

## Occurrence of sulphides on the bornite-idaite join from Vielsalm, Stavelot Massif, Belgium

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**Abstract:** Sulphides with chemical compositions between idaite and bornite have been identified from several quartz veins crosscutting Ordovician schists in Vielsalm, Belgium. Idaite is an oxidation product of bornite, and occurs in the more oxidised parts of the quartz veins. The alteration sequence bornite → “anomalous bornite” → idaite is observed, associated with the growth of chalcopyrite lamellae parallel to the {100} and {111} directions of precursor bornite. Electron microprobe analyses indicate a chemical composition significantly enriched in Cu, when compared with the ideal composition of idaite,  $\text{Cu}_3\text{FeS}_4$ , as well as a progressive compositional evolution from bornite to idaite. The X-ray powder pattern is indexed in the space group  $I\bar{4}2m$ , similar to that of stannite, and gives the unit-cell parameters  $a = 5.279(4)$  and  $c = 10.47(2)$  Å.

**Key-words:** idaite,  $\text{Cu}_3\text{FeS}_4$ , stannite structure, Vielsalm, Stavelot Massif, Belgium.

### Introduction

First described by Frenzel (1958 and 1959) as a supergene sulphide from the Ida mine, Southwest Africa, idaite has been the subject of numerous controversies about its chemical composition and crystallographic properties. Frenzel (1958 and 1959) proposed the formula  $\text{Cu}_5\text{FeS}_6$  for idaite, because the X-ray powder pattern of a sample from Tarvis Township, Ontario, Canada, was similar to that given by Merwin & Lombard (1937) for a synthetic phase with the composition  $\text{Cu}_5\text{FeS}_6$ . According to the hexagonal unit cell of this synthetic compound, Frenzel (1959) calculated the parameters  $a = 3.90$  and  $c = 16.95$  Å for idaite.

Experimental investigations by Yund (1963) led to the more general formula  $\text{Cu}_{5.5x}\text{Fe}_x\text{S}_{6.5x}$  for the synthetic idaite-like phase. Yund (1963) also compared the X-ray powder-diffraction pattern of natural idaite, given in Frenzel (1959), and of its synthetic analogue. In particular, Yund (1963) noted three additional Bragg peaks at 11.17, 5.58, and 3.723 Å.

Electron microprobe chemical analyses of natural idaite samples, reported by Lévy (1967), Sillitoe & Clark (1969), and Constantinou (1975), indicated an ideal composition  $\text{Cu}_3\text{FeS}_4$ , different of that previously reported for the synthetic idaite-like compound. Lévy (1967) also noticed that the  $d$ -spacings given by Frenzel (1959) better correspond to those of mawsonite,  $\text{Cu}_6\text{Fe}_2\text{SnS}_8$ . Lévy (1967) suggested that idaite, mawsonite, and stannite were members of the solid solution  $\text{Cu}_{2+x}\text{FeSn}_{1-x}\text{S}_4$ , with a tetragonal framework. Following this hypothesis, Wang (1976) re-calculated

the unit-cell parameters of idaite from Tarvis Township,  $a = 10.50$  and  $c = 10.72$  Å, which are close to those of mawsonite.

Frenzel & Otteman (1967) and Clark (1970) observed a hypogene sulphide with optical properties similar to those of idaite, but exhibiting a chemical composition corresponding to  $\text{Cu}_5\text{FeS}_6$ . This mineral was the natural analogue of the compounds previously synthesised by Merwin & Lombard (1937) and later by Yund (1963). Consequently, Sillitoe & Clark (1969) and Clark (1970) concluded to the existence of two distinct mineral species: supergene idaite,  $\text{Cu}_3\text{FeS}_4$  (Frenzel, 1958 and 1959), and a hypogene sulphide with the composition  $\text{Cu}_5\text{FeS}_6$  (Frenzel & Otteman, 1967; Clark, 1970). Rice *et al.* (1979) named this mineral nukundamite, after its occurrence in the Undu mine, Nukundamu, Fiji Islands.

Synthetic idaite was prepared by Ugarte & Burkin (1977) by leaching bornite with ferric sulphate solution. These authors considered idaite to have a crystal structure related to that of chalcopyrite, where the Fe atoms ( $0, \frac{1}{2}, \frac{3}{4}$ ) are replaced by Cu. The unit-cell parameters of synthetic idaite,  $a = 5.26$  and  $c = 10.46$  Å, are very close to those of chalcopyrite.

The data on the sulphides of the bornite-idaite join from Vielsalm were obtained during a detailed mineralogical study of the sulphides occurring in the Salm Valley, Belgium (Hatert, 1996). The aim of this paper is to describe the optical, chemical, and crystallographic properties of idaite from Vielsalm, leading to a better characterisation of this inadequately defined mineral species.

## Geological setting

The Venn-Stavelot Massif, Belgian Ardennes, represents a large unit of Cambro-Ordovician metasediments belonging to the Rhenohercynian of the Variscan orogenic belt. Localised in the SE portion of the massif, the Salm syncline shows, between Vielsalm and Salmchâteau, a geological section covering the Salmian stage of Lower Ordovician age. During the Hercynian orogeny, this area was affected by a low-grade metamorphism (Schreyer, 1975). The manganese-rich sediments of Middle Salmian contain P-T indicative mineral assemblages, and Kramm (1982) and Kramm *et al.* (1985) estimated P-T conditions of 2 kbar / 360–420°C, based on the presence of andalusite, chloritoid, spessartine, and white K-mica composition.

The quartz veins crystallised at the end of this metamorphic phase. Fluid circulations through the rock fractures, coupled with metasomatic processes, resulted in concentration of numerous chemical elements, such as Cu, Mn, Te, or Mo, within the quartz veins (Michot, 1954–1955; de Béthune & Fransolet, 1985; Hatert, 1996). This explains the diversity of minerals discovered in this area (Hatert *et al.*, 2002): copper sulphides, such as bornite, chalcopyrite, idaite, covellite, digenite, djurleite, and chalcocite (du Ry *et al.*, 1976; Hatert, 1996), Mn-bearing aluminosilicates, such as davreuxite and ottrelite (Fransolet & Bourguignon, 1976; Fransolet, 1978; Hanson, 1983; Fransolet *et al.*, 1984; Theye & Fransolet, 1994), and manganese oxides, such as lithiophorite, cryptomelane, nsutite, and hollandite-strontiomelane (Fransolet, 1979; Schreyer *et al.*, 2001; Gustine, 2002).

Idaite occurs in several quartz veins in the region of Vielsalm and Fraiture, but this mineral is particularly abundant in a vein localised in the old quarry of Thier des Carrières, Cahay. This centimetre-sized vein, crosscutting the green to violet chloritoid-bearing schists related to the upper part of Middle Salmian (Sm2c; Geukens, 1999), contains quartz, with accessory chlorite, hematite, rutile, and copper sulphides. Under oxidising superegene conditions, secondary copper-bearing carbonates, sulfates, phosphates, and arsenates have also crystallised (Hatert *et al.*, 2002).

## Analytical methods

Chemical analyses were obtained using a CAMECA SX50 electron microprobe operating in WDS mode, at the Louvain-la-Neuve University (Belgium). Measurement conditions were 15 kV acceleration voltage, 20 nA beam current, 16–20 s counting time per element. The standards used were pyrite (Fe, S) or the pure metals (Cu, Zn, Ag, Se). The elements occurring in very low amounts, below the detection limits of the instrument, were not considered in the chemical analyses. As, Au, Bi, Cd, Co, Ge, Ni, Pb, Sn, and Te were also checked but not detected in significant amounts.

X-ray measurements were performed with a Debye-Scherrer camera (diameter 114.6 mm) using  $\text{CuK}\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). The unit-cell parameters were calculated with the least-squares refinement program LCLSQ 8.4 (Burnham, 1991), using  $d$ -spacings corrected by the Straumanis method.

## Mineralogy of idaite

### Ore petrography

Idaite occurs in the more oxidised part of the Cahay quartz vein, where it forms sub-metallic grains up to 5 mm, of bronze colour. In reflected light, the mineral shows yellowish to orange colours and includes lamellae of chalcopyrite (Fig. 1). The very strong anisotropy, with colours ranging from pale green to yellow green, is also characteristic (Fig. 3). Idaite grains are successively rimmed by an amorphous Cu-Fe-oxide, covellite, brochantite, and malachite (Fig. 2 and 3).

Idaite is associated with bornite, and with a second mineral exhibiting pinkish to orange colours. The latter shows a low yellowish to green anisotropy and contains chalcopyrite lamellae, smaller than those observed in idaite. The optical characters of this phase indicate that it probably corresponds to the “anomalous bornite” described by Sillitoe & Clark (1969).

### Chemical composition

Electron microprobe analyses, performed on idaite from several quartz veins localised in the Stavelot Massif, are presented in Table 1. The chemical composition of these samples varies between  $\text{Cu}_{3.85}\text{Fe}_{0.98}\text{S}_4$  and  $\text{Cu}_{3.25}\text{Fe}_{0.97}\text{S}_4$ , thus indicating a distinct enrichment in copper, when compared with the ideal composition  $\text{Cu}_3\text{FeS}_4$ . This observation shows that the mineral from the Stavelot Massif does not correspond exactly to the pure idaite end-member.

The analyses of idaite, “anomalous bornite”, and bornite, taken from the literature, are also plotted into the Cu-Fe-S diagram and compared with idaite from the Stavelot Massif (Fig. 4). The chemical compositions of these three phases range from  $\text{Cu}_3\text{FeS}_4$  to  $\text{Cu}_5\text{FeS}_4$ , thus confirming the composition  $\text{Cu}_3\text{FeS}_4$  for idaite, and indicating a progressive evolution from bornite to idaite.

### X-ray powder diffraction

The X-ray powder-diffraction pattern of idaite from Vielsalm (Table 2) is rather similar to that of idaite from Tarvis Township (Frenzel, 1959) and to that of synthetic idaite (Ugarte & Burkin, 1977). Assuming an isostructural relationship between idaite and stannite (Lévy, 1967; Ugarte & Burkin, 1977), we calculated the theoretical powder pattern of idaite with the program LAZY PULVERIX (Yvon *et al.*, 1977). According to the indexation provided by this program, the unit-cell parameters of idaite from Vielsalm were also calculated, giving  $a = 5.279(4)$  and  $c = 10.47(2) \text{ \AA}$ .

The space group of idaite, deduced from the crystal structure proposed by Ugarte & Burkin (1977), must be  $\bar{I}42m$ , identical to that of stannite. The very weak 002 reflection ( $d = 5.146 \text{ \AA}$ ), observed on the powder diffraction pattern, is in good agreement with this space group, but not with that of chalcopyrite,  $\bar{I}42d$ .

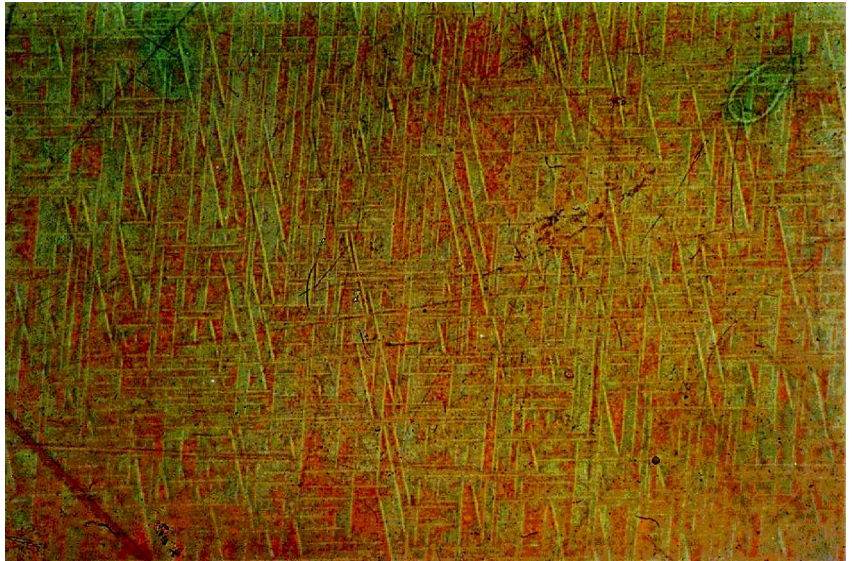


Fig. 1. Idaite from Vielsalm, containing lamellae of chalcopyrite parallel to the  $\{100\}$  and  $\{111\}$  directions of parent bornite. Reflected light microscopy, 150  $\mu\text{m}$  (BOR1-1).

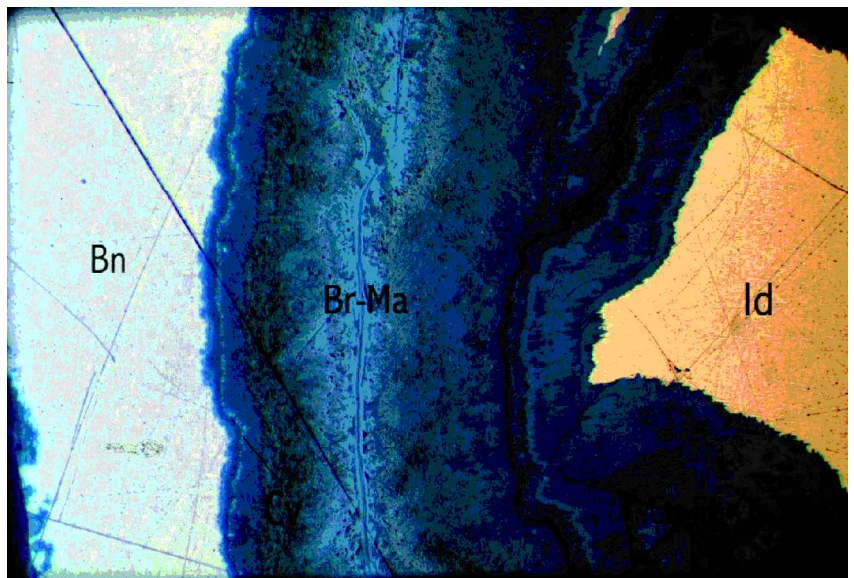


Fig. 2. Idaite (Id) and bornite (Bn) from Vielsalm, surrounded by covellite grains (Cv), an amorphous Cu-Fe-bearing oxide (Cu-Fe-Ox), brochantite, and malachite (Br-Ma). The orange colour of idaite is clearly visible, when compared to bornite. Reflected light microscopy, 300  $\mu\text{m}$  (BOR1-1).

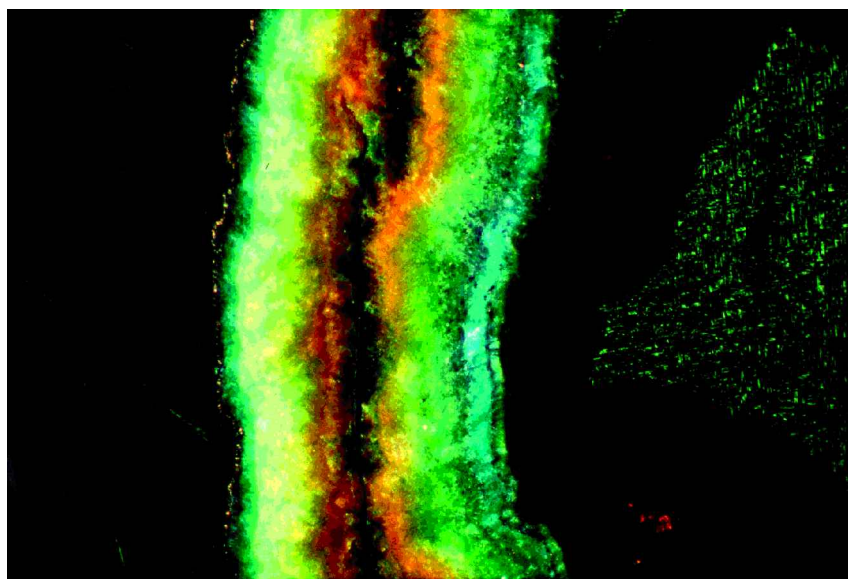


Fig. 3. Same section as in Fig. 2, in crossed nicols. The strong anisotropy of idaite is clearly visible, as well as the green internal reflections of brochantite and malachite. Reflected light microscopy, crossed nicols, 300  $\mu\text{m}$  (BOR1-1).

Table 1. Results of electron microprobe analyses of idaite from the Stavelot Massif.

	BOR1-1a (n = 3)	BOR1-1b (n = 2)	BOR1-1c (n = 2)	M402-a (n = 3)	M402-b (n = 2)	VIEL-1 (n = 2)	FRAI-2 (n = 3)
Cu	54.30 (39)	53.24 (2)	55.30 (75)	52.81 (8)	56.66 (12)	52.12 (97)	55.70 (64)
Fe	12.98 (62)	13.89 (5)	12.71 (42)	13.80 (24)	12.67 (16)	14.45 (65)	12.62 (12)
S	31.84 (24)	31.66 (27)	30.54 (35)	32.79 (29)	29.71 (10)	30.95 (18)	29.86 (29)
Zn	0.04 (7)	0.00	0.08 (11)	0.04 (7)	0.00	0.00	0.00
Ag	0.05 (8)	0.05 (8)	0.00	0.00	0.00	0.00	0.00
Se	0.00	0.00	0.00	0.00	0.00	0.07 (1)	0.00
Total	99.21	98.84	98.63	99.44	99.04	97.59	98.18
Cation numbers							
Cu	3.44	3.39	3.65	3.25	3.85	3.40	3.76
Fe	0.94	1.01	0.96	0.97	0.98	1.07	0.97
S	4.00	4.00	4.00	4.00	4.00	4.00	4.00
Zn	0.00	0.00	0.01	0.00	0.00	0.00	0.00
Ag	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Se	0.00	0.00	0.00	0.00	0.00	0.00	0.00

Analyst: J. Wautier. The number of point analyses is indicated in parentheses, as well as the standard deviations. Cation numbers were calculated on the basis of 4 sulfur atoms per formula unit. BOR1 and M402: Idaite from the Cahay quartz vein. VIEL: Idaite associated with arsenopyrite, Cahay. FRAI: Idaite, Fraiture.

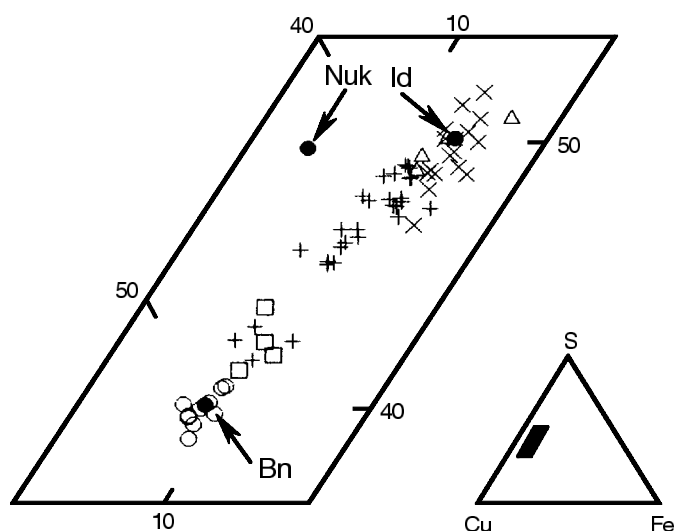


Fig. 4. Chemical analyses of idaite, anomalous bornite, and bornite from several occurrences. o: Bornite, Stavelot Massif. +: Idaite and “anomalous bornite”, Stavelot Massif. Δ: Synthetic idaite (Ugarte & Burkin, 1977). x: Natural idaite (Lévy, 1967; Sillitoe & Clark, 1969; Otteman & Frenzel, 1971; Constantinou, 1975; Rice *et al.*, 1979). □: “Anomalous bornite” (Sillitoe & Clark, 1969). ●: Ideal compositions of idaite,  $\text{Cu}_3\text{FeS}_4$  (Id), bornite,  $\text{Cu}_5\text{FeS}_4$  (Bn), and nukundamite,  $\text{Cu}_5\text{FeS}_6$  (Nuk).

## Discussions

Good-quality samples of idaite from the Stavelot Massif, Belgium, give us new chemical and crystallographic data for this poorly defined mineral species. Idaite, with a chemical composition close to  $\text{Cu}_3\text{FeS}_4$ , exhibits a crystal structure similar to that of stannite,  $\text{Cu}_2\text{FeSnS}_4$ , where Sn is re-

placed by Cu. The similarity between the powder patterns of idaite and chalcopyrite probably results from the structural relationships between chalcopyrite and the stannite mineral group. Rice *et al.* (1979) did not succeed in the interpretation of the X-ray powder pattern of idaite from the Ida mine, but mentioned the possibility that this mineral could give a powder pattern similar to that of chalcopyrite.

The occurrence of small amounts of Ag and Zn in idaite (Table 1) is also fully consistent with a stannite-type structure, because hocartite,  $\text{Ag}_2\text{FeSnS}_4$ , pirkuitasite,  $\text{Ag}_2\text{ZnSnS}_4$ , kesterite,  $\text{Cu}_2\text{ZnSnS}_4$ , and ferrokesterite,  $\text{Cu}_2(\text{Fe,Zn})\text{SnS}_4$ , also belong to the stannite mineral group (Gaines *et al.*, 1997).

Petrographic relations between the sulphides from Cahay indicate that idaite is a supergene oxidation product of bornite. The first step of the oxidation process is the development of digenite along fractures within primary bornite. This association is observed in the parts of the quartz vein affected by a low oxidation. Under more oxidising supergene conditions, the bornite grains, situated between digenite fractures, transform progressively to “anomalous bornite” and to idaite. This solid-state transformation, associated with the growth of chalcopyrite lamellae along the {100} and {111} directions of original bornite (Fig. 1), is demonstrated by the progressive intensification of the orange colour and anisotropy, relative to parent bornite. These characteristics have been previously observed in several localities (Frenzel, 1958 and 1959; Krause, 1965; Sillitoe & Clark, 1969; Ramdohr, 1969) and probably result from the leaching of Cu atoms from bornite (Ugarte & Burkin, 1977).

Further oxidation of idaite grains, containing chalcopyrite, result in the formation of an amorphous Cu-Fe-bearing oxide, covellite, malachite, and brochantite (Fig. 2 and 3). The oxidation processes, involved in the formation of idaite in the Stavelot Massif, are similar to those described by Sillitoe & Clark (1969) in Copiapó, Chile.



Table 2. X-ray powder data for idaite.

Tarvis Township (Frenzel, 1959)		Synthetic (Ugarte & Burkin, 1977)		Vielsalm, Belgium (this work)				
<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> <sub>obs.</sub> (Å)	<i>I</i>	<i>d</i> <sub>calc.</sub> (Å)	<i>I</i> / <i>I</i> <sub>0</sub>	<i>hkl</i>
-	-	-	-	5.146	vw	5.233	5	002
3.35	vw	-	-	-	-	-	-	-
3.20	vw	3.220	w	-	-	-	-	-
3.05	VS	3.018	VS	3.018	VS	3.039	80	112
2.81	S	-	-	2.787	w	→ Covellite?	-	-
2.68	S	2.622	w	2.630	vw	2.640	5	200
1.92	VS	1.903	w	1.934	w	1.946	5	105
1.85	m	1.859	S	1.868	VS	1.858	100	204
1.636	vw	-	-	-	-	-	-	-
1.571	w	1.588	m	1.588	S	1.591	20	312
-	-	1.316	w	1.321	vw	1.320	20	400
1.205	vw	1.209	w	1.207	w	1.206	10	316
1.108	vw	-	-	-	-	-	-	-
1.080	w	1.078	w	1.078	S	1.076	80	424
-	-	1.016	vw	1.015	w	1.016	15	512

vw = very weak; w = weak; m = medium; S = strong; VS = very strong.

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