

Ordered distribution of Au and Ag in the crystal structure of muthmannite, AuAgTe₂, a rare telluride from Sacarîmb, western Romania

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ABSTRACT

Muthmannite, AuAgTe₂, a rare gold-silver telluride was discovered in a sample from the historical mineralogical collection of the Naturhistorisches Museum of Vienna. The sample is from the gold-telluride deposit of Sacarîmb, Metaliferi Mountains, western Romania. Muthmannite occurs as anhedral grains up to 200 µm associated with large sylvanite crystals and does not show any inclusions or intergrowths of other minerals. The associated minerals are sylvanite, calaverite, and petzite, whereas the gangue mineral is quartz. Muthmannite is pale bronze in color and shows a gray-black streak. No cleavage was observed, the fracture is uneven, and the Vickers hardness (VHN₁₅) is 186 kg/mm². Muthmannite is grayish white in reflected light, with very low bireflectance and pleochroism. When observed near sylvanite it is darker and shows a gray color with a slightly bluish tint. Reflectance percentages for R_{\min} and R_{\max} were found to be 40.1, 40.8 (471.1 nm), 38.3, 38.6 (548.3 nm), 37.9, 38.3 (586.6 nm), and 37.7, 38.1 (652.3 nm), respectively. Muthmannite is monoclinic, space group $P2/m$, with the following unit-cell parameters: $a = 5.124(2)$, $b = 4.419(1)$, and $c = 7.437(2)$ Å, $\beta = 89.96(1)^\circ$, $V = 168.4(4)$ Å³, and $Z = 2$. Electron microprobe analyses gave the chemical formula Au_{0.97}Ag_{0.99}Te_{2.03}. The calculated density (from the ideal formula) is 11.04 g/cm³. The crystal structure was solved and refined to $R = 5.52\%$. It is based on the NiAs-type structure, with a distorted hexagonal closest-packed array of Te²⁻ atoms with Au³⁺ and Ag⁺ occupying all the octahedral sites. The crystal-chemical relationships with other gold-silver tellurides are outlined.

INTRODUCTION

Muthmannite, AuAgTe₂, was identified as mineral species by Zambonini (1911) during a study of gold-silver tellurides from the Sacarîmb (formerly Nagyag) deposit, western Romania, and was named in honor of Friedrich W. Muthmann (1861–1913), a German chemist and crystallographer. On the basis of the chemical composition Zambonini (1911) proposed the formula (Au,Ag)Te with Au:Ag = 1:1. More recently, Spiridonov and Chvileva (1985) described the second occurrence of muthmannite from Baia-de-Arieş (formerly Aranyosbánya), Romania, giving the following chemical formulae (for two grains): Au_{0.97}Ag_{0.92}Cu_{0.03}Fe_{0.01}Hg_{0.01}Te_{2.06} and Au_{0.95}Ag_{0.97}Cu_{0.02}Fe_{0.01}Hg_{0.01}Te_{2.04}. These authors reported that the mineral replaces calaverite along the margin of the grains forming pseudomorphs after small calaverite grains. Powder X-ray study data in Spiridonov and Chvileva (1985) show strong similarities between muthmannite and calaverite, the only differences being the presence of three additional lines in the muthmannite powder pattern [i.e., d (Å): 4.17, 2.79, and 2.57]. However, owing to the scarcity of suitable crystals for a full structural study and to the presence of complex intergrowths with calaverite, Spiridonov and Chvileva (1985) were unable to determine the crystal structure of muthmannite.

In the course of a research project on tellurium-bearing miner-

als in the museum's historical mineralogical collections (Bindi and Cipriani 2003a, 2003b, 2003c; Bindi et al. 2004; Cipriani and Bindi 2003) we discovered another muthmannite sample from the type locality. In this paper we report on the crystal structure of muthmannite together with physical and chemical data for the mineral.

OCCURRENCE

The Sacarîmb gold-telluride deposit is located in the southeastern part of the Metaliferi Mountains, western Romania. Although now close to exhaustion, it is one of the most famous Neogene epithermal deposits in the world. As reported by Simon et al. (1994), the telluride-bearing veins are located in a volcanic body consisting of hornblende- and pyroxene-bearing quartz-andesites of Neogene age. Geological and metallogenic data concerning the Sacarîmb deposit are summarized by Udubasa et al. (1992).

As described in the former section, the sample containing muthmannite was not found in situ but comes from the historical mineralogical collection of the Naturhistorisches Museum of Vienna, where it was labeled "sylvanite, Nagyag" (catalogue number L2138). The sample consists of sylvanite with associated muthmannite, calaverite, petzite, and quartz. Muthmannite occurs as anhedral grains up to 200 µm associated with large sylvanite crystals (Fig. 1), and it does not show inclusions or intergrowths of other minerals.

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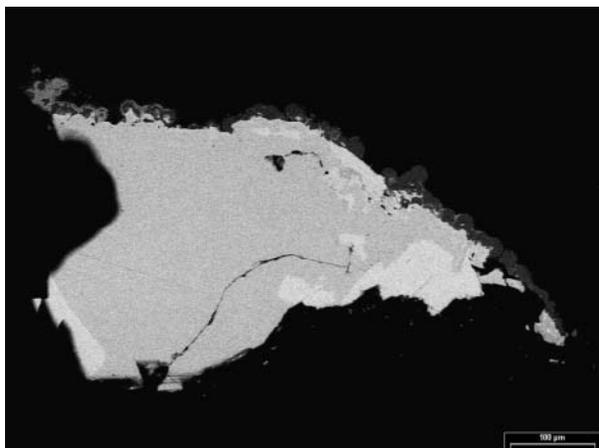


FIGURE 1. BSE microphotograph of muthmannite (light gray) associated with sylvanite (dark gray).

PHYSICAL AND OPTICAL PROPERTIES

Muthmannite is pale bronze in color and shows a gray-black streak. The mineral is opaque with a metallic luster. No cleavage was observed and the fracture is uneven. Muthmannite is granular and sometimes forms compact masses. The calculated density for ideal AuAgTe₂ ($Z = 2$) is 11.04 g/cm³. Unfortunately, the density could not be measured because of the small grain size. Micro-indentation measurements carried out with a VHN load of 15 g gave a mean value of 186 kg/mm² (range: 180–196) corresponding to a Mohs hardness of about 2.5. This value is in good agreement with that determined by Spiridonov and Chvileva (1985) for muthmannite from Baia-de-Arieş, Romania (194 kg/mm²).

In plane-polarized incident light muthmannite is grayish white in color, with very low birefractance and pleochroism. When observed near sylvanite it is darker and shows a gray color with a slightly bluish tint. With crossed polars, muthmannite shows distinct anisotropism, similar to petzite, but slightly lower. Internal reflections are absent. No evidence of growth zonation was observed.

Reflectance measurements were performed in air by means of an MPM-200 Zeiss microphotometer equipped with a MSP-20 system processor on a Zeiss Axioplan ore microscope. The filament temperature was approximately 3350 K. An interference filter was adjusted, in turn, to select four wavelengths for measurement (471.1, 548.3, 586.6, and 652.3 nm). Readings were taken for specimen and standard (SiC) maintained under the same focus conditions. The diameter of the circular measuring area was 0.1 mm. Reflectance percentages for R_{\min} and R_{\max} were 40.1, 40.8 (471.1 nm), 38.3, 38.6 (548.3 nm), 37.9, 38.3 (586.6 nm), and 37.7, 38.1 (652.3 nm), respectively.

As made evident in Figure 2, the reflectance percentages obtained for muthmannite from the type locality are very similar to the values measured by Spiridonov and Chvileva (1985) for muthmannite from Baia-de-Arieş, Romania.

X-RAY CRYSTALLOGRAPHY AND CRYSTAL-STRUCTURE DETERMINATION

A small crystal fragment, hand-picked under a reflected light microscope from the polished section, was selected for the X-ray single-crystal diffraction study. Unit-cell parameters, determined by centering 25 high- θ (18–25°) reflections with an automated diffractometer (Nonius-Mach3), are shown in Table 1. In order to determine the real symmetry of the selected crystal, we decided to perform a test data collection of the whole Ewald sphere to $2\theta_{\text{MoK}\alpha} = 30^\circ$. Analysis of the reflections indicated three possible space groups: $P2$, Pm , and $P2/m$. Statistical tests on the distribution of $|E|$ values strongly indicated the presence of an inversion centre, thus suggesting the choice of space group $P2/m$. Intensity data were collected using MoK α radiation monochromatized by a flat graphite crystal in ω scan mode. Intensities were corrected for Lorentz-polarization effects and subsequently for absorption

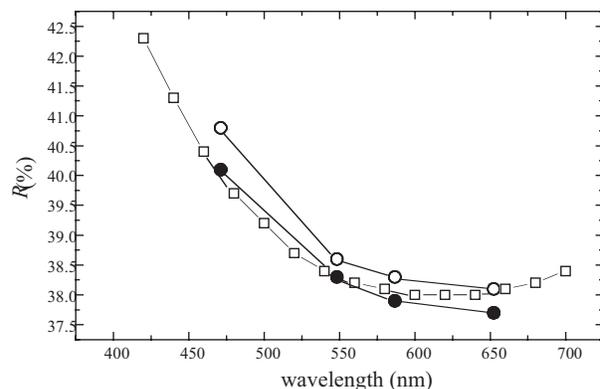


FIGURE 2. Reflectivity curves for muthmannite. Circles refer to muthmannite from the Sacarimb deposit (this study), squares refer to muthmannite from Baia-de-Arieş (Spiridonov and Chvileva 1985). Filled and open symbols refer to R_{\min} and R_{\max} values, respectively.

TABLE 1. Data and experimental details for the selected muthmannite crystal

Space group	$P2/m$ (no. 10)
Cell parameters	$a = 5.124(2)$ (Å) $b = 4.419(1)$ (Å) $c = 7.437(2)$ (Å) $\beta = 89.96(1)$ (°) $V = 168.4(4)$ (Å ³)
Crystal size (μm)	$20 \times 20 \times 30$
Wavelength (32 mA \times 50 kV)	MoK α
Theta-range (°)	1–35
Scan mode	ω
Scan width (°)	2.50
Scan speed (°/min)	1.65
Independent refl.	431
Refl. with $F_o > 4\sigma(F_o)$	386
R_{obs} (%)	5.52
R_{int} (%)	5.59

following the semi-empirical method of North et al. (1968); the values of the equivalent pairs hkl and $h\bar{k}l$ were averaged. The merging R for the ψ -scan data set decreased from 14.34% before the absorption correction to 2.49% after the correction.

The position of Au and Ag atoms was determined from the three-dimensional Patterson synthesis (Sheldrick 1997a), which was performed in the centrosymmetric space group $P2/m$. A least-squares refinement using these heavy-atom positions and isotropic temperature factors yielded an R factor of 10.28%. Three-dimensional difference Fourier synthesis yielded the position of the remaining tellurium atoms. The full-matrix least-squares program SHELXL-97 (Sheldrick 1997b) was used for the refinement of the structure. Introduction of anisotropic temperature factors for all atoms led to $R = 5.52\%$ for 386 observed reflections [$F_o > 4\sigma(F_o)$] and $R = 5.59\%$ for all 431 independent reflections. Neutral scattering curves for Au, Ag, and Te were taken from *The International Tables of X-ray Crystallography*, volume IV (Ibers and Hamilton 1974). Inspection of the difference Fourier map revealed that the maximum positive and negative peaks were 3.41 and 3.44 $e^{-}/\text{Å}^3$, respectively. Experimental details and R indices are given in Table 1. Fractional atomic coordinates and anisotropic-displacement parameters are shown in Table 2. Table 3¹ lists the observed and calculated structure factors.

TABLE 2. Fractional coordinates and anisotropic displacement parameters for muthmannite

	Position	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃	<i>U</i> _{eq}
Au1	1(a)	0	0	0	0.0225(9)	0.0241(9)	0.0234(9)	0	-0.0003(5)	0	0.0233(6)
Au2	1(c)	0	0	½	0.0256(9)	0.0271(9)	0.0267(9)	0	0.0001(6)	0	0.0265(6)
Ag1	1(e)	½	½	0	0.0281(9)	0.0321(9)	0.0308(8)	0	-0.0002(8)	0	0.0303(8)
Ag2	1(h)	½	½	½	0.0291(9)	0.0324(8)	0.0312(9)	0	-0.0007(9)	0	0.0309(9)
Te1	2(m)	0.6277(4)	0	0.2509(3)	0.0255(8)	0.0284(9)	0.0272(9)	0	-0.0003(7)	0	0.0270(6)
Te2	2(n)	0.1089(4)	½	0.2582(3)	0.0282(9)	0.0311(8)	0.0299(9)	0	-0.0002(7)	0	0.0298(7)

CHEMICAL COMPOSITION

The same crystal fragment used for the structural study was analyzed by means of a Jeol JXA-8600 electron microprobe. Major and minor elements were determined at 20 kV accelerating voltage and 40 nA beam current, with variable counting times: 30 s were used for Au, Ag, and Te and 60 s for the minor elements Fe, As, Cu, Bi, Hg, S, Pb, and Sb. For WDS analyses the following lines were used: AuMα, AgLα, TeLα, FeKα, AsLα, CuKα, BiMβ, HgMα, SKα, PbMα, and SbLβ. The estimated analytical precision is ±0.30 for Au, ±0.20 for Ag and Te, ±0.02 for Fe, As, Cu, and Hg, and ±0.01 for Bi, Pb, S, and Sb. The standards employed were Au (Au), Ag (Ag), synthetic Sb₂Te₃ (Sb, Te), marcasite (Fe), synthetic GaAs (As), Cu (Cu), Bi (Bi), coloradoite (Hg), and galena (Pb, S). The crystal fragment was found to be homogeneous within the analytical error. The average chemical composition (15 analyses on different spots), together with ranges of wt% of elements, is reported in Table 4. On the basis of 4 atoms, the formula of muthmannite is Au_{0.97}Ag_{0.99}Te_{2.03}, ideally AuAgTe₂.

DESCRIPTION OF THE STRUCTURE AND DISCUSSION

The structure of muthmannite is simple and densely packed. It is based on the NiAs-type structure, with a distorted hexagonal closest-packed array of Te²⁻ anions with Au³⁺ and Ag⁺ cations occupying all the octahedral sites (Fig. 3). The structure can be divided into two slabs: a gold slab (sheet at *x* = 0, Au1 and Au2 atoms) and a silver slab (sheet at *x* = 0.5, Ag1 and Ag2 atoms). In the gold slab, both Au1 and Au2 form edge-sharing octahedral chains parallel to *b*, and each Au1 shares two faces with the Au2 atoms making chains parallel to *c*, thus forming sheets parallel to (100) (Fig. 4a). In the same way, in the silver slab, both Ag1 and Ag2 form edge-sharing octahedral chains parallel to *b*, and

TABLE 4. Electron microprobe analyses (means and ranges in wt%, based on 15 analyses) for muthmannite

Element	wt%	Range
Pb	0.01	0.01–0.03
Au	34.21	35.41–34.12
Ag	19.06	19.88–18.79
As	0.02	0.02–0.04
Cu	0.00	0.00–0.01
Hg	0.00	0.00–0.01
Fe	0.01	0.01–0.02
Sb	0.00	0.00–0.01
Bi	0.01	0.00–0.02
Te	46.41	47.12–46.01
S	0.01	0.00–0.02
Total	99.74	

¹For a copy of Table 3, document item AM-04-072, contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. Deposit items may also be available on the American Mineralogist web site at <http://www.minsocam.org>.

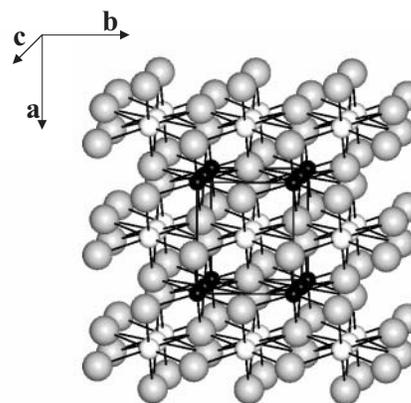
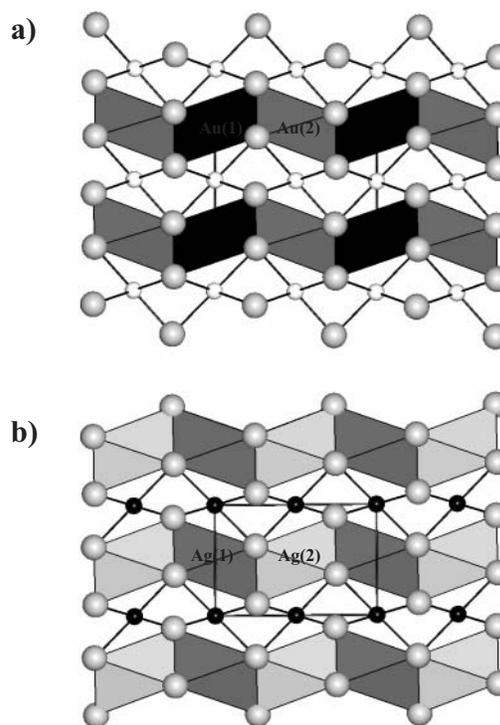

FIGURE 3. The crystal structure of muthmannite. Black, white, and gray circles refer to the Au, Ag, and Te atoms, respectively. The unit cell and the orientation of the structure are outlined.

FIGURE 4. The structure of muthmannite projected down [010]. (a) Gold slab containing Au1 and Au2 (see text). (b) Silver slab containing Ag1 and Ag2 (see text). Symbols as in Figure 3. The unit cell is outlined.

TABLE 5. Selected bond distances (Å) and interatomic angles (°) for muthmannite

Au1i - Te1ii,xiv	2.668(2)	Te1ii - Au1i - Te1xiv	180.00
- Te2i,iii,xii,xiii	2.980(2)	Te1ii - Au1i - Te2i,iii	71.54(5)
Au1i - Ag1i,ii,iii,iv	3.383(3)	Te1xiv - Au1i - Te2xii,xiii	71.54(5)
- Au2i,v	3.718(2)	Te1ii - Au1i - Te2xii,xiii	108.46(5)
Au1i - Au1iii,vi	4.419(3)	Te1xiv - Au1i - Te2i,iii	108.46(5)
		Te2i - Au1i - Te2xiii	180.00
		Te2i - Au1i - Te2iii	95.70(6)
		Te2xii - Au1i - Te2xiii	95.70(6)
		Te2i - Au1i - Te2xii	84.30(6)
		Te2iii - Au1i - Te2xiii	84.30(6)
		Te2iii - Au1i - Te2xii	180.00
Au2i - Te1ii,xviii	2.660(2)	Te1ii - Au2i - Te1xviii	180.00
- Te2i,iv,v,vi	2.903(2)	Te1ii - Au2i - Te2i,iii	72.91(5)
Au2i - Ag2i,iii,xv,xvi	3.383(3)	Te1xviii - Au2i - Te2xv,xvi	72.91(5)
- Au1i,vii	3.718(2)	Te1ii - Au2i - Te2xv,xvi	107.09(5)
Au2i - Au2iii,vi	4.419(3)	Te1xviii - Au2i - Te2i,iii	107.09(5)
		Te2i - Au2i - Te2xvi	180.00
		Te2i - Au2i - Te2iii	99.14(6)
		Te2xv - Au2i - Te2xvi	99.14(6)
		Te2i - Au2i - Te2xv	80.87(6)
		Te2iii - Au2i - Te2xvi	80.87(6)
		Te2iii - Au2i - Te2xv	180.00
Ag1i - Te2i,xiv	2.775(2)	Te2i - Ag1i - Te2xiv	180.00
- Te1i,vi,xiv,xvii	2.965(1)	Te2i - Ag1i - Te1i,vi	73.98(5)
Ag1i - Au1i,vi,viii,ix	3.383(3)	Te2xiv - Ag1i - Te1xiv,xvii	73.98(5)
- Ag2i,v	3.718(2)	Te2i - Ag1i - Te1xiv,xvii	106.02(5)
Ag1i - Ag1iii,vi	4.419(3)	Te2xiv - Ag1i - Te1i,vi	106.02(5)
		Te1vi - Ag1i - Te1xiv	180.00
		Te1vi - Ag1i - Te1i	96.34(6)
		Te1xvii - Ag1i - Te1xiv	96.34(6)
		Te1vi - Ag1i - Te1xvii	83.66(6)
		Te1i - Ag1i - Te1xiv	83.66(6)
		Te1i - Ag1i - Te1xvii	180.00
Ag2i - Te2i,xviii	2.693(2)	Te2i - Ag2i - Te2xviii	180.00
- Te1i,vi,xviii,xix	2.956(1)	Te2i - Ag2i - Te1i,vi	75.29(5)
Ag2i - Au2i,vi,viii,ix	3.383(3)	Te2xviii - Ag2i - Te1xviii,xix	75.29(5)
- Ag1i,vii	3.718(2)	Te2i - Ag2i - Te1xviii,xix	104.71(5)
Ag2i - Ag2iii,vi	4.419(3)	Te2xviii - Ag2i - Te1i,vi	104.71(5)
		Te1vi - Ag2i - Te1xviii	180.00
		Te1vi - Ag2i - Te1i	96.72(6)
		Te1xix - Ag2i - Te1xviii	96.72(6)
		Te1vi - Ag2i - Te1xix	83.28(6)
		Te1i - Ag2i - Te1xviii	83.28(6)
		Te1i - Ag2i - Te1xix	180.00
Te1i - Te2ix,x	3.311(2)	Te2ix - Te1i - Te2x	83.71(5)
- Te2i,iii	3.457(2)	Te2ix - Te1i - Te2i	98.39(6)
- Te1xviii	3.929(2)	Te2x - Te1i - Te2iii	98.39(5)
- Te1xiv	3.956(2)	Te2ix - Te1i - Te1xviii	103.42(5)
Te2i - Te2xv	3.765(2)	Te2x - Te1i - Te1xviii	103.42(5)
- Te2xii	4.000(3)	Te1xviii - Te1i - Te1xiv	141.22(6)
		Te1vi - Te2i - Te2xv	104.03(5)
		Te1i - Te2i - Te2xv	104.03(5)
		Te2xv - Te2i - Te2xii	146.56(6)

Note: Symmetry codes are: (i): x, y, z; (ii): -1 + x, y, z; (iii): x, -1 + y, z; (iv): -1 + x, -1 + y, z; (v): x, y, -1 + z; (vi): x, 1 + y, z; (vii): x, y, 1 + z; (viii): 1 + x, 1 + y, z; (ix): 1 + x, y, z; (x): 1 + x, -1 + y, z; (xi): -1 + x, 1 + y, z; (xii): -x, y, -z; (xiii): -x, -1 + y, -z; (xiv): 1 - x, y, -z; (xv): -x, y, 1 - z; (xvi): -x, -1 + y, 1 - z; (xvii): 1 - x, 1 + y, -z; (xviii): 1 - x, y, 1 - z; (xix): 1 - x, 1 + y, 1 - z.

each Ag1 shares two faces with the Ag2 atoms making chains parallel to c, thus forming sheets parallel to (100) (Fig. 4b). In addition, edge-sharing of Au1 (Au2) with Ag1 (Ag2) and corner-sharing of Au1 (Au2) with Ag2 (Ag1) provides connectivity between the two slabs.

Selected bond distance and angles are given in Table 5. The structural model for muthmannite presented in this work strongly resembles the basic structure of calaverite (Pertlik 1984a; Schutte and de Boer 1988; Reithmayer et al. 1993). The main difference between the two structures is that in calaverite the sheets of AuTe₆

octahedra are linked by Te-Te bonds, whereas in muthmannite the AuTe₆ octahedra are connected by sheets of AgTe₆ octahedra. However, the crystal-chemical environment of noble metals is very similar in both structures. In calaverite, indeed, the Au atoms show a [2+4] coordination with Au-Te distances ranging from 2.675 to 2.970 Å (Reithmayer et al. 1993). These values are in excellent agreement with those found for Au1 and Au2 in muthmannite (Au1: 2.668–2.980 Å; Au2: 2.660–2.903 Å). On the other hand, the Au atoms in the crystal structure of krennerite (AuTe₂; Pertlik 1984b) and sylvanite (AgAuTe₄; Pertlik 1984c), show a [4+2] coordination with bond lengths ranging from 2.70–3.40 Å and 2.68–3.33 Å, respectively. Owing to the greater size of Ag with respect to Au, the AgTe₆ octahedron observed in muthmannite is slightly larger. It shows a [2+4] coordination with Ag-Te distances ranging from 2.775 to 2.965 Å for Ag1 and 2.693 to 2.956 Å for Ag2. These values are shorter than that found for the Ag position in the crystal structure of sylvanite (2.74–3.23 Å; Pertlik 1984c). The shortest Te-Te contacts are in the range 3.309–3.459 Å.

An X-ray powder pattern calculated using the structural data is shown in Table 6. It appears completely different from that reported by Spiridonov and Chvileva (1985) for muthmannite from Baia-de-Arieş, Romania. Possible reasons could be the small amount of material (very small grain size) used by these authors (Fig. 1 of their paper) and the complex intergrowths with calaverite.

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TABLE 6. Calculated X-ray powder diffraction pattern for muthmannite

hkl	d _{calc} (Å)	I/I _{calc}
100	5.124	11
010	4.419	100
110	3.346	93
012	2.845	15
200	2.562	24
112	2.488	38
112	2.487	34
201	2.423	11
201	2.422	10
210	2.216	3
020	2.210	43
211	2.125	14
211	2.124	14
202	2.110	11
202	2.109	9
004	1.859	24
203	1.782	4
203	1.781	4
014	1.714	13
220	1.673	12
213	1.653	6
213	1.652	6
221	1.633	6
221	1.632	6
114	1.626	9
114	1.625	11
310	1.593	11
222	1.526	7
222	1.525	6
204	1.505	3
204	1.504	5
030	1.473	4
312	1.465	5
312	1.464	5
024	1.423	18
130	1.416	6
223	1.387	3

Note: d values calculated on the basis of a = 5.124(2), b = 4.419(1), and c = 7.437(2) Å, β = 89.96(1)°. Intensities calculated using XPOW software version 2.0 (Downs et al. 1993). Only reflections with I/I_{calc} > 3 are listed.

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