

ON TYPE ROMARCHITE AND HYDROROMARCHITE FROM BOUNDARY FALLS, ONTARIO, AND NOTES ON OTHER OCCURRENCES

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ABSTRACT

The type material of romarchite and hydroromarchite from Boundary Falls, Winnipeg River, Ontario, Canada, was re-examined. The minerals form deposits on pewter bowls and are products of tin corrosion in a unique cold freshwater environment. Type romarchite is very fine-grained and replaces pseudotetragonal crystals of an unknown precursor. Revised X-ray powder-diffraction data are consistent with the accepted tetragonal symmetry and space group $P4/nmm$, and provide the refined unit-cell parameters a 3.8014(3), c 4.8559(6) Å. Type hydroromarchite forms euhedral single crystals. Revised X-ray powder-diffraction data are consistent with the accepted tetragonal symmetry and space group $P4_2/c$, and provide the refined unit-cell parameters a 7.9269(5), c 9.0970(9) Å. Additional Sn-bearing corrosion products are SnO₂ (cassiterite) and two unidentified phases. Residual aggregates of Pb, SbSn and Cu₆Sn₅ (η -bronze) are found in areas of deep localized corrosion. Reports of romarchite subsequent to the original description are numerous, but those of hydroromarchite are few and only quite recent.

Keywords: romarchite, hydroromarchite, tin oxide, pewter, corrosion product, X-ray data, Boundary Falls, Ontario, Canada.

SOMMAIRE

Nous avons ré-examiné les échantillons holotypes de la romarchite et de l'hydroromarchite provenant des chutes Boundary, sur la rivière Winnipeg, en Ontario, Canada. Ces minéraux se sont formés sur des bols enfouis en étain comme produits de la corrosion de l'étain dans un milieu unique d'eau fraîche froide. Le romarchite type a une granulométrie très fine et remplace des cristaux pseudotétragonaux d'un précurseur méconnu. Les données révisées obtenues en diffraction X, méthode des poudres, concordent avec la symétrie tétragonale acceptée et le groupe spatial $P4/nmm$, et mènent aux paramètres réticulaires affinés a 3.8014(3), c 4.8559(6) Å. L'hydroromarchite type forme des monocristaux idiomorphes. Les données révisées en diffraction X, méthode des poudres, concordent avec la symétrie tétragonale acceptée et le groupe spatial $P4_2/c$, et mènent aux paramètres réticulaires a 7.9269(5), c 9.0970(9) Å. Sont aussi présents comme produits de corrosion stannifères SnO₂ (cassitérite) et deux phases non identifiées. Des agrégats résiduels de Pb, SbSn et Cu₆Sn₅ (bronze- η) ont été décelés dans des endroits où la corrosion était accentuée. Les indices de romarchite connus depuis sa découverte sont nombreux, mais ceux de la hydroromarchite sont peu nombreux et assez récents.

(Traduit par la Rédaction)

Mots-clés: romarchite, hydroromarchite, oxyde d'étain, étain, produit de corrosion, données en diffraction X, chute Boundary, Ontario, Canada.

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INTRODUCTION

Early studies of tin and pewter artifacts document a long-standing difficulty in the characterization of corrosion products of tin; initially recognized were SnO₂ and “tin pest”, the destructive transformation of tetragonal metallic tin to its grey, powdery cubic allotrope. The presence of “hydrous” tin oxides, including “hydrous stannous oxide”, was also noted during the early 20th century. It was not until the work of Plenderleith & Organ (1953), Lihl (1962) and Gettens (1964) that SnO was confirmed as a common constituent of corrosion products, typically admixed with SnO₂. Several years later, discovery of pure corrosion products on Canadian pewter artifacts provided an opportunity to characterize two of these phases as the minerals romarchite and hydroromarchite (Organ & Mandarino 1971), by demonstrating equivalence to known synthetic phases. The chemistry of tin corrosion and conservation aspects were dealt with by Leidheiser (1971) and Turgoose (1985).

The type material from Boundary Falls was recently re-examined by the senior author in order to update the descriptive mineralogy of romarchite and hydroromarchite. In the process, a search of the literature revealed numerous additional reported occurrences of romarchite, but only a few, and quite recent, reported occurrences of hydroromarchite. In light of the brevity of the original descriptions of type material, we felt that publication of the updated descriptions and a review of the literature are warranted.

HISTORY OF THE TYPE MATERIAL
FROM BOUNDARY FALLS

Eighteen pewter bowls were among the artifacts recovered in 1966 by a joint Ontario–Minnesota archaeological team, in five meters of water below rapids at Boundary Falls, on the Winnipeg River, Kenora District, Ontario, at latitude 50°13'15"N, longitude 95°06'12"W. The area of the Falls was a particularly dangerous segment of a major voyageur trade route: the capsizing of a freight canoe in 1800 was documented in dramatic detail (Coues 1897). However, the bowls were not lost in that mishap, as trademark stamps on some of them indicate their manufacture in London, England between 1801 and 1811 (Wheeler *et al.* 1975). Dr. Walter Kenyon (1917–1986), archeologist with the Royal Ontario Museum (ROM) and co-leader of the archeological team, submitted those bowls that were moderately to heavily corroded to Museum staff for mineralogical study. Organ & Mandarino (1971) described two of the corrosion products as the new mineral species romarchite and hydroromarchite; the proposed minerals had been submitted to the Commission on New Minerals and Mineral Names of the International Mineralogical Association in 1969, and were approved despite their anthropogenic status. Incrusta-

tions of romarchite and hydroromarchite (gram and milligram quantities, respectively) on the two bowls registered under M28744 in the ROM mineral collections are designated holotypes. A considerable quantity of romarchite is also present on the bowl registered under M49593.

REPORTED OCCURRENCES OF ROMARCHITE
AND HYDROROMARCHITE

Subsequent to its original description, romarchite has been reported from a number of natural and anthropogenic occurrences:

- U.S. Virgin Islands [natural]; typically with cassiterite, cerussite and several lead and tin oxides, hydroxides and oxychlorides, in complex secondary assemblages derived by partial alteration of nuggets (up to 5 mm in size) of an intergrowth of native tin and native lead; found in outcrops of the weathering zone and in concentrates from both soils and stream sediments, on virtually all of the Islands (Alminas *et al.* 1994). No anthropogenic evidence was noted.

- María Teresa mine, Huari, Oruro, Bolivia [natural]; replaces herzenbergite, SnS (Ramdohr 1980, vol. 1, p. 669). Ramdohr, in his description of herzenbergite, states that “SnO, romanèchite, is observed as replacing herzenbergite...” Considering the paragenesis, it is all but certain that the species concerned is romarchite (R. Bideaux, pers. commun., 2002). Ramdohr makes further reference to romanèchite as an alteration of herzenbergite in his description of teallite at Carguaicollo (Carguaicollo), Bolivia (vol. 1, p. 672). We caution that Ramdohr did not mention Carguaicollo as a locality for herzenbergite in his description of that mineral, so it is possible that this second reference is the same as the first describing “romanèchite” in the María Teresa material, and simply misplaced. Turneaure & Gibson (1945) made no mention of either Mn oxides or SnO at Carguaicollo. The occurrence of romarchite at Carguaicollo is therefore possible but suspect.

- Bayhorse area, Idaho [natural]; tentatively identified in Cu–Au mineralization (H.V. Alminas, pers. commun., 2001).

- Duluth Complex, Minnesota [natural]; tentatively identified (H.V. Alminas, pers. commun., 2001).

- Mauna Loa, Hawaii [natural]; “SnO” was identified as a minor constituent phase, together with ZnS, CuS, CaSO₄ and CdS, in sodium sulfate (and possibly sulfur) deposited as volcanic sublimates (Bayhurst & Janecky 1986). It may quite possibly be romarchite, but we have not yet been able to confirm this.

- Sharm Abhur, a cove and submarine valley of the Red Sea, near Jiddah, Saudi Arabia [anthropogenic]; found with abhurite on tin ingots recovered from a shipwreck site (Matzko *et al.* 1985).

- Caergwrle, Wales, UK [anthropogenic]; with cassiterite, as alteration products of what were probably

gilded tin decorative elements on the Caergwrle Bowl, a carved oil shale artifact probably of the Bronze Age, which was unearthed from a bog in 1823 (Green 1985).

- corrosion product of a Roman bronze figure, Venus (Demeter) [anthropogenic]; with cassiterite and other phases (Scott 1994).

- component of black “patina” on a Chinese bronze mirror of the Han Dynasty [anthropogenic]; with cassiterite and cuprite (Williams *et al.* 1998).

Most recently, Dunkle (2002) and Dunkle *et al.* (2003) reported the anthropogenic occurrence of both romarchite and hydroromarchite on pewter artifacts from the following marine archeological sites:

- the *Queen Anne’s Revenge* (1718), Beaufort Inlet, North Carolina; with abhurite and, tentatively, herzenbergite.

- the St. Johns Bahamas wreck (~1550), Grand Bahama Island; with abhurite and possibly cassiterite.

- *La Capitana* (1715), Indian River County, Florida; with abhurite and cassiterite.

- the *San José* (1733), in the Florida Keys; with abhurite.

- Port Royal, Jamaica (1692), a submerged colony; with abhurite.

- the *Henrietta Marie* (1701), Key West, Florida; with abhurite and cassiterite.

THE BOUNDARY FALLS TYPE MATERIAL

The pewter bowls recovered at Boundary Falls were originally believed to be tin. The determination was based presumably on an analysis of an acid leach solution, details of which were given by Wheeler *et al.* (1975). They offered an opinion that the levels of Pb, Zn, Cu and Sb in the metal are consistent with those of a tin refined to the limits of technology in the early 1800s. However, our examination of the metal by scanning electron microscopy (SEM), semiquantitative energy-dispersion spectroscopy (EDS), X-ray diffraction (XRD) and inductively coupled plasma – atomic emission spectroscopy (ICP–AES) showed that it is actually a pewter with significant levels of Pb, Sb and Cu. An ICP–AES analysis of an acid solution of uncorroded metal shavings weighing 1.07 mg gave: Sn 82.0, Pb 11.0, Sb 1.1, Cu 0.7, Fe 0.3, Zn 0.1, As 0.05, Mg 0.05, Ca 0.7, total 96.0 wt%. Back-scattered electron (BSE) images show Pb as abundant blebs up to 10 μm in diameter in a tin matrix (Fig. 1), as well as what appear to be micrometric cuboid crystals of SbSn. Deep localized corrosion of the pewter has produced delicate spongy grey residual aggregates that were shown by XRD to be Pb, SbSn and Cu_6Sn_5 (η -bronze); the dendritic texture of the latter is evident locally (Fig. 2). North (1987) reported the occurrence of SbSn among such residues of corrosion. The mutual occurrence of the intermetallic phases SbSn and Cu_6Sn_5 in pewter and related alloys is widely documented (*e.g.*, Barry & Thwaites 1983); however, we have not been able to determine whether

the Cu_6Sn_5 crystals are original to the pewter or are a product of the corrosion process. The residual aggregates are, in places, accompanied by an unidentified white platy Sn-rich phase designated UTP–1, described below. It is apparent that the corrosion process involved preferential leaching and oxidation of Sn. Interestingly, SbSn and a phase similar or identical to the metallurgical Cu_6Sn_5 are also known to occur naturally with native tin, as the minerals stistaite and sorosite, respectively (Rose 1981, Alminas *et al.* 1994, Barkov *et al.* 1998).

Romarchite (Figs. 3, 4, 5) and hydroromarchite (Fig. 6) appear to have encrusted only those metal surfaces enclosed between tightly stacked bowls. In the process, they had cemented (and possibly sealed) the bowls together; judging by marks on the bowls, considerable force had been applied to separate them. Such a restricted environment in ambient cold, fresh water provided a unique and fortuitous opportunity for the development of these minerals. The most heavily corroded bowls are partially encrusted with thick deposits of cream-white to tan, very fine-grained SnO_2 (cassiterite), apparently on surfaces that were openly exposed. Remarkably, translucent trade beads of white glass are embedded in this cassiterite.

Two additional products of corrosion are in common association with both the romarchite and the fine-grained cassiterite; neither could be identified by XRD methods despite their apparently simple chemical composition. One of these Sn-rich phases (UTP–1) comprises abundant incrustations of white bladed orthogonal crystals; some of the crystals are cyclically twinned in a

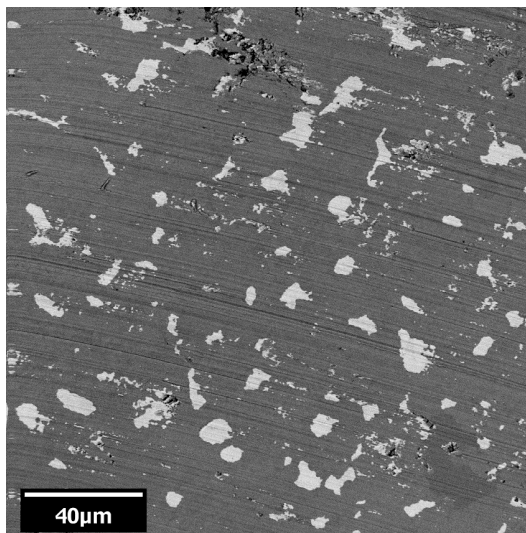


FIG. 1. Surface of a fresh pewter shaving, showing blebs of lead (white) in tin matrix (grey); Boundary Falls. SEM–BSE image.

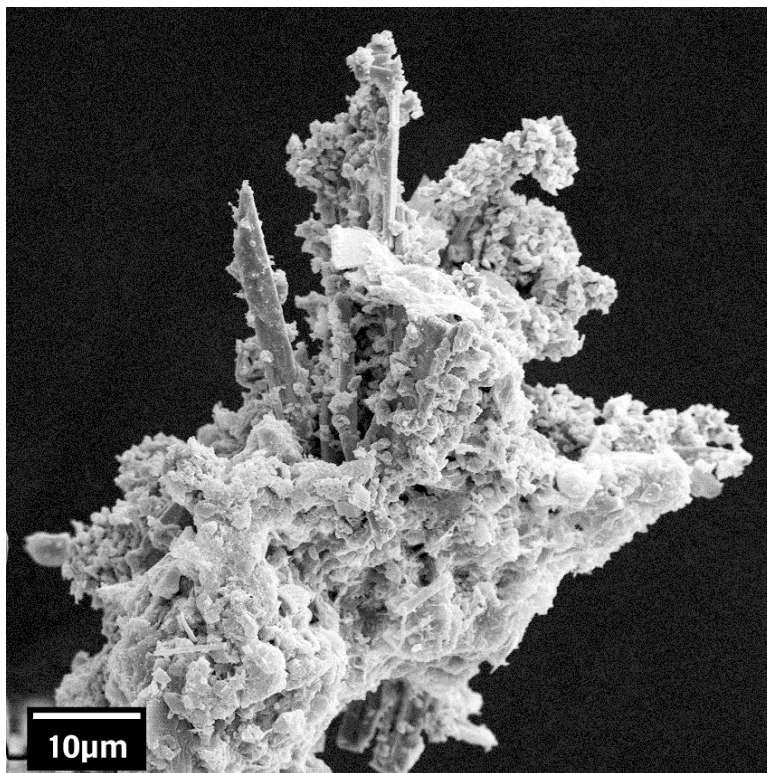


FIG. 2. Delicate residual aggregate of Pb, Cu_6Sn_5 (elongate individuals of dendrites are visible) and SbSn, from deeply corroded pewter; Boundary Falls. SEM-BSE image.

manner resembling cerussite “trillings” (Fig. 7). Electron probe micro-analysis (EPMA) showed only the presence of major Sn. The X-ray powder-diffraction pattern appears to be unique. The strongest reflections [d in Å (I)] are: 3.51 (65), 3.38 (55), 3.05 (100), 2.97 (80), 2.587 (75), 1.788 (70), 1.714 (55). The second unidentified tin-rich phase (UTP-2) comprises pale yellow to yellow crystals, hexagonal in appearance (Figs. 4, 5); crystals are solitary and in druses. SEM-EDS analyses show the presence of major Sn, minor S, and minor to very minor Si. The X-ray powder-diffraction pattern is remarkably similar to that of the sulfosalt pearceite. The strongest reflections [d in Å (I)] are: 6.42 (45), 5.66 (25), 4.38 (25), 3.09 (80), 3.00 (50), 2.823 (100), 1.845 (25), 1.694 (30).

DESCRIPTIONS OF ROMARCHITE AND HYDROROMARCHITE

Data are for type material unless otherwise stated; data for the U.S. Virgin Islands mineral are taken from Alminas *et al.* (1994), and those for the *Queen Anne's Revenge* material are taken from Dunkle (2002).

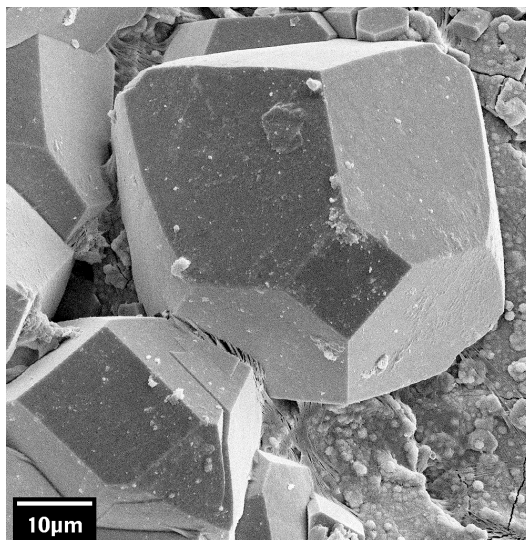


FIG. 3. Romarchite pseudomorph after an unknown precursor; Boundary Falls. SEM-BSE image.

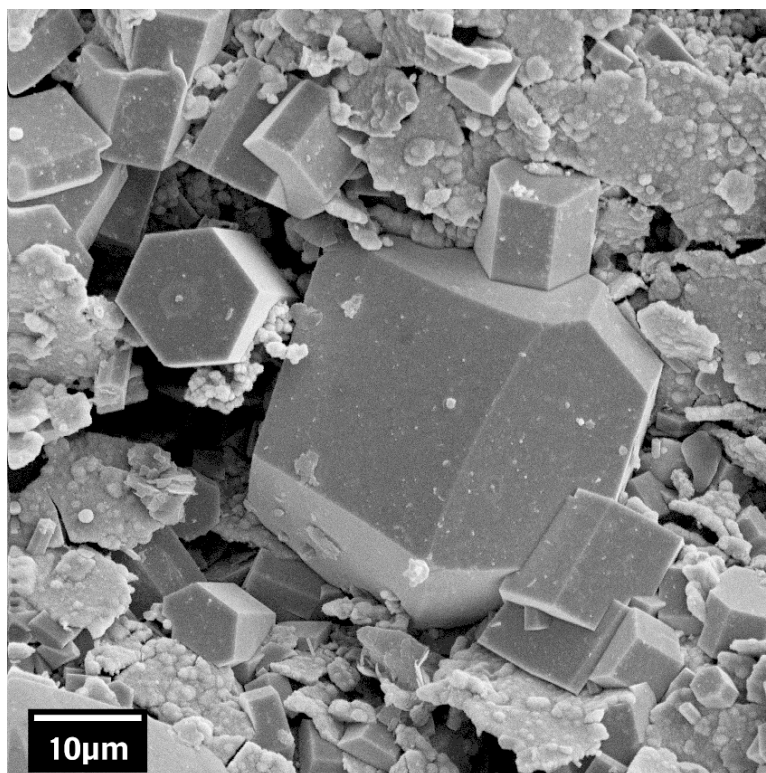


FIG. 4. Romarchite pseudomorphs after an unknown precursor, with “hexagonal” crystals of unidentified Sn-rich phase (UTP-2); Boundary Falls. SEM-SE image.

Romarchite

Romarchite, tetragonal SnO, has the space group $P4/mmm$, determined by Swanson *et al.* (1955) for synthetic SnO, and class $4/mmm$. Unit-cell parameters a 3.8014(3), c 4.8559(6) Å were refined from powder data for the type material (Table 1), giving a V of 70.170 Å³ and $c:a = 1.2774$; $D_{\text{calc}} = 6.374$ g/cm³ for $Z = 2$.

Organ & Mandarino (1971) adopted the tetragonal cell and space group of Swanson *et al.* (1955). Pannetier & Denes (1980) confirmed the space group for synthetic black SnO and reported that the unit-cell parameters are a 3.8029, c 4.8382 Å, for which $c:a = 1.2722$, and $D_{\text{calc}} = 6.39$ g/cm³. Their structure analysis of synthetic crystals revealed a Sn–O–Sn triple-layer structure. The Sn atoms lie at the apices of regular tetragonal pyramids, arising from both sides of a central layer of oxygen atoms. The triple layers are coupled by residual bonds. Romarchite is isostructural with litharge, PbO.

X-ray powder-diffraction data for type romarchite (Table 1) are consistent with the accepted tetragonal space-group. Diffraction lines are broadened by the fine

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

l	d_{obs} (Å)	d_{calc} (Å)	hkl	l	d_{obs} (Å)	d_{calc} (Å)	hkl
9	4.857 *	4.856	001	3	1.385	1.387	113
100	2.995 *	2.993	101	9	1.343	1.344	220
46	2.687 *	2.688	110	5	1.2256	1.2261	301
16	2.428 *	2.428	002	7	1.2020	1.2021	310
3	2.041	2.046	102	11	1.174	1.1759	222
15	1.901 *	1.901	200	11	1.1713	1.1723	213
33	1.802 *	1.802	112	3	1.105 †	1.1064	114
3	1.773	1.770	201	9	1.0774	1.0773	312
33	1.605 *	1.605	211	9	1.030	1.0303	321
18	1.492	1.497	202	6	1.022	1.0231	204
9	1.485	1.489	103				

with 10 additional lines to 0.78 Å

Sample M28744; 0.1 mm Ø capillary with internal Si reference; 114.6 mm Debye-Scherrer camera. Ni-filtered Cu radiation with $K\alpha_1$, stripped with the Rachinger algorithm, $\lambda(K\alpha_1) = 1.540562$ Å; scanned positions and intensities. The indexing was based on refined cell: a 3.8014(3), c 4.8559(6) Å; space group $P4/mmm$.

† interference by adjacent reflection of Si internal standard; * spacing used for refinement; } poorly resolved doublet.

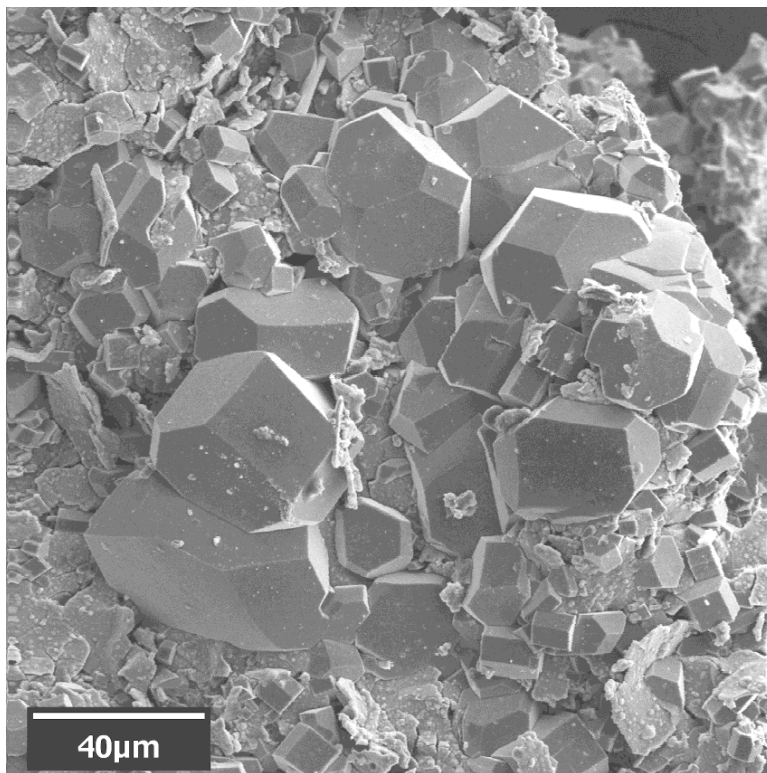


FIG. 5. Romarchite incrustation of pseudomorphic crystals, with "hexagonal" crystals of unidentified Sn-rich phase (UTP-2); Boundary Falls. SEM-SE image.

size of the crystallites. This dataset is similar to PDF 6-395, the data of Swanson *et al.* (1955) for synthetic SnO. Lower d values are reported for Pb-bearing romarchite from the U.S. Virgin Islands.

The Boundary Falls mineral typically comprises thin crusts of lustrous euhedral crystals between 10 to 50 μm in diameter (Fig. 5); exceptional individuals reach 0.7 mm. Remarkably, all crystals examined were found to be very fine-grained romarchite, pseudomorphic after an unknown precursor. There is no evidence to suggest that the precursor was hydroromarchite, which can be found as pristine crystals in close proximity to the pseudomorphs. Romarchite also occurs as fine-grained dull black coatings, in places showing concentric growth-related patterns reminiscent of "wood tin" cassiterite, and as a component of pustules. The U.S. Virgin Islands mineral occurs as crystals attaining several micrometers in diameter, as submillimetric spherules, and as a constituent of very fine-grained intimate intergrowths of complex mineralogy. Romarchite from the *Queen Anne's Revenge* is reported as irregular grains and as crystals attaining 100 μm , commonly in intimate association with hydroromarchite.

The pseudomorphic crystals from Boundary Falls (Figs. 3, 4, 5) and the crystals from the U.S. Virgin Islands, as illustrated by Alminas *et al.* (1994), appear to be of similar habit: equant to slightly elongate, bipyramidal, typically with subordinate pinacoid and equal to subordinate prism. However, minor morphological features of the Boundary Falls crystals indicate that the precursor phase was probably pseudotetragonal, *i.e.*, of a lower symmetry. Crystals from the *Queen Anne's Revenge* were described as intergrown laths; an acicular habit was also reported for "free-grown" crystals on outer surfaces of artifacts.

Romarchite is black to very dark brown in fine-grained aggregates; the streak is clove brown. Crystals are opaque, although deep blue minute internal reflections were noted in the largest crystals. Splendent crystals are adamantine in luster, approaching submetallic. Ultraviolet fluorescence was not evident. Romarchite is brittle, and SEM images of broken surfaces show an uneven fracture with no evidence of cleavage, belying the fine-grained nature, as a perfect cleavage would be

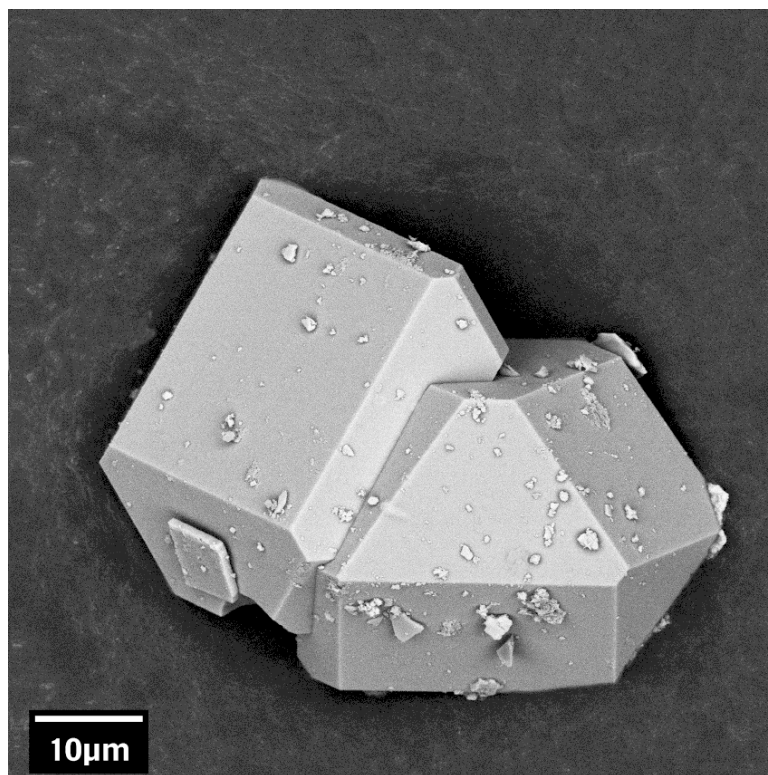


FIG. 6. Hydroromarchite; Boundary Falls. SEM-SE image.

expected for the structure (by analogy to litharge). The Mohs hardness is 4. Density has not been measured.

The original analysis of type material by X-ray fluorescence (XRF) spectrometry revealed only Sn and a trace of Fe. Subsequent scans by SEM-EDS detected only Sn. The numerous EPMA datasets given in Dunkle (2002) indicate compositions that are nominally stoichiometric, with Pb, Fe, Cu, As and Cl typically below 1 wt.% in total. The substitution of 10–20 mol.% PbO is reported for the U.S. Virgin Islands mineral. The ideal formula requires: Sn 88.12, O 11.88, total 100.00 wt%.

The mineral was named after the *Royal Ontario Museum and archeology*.

Hydroromarchite

Hydroromarchite, tetragonal $\text{Sn}_3\text{O}_2(\text{OH})_2$, has the space group $P2_1c$, determined by Abrahams *et al.* (1996) for synthetic material, and class $2m$. The unit-cell parameters, a 7.9269(5), c 9.0970(9) Å, were refined from data on powder taken from the type material (Table 2), giving a V of 571.6 Å³ and $c:a = 1.1476$; $D_{\text{calc}} = 4.904 \text{ g/cm}^3$ for $Z = 4$. Abrahams *et al.* (1996) reported simi-

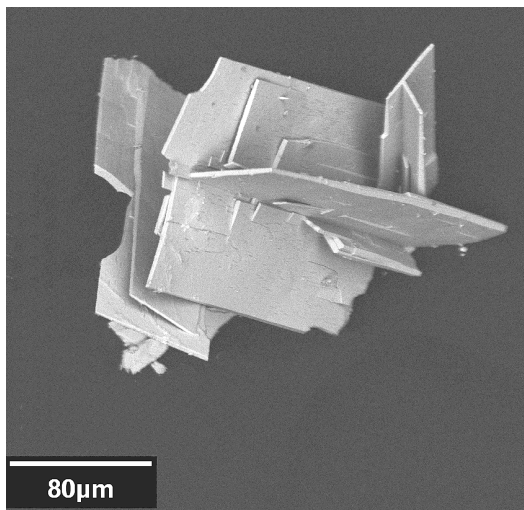


FIG. 7. Unidentified Sn-rich phase (UTP-1), twinned crystals; Boundary Falls. SEM-SE image.

lar data for their synthetic material: a 7.9268, c 9.1025 Å, V 571.94 Å³, and D_{calc} 4.904 g/cm³.

Originally, Organ & Mandarino (1971) had adopted the triclinic cell of Donaldson (1961), and Howie & Moser (1968, 1973) had determined possible tetragonal space-groups $P4/mnc$ or $P4nc$. The structure analysis of synthetic crystals by Abrahams *et al.* (1996) revealed Sn₆O₄(OH)₄ clusters in a pseudo-body-centered arrangement, with intercluster hydrogen bonding between OH and O atoms. Sn²⁺ is in a distorted tetragonal pyramidal coordination. Hydroromarchite is isostructural with synthetic Pb₃O₂(OH)₂; there are no known relationships to other mineral species.

X-ray powder-diffraction data for type hydroromarchite (Table 2) are consistent with the accepted space-group. This dataset is similar to PDF 46-1486, which was calculated from the structure of Abrahams *et al.* (1996), determined on synthetic material.

The Boundary Falls mineral comprises solitary euhedral crystals and thin incrustations that grew directly on pewter surfaces and in close association with the far more abundant romarchite. Crystals are typically smaller than 30 µm, and rarely attain 100 µm. Their morphology ranges from tabular with prominent pinacoid to nearly equant, with pinacoid, shallow bipyramid and prism all being prominent (Fig. 6).

Hydroromarchite on artifacts of the *Queen Anne's Revenge* comprises irregular grains and crystals attaining 100 µm, intimately associated with romarchite. Hydroromarchite forms have not been indexed. Twinning is not evident.

Hydroromarchite crystals are colorless, transparent to translucent, and have an adamantine luster; incrustations are pale tan in color. Fluorescence in ultraviolet light is not evident. Hydroromarchite is brittle; SEM photomicrographs of crystal fragments show at least three cleavage planes, one of which is very well developed. Hardness and density could not be measured. A pale orange-yellow color was noted in the finely ground mineral, an indication of probable grinding-induced oxidation; bruises on hydroromarchite incrustations are brown.

The original analysis of type material by XRF spectrometry revealed only Sn. Numerous EPMA datasets given in Dunkle (2002) indicate that compositions are nominally stoichiometric, with Pb, Fe, Cu, As and Cl typically below 1 wt.% in total. The ideal formula requires: SnO 95.73, H₂O 4.27, total 100.00 wt.%.

The name of the mineral reflects its hydrated nature and its chemical relationship to romarchite.

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TABLE 2. X-RAY POWDER-DIFFRACTION DATA FOR HYDROROMARCHITE

<i>l</i>	d_{obs} (Å)	d_{calc} (Å)	<i>hkl</i>	<i>l</i>	d_{obs} (Å)	d_{calc} (Å)	<i>hkl</i>
41	5.980 *	5.976	101			1.7733	105
34	5.612 *	5.605	110			1.7725	420
17	4.556	4.549	002	23	1.7724	1.7709	412
						1.7660	224
100	3.540	3.545	210			1.7398	421
		3.532	112	11	1.7404	1.7282 *	332
60	3.303 *	3.303	211	15	1.7282 *	1.7283	332
81	2.988 *	2.988	202	23	1.6845 *	1.6843	314
15	2.832	2.832	103			1.6237	413
				16	1.6189	1.6187	215
94	2.797	2.803	220			1.5618	501
		2.796	212	18	1.5621	1.5324	511
38	2.507 *	2.507	310			1.5303	423
28	2.416 *	2.417	311	8	1.5315	1.5162	006
13	2.386	2.386	222	3	1.5163	1.4971	502
3	2.305	2.304	213	3	1.4973	1.4724	315
17	2.274 *	2.274	004			1.4720	520
		2.199	320	16	1.4718	1.4711	512
4	2.197	2.195	312			1.4161	206
4	2.138	2.137	321	4	1.4161	1.4059	433
6	1.992	1.992	303	3	1.4039	1.3984	424
5	1.982	1.982	400	10	1.3984	1.3942	216
29	1.933	1.932	313	8	1.3942	1.3826	513
25	1.915	1.914	214	3	1.3826	1.3579	530
4	1.881	1.881	411	2	1.3579	1.3402	405
12	1.8170	1.8168	402	5	1.3395	1.3392	442
16	1.7795	1.7799	323				

plus 26 additional lines to 0.81 Å

Sample M28744: 0.1 mm Ø capillaries with and without internal Si reference; 114.6 mm Debye-Scherrer camera, Ni-filtered Cu radiation with $K\alpha_2$ stripped with the Rachinger algorithm, $\lambda(K\alpha_1) = 1.540562$ Å; scanned positions and intensities. The indexing is based on the refined cell: a 7.9269(5), c 9.0970(9) Å, space group $P4_2c$. * Spacing used for refinement.

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