  $T_2M_{10N+6}S_{11N+13}$  ( $T$  represents tetrahedrally co-ordinated cations,  $M$  are cations with square pyramidal, octahedral and capped-prismatic co-ordination, and  $N$  is the order number of the homologue studied) as established by Makovicky & Mumme (1985). Furthermore, they were used to establish the variable valencies and roles of the elements involved, using the formulae derived by Zakrzewski & Makovicky (1986).

Preliminary calculations based on the assumption that the order number  $N$  of giessenite in the kobellite homologous series is equal to four indicate that the minor contents of Ag in both compositions can be assigned to the tetrahedral positions. With such low contents of Ag, its re-assignment to the  $M$  sites would hardly influence the results of the calculations.

For the model calculations of the order number  $N$ , formula (14) of Zakrzewski & Makovicky (1986) was used

$$N_{\text{calc}} = x \frac{6M^{2+} + 3M^{3+}}{4M^{3+} - M^{2+}} + (1-x) \frac{5M^{2+} + 2M^{3+}}{4M^{3+} - M^{2+}}$$

where  $x = T^+ / (T^+ + T^{2+})$  and  $(1-x) = T^{2+} / (T^+ + T^{2+})$ .

$T$  and  $M$ , respectively, denote cations in the tetrahedral and octahedral or prismatic sites. This formula assumes fully occupied tetrahedral positions, the situation judged to be most probable for natural occurrences by Zakrzewski & Makovicky (1986).

With the valency of Cu not known,  $x$  was tentatively put equal to 1, 0.5 and 0; for composition (2) the model  $\Sigma T = 0.905$  ( $\text{Cu}^+ + \text{Ag}^+$ ) +  $0.095 \text{ Fe}^{2+}$

was also used. For composition (1) based on combined standards,  $N_{\text{calc}}$  is equal to 4.23 for only  $T^+$  present, 3.78 for ( $\frac{1}{2}T^{++} + \frac{1}{2}T^{2+}$ ) present and 3.34 for only  $T^{2+}$  assumed. For composition (2), considered superior to (1) and based on sulfide standards and with Fe included, the relevant values for  $N_{\text{calc}}$  are 4.19, 3.75 and 3.30. For this composition, when all Cu and Ag are assumed univalent and Fe divalent, the  $N_{\text{calc}}$  value is equal to 4.11.

Thus, giessenite clearly represents the kobellite homologue with the order number  $N$  equal to 4. If we recast its atomic ratios to 48 metal atoms in a formula unit ( $Z = 2$ ), the following empirical formulae result:  $\text{Ag}_{0.07}\text{Cu}_{1.58}\text{Fe}_{0.0}\text{Pb}_{26.72}\text{Bi}_{16.61}\text{Sb}_{3.02}\text{S}_{55.88}$  for composition (1) (combined standards), and  $\text{Ag}_{0.14}\text{Cu}_{1.56}\text{Fe}_{0.18}\text{Pb}_{26.50}\text{Bi}_{16.73}\text{Sb}_{2.90}\text{S}_{57.49}$  for composition (2) (sulfide standards). In the first case  $\Sigma T = 1.65$ , in the second case  $\Sigma T = 1.88$ . We assign the difference in  $\Sigma T$  from the ideal value of 2 to experimental and correction errors.

Using the molar ratios of large cations (and assuming  $\Sigma T = 2$ ), the theoretical value of  $x = T^+ / (T^+ + T^{2+})$  can be calculated if  $N$  is postulated to be equal to 4. Formula (15) of Zakrzewski & Makovicky (1986) is to be used:

$$x_N = \frac{(4N-2) M^{3+} - (N+5) M^{2+}}{M^{2+} + M^{3+}}$$

For composition (1), 74.4%  $T^+$  and 25.6%  $T^{2+}$  are obtained, whereas for (2), 78.6%  $T^+$  and 21.4%  $T^{2+}$  result. If we realize that Fe occupies ideally 9.5% of the tetrahedral sites, a further ~12% of these sites are supposedly occupied by divalent Cu. Considering standard errors on different elements and the presumed absolute error in the  $\Sigma T$  value, we cannot overemphasize these results; they may well lie within error bounds of the measurements. However, it can be safely stated that the giessenite from Bjørkåsen contains primarily univalent copper. Our results can be compared with those discussed in the companion paper by Zakrzewski & Makovicky (1986).

The new analyses yield a Sb/(Sb + Bi) ratio equal to 0.154 (1) and 0.148 (2), thus confirming that the giessenite from Bjørkåsen represents the most B-rich kobellite homologue with  $N = 4$  that is known at present.

#### X-RAY CRYSTALLOGRAPHY

Two crystals were extracted from polished sections for the study on a Weissenberg camera. Both gave identical results. Well-exposed photographs reveal that giessenite is monoclinic but intimately twinned on (100). The systematic extinctions,  $0k0$  present only for  $k = 2n$  and  $h0l$  present only for  $h + l = 2n$ , indicate space group  $P2_1/n$ . However, another class of reflections is found that obeys  $0kl$  generally present for  $k + l = 2n$  and generally absent or very weak for  $k + l = 2n + 1$ . In the latter category the following reflections are observed: 081, 0.10.1, 0.26.1, 0.28.1, 092 and 0.27.2; these are all absent in orthorhombic izoklakeite. Moreover, the lattice is metrically pseudo-orthorhombic, with the deviation of  $\beta$  from  $90^\circ$  so small that it is only revealed as a split of  $\pm h.0.l$  (and adjacent) reflections of the twinned crystal at high  $\theta$  values. In both cases examined, the volumes of the two twin orientations with common  $a^*$  and  $b^*$  axes, with separate  $c^*$  directions, are unequal. The inequality of the pairs  $I_{hkl}$  and  $I_{\bar{h}\bar{k}l}$  ( $k \neq 0$ ) due to monoclinic symmetry is easy to ascertain, but it relates to the details of the weighted reciprocal lattice and not to the overall distribution

of the areas of the strong and weak intensities. The latter retain the character and distribution observed for orthorhombic izoklakeite.

Summing up these observations, the observable differences from orthorhombic symmetry, space group  $Pnmm$ , are unambiguous but not very pronounced, and a cursory evaluation of somewhat underexposed single-crystal X-ray photographs will almost certainly lead to an erroneous conclusion about symmetry. On the other hand, the relatively weak deviations of the weighted reciprocal lattice from the  $Pnmm$  symmetry and its overall closeness to that of izoklakeite indicate that the distortions from the structural scheme of izoklakeite in the crystal structure of giessenite are only minor. Intimate twinning of giessenite, in one case with the related reflections of the two twin orientations interconnected by diffuse streaks (evidence of intimate twinning down to unit-cell level), suggests that giessenite might have been orthorhombic ( $Pnmm$ ) at the temperature of formation; it has possibly undergone a displacive phase-transformation, involving perhaps increase in cation ordering, to  $P2_1/n$  on cooling. The weak 8-Å superstructure observed in izoklakeite, and indicative of partial ordering along the  $c$  direction, is absent in giessenite.

The cell constants estimated from Weissenberg and rotation photographs are  $a$  34.36(2),  $b$  38.02(2),  $c$  4.07(1) Å and  $\beta$  90.33(3) $^\circ$ . The Guinier powder film of giessenite from Bjørkåsen used by Karup-Møller (1973) was remeasured taking into account possible

TABLE 2. X-RAY POWDER-DIFFRACTION DATA FOR GIESSENITE FROM BJØRKÅSEN

$\bar{l}$	est	$\bar{d}$	obs	$\bar{d}$	calc	$hk \bar{l} $	$\bar{l}$	est	$\bar{d}$	obs	$\bar{d}$	calc	$hk \bar{l} $	$\bar{l}$	est	$\bar{d}$	obs
2	4.292	4.292	800	1	3.119	3.118	2.12.0	1	2.503	2.507	881	1D	2.021				
2BD	3.942	3.941	670	3	3.048	3.042	471			2.498	6.10.1	1D	1.9976				
4	3.910	3.910	580	3	3.036	3.037	651	1	2.474	2.473	2.12.1	1D	1.9888				
2BD	3.824	3.827	301	2	3.007	3.007	7.10.0	2	2.330	2.330	10.12.0	1D	1.9686				
4	3.790	3.793	490	1	2.945	2.941	811	2	2.321	2.320	7.11.1	1D	1.9573				
4	3.650	2.649	241	1	2.933	2.936	661	2	2.255	2.254	1.14.1	2	1.9145				
4	3.610	3.611	3.10.0	2	2.922	2.918	191	1BD	2.207	2.210	13.1.1	1D	1.8923				
3	3.566	3.563	151	4	2.904	2.903	10.7.0			2.205	12.11.0	1D	1.8742				
2	3.551	3.550	341	4	2.888	2.888	11.5.0	2	2.171	2.170	13.10.0	1D	1.8491				
1	3.489	3.495	501	1	2.864	2.861	12.0.0	2	2.166	2.166	4.17.0	2D	1.8246				
3	3.477	3.478	4.10.0	4	2.838	2.837	3.9.1	6	2.152	2.151	0.15.1	1	1.8079				
7	3.433	3.434	10.0.0	1	2.829	2.830	12.2.0	2	2.134	2.135	2.15.1	1	1.8013				
3	3.416	3.415	780	2	2.815	2.817	581			2.133	16.2.0	2BD	1.7870				
6	3.400	3.301	690	2	2.804	2.805	761	2	2.125	2.124	12.12.0	2BD	1.7710				
1	3.332	3.328	5.10.0	4	2.742	2.741	2.10.1	2	2.112	2.112	9.15.0	4	1.7545				
2	3.314	3.314	10.3.0	1	2.693	2.693	5.13.0	2D	2.100	2.101	6.14.1	3BD	1.7440				
2	3.306	3.306	451	1BD	2.638	2.642	4.10.1			2.099	13.11.0	2	1.7238				
1	3.282	3.281	541			2.635	13.1.0			2.099	12.8.1	2	1.6991				
1	3.204	3.204	631	1D	2.623	2.626	1.11.1	2D	2.061	2.062	14.10.0	1BD	1.6857				
3	3.169	3.169	6.10.0	1D	2.607	2.607	691	2	2.048	2.049	1.16.1						
1	3.142	(3.130)	10.5.0)					5	2.032	2.033	16.6.0						

Guinier-Hägg camera,  $\text{CuK}\alpha$  radiation, quartz internal standard.

$\bar{d}$  calc represent approximate (orthorhombic) averages of  $\bar{d}(hkl)$  and  $\bar{d}(h\bar{k}l)$ ,  $\Delta\bar{d}$  being 0.01-0.02 Å for  $\bar{d}$  values  $\approx 3$  Å. The  $\bar{d}$  values in the last column allow multiple indexing. BD = broad line; D = double line.

splitting of the diffraction lines due to monoclinic symmetry (Table 2). Because of the large  $a$  and  $b$  cell dimensions, a conservative approach was adopted for the refinement. The strongest 24 reflections that could be unambiguously indexed from Weissenberg photographs were used, and their Miller indices remained fixed during the refinement. It was found that the splitting of  $hkl$  and  $hk\bar{l}$  reflections is too small to allow independent measurements on the two parts of the doublets (Table 2); an orthorhombic model had to be adopted, with  $\beta = 90^\circ$ . The refinement results for this model are  $a$  34.337(5),  $b$  38.050(10),  $c$  4.061(5) Å. These values confirm in principle those obtained from the single-crystal photographs. They differ significantly from the data by Karup-Møller (1973) only in the  $b$  value, for which he obtained the value of 37.37 Å. This difference stems from the ambiguity of refinement peculiar to both giessenite and izoklakeite (Zakrzewski & Makovicky 1986): several pairs of strong reflections,  $h_1k_10$  and  $h_2k_21$ , with close  $d$  values, occur in the powder patterns. It is not obvious in which order the Miller indices are to be assigned to the members of these pairs. The two alternatives yield two answers with the difference in the resulting  $b$  values of the order of  $\sim 0.5$  Å. In both cases the true assignment could be found only by comparing the results with the single-crystal data.

The above results correct the original space-group of giessenite, believed to be  $P2_12_12_1$  by Graeser (1963) and Karup-Møller (1973). To find out whether our material is the same as that previously examined by Graeser, we studied the Weissenberg photographs of one of the giessenite crystals from Binnatal, lent to us kindly by Dr. Graeser. This study revealed that the symmetry of original giessenite from Binnatal and its weighted reciprocal lattice are identical to that of giessenite from Bjørkåsen. On the other hand, Dr. Armbruster has checked his film and diffractometer data for the single crystal of "Sb-giessenite" from Zevreila, with the empirical composition  $\text{Cu}_{2.6}\text{Fe}_{1.3}\text{Ag}_{0.9}\text{Pb}_{53.8}\text{Sb}_{14.8}\text{Bi}_{22.7}\text{S}_{117.3}$  ( $Z = 1$ ) (Armbruster *et al.* 1984), and confirms the orthorhombic symmetry of his specimen, although he notes the small size of the crystal used. Thus, this phase apparently belongs to the orthorhombic part of the Bi-Sb solid solution with  $N = 4$ , *i.e.*, it is Bi-rich izoklakeite.

#### CONCLUSIONS

1. The chemical formula of giessenite from Bjørkåsen, Otoften, Norway, (for  $Z = 2$ ) can be simplified as  $\text{Cu}_{1.7}\text{Ag}_{0.1}\text{Fe}_{0.2}\text{Pb}_{26.4}\text{Bi}_{16.7}\text{Sb}_{2.9}\text{S}_{57}$ , and perhaps further simplified as  $(\text{Cu}, \text{Fe})_2\text{Pb}_{26.4}(\text{Bi}, \text{Sb})_{19.6}\text{S}_{57}$ . It is questionable whether striving for integer proportions in the formula is justified because of the extensive  $T^+ \rightleftharpoons T^{2+}$ ,  $\text{Pb} \rightleftharpoons \text{Bi}$  and  $\text{Bi} \rightleftharpoons \text{Sb}$

substitutions that were found in the crystal structure of the closely related species *izoklakeite* (Makovicky & Mumme 1985).

2. Giessenite from Bjørkåsen is monoclinic, space group  $P2_1/n$ , with the cell parameters  $a$  34.34(1),  $b$  38.05(1),  $c$  4.06(1) Å and  $\beta$  90.33(5)°. It is intimately twinned on (100). Deviations from  $Pnmm$  in systematic extinctions and in the symmetry of the weighted reciprocal lattice are definite but not conspicuous. Giessenite from Binnatal displays the same X-ray crystallography.

3. The chemical and the crystallographic data unambiguously prove that giessenite is chemically and crystallographically very close to *izoklakeite* (Harris *et al.* 1986, Zakrzewski & Makovicky 1986, Makovicky & Mumme 1985), both being members with  $N = 4$  of the kobellite homologous series. Giessenite represents the Bi-rich end of the series, with monoclinic symmetry, whereas *izoklakeite* represents the more Sb-rich member that displays orthorhombic symmetry. The structural difference between the two phases ought to consist only of small distortions and the concurrent ordering of cations, with the structural scheme essentially identical.

4. The revised chemical and crystallographic data on the material from Bjørkåsen are sufficiently close to the results of Karup-Møller (1973) to ensure that we are dealing with the same mineral.

#### ACKNOWLEDGEMENTS

The X-ray study of giessenite was carried out while E.M. was a Visiting Fellow at the Research School of Chemistry, The Australian National University, Canberra, A.C.T. The keen interest of Prof. B.G. Hyde (Canberra) and the useful discussions with Dr. W.G. Mumme (Melbourne) are gratefully acknowledged. Co-operation of Prof. S. Graeser (Basel) and the keen interest of Drs. M. Zakrzewski (Amsterdam) and D.C. Harris (Ottawa) are appreciated. The refinement of powder data was carried out with the kind assistance of lektor E. Leonardsen (Copenhagen).

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Received July 20, 1985, revised manuscript accepted October 30, 1985.