Criddleite, $TlAg_2Au_3Sb_{10}S_{10}$, a new gold-bearing mineral from Hemlo, Ontario, Canada[1]

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Abstract

Criddleite, ideally $TlAg_2Au_3Sb_{10}S_{10}$, is a rare constituent within the Hemlo gold deposit, Hemlo, Ontario, Canada. The mineral occurs as 20 to 50 μm-sized lath-like, tabular or anhedral grains usually surrounding or penetrating aurostibite, or associated with native antimony, native gold and stibnite. Criddleite is opaque with a metallic lustre and a black streak. It has been synthesized by reacting $TlSbS_2$ and high purity Ag, Au, Sb and S in an evacuated silica glass tube at 400 °C. The measured density of the synthetic material is 6.86; the calculated density is 6.57 g/cm^3. The difference is due to minor admixed aurostibite, native antimony and a dyscrasite-like phase within the charge. VHN_{25} is 94–129. Mohs hardness (calc.) = $3-3\frac{1}{2}$. In reflected plane-polarized light in air, natural criddleite is weakly bireflectant with a discernible reflectance pleochroism from grey-blue to slightly greenish grey-blue. The mineral has a distinct to moderate anisotropy with rotation tints in shades of buff to slate grey. Reflectance spectra and colour values for both natural and synthetic criddleite are given. X-ray study showed that synthetic criddleite is monoclinic (pseudotetragonal) with refined unit-cell parameters $a = 20.015(2)$, $b = 8.075(2)$, $c = 7.831(2)$ Å, $\beta = 92.01(2)°$, $V = 1264.9 \pm 1.0$ Å^3 and $a:b:c = 2.4786:1:0.9698$. The space group choices are $A2/m(12)$, $A2(5)$ or $Am(8)$, diffraction aspect $A*/*$. The seven strongest lines in the X-ray powder diffraction pattern [d in Å (I) (hkl)] are: 5.63(90) (011), 3.91(50) (002), 3.456(50) (320), 2.860(70) (700), 2.813(100) (022), 2.018(60) (040) and 1.959(70) (004). Electron microprobe analyses are reported of natural criddleite in five polished sections of drill core from four holes. The averaged empirical formulae, based on 26 atoms, are $Tl_{0.92}Ag_{1.99}Au_{2.93}Sb_{9.87}S_{10.28}$ (natural) and $Tl_{0.94}Ag_{2.03}Au_{2.89}Sb_{9.76}S_{10.38}$ (synthetic).

KEYWORDS: criddleite, new mineral, Hemlo gold deposit, Hemlo, Ontario, Canada, reflectance data, X-ray data, electron microprobe analyses, synthesis.

Introduction

CRIDDLEITE, ideally $TlAg_2Au_3Sb_{10}S_{10}$, is a newly described gold-bearing mineral from the Hemlo gold deposit, Hemlo, Ontario, Canada. The mineral is named for Alan J. Criddle (1944–), of the British Museum (Natural History), London, England, for his substantial contributions to the quantitative reflectance of ore minerals and for his research on opaque minerals. The mineral and the mineral name have been approved by the Commission on New Minerals and Mineral Names, International Mineralogical Association. Type material is preserved at: the British Museum (Natural History) as polished section E.1230, BM 1987,351; within the Systematic Reference Series of the National Mineral Collection, housed at the Geological Survey of Canada, Ottawa, Ontario,

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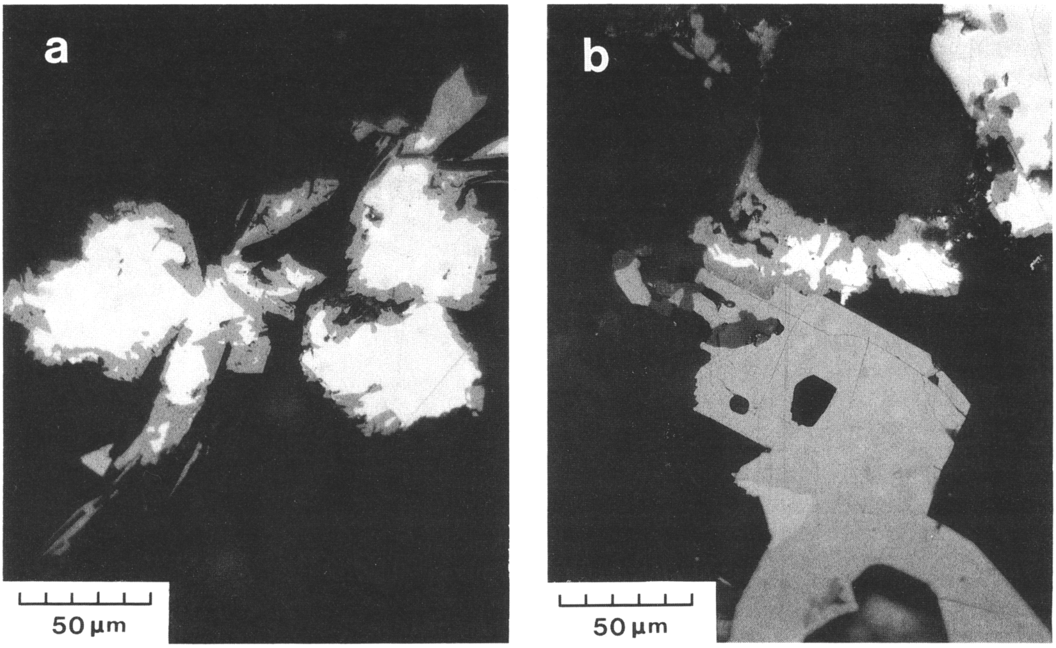

FIG. 1. (*a*) Photomicrograph of criddleite (grey) surrounding and penetrating aurostibite (white) within quartz gangue. Oil immersion. Polished section GG23, 651.6. (*b*) Photomicrograph of criddleite surrounding and partially replacing aurostibite (white) in contact with a coarse grain of chalcostibite (light grey) within quartz gangue. Oil immersion. Polished section GG23, 651.6.

Canada as a polished section, and as drill core specimens under catalogue number NMC 65186; and as a polished section at the Royal Ontario Museum, Toronto, Ontario, Canada.

Occurrence

The Hemlo gold deposit is located near the northeast shore of Lake Superior, 35 km east of Marathon, Ontario, Canada, adjacent to the Trans-Canada Highway # 17, at latitude 48° 40″ N and longitude 86° 00″ W. The deposit was discovered in 1982 and, within the last few years, three mines have been brought into production. They are, from east to west, the David Bell mine, the Golden Giant mine and the Page–Williams mine. In 1983, a mineralogical study of the deposit was initiated and preliminary reports on the mineralogy have been published by Harris (1986*a*, *b*, *c*). The gold deposit is substantially enriched in Mo, Sb, As, Hg, Tl, V and Ba and contains a diverse assemblage of minerals. The most common ore minerals are pyrite, molybdenite, stibnite, tetrahedrite-tennantite, native gold (containing up to 22 wt. % Hg), sphalerite (containing up to 27 wt. % Hg), zinkenite, realgar, arsenopyrite, cinnabar and aktashite. Minor-to-trace ore minerals are aurostibite, boulangerite, bournonite, twinnite, geocronite, chalcostibite, stibarsen, native arsenic, native antimony, arsenopyrite, orpiment, berthierite, routhierite, parapierrotite, dufrenoysite, baumhauerite, gersdorffite, ullmannite, barian tomichite, magnetite, ilmenite and chromite. The gangue minerals are principally quartz, baryte, sericite, vanadian muscovite (containing up to 8.5 wt. % V_2O_3), rutile (with as much as 5.6 wt. % V_2O_3, 6.5 wt. % Sb_2O_3, 2.3 wt. % WO_3) and barian microcline (containing up to 16.6 wt. % BaO).

Criddleite is very rare within the deposit and has been found in polished sections of drill core specimens from only four holes: GG23, 651.6 m; GG25, 324.9 m; W70, 758.6 m and W92, 642.9 m. In polished section GG23, 651.6 m, it occurs as 40 μm-sized anhedral rims around aurostibite (Fig. 1*a* and *b*), associated with chalcostibite and parapierrotite. In polished section GG25, 324.9 m, it occurs as an intergrowth with native gold and aurostibite in gangue. The largest composite grain is 20×60 μm. In polished section W70, 758.6 m, it occurs as an intergrowth with native antimony in gangue. The largest composite grain

Table 1. Electron microprobe analyses of criddleite

Polished Section	Tl	Ag	Au	Sb	S	Total
GG23, 651.6	7.1	8.8	23.1	48.2	13.1	100.3
GG23, 651.6 – 2a	7.8	8.7	22.8	47.9	13.3	100.5
GG25, 324.9	7.5	8.7	23.0	47.4	13.1	99.7
W92, 642.9	7.5	8.7	23.2	47.5	13.1	100.0
W70, 758.6	7.4	7.8	22.3	47.3	12.8	97.6
Atomic proportions total = 26						
GG23, 651.6	0.87	2.04	2.94	9.92	10.23	
GG23, 651.6 – 2a	0.95	2.01	2.89	9.81	10.34	
GG25, 324.9	0.92	2.03	2.94	9.81	10.29	
W92, 642.9	0.92	2.03	2.96	9.81	10.28	
W70, 758.6	0.93	1.86	2.91	10.01	10.28	
Average	0.92	1.99	2.93	9.87	10.28	

is 30 × 70 μm. Other ore minerals observed in this polished section are native gold, stibnite, molybdenite, aurostibite, zinkenite and pyrite. In polished section W92, 642.9 m, it occurs as 20 μm-sized grains in gangue associated with, but not in contact with, stibnite, tetrahedrite, native antimony and aurostibite. In all sections, criddleite commonly forms lath-like or anhedral grains and is a primary mineral formed from the same hydrothermal fluids that have resulted in the diverse assemblage of ore minerals.

Electron microprobe analyses

The quantitative analyses were performed with a CAMEBAX electron microprobe operated at 20 kV and with a beam current of 30 nA. The analytical results are given in Table 1. The X-ray lines and standards used were Au-*Mα*, Ag-*Lα* (synthetic $Au_{0.678}Ag_{0.322}$); Tl-*Mα*, Sb-*Lα*, S-*Kα* (synthetic $TlSbS_2$). The averaged empirical formula of the five analyses, based on 26 atoms, is $Tl_{0.92}Ag_{1.99}Au_{2.93}Sb_{9.87}S_{10.28}$ or, ideally, $TlAg_2Au_3Sb_{10}S_{10}$.

Synthesis

Owing to the small size of the natural criddleite grains, syntheses were undertaken: (*a*) to verify the composition of the natural material as determined by the electron microprobe analyses, (*b*) to obtain synthetic material suitable for single-crystal X-ray studies, and (*c*) to use the synthetic material as a potential electron microprobe standard. A 200 mg charge, equivalent to the formula $TlAg_2Au_3Sb_{10}S_{10}$, was synthesized by reacting $TlSbS_2$ and high-purity Au, Ag, Sb and S in an evacuated silica glass tube. The starting materials were heated at 350 °C for three days and then heated at 400 °C for an additional three days. The charge was then examined megascopically and, although it appeared to have melted, it did not appear homogeneous; a small amount of unreacted material (Au?) was present on the surface of the charge. It was, therefore, decided to increase the temperature to 450 °C and examination of the charge after one day at this higher temperature indicated a more complete reaction. Thereafter, the charge was annealed for a period of two months at 400 °C and then subjected to grinding, pelletizing and reannealing at 400 °C for an additional period of two months. This synthesis was successful because microscopic and electron microprobe examination of a polished section containing a portion of the synthetic charge showed approximately 96% criddleite, 2% aurostibite, 1% native antimony and 1% of a dyscrasite-like phase which has the composition Ag 35.2, Au 44.0, Sb 22.8 wt. %.

Electron microprobe analyses of synthetic criddleite, using the same standards and operating conditions that were applied to the analyses of the natural grains, gave Tl 7.7, Ag 8.8, Au 22.9, Sb 47.8, S 13.4, total 100.6 wt. %, which corresponds to $Tl_{0.94}Ag_{2.03}Au_{2.89}Sb_{9.76}S_{10.38}$. Subtracting the extra phases from the starting composition of Tl 8.0, Ag 8.5, Au 23.2, Sb 47.7, S 12.6, total 100.0 wt. %, gives a calculated composition for the synthetic criddleite of Tl 8.3, Ag 8.5, Au 22.7, Sb 47.5, S 13.0, total 100.0 wt. %, in reasonable agreement with the electron microprobe results.

Optical properties

The polished sections containing the natural criddleite and the synthetic equivalent were buffed with a paste of MgO in distilled water on Lamplan 'velvet' immediately before making observations and measurements.

Criddleite takes an excellent polish, although its polishing hardness is low. In reflected plane-polarized light (colour temperature ~ 3200 K) in air it is weakly bireflectant with a discernible reflectance pleochroism from grey-blue to slightly greenish grey-blue. In oil, this subtle reflectance pleochroism is enhanced. Criddleite has a distinct to moderate anisotropy with rotation tints in shades of buff to slate grey. Simple twinning is common. The synthetic criddleite has identical optical characteristics but, in addition to simple twinning, polysynthetic twinning is also common.

Reflectance measurements were made relative to a WTiC standard (Zeiss no. 314) at intervals of 10 nm from 400 to 700 nm in air and in oil (Zeiss N_D 1.515, DIN 58.884) at 21 °C. A plane glass

TABLE 2. Reflectance data and colour values in air and oil for natural and synthetic criddleite.

	GG 23 651.6		*synthetic*		*GG 23 651.6*		*synthetic*	
λ*nm*	R_1	R_2	R_1	R_2	${}^{im}R_1$	${}^{im}R_2$	${}^{im}R_1$	${}^{im}R_2$
400	40.6	41.85	41.4	42.5	26.3	28.3	27.0	29.8
410	40.4	41.4	41.05	42.1	25.95	27.5	26.6	28.75
420	40.1	41.0	40.7	41.75	25.4	26.7	26.15	27.9
430	39.7	40.5	40.15	41.3	25.1	26.2	25.7	27.2
440	39.4	40.1	39.7	40.9	24.7	25.9	25.2	26.5
450	39.0	39.8	39.3	40.55	24.4	25.6	24.7	26.0
460	38.6	39.4	39.0	40.25	24.1	25.4	24.3	25.7
470	38.4	39.3	38.6	39.9	23.8	25.2	23.9	25.5
480	38.1	39.2	38.2	39.75	23.6	25.1	23.6	25.5
490	37.9	39.2	37.9	39.65	23.3	25.0	23.3	25.45
500	37.65	39.1	37.6	39.6	23.0	24.8	23.1	25.4
510	37.5	39.0	37.5	39.6	22.8	24.7	22.9	25.3
520	37.3	38.9	37.2	39.45	22.6	24.6	22.6	25.1
530	37.0	38.7	37.0	39.3	22.3	24.3	22.35	24.9
540	36.7	38.4	36.8	39.1	22.1	24.2	22.2	24.6
550	36.4	38.2	36.4	38.9	21.8	23.9	21.95	24.4
560	36.1	38.0	36.15	38.8	21.6	23.7	21.7	24.2
570	35.9	37.9	35.8	38.55	21.3	23.5	21.4	24.0
580	35.55	37.65	35.6	38.4	21.1	23.3	21.1	23.8
590	35.3	37.4	35.2	38.1	20.85	23.1	20.8	23.6
600	35.0	37.2	34.8	37.8	20.6	22.9	20.5	23.3
610	34.7	36.95	34.45	37.5	20.35	22.6	20.2	23.0
620	34.35	36.6	34.1	37.2	20.0	22.3	19.85	22.6
630	34.0	36.3	33.7	36.8	19.8	22.0	19.5	22.2
640	33.7	36.0	33.3	36.3	19.6	21.7	19.2	21.8
650	33.4	35.6	33.0	36.0	19.3	21.4	18.9	21.4
660	33.1	35.3	32.7	35.6	19.1	21.1	18.7	21.1
670	32.8	35.0	32.45	35.1	18.9	20.75	18.5	20.8
680	32.65	34.6	32.3	34.8	18.9	20.5	18.5	20.4
690	32.6	34.4	32.3	34.5	18.8	20.3	18.5	20.25
700	32.5	34.1	32.35	34.3	18.9	20.1	18.6	20.05
COLOUR VALUES								
Illuminant C								
x	.300	.303	.299	.303	.295	.299	.293	.298
y	.308	.312	.307	.312	.303	.308	.302	.308
Y%	36.2	38.0	36.1	38.7	21.6	23.7	21.7	24.2
λ_d	481	483	481	482	480	482	480	482
P_e%	4.5	3.0	5.0	3.0	7.0	4.9	7.7	5.1
Illuminant A								
x	.437	.440	.436	.440	.432	.436	.430	.435
y	.407	.408	.406	.408	.405	.407	.405	.407
Y%	35.8	37.7	35.7	38.3	21.3	23.4	21.3	23.9
λ_d	493	495	493	495	492	494	492	495
P_e%	2.5	1.8	2.8	1.8	3.8	2.9	4.2	3.0

reflector was used with × 40 objectives, the effective numerical apertures of which were adjusted to 0.35. As the extinction positions could not be established with certainty, the maximum and minimum positions for the measured grains were determined photometrically at 560 nm. Otherwise, the equipment used and the measurement procedures were as described by Criddle *et al.* (1983).

Owing to the small grain size and paucity of natural material, the measurements (Table 2 and Fig. 2) were obtained on a single lath-shaped (25 × 15 μm) grain included in aurostibite in polished section GG23, 651.6. Other grains of criddleite were examined but gave less reliable results because of twinning or because of difficulties encountered in levelling grains adjacent to the much harder quartz gangue. Check measurements on these grains, however, showed no significant differences in either the magnitude or dispersion of the spectra.

The most strongly anisotropic and bireflectant grain of the synthetic preparation was selected for measurement. Table 2 shows that the differences in both reflectance and bireflectance between this and the most bireflectant grain in section GG23, 651.6 are minimal. All spectra in Fig. 2 were smoothed graphically between 400 and 450 nm.

The mineral is monoclinic and, because the grains could not be oriented, the reflectance spectra are labelled R_1 and R_2 (Criddle, 1980). One sectioned grain of the synthetic mineral is isotropic, and its measured reflectance R_0 is very close to R_1.

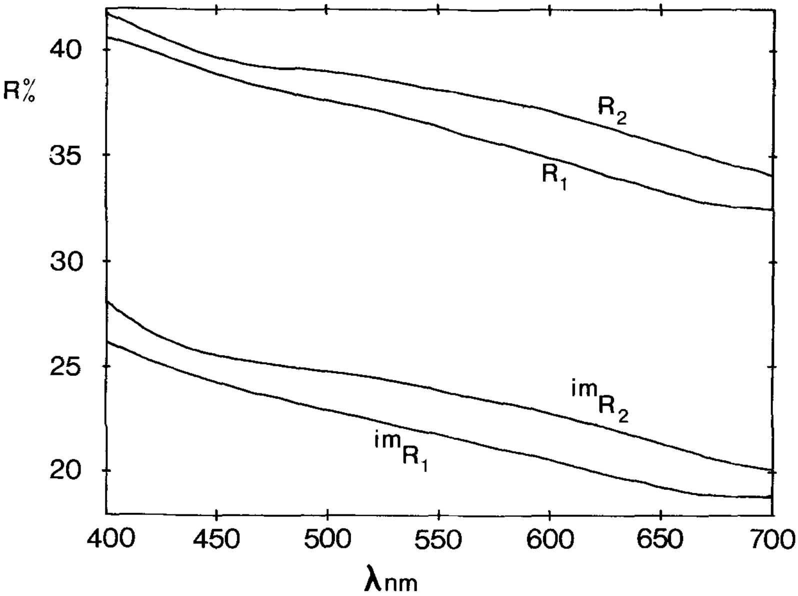

FIG. 2. Reflectance spectra for criddleite in air and in oil. (Specimen GG23, 651.6.)

It follows that the mineral is effectively uniaxial, as well as being pseudotetragonal.

Dominant wavelength values (λd in Table 2) for the CIE A illuminant (close to the colour temperature of the light source used for the qualitative description) confirm the overall grey-blue colour of the mineral. The perceived slightly more greenish grey-blue hue for R_2 is a function of its lower excitation purity and slightly higher luminance (Y%) compared with R_1.

Microhardness and physical properties

Synthetic criddleite has a microhardness VHN_{25} in the range 94–129 based on 10 indentations. This corresponds to a calculated Mohs hardness of 3–3½. In shape, these indentations were perfect to slightly deformed with minor fracturing at the corners. The grain size of the natural material was too small for VHN measurements. The mineral is opaque with a metallic lustre and a black streak.

X-ray powder and single-crystal study

The small grain size and twinning characteristics of natural criddleite prevented extraction of a suitable fragment from a polished section for precession single crystal study. However, a 57.3 mm Debye-Scherrer powder film of natural criddleite showed a sufficient number of observable diffraction lines for comparison to be made with a powder pattern of synthetic material obtained with the same diameter camera; the two patterns were for one and the same phase. The measurable reflections for natural criddleite (in Å), 5.66 (weak), 2.85 (strong), 2.81 (strong), 2.02 (medium) and 1.96 (medium) are consistent with the strongest reflections presented in Table 3 for synthetic material. Other weak reflections were identified but, because of the spotty nature of the film, were unmeasurable. Therefore, two fragments of synthetic material were mounted and studied by precession single-crystal methods employing Zr-filtered Mo radiation. One fragment was orientated such that a^* was coincident with the dial axis and the other fragment mounted with 011^* coincident with the dial axis. The levels collected were: $hk0 \rightarrow hk2$, $h0l \rightarrow h2l$, $0kl \rightarrow 4kl$ and $011^* \wedge a^*$. No evidence of twinning was observed on the precession single-crystal films. Criddleite is pseudotetragonal, monoclinic with measured (and calculated) cell parameters derived from zero-level precession films of: $a = 19.96$, $b = 8.057$, $c = 7.809$ Å, $\beta = 92.08°$. The space-group absence conditions; hkl with $k+l \neq 2n$, $hk0$ with $k \neq 2n$, $h0l$ with $l \neq 2n$, $0kl$ with $k+l \neq 2n$ and $0k0$ with $k \neq 2n$, indicate that $A2/m$ (12), $A2$ (5) or Am (8) are the permissible space-group choices. The diffraction aspect is $A^*/*$. A fully indexed Guinier–DeWolff powder pattern of synthetic material is presented in Table 3. Admixed end-member aurostibite was used as an internal standard.

Table 3. X-ray powder data for synthetic criddleite

Iest.	dÅmeas.	dÅcalc.	*hkl*
5	10.01	10.00	200
30	6.67	6.67	300
90	5.63	5.62	011
10	5.45	5.45	$\bar{1}11$
40	5.00	5.00	400
5	4.94	4.95	$\bar{2}11$
20	4.85	4.85	211
30	4.34	4.35	$\bar{3}11$
40	4.25	4.25	311
40	4.03	4.04	020
15	4.00	4.00	500
3	3.95	3.96	120
50	3.91	3.91	002
1	3.75	3.74	220
10	3.69	3.69	$\bar{2}02$
50	3.456	3.454	320
10	3.431	3.428	$\bar{3}02$
10	3.331	3.334	600
		3.324	302
1	3.298	3.299	$\bar{5}11$
3	3.225	3.221	511
5	3.142	3.141	420
5	2.896	2.899	$\bar{6}11$
70	2.860	2.858	700
100	2.813	2.810	022
3	2.774	2.773	122
1	2.689	2.688	222
3	2.612	2.613	$\bar{3}22$
30	2.569	2.573	$\bar{7}11$
		2.566	322
40	2.545	2.545	031
15	2.522	2.522	711
10	2.500	2.500	800
30	2.480	2.482	013
20	2.473	2.476	$\bar{4}22$
		2.474	$\bar{1}13$
		2.473	$\bar{2}31$
3	2.426	2.424	422
3	2.392	2.391	213
5	2.385	2.387	$\bar{3}31$
10	2.366	2.369	331
3	2.351	2.352	$\bar{3}13$
1	2.329	2.332	720
		2.327	$\bar{5}22$
5	2.305	2.302	313
3	2.278	2.273	522
5	2.225	2.223	900
10	2.125	2.126	820
		2.123	622
30	2.030	2.029	$\bar{7}22$
60	2.018	2.019	040
3	2.001	2.000	$\underline{10}00$
35	1.980	1.979	722
70	1.959	1.957	004
1	1.932	1.932	340
5b	1.909	1.911	$\bar{7}31$
		1.906	$\bar{7}13$
10	1.891	1.890	731
10	1.873	1.872	440
10	1.847	1.844	$\bar{4}04$
		1.844	713
1	1.815	1.815	$\bar{3}33$
1	1.802	1.802	540
15	1.793	1.792	$\underline{10}20$
		1.792	333
10	1.761	1.765	$\bar{9}22$
		1.761	024
10b	1.741	1.739	$\bar{3}42$
10	1.726	1.725	342
10	1.715	1.716	$\bar{3}24$
3	1.697	1.697	$\bar{4}42$
3	1.689	1.689	324
3	1.679	1.680	533
		1.680	442
		1.677	$\bar{4}24$
1	1.662	1.662	604
30	1.648	1.649	740
5	1.640	1.641	$\bar{7}04$
3b	1.610	1.611	$\underline{10}22$
5	1.588	1.589	704
5	1.580	1.585	$\bar{7}33$
		1.582	051
3	1.567	1.568	$\bar{8}04$
3	1.548	1.549	733
10	1.536	1.537	015
5	1.486	1.486	315
15	1.425	1.429	$\underline{14}00$
		1.421	$\underline{10}40$
3	1.415	1.414	$\bar{6}15$
40	1.399	1.405	044
		1.404	$\bar{1}44$
		1.388	$\bar{7}51$

- Guiner-DeWolff camera employing Fe filtered Co radiation (λ Co Kα_1 = 1.78892 Å)
- b = broad line
- intensities estimated visually
- pattern run at CANMET by Mr P. Carrière
- pattern corrected for shrinkage
- indexed on a = 20.015, b = 8.075, c = 7.831 Å, β = 92.01°

Powder lines solely ascribable to admixed aurostibite, the dyscrasite-like phase and native antimony have been deleted. The refined unit-cell parameters for synthetic criddleite, based on 36 reflections between 4.04 and 1.548 Å for which unambiguous indexing, aided by visual inspection of precession single-crystal films, was possible, are: $a = 20.015(6)$, $b = 8.075(2)$, $c = 7.831(2)$ Å, $\beta = 92.01(2)°$, $V = 1264.9 \pm 1.0$ Å^3 and $a{:}b{:}c = 2.4786{:}1{:}0.9698$. With Z = 2, the calculated density for the synthetic empirical formula is 6.57 g/cm^3, about 4.4% lower than the measured Berman balance density of 6.86(7) g/cm^3. The difference is ascribable to minor admixed aurostibite, a dyscrasite-like phase and native antimony in the synthetic charge.

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