



Luxembourgite, $\text{AgCuPbBi}_4\text{Se}_8$, a new mineral species from Bivels, Grand Duchy of Luxembourg

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Abstract. Luxembourgite, ideally $\text{AgCuPbBi}_4\text{Se}_8$, is a new selenide discovered at Bivels, Grand Duchy of Luxembourg. The mineral forms tiny fibres reaching 200 μm in length and 5 μm in diameter, which are deposited on dolomite crystals. Luxembourgite is grey, with a metallic lustre and without cleavage planes; its Mohs hardness is 3 and its calculated density is 8.00 g cm^{-3} . Electron-microprobe analyses indicate an empirical formula $\text{Ag}_{1.00}(\text{Cu}_{0.82}\text{Ag}_{0.20}\text{Fe}_{0.01})_{\Sigma 1.03}\text{Pb}_{1.13}\text{Bi}_{4.11}(\text{Se}_{7.72}\text{S}_{0.01})_{\Sigma 7.73}$, calculated on the basis of 15 atoms per formula unit. A single-crystal structure refinement was performed to $R_1 = 0.0476$, in the $P2_1/m$ space group, with $a = 13.002(1)$, $b = 4.1543(3)$, $c = 15.312(2)$ Å, $\beta = 108.92(1)^\circ$, $V = 782.4(2)$ Å³, $Z = 2$. The crystal structure is similar to that of lithochlebite and watkinsonite and can be described as an alternation of two types of anionic layers: a pseudotetragonal layer four atoms thick and a pseudo-hexagonal layer that is one atom thick. In the pseudotetragonal layers the Bi1, Bi2, Bi3, Pb, and Ag1 atoms are localised, while the Cu2 and Bi4 atoms occur between the pseudotetragonal and the pseudo-hexagonal layers. Bi1, Bi2, and Bi3 atoms occur in weakly distorted octahedral sites, whereas Bi4 occurs in a distorted 7-coordinated site. Ag1 occupies a fairly regular octahedral site, Cu2 a tetrahedral position, and Pb occurs on a very distorted 8-coordinated site.

1 Introduction

The term “Luxembourg” designates a natural region composed of the Grand Duchy of Luxembourg and by the Luxembourg Province in the South of Belgium. The Ardennes mountains are in the north of this region and are named “Oesling” in the Grand Duchy. This massif is constituted of Lower Devonian rocks, mainly schists and quartzites, which were affected by the Variscan metamorphism. Pressures and temperatures were estimated to be 500 °C and 3–4 kbar in the region of Libramont, but the metamorphic conditions progressively decrease to the east, reaching 400 °C and 2 kbar at Bastogne (Beugnies, 1986; Theye and Fransolet, 1993; Hatert, 2005).

During winter 2012, excavation works were realised by the “Société électrique de l’Our” at Bivels, in the north of the Grand Duchy, exposing some fragments of reddish schists containing mineralised veins. The veins were constituted by translucent dolomite crystals reaching 2 mm in diameter, as well as by siderite. A careful examination under the binocular microscope also showed needles of a grey metallic mineral, with a habit similar to that of millerite. Preliminary Energy-Dispersive X-ray Spectrometry (EDS) measurements indicated that these fibres contained Cu, Bi, Ag, Pb, and Se as essential constituents, thus corresponding to an intermediate between lithochlebite, $\text{Ag}_2\text{PbBi}_4\text{Se}_8$ (Sejkora et al., 2011) and watkinsonite, $\text{Cu}_2\text{PbBi}_4\text{Se}_8$ (Topa et al., 2010).

Electron-microprobe analyses indicated a Cu/Ag ratio very close to 1.0 in the mineral, in agreement with

the formula AgCuPbBi₄Se₈. A single-crystal X-ray diffraction study showed a crystal structure similar to that of watkinsonite but with significant differences compared to lithochlebite. Moreover, the ordering of Cu and Ag at two different crystallographic sites indicated that the mineral was not only an intermediate member between lithochlebite and watkinsonite but a separate mineral species. This assumption was confirmed by the very limited solid solution between the new species and lithochlebite.

The mineral was named luxembourgite for the city of Luxembourg, close to the locality where it was discovered; it is the first new species found in the Grand Duchy of Luxembourg. The new mineral and its name were approved by the IMA-CNMNC under the number IMA 2018-154. A part of the type sample used for electron-microprobe analyses is stored in the collection of the Natural History Museum of Luxembourg (catalogue number FD040). Another part of the type used for the single-crystal structure determination is stored in the collection of the Laboratory of Mineralogy, University of Liège, catalogue number 21302. A detailed mineralogical characterisation of this new species is given in the present paper.

2 Occurrence

Luxembourgite was found on dumps produced by the construction of a tunnel by the “Société Electrique de l’Our”, at Bivels, north of the Grand Duchy of Luxembourg (coordinates: 49°57'8" N, 6°10'41" E). Associated minerals are dolomite and siderite. The mineral occurs in red schists containing veins of finely crystallised dolomite and siderite. The dolomite crystals reach 2 mm in diameter and sometimes show tiny fibres of luxembourgite. The veins were formed by a hydrothermal process, which is also responsible for other famous sulfide mineralisations in the area, such as the copper mine of Stolzenbourg (Philippo et al., 2007) and the antimony mine of Goesdorf (Philippo and Hanson, 2007; Philippo and Hatert, 2018).

3 Physical properties

Luxembourgite forms tiny fibres deposited on dolomite crystals; the fibres reach 200 μm in length but only 5 μm in diameter (Fig. 1). The colour is grey, the lustre is metallic, and the streak is black. No cleavage has been observed, and the tenacity is brittle. The Mohs hardness, estimated by comparison with lithochlebite (Sejkora et al., 2011), is 3. The density, calculated from the chemical analysis and the single-crystal unit cell parameters, is 8.00 g cm⁻³. Optical properties were not determined due to small grain size.

Table 1. Electron-microprobe analysis of luxembourgite.

	Wt. %	Range (wt. %)	Cation numbers
S	0.01	0.00–0.02	0.005
Fe	0.02	0.00–0.05	0.006
Pb	11.95	10.50–12.79	1.133
As	0.00	0.00	0.000
Ag	6.60	5.68–9.04	1.202
Cu	2.66	2.23–2.81	0.821
Bi	43.73	42.66–44.26	4.111
Se	31.04	30.37–32.04	7.722
Total	96.01		

Average of five-point analyses. Cation numbers were calculated on the basis of 15 atoms per formula unit.

4 Chemical composition

Electron-microprobe analyses of luxembourgite (Table 1) were performed with a Jeol JXA-8200 WDS instrument located in Milan, Italy. Acceleration voltage was 15 kV, with a probe current of 5 nA and a beam diameter of 1 μm. Standards used were galena (Pb, S), nickeline (As), and pure metals (Fe, Ag, Cu, Bi, Se). The empirical formula, calculated on the basis of 15 atoms per formula unit, is Ag_{1.00}(Cu_{0.82}Ag_{0.20}Fe_{0.01})_{Σ1.03}Pb_{1.13}Bi_{4.11}(Se_{7.72}S_{0.01})_{Σ7.73}. The ideal formula is AgCuPbBi₄Se₈, which requires Ag 5.58, Cu 3.44, Pb 11.22, Bi 45.28, and Se 34.22 for a total of 100.00 wt %.

5 X-ray diffraction and crystal structure

X-ray powder diffraction data of luxembourgite (Table 2), as well as X-ray single-crystal data, were collected on a Rigaku Xcalibur four-circle diffractometer using the MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) and equipped with an EOS Charge-Coupled Device (CCD) detector. Unit cell parameters were refined from the powder diffraction data, using the LCLSQ software (Burnham, 1962): $a = 13.01(13)$, $b = 4.15(2)$, $c = 15.36(14) \text{ \AA}$, $\beta = 109.4(6)^\circ$, $V = 783(16) \text{ \AA}^3$, $Z = 2$, space group $P2_1/m$.

The X-ray structural study was carried out on a fibre of luxembourgite measuring $0.005 \times 0.005 \times 0.150 \text{ mm}$. A total of 361 frames with a spatial resolution of 1° were collected by the φ/ω scan technique, with a counting time of 20 s per frame, in the range $5.00^\circ < 2\theta < 57.68^\circ$. A total of 5904 reflections were extracted from these frames, corresponding to 2117 unique reflections. The unit cell parameters refined from these reflections are in very good agreement with those refined from the X-ray powder data (see above). Data were corrected for Lorentz, polarisation and absorption effects, the latter with an empirical method using the SCALE3 ABSPACK scaling algorithm included in the CrysAlisRED package (Oxford Diffraction, 2007). More de-

Table 2. X-ray powder diffraction pattern of luxembourgite (d in Å).**Figure 1.** Scanning Electron Microscope (SEM) images of luxembourgite fibres occurring on rhombohedral dolomite crystals.

I_{obs}	d_{obs}	d_{calc}^*	I_{calc}^*	hkl
20	4.61	4.588	11	2 0 -3
-	-	4.330	5	3 0 -1
-	-	3.790	8	3 0 -3
-	-	3.647	22	3 0 1
-	-	3.621	12	0 0 4
20	3.59	3.604	37	0 1 2
-	-	3.443	4	2 1 0
-	-	3.296	5	3 0 -4
-	-	3.204	20	1 0 4
100	2.984	2.998	100	3 1 -1
-	-	2.953	60	3 1 -2
-	-	2.897	44	0 0 5
-	-	2.800	16	3 1 -3
-	-	2.582	4	3 1 -4
20	2.425	2.446	21	2 1 -5
-	-	2.344	4	3 1 -5
-	-	2.276	30	3 1 3
-	-	2.172	6	4 1 2
-	-	2.165	6	6 0 -2
-	-	2.155	25	6 0 -3
-	-	2.115	23	3 1 -6
60	2.085	2.077	36	0 2 0
-	-	2.053	16	3 1 4
-	-	1.969	6	1 1 6
-	-	1.920	5	6 1 -2
20	1.916	1.909	9	3 1 -7
		1.894	12	6 1 -1
-	-	1.824	10	6 0 2
-	-	1.805	7	3 2 1
-	-	1.772	6	6 0 -7
-	-	1.743	7	1 2 4
-	-	1.688	18	0 2 5
20	1.490	1.496	16	6 2 -3
-	-	1.475	5	6 1 4
-	-	1.431	6	2 1 -10
-	-	1.415	5	4 1 7
-	-	1.370	8	6 2 2
		1.363	12	9 1 -4
30	1.355	1.348	5	6 2 -7
		1.345	4	8 1 -8
30	1.188	1.188	2	0 2 10

Data collected with a Rigaku Xcalibur four-circle diffractometer, MoK α radiation ($\lambda = 0.71073$ Å), EOS Rigaku detector. Intensities were estimated visually. Calculated intensities were obtained from the structural data with LAZY PULVERIX (Yvon et al., 1977). Calculated d values were refined with LCLSQ (Burnham, 1962); the refined unit cell parameters are $a = 13.01(13)$, $b = 4.15(2)$, $c = 15.36(14)$ Å, $\beta = 109.4(6)^\circ$ (space group $P2_1/m$). * Only lines with a calculated intensity higher than 4 rel. % are shown.

Table 3. Experimental details for the single-crystal X-ray diffraction study of luxembourgite from Bivels, Luxembourg.

Ideal structural formula	AgCuPbBi ₄ Se ₈
<i>a</i> (Å)	13.002(1)
<i>b</i> (Å)	4.1543(3)
<i>c</i> (Å)	15.312(2)
β (°)	108.92(1)
<i>V</i> (Å ³)	782.4(2)
Space group	<i>P2</i> ₁ / <i>m</i>
<i>Z</i>	2
<i>D</i> _{calc} (g cm ⁻³)	7.397
Absorption coefficient (mm ⁻¹)	67.46
<i>F</i> (000)	1453
Radiation	MoK α , 0.71073
Crystal size (mm)	0.005 × 0.005 × 0.150
Colour and habit	Grey metallic needle
Temperature (K)	293(2)
θ range (°)	2.50–28.84
Reflection range	–16 ≤ <i>h</i> ≤ 17, –4 ≤ <i>k</i> ≤ 5, –19 ≤ <i>l</i> ≤ 14
Total no. of reflections	5904
Unique reflections	2117
Refined parameters	94
<i>R</i> ₁ , <i>F</i> ² > 2 σ (<i>F</i> ²)	0.0476
<i>R</i> ₁ , all data	0.0730
w <i>R</i> ₂ (<i>F</i> ²), all data	0.1069
Goodness of fit	1.077
$\Delta\sigma_{\min}$, $\Delta\sigma_{\max}$ (e/Å ³)	–2.42, 5.32

tails on the single-crystal structure refinement are given in Table 3.

The crystal structure of luxembourgite (Fig. 2; Table 4) was refined in space group *P2*₁/*m*. Scattering curves for neutral atoms, together with anomalous dispersion corrections, were taken from the International Tables for X-ray Crystallography, Vol. C (Wilson, 1992). In the final refinement cycle, all atoms were refined anisotropically, leading to the *R*₁ value 0.0476. The site occupancy factors were refined for the Cu₂, Pb, and Bi₄ sites and were constrained to full occupancy for the Ag₁, Bi₁, Bi₂, and Bi₃ sites (Table 4). A high residual electron density of 5.32e/Å³ (Table 3) was observed at 0.94 Å from Bi₄, and this atom shows a higher isotropic displacement parameter than other Bi atoms (Table 4). A double occupancy by Bi and Pb was therefore tested for this site, but this model did not improve the *R*₁ value or the displacement parameter of Bi₄. Difference Fourier maps indicated an electron density at 0.385 Å from Ag₁, so a split-atom model was also tested. The low occupancy factor of the second component of the split Ag atom, 0.015, indicated that this split-atom model was not necessary here.

The crystal structure of luxembourgite is similar to those of lithochlebite (Sejkora et al., 2011) and watkinsonite (Topa et al., 2010), and is based on the stacking of two types of an-

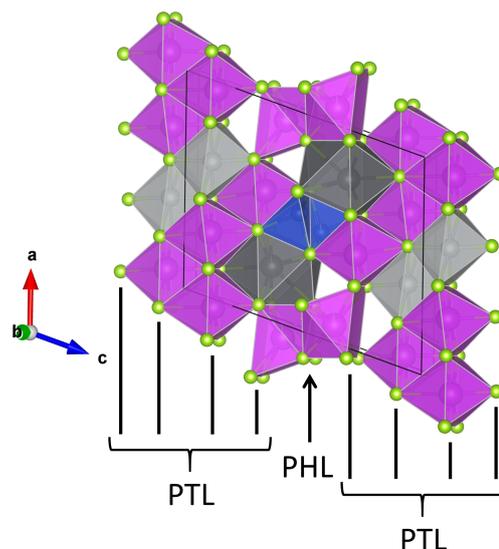


Figure 2. The crystal structure of luxembourgite viewed approximately perpendicular to the *b* axis. Se atoms are yellow, Bi polyhedra are purple, Cu tetrahedra are blue, Ag polyhedra are light grey, and Pb polyhedra are dark grey. Vertical black lines indicate the directions of pseudotetragonal layers (PTLs) and pseudo-hexagonal layers (PHLs) constituted by Se atoms.

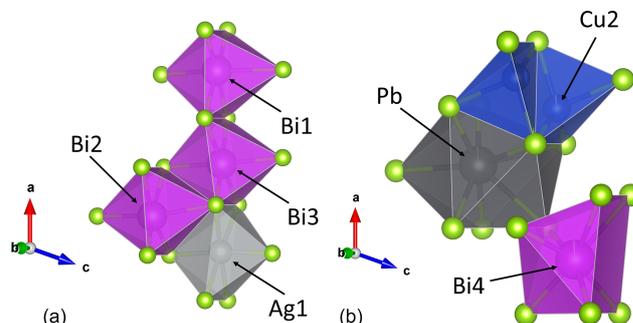


Figure 3. Morphologies of the coordination polyhedra in luxembourgite. (a) The Bi₁, Bi₂, Bi₃, and Ag₁ octahedra occur in the PbS-like pseudotetragonal layers. (b) The Pb [8], Bi₄ [7], and Cu₂ [3 + 1] sites occur between the pseudotetragonal and pseudo-hexagonal anionic layers.

ionic layers: a pseudotetragonal layer four anions thick and a pseudo-hexagonal layer one anion thick (Fig. 2). In the pseudotetragonal layers the Bi₁, Bi₂, Bi₃, and Ag₁ atoms are localised and are each coordinated by six Se anions to form weakly distorted octahedra (Table 5, Fig. 3a). The topology of this layer is identical to that observed in galena, thus explaining why some authors describe the pseudotetragonal layer as a “PbS-like slab” (Topa et al., 2006). The Cu₂ atoms are located very close to the pseudo-hexagonal layer, producing distorted Cu₂–Se₄ tetrahedra with a [3 + 1] coordination (Table 5, Fig. 3b). Finally, the Pb and Bi₄ atoms are located between the pseudotetragonal and pseudo-hexagonal slabs, forming coordination polyhedra with rather complex

Table 4. Atom coordinates and anisotropic displacement parameters (\AA^2) for luxembourgite from Bivels, Luxembourg.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₂₃	<i>U</i> ₁₃	<i>U</i> ₁₂
Ag1	0.62177(17)	0.75	0.09956(16)	0.0365(5)	0.0357(13)	0.0582(14)	0.0143(12)	0	0.0063(10)	0
Cu2*	0.4701(3)	0.25	0.5342(3)	0.0302(12)	0.033(2)	0.0180(17)	0.047(3)	0	0.0230(19)	0
Pb**	0.23164(8)	0.75	0.32905(9)	0.0303(4)	0.0280(6)	0.0190(5)	0.0466(9)	0	0.0161(6)	0
Bi1	0.95615(7)	0.75	0.11227(7)	0.0155(2)	0.0164(4)	0.0131(4)	0.0170(6)	0	0.0055(4)	0
Bi2	0.56485(7)	0.25	0.29515(7)	0.0162(2)	0.0163(5)	0.0143(4)	0.0169(6)	0	0.0038(4)	0
Bi3	0.29452(7)	0.75	0.09483(7)	0.0159(2)	0.0150(4)	0.0128(4)	0.0199(6)	0	0.0056(4)	0
Bi4***	0.92273(8)	0.25	0.37980(8)	0.0216(3)	0.0172(5)	0.0179(4)	0.0277(7)	0	0.0045(4)	0
Se1	0.87488(18)	0.75	0.49204(17)	0.0149(5)	0.0156(11)	0.0138(10)	0.0145(14)	0	0.0037(10)	0
Se2	0.13389(18)	0.75	0.11537(17)	0.0149(5)	0.0122(11)	0.0124(10)	0.0213(15)	0	0.0071(11)	0
Se3	0.70090(18)	0.75	0.27334(18)	0.0157(5)	0.0119(11)	0.0139(10)	0.0199(15)	0	0.0034(10)	0
Se4	0.43973(18)	0.25	0.07742(17)	0.0140(5)	0.0145(11)	0.0135(11)	0.0148(14)	0	0.0061(10)	0
Se5	0.38321(18)	0.75	0.29286(17)	0.0144(5)	0.0139(11)	0.0135(10)	0.0159(14)	0	0.0050(10)	0
Se6	0.63688(18)	0.25	0.48353(18)	0.0166(5)	0.0158(12)	0.0177(11)	0.0145(14)	0	0.0024(10)	0
Se7	0.80747(18)	0.75	0.11389(17)	0.0150(5)	0.0139(11)	0.0141(11)	0.0176(14)	0	0.0059(10)	0
Se8	0.04394(17)	0.75	0.30193(17)	0.0139(5)	0.0147(11)	0.0137(10)	0.0133(14)	0	0.0043(10)	0

Occupancies: * = 0.965(10) Cu; ** = 0.989(4) Pb; *** = 0.977(4) Bi.

morphologies. Pb is coordinated by five Se anions from the pseudotetragonal layer in a pyramidal arrangement, and by three Se from the pseudo-hexagonal layer in a triangular arrangement, producing a complex 8-fold polyhedron resembling a pentagonal bipyramid with one supplementary square face (Table 5, Fig. 3b). Such very distorted seven-coordinated site are very typical for Pb, a cation with lone-electron pairs (Theye et al., 2010; Krivovichev and Burns, 2000). Bi4 is coordinated by four Se anions from the pseudotetragonal layer and by three Se anions from the pseudo-hexagonal layer, leading to a 7-fold polyhedron that can be described as a pyramid with its square face adjusted on a trigonal prism (Table 5, Fig. 3b).

Site occupancy factors (Table 4) indicate that the Ag1, Bi1, Bi2, and Bi3 sites are fully occupied, while the Cu2 site contains 0.965(10) Cu, the Pb site contains 0.989(4) Pb, and the Bi4 site contains 0.977(4) Bi. Bond distances (Table 5) were used to calculate bond valence sums, according to the empirical relationship established by Brown and Altermatt (1985) and Brese and O’Keeffe (1991). These bond valence sums are 2.08 for Pb, 1.33 for Ag1, 1.00 for Cu2, and between 3.08 and 3.25 for Bi atoms, thus confirming the cationic distributions.

6 Discussion

Luxembourgite is isostructural with watkinsonite, Cu₂PbBi₄Se₈, and is closely related to lithochlebite, Ag₂PbBi₄Se₈. It also shows similarities with berryite, Cu₃Ag₂Pb₃Bi₇S₁₆, a sulfide showing PbS-like slabs containing four Bi and two Ag sites, as well as hexagonal slabs containing Cu atoms coordinated by three S atoms in triangular environments (Topa et al., 2006). In this structure,

Table 5. Selected interatomic distances (\AA) for luxembourgite from Bivels, Luxembourg.

Ag1-Se4	3.084(2) × 2	Cu2-Se5	2.716(5)
Ag1-Se4	2.565(3)	Cu2-Se6	2.465(3) × 2
Ag1-Se3	2.523(3)	Cu2-Se6	2.529(6)
Ag1-Se7	3.138(3) × 2	<Cu2-Se>	2.5438
<Ag1-Se>	2.9219		
Pb-Se5	3.035(3) × 2	Bi1-Se2	3.096(2) × 2
Pb-Se2	3.102(3)	Bi1-Se7	2.843(2) × 2
Pb-Se8	3.128(2) × 2	Bi1-Se8	2.754(3)
Pb-Se6	3.506(3) × 2	Bi1-Se2	3.298(3)
Pb-Se1	3.447(4)	<Bi1-Se>	2.9885
<Pb-Se>	3.2361		
Bi2-Se6	2.729(3)	Bi3-Se5	2.874(3)
Bi2-Se3	2.817(2) × 2	Bi3-Se2	3.033(2) × 2
Bi2-Se4	3.201(3)	Bi3-Se4	2.877(2) × 2
Bi2-Se5	3.137(3) × 2	Bi3-Se7	3.038(3)
<Bi2-Se>	2.9370	<Bi3-Se>	2.9554
Bi4-Se1	2.889(3) × 2		
Bi4-Se1	2.725(3)		
Bi4-Se8	3.072(3) × 2		
Bi4-Se3	3.499(1) × 2		
<Bi4-Se>	3.0923		

Pb sites, as well as two Bi sites, occur on the surface of the PbS-like slabs, as observed in the structure of luxembourgite (see above). Table 6 gives a comparison between the properties of luxembourgite, watkinsonite, and lithochlebite. In mineralogical classification systems, luxembourgite has a Strunz number 02.HB.20 and a Dana number 3.7.21.

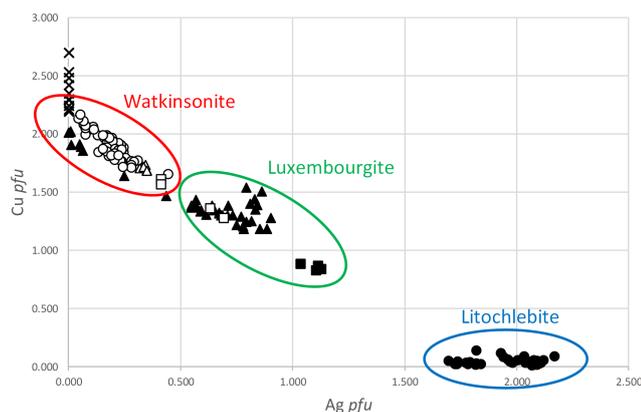


Figure 4. Ag / Cu plot, showing the compositions of luxembourgitte, watkinsonite, and lithochlebite. Crosses represent watkinsonite from Otish Mountains (Johan et al., 1987). Black triangles represent watkinsonite–luxembourgitte from Zálesí (Sejkora, unpublished data). White triangles represent watkinsonite from Zálesí (Sejkora et al., 2012). Black circles represent a lithochlebite holotype from Zálesí (Sejkora et al., 2011). White circles represent watkinsonite from Zálesí (Topa et al., 2010). Black squares represent luxembourgitte from Bivels (this paper). White squares represent watkinsonite–luxembourgitte from Niederschlema (Förster et al., 2005).

As underlined by Sejkora et al. (2011), the crystal structures of watkinsonite and lithochlebite show significant differences, mainly concerning the Ag / Cu crystallographic sites. In watkinsonite, Cu occurs in both octahedral (Cu1) and tetrahedral (Cu2) coordinations, but in lithochlebite, all Ag sites occur in octahedral environments. The Ag positions in lithochlebite are split on two very close positions, leading to Ag1a–Ag1b and Ag2a–Ag2b pairs of sites, as well as the Cu1 site of watkinsonite, which is split into a Cu1–Ag pair of sites. In luxembourgitte, the situation is more ordered, with Ag occurring on the octahedral Ag1 site and Cu on the tetrahedral Cu2 site, without any splitting of atoms. In watkinsonite, Topa et al. (2010) already observed the presence of 0.32 Ag on the Cu1/Ag site; in luxembourgitte, this site is fully occupied by Ag (Table 4).

Chemical analyses from the literature (Fig. 4) indicate that a complete solid solution exists between watkinsonite and luxembourgitte, but the replacement of Cu by Ag on the Cu2 site is more difficult, certainly due to the small size of this site in watkinsonite and luxembourgitte (average *M*-O bond lengths = 2.544 Å in luxembourgitte and 2.730 Å in lithochlebite). Consequently, the solid solution between luxembourgitte and lithochlebite is very limited, due to the significant structural changes between these minerals, and luxembourgitte cannot be considered an intermediate phase between lithochlebite and watkinsonite. Luxembourgite is the analogue of watkinsonite, with Ag and Cu ordered on the Ag1 and Cu2 sites, respectively.

Table 6. Comparison of the physical properties of luxembourgitte, watkinsonite, and lithochlebite.

Phase	Luxembourgite	Watkinsonite	Lithochlebite
Reference	This work	(a), (b)	(c)
Ideal formula	AgCuPbBi ₄ Se ₈	Cu ₂ PbBi ₄ Se ₈	Ag ₂ PbBi ₄ Se ₈
Space group	<i>P</i> 2 ₁ / <i>m</i>	<i>P</i> 2 ₁ / <i>m</i>	<i>P</i> 2 ₁ / <i>m</i>
<i>a</i> (Å)	13.002 (1)	12.952 (4)	13.203 (4)
<i>b</i> (Å)	4.1543 (3)	4.152 (1)	4.186 (1)
<i>c</i> (Å)	15.312 (2)	15.155 (5)	15.280 (4)
β (°)	108.92 (1)	108.931 (5)	109.164 (4)
<i>Z</i>	2	2	2
Strong X-ray	4.61 (20)	4.514 (40)	4.610 (25)
Lines	–	–	3.684 (53)
	3.59 (20)	3.573 (90)	3.625 (39)
	–	–	3.201 (76)
	–	–	3.028 (100)
	2.984 (100)	2.976 (100)	2.980 (88)
	–	2.929 (100)	2.892 (95)
	2.425 (20)	2.407 (70)	2.453 (22)
	–	2.245 (60)	2.288 (22)
	–	2.140 (70)	2.123 (29)
	2.085 (60)	2.065 (70)	2.094 (31)
	1.916 (20)	1.935 (10)	1.912 (17)
	1.490 (20)	1.484 (70)	1.510 (10)
	1.355 (30)	–	–
	1.188 (30)	–	–
Cleavage	None	None	None
Density	8.00 (calc.)	7.76 (calc.)	7.87 (calc.)
Hardness	3	–	3
Colour	Grey	Black	Dark grey to black

References: (a) Johan et al. (1987); (b) Topa et al. (2010); (c) Sejkora et al. (2011).

Data availability. All data are available upon request to Frédéric Hatert.

Supplement. The supplement related to this article is available online at: <https://doi.org/10.5194/ejm-32-449-2020-supplement>.

Author contributions. SP discovered the sample and realised a preliminary EDS analysis. FH performed the single-crystal structure determination with YB, obtained a powder pattern, and wrote the IMA-CNMNC proposal and the final manuscript. PV analysed the species by electron microprobe, and JS indexed the X-ray powder pattern and gave unpublished electron-microprobe analyses of similar samples.

Competing interests. The authors declare that they have no conflict of interest.

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