

**TERLINGUACREEKITE, $\text{Hg}^{2+}_3\text{O}_2\text{Cl}_2$, A NEW MINERAL SPECIES
FROM THE PERRY PIT, MARIPOSA MINE, TERLINGUA MINING DISTRICT,
BREWSTER COUNTY, TEXAS, U.S.A.**

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ABSTRACT

Terlinguacreekite, ideally $\text{Hg}^{2+}_3\text{O}_2\text{Cl}_2$, has a very pronounced subcell that is orthorhombic, space-group choices *Imam*, *Imcm*, *Ima2* and *I2cm*, with unit-cell parameters refined from powder data: *a* 6.737(3), *b* 25.528(10), *c* 5.533(2) Å, *V* 951.6(6) Å³, *a*:*b*:*c* 0.2639:1:0.2167, *Z* = 8. The true symmetry, supercell unit-cell parameters, and details regarding the crystal structure are unknown. The strongest nine lines of the X-ray powder-diffraction pattern [*d* in Å (*I*(subcell *hkl*))] are: 5.413(30)(011), 4.063(80)(121), 3.201(50)(080), 3.023(50)(161), 2.983(60)(240), 2.858(30)(211), 2.765(50)(002), 2.518(100b)(091, 251) and 2.026(30)(242). The mineral is found in an isolated area measuring approximately 1 × 0.5 m in the lower level of the Perry pit, Mariposa mine, Terlingua mining district, Brewster County, Texas (type locality), as mm-sized anhedral dark orange to reddish orange crusts of variable thickness on calcite, and rarely as 0.5 mm-sized aggregates of crystals of the same color. It has also been identified at the McDermitt mine, Humboldt County, Nevada, U.S.A., where it occurs with kleinite and calomel in silicified volcanic rocks and sediments. Terlinguacreekite is a secondary phase, most probably formed from the alteration of primary cinnabar or native mercury. At Terlingua, most crusts are thin, almost cryptocrystalline, with no discernable forms, and are resinous and translucent to opaque. Crystals are up to 0.2 mm in length, subhedral, acicular to prismatic, elongation [001], with a maximum length-to-width ratio of 4:1. They are vitreous, transparent, and some crystals have brightly reflecting faces, which may be {010} and {110}. The streak is yellow, and the mineral is brittle with an uneven fracture, no observable cleavage, and is soft, nonfluorescent under both long- and short-wave ultraviolet light. *D* (calc.) is 9.899 g/cm³ (empirical formula). Material from the McDermitt mine

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is reversibly photosensitive, and turns from vivid orange to black in strong light. In reflected plane-polarized light, it is bluish grey, with very weak (in air) to distinct (in oil) bireflectance, nonpleochroic, and distinctly anisotropic, with colors masked by ubiquitous yellowish orange to orange internal reflections. Measured values of reflectance obtained in air and in oil are tabulated. Averaged results of electron-microprobe analysis give HgO 92.03, Cl 9.54, Br 1.22, sum 102.79, less O = Cl + Br 2.28, total 100.51 wt.%. The empirical formula is $\text{Hg}^{2+}_{3.00}\text{O}_{2.00}(\text{Cl}_{1.90}\text{Br}_{0.11})_{\Sigma 2.01}$, based on O + Cl + Br = 4 atoms per formula unit. The mineral name recalls the creek that flows through the Terlingua mining district and into the Rio Grande River.

Keywords: terlinguacreekite, new mineral species, mercury oxychloride, X-ray data, electron-microprobe data, reflectance data, Perry pit, Mariposa mine, Terlingua mining district, Brewster County, Texas, McDermitt mine, Humboldt County, Nevada.

SOMMAIRE

La terlinguacreekite, de formule idéale $\text{Hg}^{2+}_3\text{O}_2\text{Cl}_2$, possède une sous-maille orthorhombique très prononcée; les groupes spatiaux possibles en seraient *Imam*, *Imcm*, *Ima2* et *I2cm*. Les paramètres réticulaires suivants ont été affinés à partir des données obtenues sur poudre: *a* 6.737(3), *b* 25.528(10), *c* 5.533(2) Å, *V* 951.6(6) Å³, *a:b:c* 0.2639:1:0.2167, *Z* = 8. La symétrie correcte, les paramètres de la sur-maille, et les détails de la structure cristalline demeurent méconnus. Les neuf raies les plus intenses du spectre de diffraction obtenu sur poudre [*d* en Å (*hkl* de la sous-maille)] sont: 5.413(30)(011), 4.063(80)(121), 3.201(50)(080), 3.023(50)(161), 2.983(60)(240), 2.858(30)(211), 2.765(50)(002), 2.518(100b)(091, 251) et 2.026(30)(242). Ce minéral se trouve à un endroit isolé mesurant environ 1 × 0.5 m au niveau inférieur du puit Perry, à la mine Mariposa, district minier de Terlingua, comté de Brewster, au Texas (la localité type); il se présente en encroûtements millimétriques xénomorphes orange foncé à rougeâtre, d'une épaisseur variable sur la calcite, et plus rarement en agrégats de cristaux de la même couleur atteignant 0.5 mm. On l'a aussi identifiée à la mine McDermitt, comté de Humboldt, Nevada, en présence de kleinite et calomel dans des roches volcaniques silicifiées et des sédiments. La terlinguacreekite serait une phase secondaire, tout probablement formée par altération du cinabre ou du mercure natif primaire. A Terlingua, la plupart de ces croûtes sont minces, presque cryptocristallines, sans formes décelables, résineuses et, translucides à opaques. Les cristaux atteignent 0.2 mm en longueur; ils sont subidiomorphes, allant de aciculaires à prismatiques, avec un allongement [001] et un rapport maximum de longueur à largeur de 4:1. Ils sont vitreux et transparents, et certains montrent des faces à forte réflectivité qui pourraient être {010} et {110}. La rayure est jaune; le minéral est cassant, avec une fracture inégale, mais sans clivage observable. Les cristaux sont mous, et ne montrent aucune fluorescence en lumière ultra-violet (longueurs d'ondes courtes ou longues). La densité calculée est 9.899 g/cm³ (formule empirique). Le matériau de la mine McDermitt est photochromique de façon réversible, passant d'un orange vif à noir dans une lumière intense. En lumière réfléchie polarisée, la terlinguacreekite est gris bleuâtre, avec une bireflectance très faible dans l'air à distincte dans l'huile; elle est non pleochroïque, et distinctement anisotrope, mais les couleurs sont masquées par les réflexions internes orange jaunâtre à orange ubiquistes. Nous présentons les valeurs mesurées de réflectance dans l'air et dans l'huile. En moyenne, les analyses effectuées avec une microsonde électronique ont donné HgO 92.03, Cl 9.54, Br 1.22, somme 102.79, moins O = Cl + Br 2.28, pour un total de 100.51% (poids). La formule empirique, $\text{Hg}^{2+}_{3.00}\text{O}_{2.00}(\text{Cl}_{1.90}\text{Br}_{0.11})_{\Sigma 2.01}$, est fondée sur O + Cl + Br = 4 atomes par unité formulaire. Le nom du minéral rappelle le cours d'eau qui traverse le district minier de Terlingua avant de se jeter dans le Rio Grande.

(Traduit par la Rédaction)

Mots-clés: terlinguacreekite, nouvelle espèce minérale, oxychlorure de mercure, données de diffraction X, données de microsonde électronique, réflectance, puit Perry, mine Mariposa, district minier de Terlingua, comté de Brewster, Texas, mine McDermitt, comté de Humboldt, Nevada.

INTRODUCTION

Terlinguacreekite, ideally $\text{Hg}^{2+}_3\text{O}_2\text{Cl}_2$, is a newly recognized mineral species. It was first encountered by the senior author during megascopic examination and routine X-ray powder-diffraction characterization of a suite of secondary Hg-bearing minerals submitted by one of us (SC) in 1997 for identification. These samples were originally collected in 1995 by Mr. Ray Stegemoller of Marble Falls, Texas, from the lower level of the Perry pit, Mariposa mine, Terlingua, Brewster County, Texas, U.S.A. The mineral has been megascopically misidentified as mosesite, terlinguaite, kleinite and comancheite. The same mineral was previously identified in the 1980s and partly characterized from the McDermitt mine, Humboldt County, Nevada, U.S.A.,

by one of the authors (EEF) before his untimely death. Unfortunately, we have only several pieces of written correspondence and emails at our disposal regarding the McDermitt mine occurrence; no samples are available for further study.

The mineral is named *terlinguacreekite* after Terlingua Creek, which flows through the Terlingua mining district of Texas and into the Rio Grande River. The mineral and mineral name have been approved by the Commission on New Minerals and Mineral Names, IMA (#2004-18). Holotype material (seven specimens) is housed in the Systematic Reference Series of the National Mineral Collection at the Geological Survey of Canada, Ottawa, Ontario, under catalogue number NMC68100. The polished section used for both quantitative electron-microprobe analysis and reflectance

measurements is deposited in the mineral collections at The Natural History Museum, London, U.K., as BM2004, 54.

OCCURRENCE

Terlinguacreekite is a secondary mineral found in the lower level of the Perry pit, The Mariposa mine, Terlingua mining district, Brewster County, Texas, U.S.A. [Origlieri (1990) and references therein]. The Mariposa mine is considered the type locality, as virtually all data reported in this paper are based on material collected from that occurrence. Terlinguacreekite was found on massive calcite in an isolated area approximately 1×0.5 m within the pit, and is not associated with any other secondary minerals. At the McDermitt mine, it is associated with kleinite and calomel in silicified volcanic rocks and sediments.

It should be considered a rare species at the Mariposa mine; several hundred specimens of calcite are known with sparse amounts of terlinguacreekite on matrix, but Mr. Stegemoller estimates there is less than 5 grams of pure material available for further study. The mineral most likely formed at the expense of primary cinnabar or native mercury.

Centimeter-sized powdery aggregates of lemon yellow terlinguacreekite have recently (February 2004) been collected *in situ* in a calcite–gypsum vein within the lower level of the Perry pit (Stefan Ansermet & Nicolas Meisser, pers. commun., 2004). It is associated with olive-brown light-tarnished calomel. The mineral was identified by Gandolfi X-ray powder pattern combined with energy-dispersion analysis.

PHYSICAL PROPERTIES

Terlinguacreekite occurs as mm-sized anhedral dark orange to reddish orange crystalline crusts (Fig. 1). These crusts have a variable thickness and a resinous luster, and are translucent to opaque. Most crusts are thin and almost cryptocrystalline with no discernable crystal forms. Crystal aggregates (Fig. 2), up to 0.5 mm, are rare. Individual crystals are up to 0.2 mm in length, subhedral, acicular to prismatic, elongation [001], and have a maximum length-to-width ratio of 4:1. They are the same color as the crusts, vitreous, and transparent; some crystals have very small brightly reflecting faces, which may be {010} and {110} (orthorhombic cell). The mineral has a yellow streak, is brittle with an uneven fracture, no observable cleavage, and is nonfluorescent under both long- and short-wave ultraviolet light. The crystals are too small to properly ascertain hardness, but the mineral is soft, as grains are easily crushed between two glass slides. The density could not be measured because of the small size of the crystals available and the dearth of material. The calculated density, on the basis of the empirical formula and subcell unit-cell parameters refined from powder data, is 9.899 g/cm^3 .

Attempts to solve the crystal structure indicate that the mineral has some form of complex twinning. The material from the McDermitt mine is photosensitive, and changes from vivid orange to black in strong light; this phenomenon is reversible.

X-RAY CRYSTALLOGRAPHY

Two crystals of terlinguacreekite were examined by single-crystal precession methods using Zr-filtered MoK α X-radiation. One crystal was mounted such that a^* , and the other such that c^* , is parallel to the dial axis. The following levels were collected: $0kl \rightarrow 2kl$, $h0l \rightarrow h4l$, $hk0$ and hkl . Precession films indicate orthorhombic symmetry, with measured unit-cell parameters a 6.706, b 25.56, c 5.611 Å. Systematic absences indicate that the space-group choices are *Imam*, *Imcm*, *Ima2* and *I2cm*. The refined unit-cell parameters, a 6.737(3), b 25.528(10), c 5.533(2) Å, V 951.6(6) Å³, $a:b:c$ 0.2639:1:0.2167, and $Z = 8$, are based on the d values of 25 X-ray powder lines between 4.063 and 1.384 Å for which unambiguous indexing was possible, based on visual inspection of single-crystal precession films. A fully indexed powder pattern is presented in Table 1. The powder data are unique and bear no resemblance to any other inorganic phase listed in the Powder Diffraction File (PDF).

We are unable to solve the crystal structure at this time. Numerous attempts have been made since the late 1980s, using various combinations of hardware and software for data collection and crystals from both the McDermitt and Mariposa mines, but none has yielded a sensible solution. The last two attempts (on material from the Mariposa mine) by coauthors PCB and MAC, using CCD-equipped diffractometers, may be summarized as follows:

PCB (University of Notre Dame 1997): monoclinic, probable space-group $P2_1$, a 11.124, b 6.692, c 26.189 Å, β 102.25°. Crystals are twinned to mimic an orthorhombic B -lattice with unit-cell dimensions a 11.124, b 6.692, c 51.186 Å, and there is a very pronounced subcell resembling an orthorhombic I -lattice.

MAC (University of Manitoba 2001): orthorhombic P -lattice, a 6.74, b 11.05, c 51.02 Å. All weak reflections can be accounted for by this cell, but solution of the crystal structure was unsuccessful. The majority of the reflections can be indexed on a monoclinic I or C cell with a 26.1, b 6.7, c 11.1 Å, β 102.2°.

Given the ambiguity of the structural results, we have chosen to give in this paper crystallographic and indexed powder-diffraction data based on the results of a detailed single-crystal precession study done in 1997. Clearly, the orthorhombic I -lattice given above is a (twinned?) subcell of the true cell, whether it is orthorhombic or monoclinic. However, all diffraction lines of the powder pattern index quite satisfactorily on this subcell, and the zero- and upper-level precession films do not show any of the weak reflections evident on the

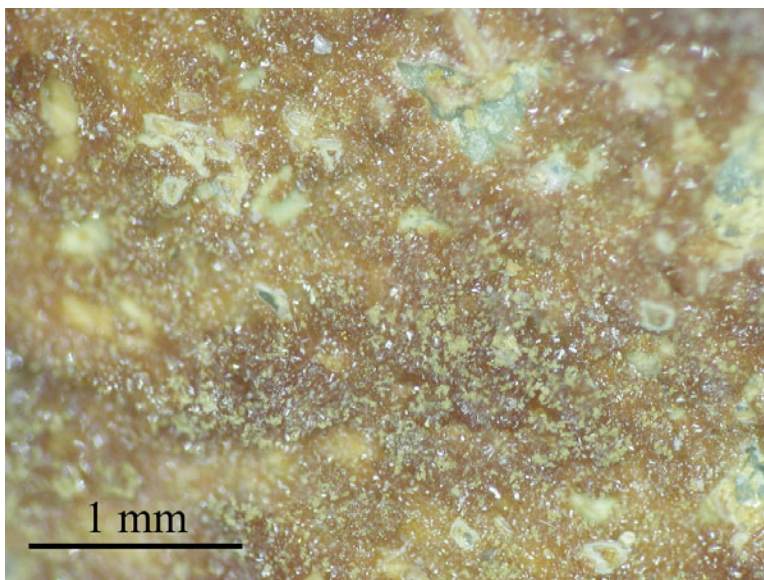


FIG. 1. Photomicrograph of a typical crust of terlinguacreekite. Scale bar: 1.0 mm.

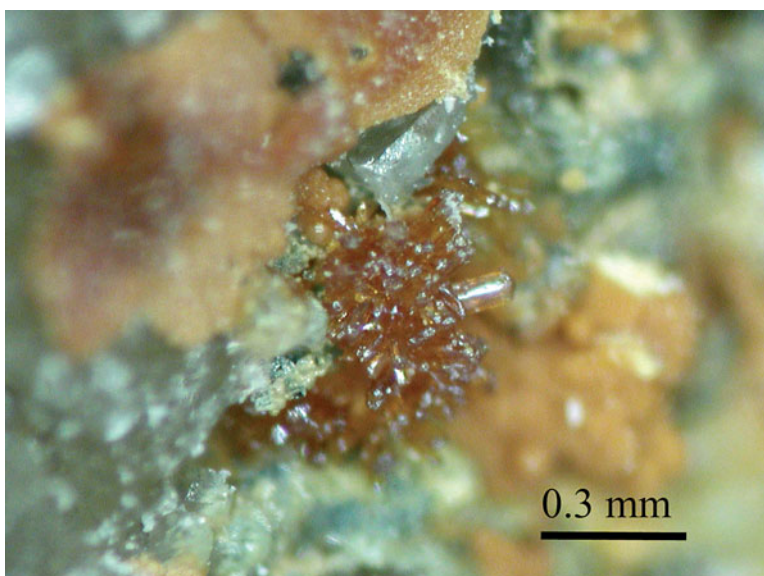


FIG. 2. Photomicrograph of an aggregate of terlinguacreekite crystals. Scale bar: 0.3 mm.

CCD-equipped diffractometers. These reflections are obviously very weak and do not contribute substantially to the powder data of Table 1. We will pursue further

structural studies of terlinguacreekite if better-quality material becomes available.

CHEMICAL COMPOSITION

An anhedral aggregate of terlinguacreekite was analyzed with a JEOL 733 electron microprobe (with Tracor Northern 5500 and 5600 automation), using an operating voltage of 15 kV, a beam current of 20 nA, and a beam diameter of 40 μm . Data reduction was done with a PAP routine in XMAQNT (C. Davidson, CSIRO, pers. commun.). The following standards were used: cinnabar ($\text{Hg}M\alpha$), scapolite ($\text{Cl}K\alpha$) and synthetic KBr ($\text{Br}L\alpha$). A 100-s energy-dispersion scan showed no elements with $Z > 8$ other than those reported; F and S were sought, but are below detection limits. The valence state for mercury was assumed to be Hg^{2+} , based on the behavior of the Hg atoms in the most recent crystal-structure attempt by one of us (MAC). The average of eight determinations (and ranges) gave HgO 92.03 (90.44–93.03), Cl 9.54 (8.77–9.92), Br 1.22 (1.18–1.31), sum 102.79, less $\text{O} = \text{Cl} + \text{Br}$ 2.28, total 100.51 wt.%. With $\text{O} + \text{Cl} + \text{Br} = 4$ atoms per formula unit (*apfu*), the empirical formula is $\text{Hg}_{2.3}^{2+}\text{O}_{2.00}(\text{Cl}_{1.90}\text{Br}_{0.11})_{\Sigma 2.01}$. Thus the analyzed material is a slightly bromian terlinguacreekite. The ideal formula, $\text{Hg}_{2.3}^{2+}\text{O}_2\text{Cl}_2$, requires HgO 92.21, Cl 10.06, sum 102.27, less $\text{O} = \text{Cl}$ 2.27, total 100.00 wt.%.

TABLE 1. X-RAY POWDER-DIFFRACTION DATA FOR TERLINGUACREEKITE

<i>I</i> est.	<i>d</i> Å meas.	<i>d</i> Å calc.	<i>hkl</i>	<i>I</i> est.	<i>d</i> Å meas.	<i>d</i> Å calc.	<i>hkl</i>
20	12.764	12.764	020				
10	6.382	6.382	040	3	1.906	1.910	262
30	5.413	5.407	011	* 15	1.869	1.869	361
20	5.277	5.282	130	3	1.847	1.851	0131
15	4.629	4.639	031	3	1.828	1.823	0140
5	4.255	4.255	060	* 25	1.798	1.799	2120
* 80	4.063	4.054	121	3	1.774	1.776	282
10	3.766	3.752	051	3	1.759	1.762	123
* 15	3.376	3.369	200	3	1.740	1.735	053
* 50	3.201	3.191	080	5	1.718	1.719	1112
* 50	3.023	3.016	161	3	1.706	1.708	332
* 60	2.983	2.979	240	* 20	1.684	1.684	400
* 30	2.858	2.859	211	3	1.649	1.650	1150
* 50	2.765	2.767	002	* 15	1.639	1.641	163
* 20	2.726	2.726	231	* 10	1.621	1.622	2131
3	2.646	2.641	260	* 15	1.606	1.604	2140
3	2.551	2.553	0100	5	1.586	1.589	233
100b	2.518	2.524	091	3	1.562	1.566	460
* 15	2.447	2.451	132	25	1.542	1.546	093
10	2.318	2.319	062	* 20	1.507	1.508	2122
			280	5	1.490	1.490	480
* 15	2.259	2.259	271	3	1.473	1.474	471
* 20	2.191	2.194	1110	* 15	1.450	1.449	0121
5	2.175	2.171	330	* 15	1.438	1.439	402
* 10	2.139	2.138	202	10	1.419	1.418	0180
* 20	2.090	2.090	082				1152
5	2.056	2.054	321	* 20	1.402	1.401	491
* 30	2.026	2.027	242	* 10b	1.384	1.383	004

114.6 mm Debye-Scherrer powder camera using Ni-filtered Cu radiation (λ $\text{Cu}K\alpha = 1.54178 \text{ \AA}$). Intensities estimated visually, b = broad. Not corrected for shrinkage, and no internal standard was used. *: lines used for unit-cell refinement. Indexed on a 6.737(3), b 25.528(10), c 5.533(2) \AA .

OPTICAL PROPERTIES

In plane-polarized reflected light ($\sim 3200 \text{ K}$), terlinguacreekite is bluish grey, with a very weak bireflectance in air and a distinct bireflectance in oil, but it is not pleochroic. It is distinctly anisotropic, but the rotation tints are masked by ubiquitous yellowish orange to orange internal reflections. Reflectance measurements were made in the visible region (400–700 nm) at intervals of 20 nm using a Leitz MPV-SP microscope-spectrophotometer. A WTiC reflectance standard (Zeiss 314) was used as a reference for measurements in air and oil (Leica $N_D = 1.518$). These were done with $20\times$ objectives, the numerical apertures of which were confined to 0.40, and the diameter of the measured discs was 9 μm . The reflectance curves (R_1 , R_2) are more or less parallel and descend continuously from short to long wavelength. In Table 2, we summarize the data collected from the aggregate in the polished section previously used for quantitative electron-microprobe analysis. If we assume that K_P (the physical molar refractivity) equals K_C (the chemical molar refractivity) ($= 0.141$ for the empirical formula) and use the calculated density, 9.899 g/cm^3 , then the calculated average index of refraction (n) for terlinguacreekite is 2.40. Applying the equation of Fresnel for minerals with low reflectivity and weak absorption, $n = 1 + \sqrt{R/1 - \sqrt{R}}$, and using a mean value of 16.2% for the reflectance, the average index of refraction (n) is 2.34, in good agreement with the value (2.40) obtained by the previous calculation.

RELATION TO OTHER SPECIES

There is no direct relation between terlinguacreekite and any known Hg-bearing mineral species. It is the seventh Hg–O–Cl-bearing phase found in

TABLE 2. REFLECTANCE DATA FOR TERLINGUACREEKITE

λ nm	R_1 %	R_2 %	mR_1 %	mR_2 %
400	16.7	19.0	4.2	5.0
420	16.2	18.7	4.0	4.9
440	15.9	17.4	3.9	4.6
460	15.6	16.9	3.7	4.5
470	15.4	16.5	3.6	4.4
480	15.1	16.2	3.5	4.3
500	14.9	16.0	3.5	4.2
520	14.6	15.8	3.4	4.1
540	14.5	15.6	3.4	4.1
546	14.5	15.6	3.4	4.1
560	14.4	15.5	3.4	4.1
580	14.4	15.4	3.4	4.2
589	14.3	15.4	3.4	4.2
600	14.2	15.4	3.4	4.2
620	14.2	15.3	3.4	4.2
640	14.2	15.2	3.5	4.3
650	14.2	15.2	3.5	4.3
660	14.1	15.2	3.5	4.3
680	14.0	15.1	3.5	4.3
700	14.0	15.1	3.5	4.3

nature, after comancheite $\text{Hg}^{2+}_{13}\text{O}_9(\text{Cl},\text{Br})_8$, eglestonite $\text{Hg}^{1+}_4\text{OCl}_2$, hanawaltite $\text{Hg}^{1+}_6\text{Hg}^{2+}_3[\text{Cl},(\text{OH})]_2$, pinchite $\text{Hg}^{2+}_5\text{O}_4\text{Cl}_2$, poyarkovite $\text{Hg}^{1+}_3\text{OCl}$ and terlinguaite $\text{Hg}^{1+}\text{Hg}^{2+}\text{OCl}$. Interestingly, of these seven, five have their type locality listed as Terlingua, Texas. Assuming that the chemical formula is correct as given, there are two polymorphs of $\text{Hg}^{2+}_3\text{O}_2\text{Cl}_2$ known from the chemical literature: the α form [Aurivillius & Stålhandske (1974) and references therein] and the β form (Neuman *et al.* 1976). Both of these modifications are monoclinic, and their X-ray powder patterns differ from each other and from that of terlinguaite. PDF cards for the α form are 76–1475 (calculated), 74–2312 (calculated), 70–1044 (calculated) and 26–1268 (experimental). The PDF card for the β form is 71–1837 (calculated).

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REFERENCES

- AURIVILLIUS, K. & STÅLHANDSKE, C. (1974): The crystal structure of $\text{Hg}_3\text{O}_2\text{Cl}_2$, a compound with three-coordinated mercury (II). *Acta Crystallogr.* **B30**, 1907-1910.
- NEUMAN, M.A., PETERSEN, D.R. & LO, G.Y.S. (1976): Crystal structure of a second modification of the mercury oxychloride $\text{Hg}_3\text{O}_2\text{Cl}_2$. *J. Crystal Mol. Struct.* **6**, 177-203.
- ORIGLIERI, M. (1990): Famous mineral localities: Terlingua, Texas. *Mineral. Rec.* **21**, 221-234.

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