HOLFERTITE

A New Hydroxyl-Hydrated Uranium Titanate

from

Starvation Canyon, Thomas Range, Utah

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ABSTRACT

Holfertite is a new mineral from Starvation (formerly Searle) Canyon, Thomas Range, Utah. It occurs as a pneumatolitic phase in cavities and fractures in a rhyolite, together with hematite, bixbyite, fluorite, topaz and red beryl. It forms elongated prismatic crystals up to 1×5 mm, and these crystals are commonly hollow, consisting of a hexagonal outer surface with a hexagonal prismatic cavity in the interior of the crystal. Crystals are found perched on hematite or topaz, piercing hematite crystals, and as inclusions in red beryl. Holfertite is canary-yellow to orange-yellow with a pale yellow streak and adamantine luster. It is brittle, $H = 4$, $D_{obs} > 4.22$, and does not fluoresce in ultraviolet light. Holfertite has perfect cleavage on {110} and an uneven to conchoidal fracture. In transmitted plane-polarized light, holfertite is non-pleochroic, colorless, uniaxial positive, $\omega = 1.815 \pm 0.008$, $\varepsilon = 1.910 \pm 0.008$. Holfertite is hexagonal, space group $P3$, $a = 10.824(2)$, $c = 7.549(2)$ Å, $V = 765.94(9)$ Å^3, $Z = 3$. The strongest five X-ray diffraction lines in the powder pattern are [$d(I)(hkl)$]: 4.60(10)(020), 3.05(2)(112), 2.90(8)(022), 1.87(3)(004/050), 1.747(3)(024). Chemical analysis by a combination of electron microprobe and crystal-structure refinement gave UO_3 76.03, TiO_2 11.89, CaO 2.29, Fe_2O_3 0.44, K_2O 0.30, H_2O (by weight loss on heating) 8.09, sum 99.04 weight %. Because of the very disordered nature of the crystal structure of holfertite, there is some ambiguity as to the chemical formula, depending on whether or not (OH) is present in the structure. The following two formulae are possible:
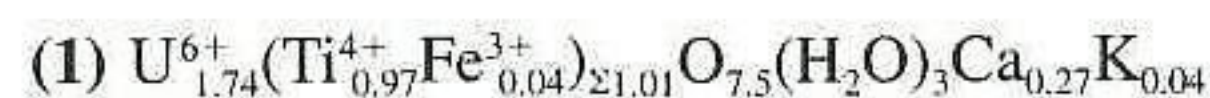
(1) $U^{6+}_{1.74}(Ti^{4+}_{0.97}Fe^{3+}_{0.04})_{\Sigma1.01}O_{7.5}(H_2O)_3Ca_{0.27}K_{0.04}$

In this case it is calculated on the basis of 10.5 O-atoms with (OH) = 0 and (H_2O) = 3 apfu ($D_{calc} = 4.22$ g/cm^3; H_2O 8.27 weight % by structure refinement).

(2) $U^{6+}_{1.74}(Ti^{4+}_{0.97}Fe^{3+}_{0.04})_{\Sigma1.01}O_{7.17}(OH)_{0.67}(H_2O)_3Ca_{0.27}K_{0.04}$
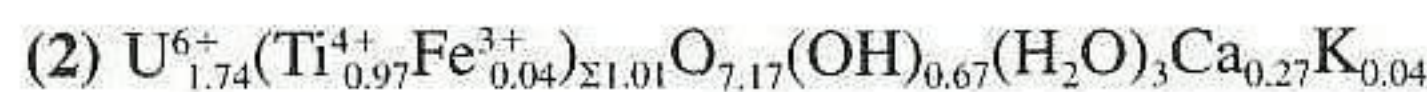
In this case it is calculated on the basis of 10.84 O-atoms with (OH) = 0.67 and (H_2O) = 3 apfu ($D_{calc} = 4.26$ g/cm^3; H_2O 9.19 weight % by structure refinement).

The corresponding simplified formulae are:

(1) $(UO_2)_{1.75}TiO_4\{(H_2O)_3Ca_{0.25}\}$ and
(2) $(UO_2)_{1.75}TiO_{3.67}(OH)_{0.67}\{(H_2O)_3Ca_{0.25}\}$, respectively.

INTRODUCTION

During a 1998 trip to topaz occurrences in rhyolites of the Thomas Range, Utah, one of the authors (DB) visited one of John Holfert's claims at Starvation (formerly Searle) Canyon in the western part of the Thomas Range. A few very small, bright-yellow acicular crystals were found while collecting smoky topaz. John Holfert told us that he had previously noticed such crystals at this and a few other places in the Thomas Range, and also mentioned that the late Eugene Foord worked on this material as a possible new mineral, but did not finish this work because of his illness and death.

In 1998, we collected only a few crystals. We subsequently visited the locality again in 2000 and 2001. With the help of John Holfert, his

Figure 1. **Holfertite type locality at Starvation (formerly Searle) Canyon, Thomas Range, Utah; the senior author is sitting in the center of the photograph (for scale). Photograph by N. Pekova.**

wife Galina, Mikhail Generalov and Natalia Pekova, we spent four days collecting about 4 mg of material. Thanks to the bright yellow or orange color of the mineral, it is relatively easy to find individual needles. However, it was not possible to get much pure material because the average crystal size is 0.1 × 1.5 mm, most crystals are hollow, and many crystals contain inclusions of associated minerals; approximately a hundred such crystals weigh only about 1 mg. In 2002, Patrick Haynes collected 2 mg of crystals from Starvation Canyon at our request, and this allowed us to finish this study.

The yellow crystals proved to be a new mineral species. It is hereby named for John W. Holfert (born 1949), who has made significant contributions to the mineralogy, geology and mineral occurrences of the Thomas Range (Holfert *et al.*, 1996). The species and name were approved by the International Mineralogical Association Commission on New Minerals and Mineral Names (IMA 2003-009). Holotype material is deposited at the Fersman Mineralogical Museum (#91374), Russian Academy of Sciences, Moscow, Russia, and at the Smithsonian Institution.

THE THOMAS RANGE and STARVATION CANYON

According to John Holfert, Starvation (formerly Searle) Canyon (Fig. 1) has the most abundant concentration of the mineral. The material used in this study all came from this site, which is designated as the type locality. Holfert has also found crystals at the end of that canyon, at the Maynard Topaz mine (on the western slope of Topaz Valley at the southern end of Topaz Mountain), and at the pseudobrookite location 1 mile south of Pismir Wash (western part of the Thomas Range).

At the type locality (Fig. 2) the rocks consist primarily of rhyolites and other volcanic rocks forming five distinct lava flows a few hundred meters thick. Details of the mineralogy and geology of the Thomas Range are given by Holfert *et al.* (1996). The rhyolites contain numerous cavities (gas bubbles) ranging from a few millimeters to a few meters in size. The most common mineral in the cavities is hematite, occurring as thin plates and as flat pinacoidal-rhombohedral crystals up to 1 cm. Certain areas in these rhyolites are well-known for smoky topaz which can nearly completely fill the original cavities and is commonly intergrown with quartz and alkali feldspar, forming what are locally known as "clinkers." Smoky topaz crystals can be transparent and up to 10 cm in size. Topaz freed from the rock by weathering fades in the sunlight. They are easily seen in the morning sunlight when numerous faded topaz crystals sparkle on the mountain slopes, making it crystal clear how Topaz Mountain got its name.

The Thomas Range is also famous for black cubic crystals of bixbyite (up to 2 cm) and flattened hexagonal prisms of red beryl up to 1 cm. Also common are columnar crystals of pseudobrookite, niobian rutile, cristobalite, magnetite and cryptocrystalline cassiterite. Rare zircon and thorianite have also been reported. At the holfertite occurrence, we also found cerianite-(Ce). In most cases, the larger crystals occur in fractures in rhyolite which served as channels for percolating fluids.

PHYSICAL PROPERTIES

Holfertite occurs as elongated prismatic crystals up to 1 × 5 mm with an average size of 0.15 × 1.5 mm. These crystals are typically

Figure 2. The holfertite pit, Starvation Canyon,
Thomas Range, Utah. Photograph by N. Pekova.

hollow, consisting of a hexagonal outer surface with a hexagonal prismatic cavity in the interior of the crystal, and occur as isolated crystals, parallel intergrowths and radial sprays of three or four crystals.

Holfertite is translucent to transparent, and canary-yellow to orange-yellow with a pale yellow streak. It is brittle, has a Mohs hardness of 4, $D_{obs} > 4.22$ (crystals sink slowly in concentrated Clerici solution), $D_{calc} = 4.22$; 4.26 g/cm³ (calculated for different crystal-structure models based on analysis H(2), Table 1). It does not fluoresce in ultraviolet light. Holfertite has perfect cleavage on {110} and an uneven to conchoidal fracture. In transmitted plane-polarized light, holfertite is non-pleochroic, colorless, uniaxial positive, $\omega = 1.815 \pm 0.008$, $\varepsilon = 1.910 \pm 0.008$ ($\lambda = 590$ nm).

OCCURRENCE

Holfertite occurs on fracture surfaces in rhyolite, on topaz "clinkers" and in cavities. Very commonly, holfertite is intergrown with, or penetrates, hematite crystals. Holfertite needles also occur on topaz crystals, sometimes partly (but never completely) included in topaz. Holfertite does occur as inclusions in crystals of red beryl (Fig. 3), but only rarely penetrates the surface of the crystals. Holfertite can also be found intergrown with bixbyite and niobian rutile. In SEM images, small (5–10 mm) octahedral crystals of cerianite-(Ce) are commonly associated with, but never contact, holfertite (Fig. 4). Many holfertite crystals are covered by a crust of deep purple to colorless fluorite crystals (Fig. 5a) or secondary calcite, and their hollow nature is easily apparent (Figs. 5a, b). The spatial relations with other minerals indicate that holfertite crystallized after quartz and Na-K-feldspar, during and after crystallization of hematite, at the very last stage of and after topaz growth, and

before red beryl, bixbyite, most hematite and fluorite. Calcite and (probably) fluorite are very late secondary minerals. The rest of the minerals are probably pneumatolitic in origin.

CHEMICAL COMPOSITION

Chemical data were obtained using a JXA-50 electron microprobe operating in EDS and WDS mode and an SEM with EDS at the Fersman Museum (RAS) and IGEM (RAS), respectively. Two chemical compositions are given in Table 1; composition 1 was determined on material used to define indices of refraction, density and H₂O content; composition 2 was determined on material used to obtain X-ray diffraction data. A special effort was made to determine Y and REEs, but neither were detected. The weight loss on heating was measured at the Faculty of Geology, Moscow State University, in a microthermocamera (built by A. E. Zadov) and using a sample of 1.483 mg. The total loss on heating was 8.09 weight %: 7.75 weight % was lost on heating to 300°C for 10 minutes and the rest (0.34 weight %) on heating to 500°C for 10 minutes. Further heating up to 900°C did not produce any additional weight loss. The infrared spectrum (Fig. 6) was recorded using a Specord-75 infrared-spectrometer, and shows the presence of uranyl groups (892 cm⁻¹), (H₂O) and (OH) groups (1626 and 3390 cm⁻¹) and overtone and fundamental stretching vibrations of Ti-O₄ (3420 cm⁻¹) and Ti-O (622 cm⁻¹). The lack of $(CO_3)^{2-}$ absorption bands in the infrared spectrum allow us to assign the total weight-loss on heating to H₂O: 8.09 weight %.

Sokolova *et al.* (2005) found that the structure of holfertite is very disordered, resulting in a significant degree of ambiguity in the formula of holfertite. Consequently, rather than a definitive formula, they produced two alternative formulae:
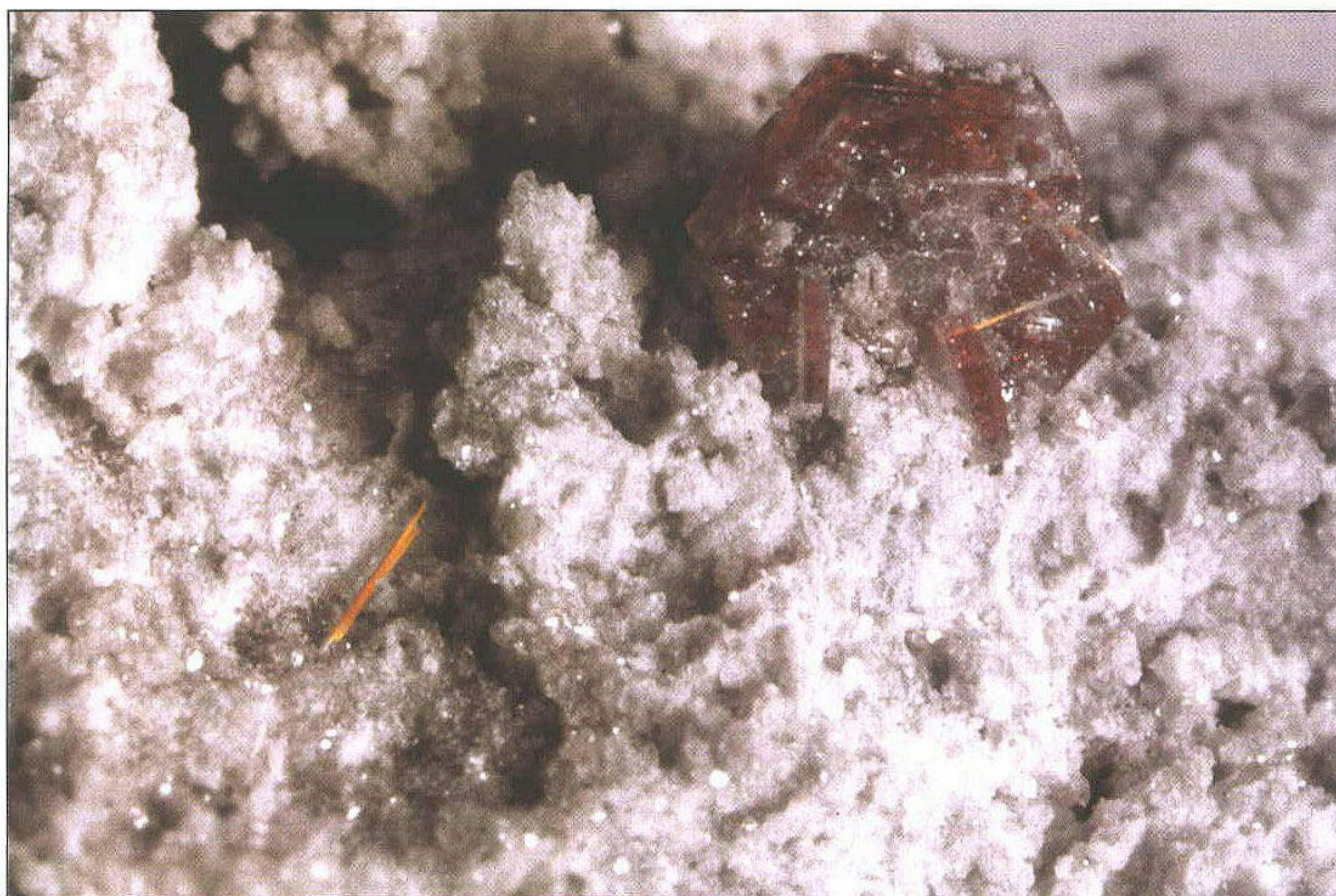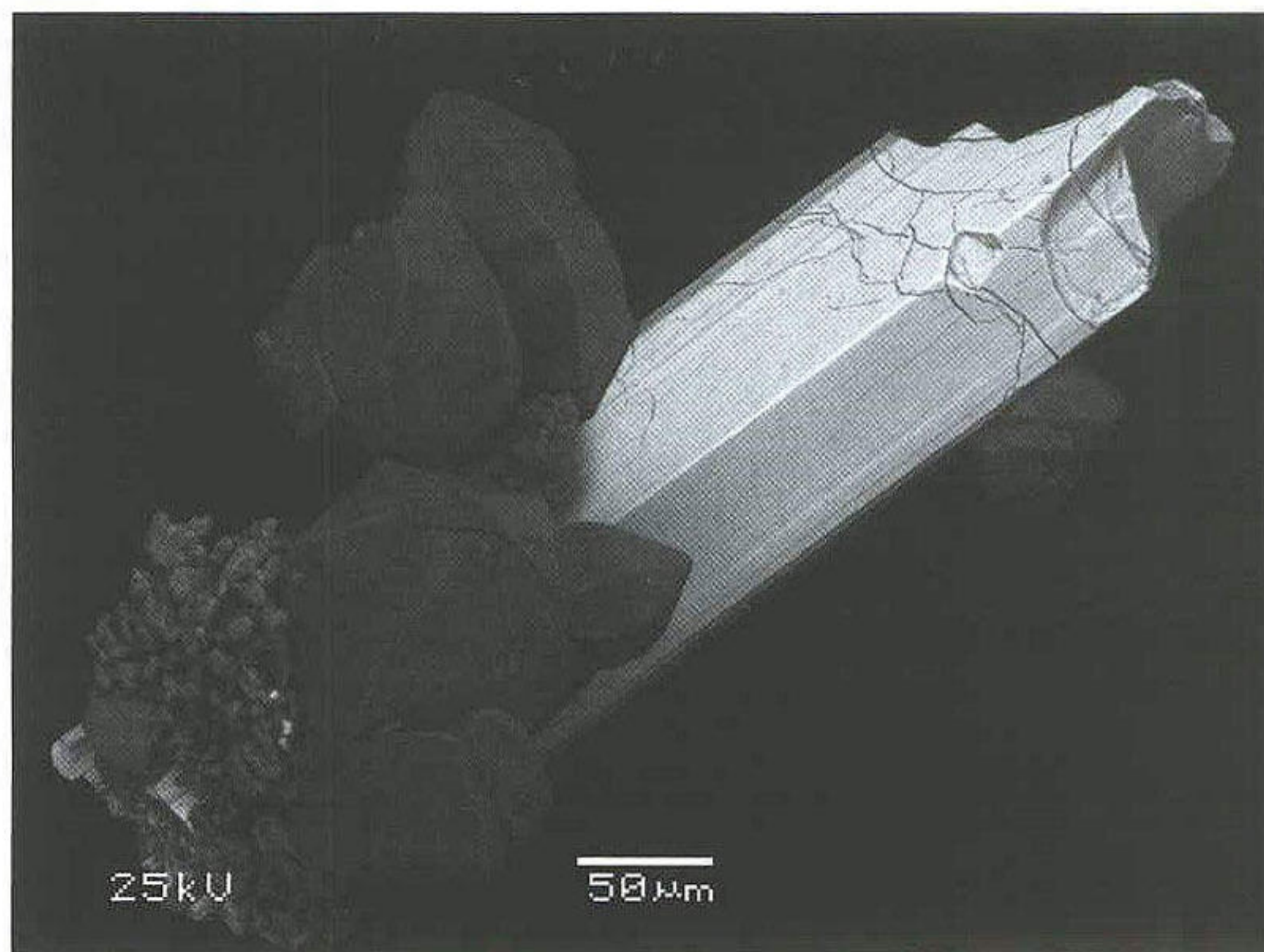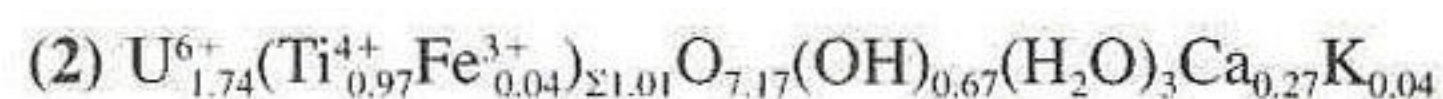
Figure 3. Holfertite crystals on rhyolite and in red beryl; the beryl crystal is about 0.6 cm across.

Figure 4. A crystal of holfertite associated with quartz crystals (dark gray). At the lower left corner, there is a cuboctahedral crystal of bixbyite and an aggregate of Nb-rutile decorated with bright specks of cerianite-(Ce).


(1) $U^{6+}_{1.74}(Ti^{4+}_{0.97}Fe^{3+}_{0.04})_{\Sigma1.01}O_{7.5}(H_2O)_3Ca_{0.27}K_{0.04}$

This was calculated on the basis of 10.5 O-atoms with (OH) = 0 and (H_2O) = 3 apfu, D_{calc}=4.22 g/cm³; (H_2O 8.27 weight % by structure refinement).

(2) $U^{6+}_{1.74}(Ti^{4+}_{0.97}Fe^{3+}_{0.04})_{\Sigma1.01}O_{7.17}(OH)_{0.67}(H_2O)_3Ca_{0.27}K_{0.04}$

This was calculated on the basis of 10.84 O-atoms with (OH) = 0.67 and (H_2O) = 3 apfu, D_{calc} = 4.26 g/cm³ (H_2O 9.19 weight % by structure refinement).

The corresponding simplified formulae are:

(1) $(UO_2)_{1.75}TiO_4[(H_2O)_3Ca_{0.25}]$ and
(2) $(UO_2)_{1.75}TiO_{3.67}(OH)_{0.67}[(H_2O)_3Ca_{0.25}]$.
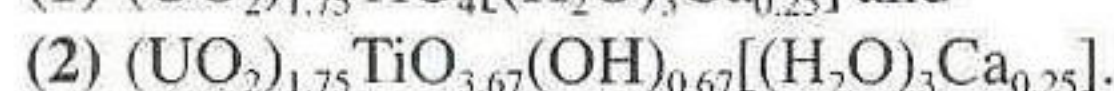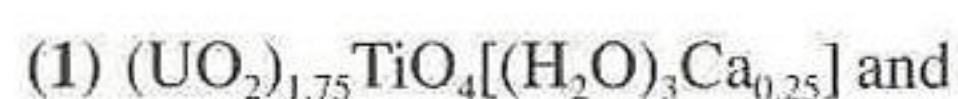
The key issue here is whether or not holfertite contains significant (OH). The infrared spectrum (Fig. 6) is rather ambiguous in this regard. There is a very strong broad band centered on 3390 cm⁻¹ and a strong sharper band at 1621 cm⁻¹, both attributable to (H_2O).

The broad band centered on 3390 cm⁻¹ has a broad subsidiary band at 3240 cm⁻¹. It is not clear if this band is due to disordered (OH), the disorder accounting for the unusual breadth of a band attributed to (OH), or another (H_2O) band. On the other hand, the amount of H_2O calculated in the chemical composition for each of these formulae (Table 1) shows closer agreement for the OH-free composition, with the amount of H_2O determined by weight loss on heating [8.09 weight %]. In our opinion, the evidence to distinguish between these two possible formulae for holfertite is not definitive, and hence we give both possibilities.

X-RAY AND ELECTRON-DIFFRACTION DATA

Table 3 gives X-ray powder-diffraction and microdiffraction data for holfertite. X-ray powder-diffraction data for holfertite were obtained on a Debye-Scherrer camera as the pattern obtained on an X-ray diffractometer exhibited strong preferred orientation and has only a small number of lines. The best result was obtained with a 57.3-mm (rather than with 114.6-mm) camera.
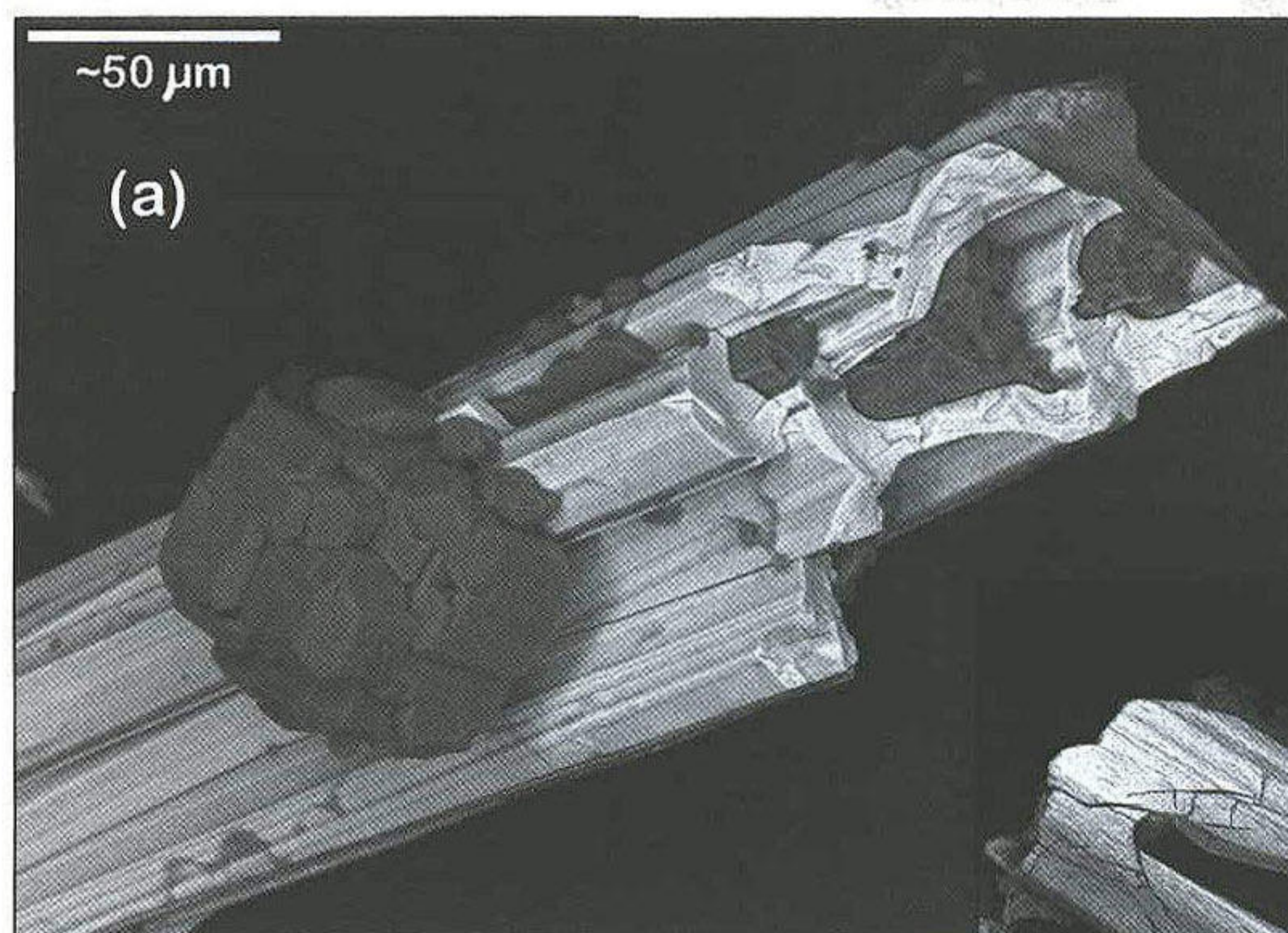
Figure 5. SEM images of holfertite; (a) a crystal of holfertite (bright) with an aggregate of fluorite crystals attached; (b) a crystal of holfertite (bright) on hematite; both images show the hollow nature of the holfertite crystals. The small crystals on the left below holfertite are cerianite-(Ce).
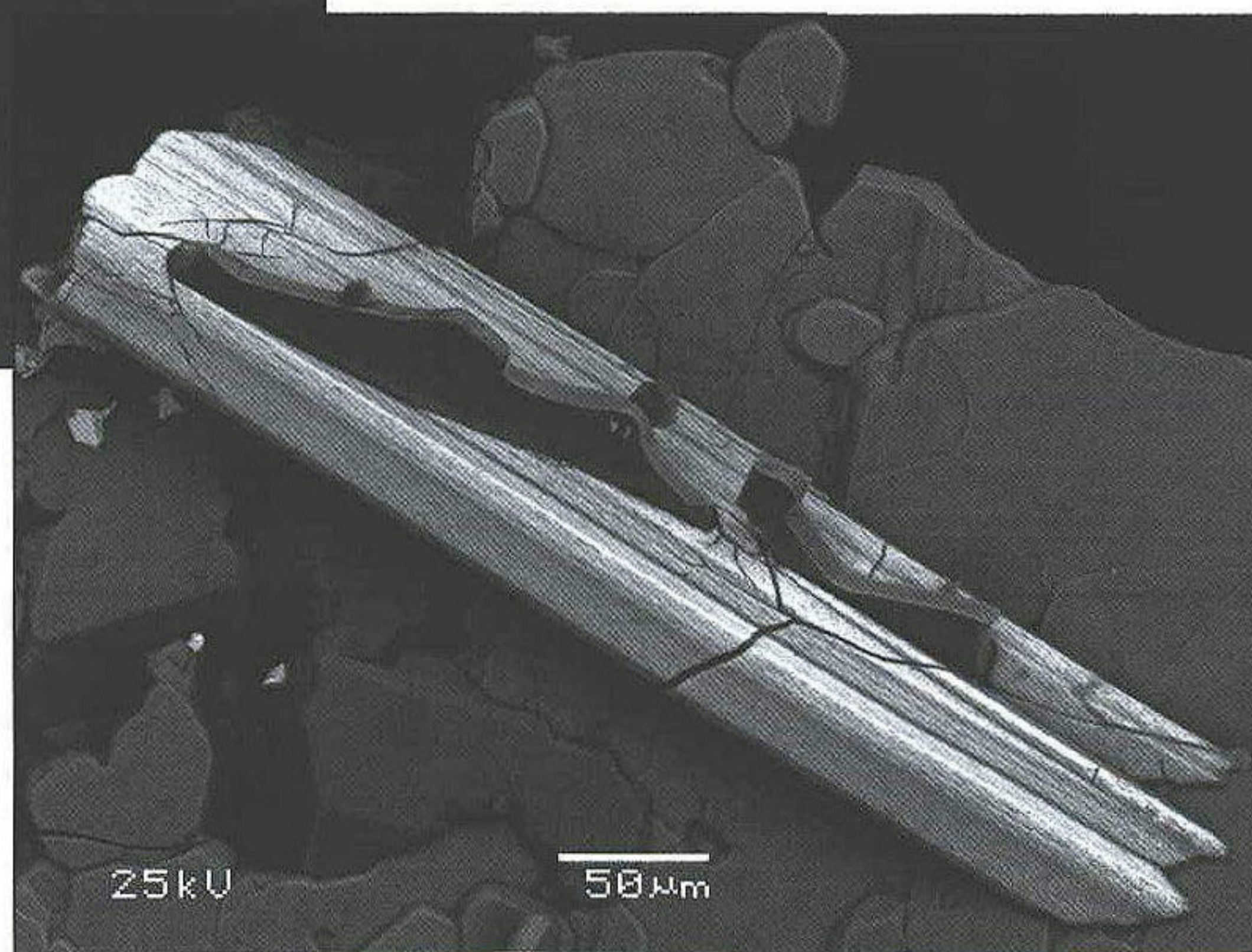
Table 1. Chemical composition (weight %) of holfertite H(1,2)* and the Ti-U-Ca-HREE oxide, F, of Foord *et al.* (1995) and the formula (apfu) of holfertite.

	H(1)	H(2)	F		H(1)	H(2)
UO₃	75.97	76.03	18.5	U	1.68	1.74
TiO₂	13.02	11.89	45.5	Ti	1.03	0.97
Fe₂O₃	0.47	0.44	n.d.	Fe³⁺	0.04	0.04
CaO	3.01	2.29	3.25	Ca	0.34	0.27
Y₂O₃	n.d.	n.d.	3.1	K	0.04	0.04
RE₂O₃	n.d.	n.d.	6.93			
K₂O	0.31	0.30	n.d.	O₇.₅	(OH)₀	(H₂O)₃
H₂O**	8.09	8.09	22.00			
Total	100.87	99.04	100.03***	O₇.₁₇	(OH)₀.₆₇	(H₂O)₃

UO_3 75.97 76.03 18.5 U 1.68 1.74
TiO_2 13.02 11.89 45.5 Ti 1.03 0.97
Fe_2O_3 0.47 0.44 n.d. Fe^{3+} 0.04 0.04
CaO 3.01 2.29 3.25 Ca 0.34 0.27
Y_2O_3 n.d. n.d. 3.1 K 0.04 0.04
RE_2O_3 n.d. n.d. 6.93
K_2O 0.31 0.30 n.d. $O_{7.5}$ $(OH)_0$ $(H_2O)_3$
H_2O** 8.09 8.09 22.00
Total 100.87 99.04 100.03*** $O_{7.17}$ $(OH)_{0.67}$ $(H_2O)_3$

H_2O from structure refinement for OH = O apfu: H(1) 8.56; H(2) 8.27 weight %

H_2O from structure refinement for OH = 0.67 apfu: H(1) 9.51; H(2) 9.19 weight %

* mean of 14 and 10 points respectively;
** H_2O by weight loss on heating;
*** Includes Na_2O 0.1, MgO 0.1, BaO 0.55 weight %; U as UO_2.

JXA-50 microprobe with Link EDS (Si-Li detector). U = 20kV, I = 2na. Beam diameter = 2 microns. K, U, Y and REE were measured using WDS U = 20kV, I = 20na. K was measured separately (PET crystal) using a slightly defocused beam. Standards: Ca—Diopside USNM 117733, U—Synthetic UO_2, Ti and Fe—Ilmenite USNM 96189, K—Microcline USNM 1439.

Table 2. Physical properties of holfertite in comparison with Ca-U-Ti-HREF mineral of Foord *et al.* (1995).

	Our material	Ca-U-Ti-HREF mineral of Foord et al. (1995)
Color	yellow or orange yellow	Yellow or orange yellow sometimes white
Optical properties	unixial + parallel extinction $\omega = 1.815(8)$; $\varepsilon = 1.910(8)$	uniaxial − parallel extinction $\omega, \varepsilon < 1.7$
Density	observed > 4.22 g/cm³ calculated 4.22–4.26 g/cm³	2.64 for yellow 2.62 for white

Electron diffraction showed even single crystals of holfertite to be quite heterogeneous with respect to their diffraction behavior. For example, a holfertite crystal (1–2 microns long) gives very good spots at one point and rings in another part, indicating local violation of long-range order. The character of the disorder in holfertite, as indicted by the electron-diffraction results, is not clear. However, the single-crystal X-ray diffraction results (Sokolova *et al.*, 2005) indicate that crystals of holfertite have strong OD (Order-Disorder) character (Merlino, 1997) associated with translational disorder of uranyl-titanate chains in the *a*- and *b*-directions. The disorder indicated by electron diffraction may thus be the result of this chain disorder. It is difficult to decide if this chain disorder is the result of radiation damage, inasmuch as many minerals show OD behavior (Merlino, 1997), and yet are not associated with any radioactive materials.
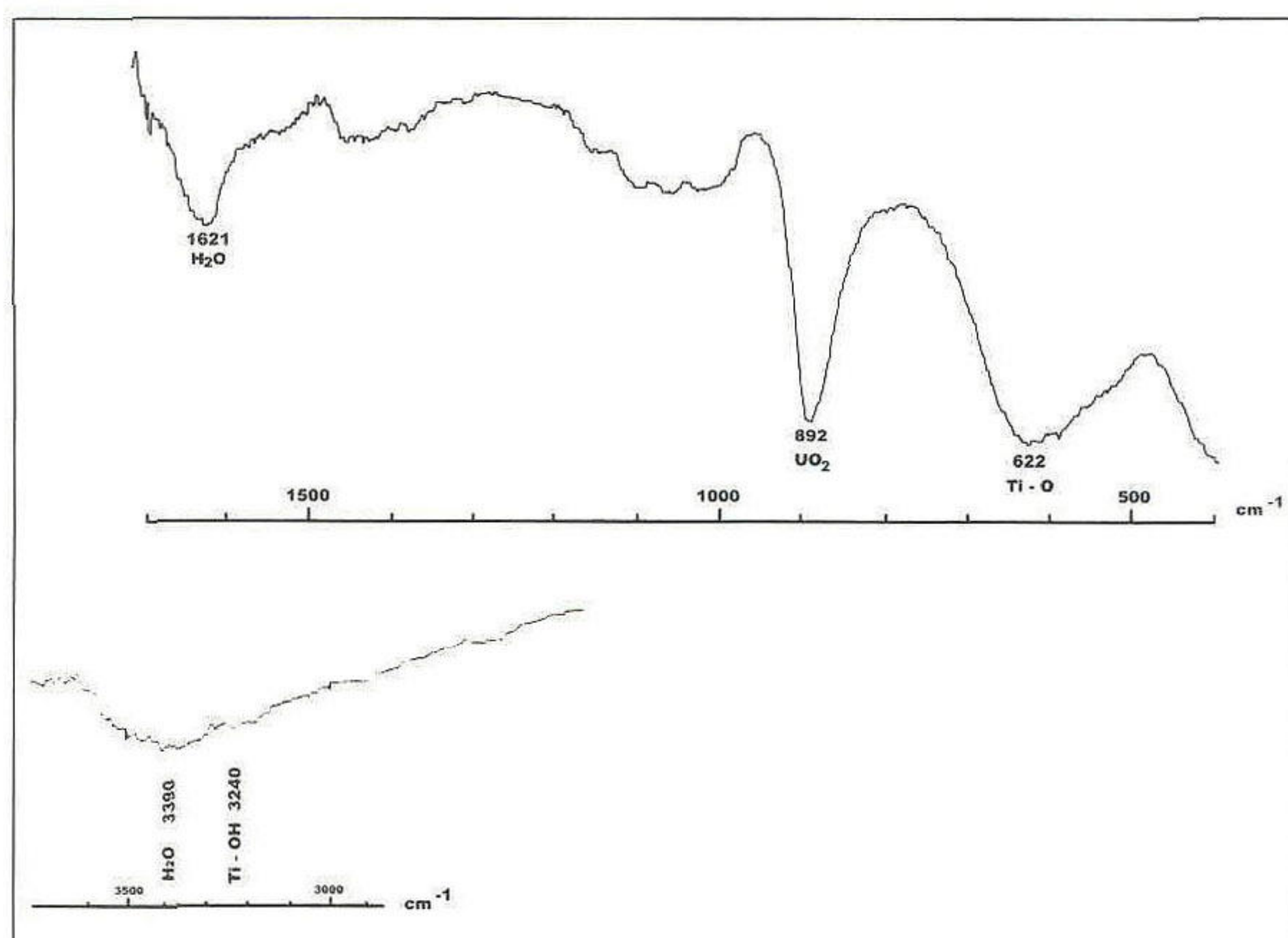
Figure 6. Infrared spectrum of holfertite.

Table 3. X-ray data for U-Ti mineral.

Camera*		Diffractometer**		Electron diffraction***				Ca-U-Ti-HREE mineral of Foord et al. (1995)	
d (Å)	I/Io	d (Å)	I/Io	d (Å)	d_{calc} (Å)	hkl		I/Io	d (Å)
		9.456	50	9.7	9.37	010		S	9.40
4.60	10	4.660	100	4.6	4.687	020		S	4.70
3.77	1			3.8	3.774	002		W	3.74
3.38	1				3.501	012		Vw	3.52
3.05	2			3.05	3.096	112		W	3.05
2.90	8			2.85	2.940	022		S	2.92
2.61	1w				2.600	130			
2.35	1w				2.343	040		M	2.34
2.12	<1				2.151	230		Vw	2.15
					2.141	132			
1.99	<1				2.046	140		Vw	2.03
					1.991	042			
1.87	3				1.887	004		S	1.87
					1.875	050			
1.747	3				1.751	024		W	1.74
1.593	1				1.604	242			
					1.587	143			
1.531	2				1.538	152			
1.409	1w				1.437	025		W	1.435
					1.405	161			
1.246	1w				1.258	006			
1.211	3				1.229	262		Vw	1.225
					1.215	026			
1.047	1				1.054	182			

a = 10.824   a = 10.756
c = 7.549 Å   c = 7.496 Å

* Debye-Scherrer camera 57.3 mm. FeKα (Mn-filtered) radiation, U = 20 kV, I = 25 mA, exposure 10 hours.
** Diffractometer DRON 2.0 U = 30kv, I = 30 mA. Scanning velocity 1 /min, sample had strong preferred orientation parallel to the cleavage.
*** TEM JEM-100C.

CRYSTAL STRUCTURE

The unit cell parameters for holfertite were determined with a Bruker P4 single-crystal diffractometer equipped with a CCD detector: hexagonal, a 10.824(2), c 7.549(2) Å. The crystal structure of holfertite, space group $P3$, $Z = 3$, was solved by direct methods and refined to R_1 21.1%; the high value of R_1 is due to the OD character of the structure. Full details are reported by Sokolova et al. (2005).

In the generalized structure model, chains of U and Ti polyhedra link through common vertices to form a framework with channels along [001]. The channels have a diameter of ~8 Å and contain eight sites which are partly occupied by Ca atoms and (H_2O) groups, giving the total channel constituents as $\{(H_2O)_{3.04}Ca_{0.25}\}$ apfu. In the crystal structure of holfertite, many of the 28 sites are only partly occupied. These partial occupancies are almost certainly related to the occurrence of vacancies at the U and Ca sites, and short-range coupling of these occupancies is almost certainly the cause of the OD nature of the structure.

COMPARISON WITH THE
MATERIAL OF FOORD et al. (1995)

Several years ago, Foord et al. (1995) reported on a possible new mineral species from Topaz Valley, Utah. They described the geology and mineralogy of the Topaz Valley area, and partially characterized a novel hydrated oxide of U-Ti-Ca heavy rare-earth elements from a locality in Starvation Canyon (the locality was mentioned by Robinson, 1993; Robinson and King, 1993) and from a locality about half a mile from Pismir Knolls. The amount of material available was small and the crystals did not diffract well. Moreover, low totals from electron-microprobe analyses suggested an H_2O content of 22 weight % by difference, and a TGA study indicated a weight loss of 35 weight % up to 1000°C. Perhaps because of these difficulties, a formula was not proposed for this material.

The material described by Foord et al. (1995) strongly resembles holfertite, and at least some of the material collected by them came from the same small pit in Starvation Canyon (Fig. 2) where we collected holfertite (although the pit has deepened in the intervening years, and holfertite was collected approximately 10 feet deeper than the material of Foord et al., 1995). The X-ray diffraction pattern and calculated cell-parameters reported by Foord et al. (1995) correspond to those reported here for holfertite. However, the chemical compositions, optical properties and densities of holfertite and the material of Foord et al. (1995) are completely different (Tables 1, 2). We analyzed a couple of dozen crystals and never found chemical compositions even close to the analysis given in Foord et al. (1995); indeed, all chemical compositions obtained by us are very similar to those reported in Table 1. Analysis of crystals provided to us by Forest Cureton (obtained by him from E. Foord) corresponds closely to the compositions obtained for type holfertite.

Comparison of the properties of holfertite and the Ca-U-Ti-HREE phase of Foord et al. (1995) (Table 1) provides us with a conundrum: (1) the color, habit, X-ray diffraction pattern, crystal class and cell dimensions are consistent with the two phases being identical; (2) on the other hand, the chemical composition, optical properties and density (reported by Foord et al.) indicate that the materials are completely different. It seems inconceivable that the differences in the two sets of data can be ascribed to errors in data measurement. Perhaps Foord et al. (1995) examined two completely different materials when measuring the properties that they report. In particular, Foord et al. (1995) report the Gandolfi pattern of their yellow crystals, and this is virtually identical to the pattern reported here for holfertite (Table 3). However, they also report the

density of the yellow needles as 2.64 g/cm^3 whereas we obtain >4.22 g/cm^3 for holfertite (which is compatible with the refined crystal-structure, Sokolova et al., 2005). The chemical, optical and density data are very different (e.g., $UO_3 \approx 76$ and 19 weight %, respectively). Also it seems unlikely that the 9 weight % Y_2O_3 + REE_2O_3 in Foord's material is a mistake. However, no other Y-bearing or REE-bearing mineral was found at the location so far except cerianite-(Ce) reported above (which contains only Ce^{4+} and no other REE or Y). Foord et al. (1995) do report the presence of whitish crystals from the pit at Starvation Canyon, but we did not find any of this material. As noted above, our crystals came from a point 10 feet deeper on the pit than the material collected by Foord et al. (1995). Resolution of this conundrum must await rediscovery of the unknown white crystals and composite white-yellow crystals.

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