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ADDITIONAL STUDIES ON MIXED URANYL OXIDE–HYDROXIDE HYDRATE ALTERATION PRODUCTS OF URANINITE FROM THE PALERMO AND RUGGLES GRANITIC PEGMATITES, GRAFTON COUNTY, NEW HAMPSHIRE

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ABSTRACT

Additional studies on an incompletely characterized secondary uranium "mineral" from the Ruggles and Palermo granitic pegmatites, New Hampshire, referred to as mineral "A" by Frondel (1956), reveal a mixture of schoepite-group minerals and related uranyl oxide–hydroxide hydrated compounds. A composite chemical analysis yielded (in wt.%): PbO 4.85 (EMP), UO_3 83.5 (EMP), BaO 0.675 (av. of EMP and ICP), CaO 0.167 (av. of EMP and ICP), K_2O 2.455 (av. of EMP and ICP), SrO 0.21 (ICP), ThO_2 0.85 (ICP), H_2O 6.9, Σ99.61. Powder-diffraction X-ray studies indicate a close resemblance in patterns between mineral "A" and several uranyl oxide–hydroxide hydrated minerals, including the schoepite family of minerals and $UO_2(OH)_2$. The powder-diffraction data for mineral "A" are most similar to those for synthetic $UO_{2.86}{\cdot}1.5H_2O$ and $UO_2(OH)_2$, but other phases are likely present as well. TGA analysis of both mineral "A" and metaschoepite show similar weight-loss and first derivative curves. The dominant losses are at 100°C, with secondary events at 400° and 600°C. IR spectra show the presence of (OH) and H_2O. Uraninite from both pegmatites, analyzed by LAM–ICP–MS, shows the presence of Th, Pb, K and Ca.

Keywords: mineral "A", uraninite, schoepite, $UO_2(OH)_2$, Ruggles pegmatite, Palermo pegmatite, New Hampshire.

SOMMAIRE

Des études additionnelles portant sur un minéral secondaire d'uranium des pegmatites granitiques de Ruggles et de Palermo, au New Hampshire, dont la caractérisation demeure incomplète [c'est le minéral "A" de Frondel (1956)], montrent qu'il s'agit d'un mélange de minéraux du groupe de la schoepite et de composés oxydes–hydroxydes hydratés d'uranyle. Une analyse chimique composée a donné, en poids, PbO 4.85 (microsonde électronique, MSE), UO_3 83.5 (MSE), BaO 0.675 (moyenne de MSE et plasma à couplage inductif, PCI), CaO 0.167 (moyenne de MSE et PCI), K_2O 2.455 (moyenne de MSE et PCI), SrO 0.21 (PCI), ThO_2 0.85 (PCI), H_2O 6.9, Σ99.61. Les études par diffraction X (méthode des poudres) révèlent une grande ressemblance entre les spectres du minéral "A" et plusieurs oxydes–hydroxydes hydratés d'uranyle, y inclus les minéraux du groupe de la schoepite et $UO_2(OH)_2$. D'après les données de diffraction X, le minéral "A" ressemble davantage aux composés synthétiques $UO_{2.86}{\cdot}1.5H_2O$ et $UO_2(OH)_2$, mais il est probable que d'autres phases sont aussi impliquées. Une analyse thermogravimétrique du minéral "A" et de la métashoepite montrent un taux de perte de poids et une courbe du premier dérivé assez semblables. Les pertes importantes sont à 100°C, avec des événements secondaires à 400° et à 600°C. Le spectre infra-rouge révèle la présence de (OH) et de H_2O. Des échantillons d'uraninite provenant des deux pegmatites, analysés par PCI avec ablation au laser et spectrométrie de masse, contiennent Th, Pb, K et Ca.

(Traduit par la Rédaction)

Mots-clés: minéral "A", uraninite, schoepite, $UO_2(OH)_2$, pegmatite de Ruggles, pegmatite de Palermo, New Hampshire.

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INTRODUCTION

An unknown and incompletely characterized "mineral" of uranium was originally described as phase "A" by Frondel (1956) as part of a study on "gummite". Additional studies were conducted on mineral "A" in an attempt to fully characterize it as a new species. Uraninite, from four localities, also was chemically studied, and their ages determined. Metaschoepite from Shinkolobwe, Zaire, was chemically analyzed and studied by thermogravimetric analysis (TGA).

Samples of phase "A" from two bodies of granitic pegmatite, the Palermo No. 1 and Ruggles, were used in this investigation. The Palermo No. 1 pegmatite is located near North Groton Village, Groton, Grafton County, New Hampshire, and the Ruggles pegmatite is located in Grafton, Grafton County, New Hampshire [see Fig. 1 of Korzeb *et al.* (1997)].

METHODS OF INVESTIGATION

Specimens of phase "A" were obtained from the collection of the Harvard University Mineralogical Museum (HUMM) collection and from Mr. Robert Whitmore, Weare, New Hampshire. Electron-microprobe, TGA, (LAM–ICP–MS) laser-ablation microprobe – inductively coupled plasma – mass spectrometry, and other analytical methods, including gas chromatography, X-ray diffraction (XRD), and transmission infrared spectrophotometry, were utilized during the study of mineral "A", uraninite and schoepite, and the resultant data were used to determine the nature of mineral "A". Unsuccessful attempts were made to determine unit-cell dimensions from powder-diffraction data using the zone-axis method of Visser (1969), and to interpret these data by Rietveld structure analysis by B.C. Chakoumakos at the Oak Ridge National Laboratory, Oak Ridge, Tennessee.

THE NATURE OF UNKNOWN "A"

Mineral "A" is one of a number of minerals identified in "gummite" (Frondel 1956). This mineral was found at six localities, all in granitic pegmatites, at North Wilton, Palermo, Ruggles, Alstead, and Beryl Mountain, New Hampshire and at Grassy Creek, Mitchell County, North Carolina. We believed it to be a new mineral species based on the X-ray patterns obtained. However, chemical and structural studies were not completed. Additional unpublished work was done on mineral "A" by Frondel (C. Frondel, pers. comm., 1994 and C.A. Francis, pers. comm., 1993). We have attempted to further clarify the status of Mineral "A" using material from Palermo No. 1 mine and Ruggles mine.

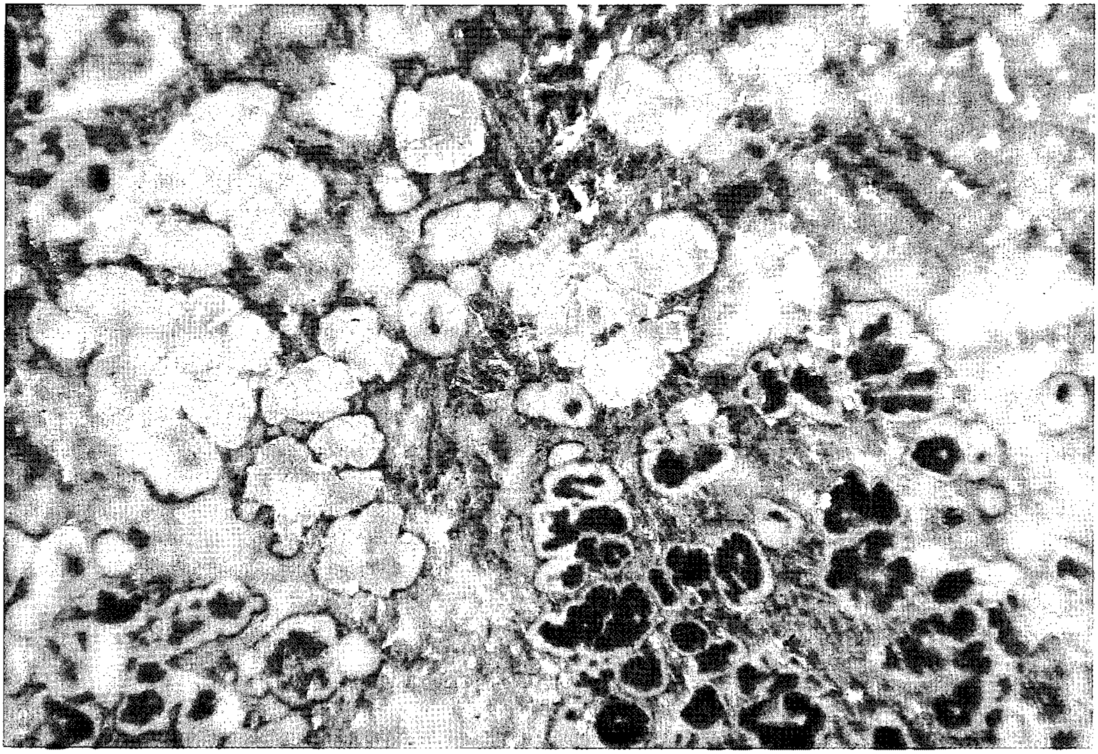

FIG. 1. Photograph showing the variable alteration of uraninite to mineral "A". Palermo no. 1. HUMM# 132005. The width of the field of view is 1.8 cm.

TABLE 1. CHEMICAL COMPOSITION OF MINERAL "A" FROM THE PALERMO MINE, GROTON, NEW HAMPSHIRE

Element or oxide	EMP and GC analyses (wt. %)	LA-ICP-MS analysis (wt. %)	Composite analysis (wt. %)
Pb	4.5[4]		
U	69.5[3]		
K	0.8[3]		
Ca	0.05[4]		
Ba	1.2[4]		
O	21.5[2]		
H	0.72[1]		
C	0.07[1]		
N	0.01[1]		
Total	98.35		
UO_3		85.3	83.5[5]
ThO_2		0.85	0.85[7]
PbO		3.00	4.85[5]
BaO		0.01	0.675[6]
ZrO_2		0.07	
Y_2O_3		0.02	
CaO		0.26	0.167[6]
K_2O		3.77	2.455[6]
SrO		0.21	0.21[7]
H_2O		6.9	6.9
Total		100.39	99.607

TGA total weight loss to 1,000°C = 8.75 wt.%; subtracting 1.86 wt.% because of conversion of UO_3 to U_3O_8 between 800° and 1000°C results in a total of 6.89 wt.%. Total water (780°C): 6.9 wt.%; agrees with TGA data. 0.72 wt.% H converted to H_2O is 6.5 wt.%, a value in reasonable agreement with the direct determination of water content. Chemical analyses were done by a number of methods: electron-microprobe analyses for U, O, Ba, K, Pb and Ca were done by K.L. Wenrich (USGS, Denver); gas chromatography analyses for H, C, and N, done on sample weights of 14.2, 3.8, and 23.4 mg, respectively, were done by C.J. Steen (USGS, Reston), and the water determination (total, +, and -) (Carl Fisher titration) was done by L.L. Jackson (USGS, Denver). [1] average of 3 determinations; [2] average of 4 analyses; [3] average of 5 analyses; [4] average of 6 analyses; [5] EMP; [6] average of EMP and ICP results; [7] ICP.

Mineral "A" is the first "mineral" to form as an alteration product of uraninite (Figs. 1, 2). We also have identified this "mineral" from several pegmatites in Maine and North Carolina, and from the Monticello fluorite district, New Mexico. In all cases, the "mineral" is a distinctive orange to yellow-orange color.

Results of a composite chemical analysis for mineral "A" are given in Table 1. Additional analytical data were provided by TGA for weight loss as a function of temperature, and by LAM–ICP–MS for more than 70 elements.

The analytical data in Table 1 indicate a composition very similar to that of two samples of "gummite" from Mitchell County, North Carolina, which yielded X-ray powder patterns for mineral "A" in Frondel's study. An average of the two compositions after subtraction for uranophane and Fe_2O_3 is: BaO 1.6 wt.%, PbO 8.25%, UO_3 81.5%, H_2O 8.65%, Σ100.0 wt.%.

Results of a LAM–ICP–MS analysis of mineral "A" from the Palermo mine are given in Table 2. All of the lead present is radiogenic, and the $^{206}Pb/^{207}Pb$ ratio is 0.053, yielding a calculated age of 329 Ma.

There are several significant differences between the electron-microprobe (EMP) results and the LAM–ICP–MS results. The LAM–ICP–MS showed no Ba, as opposed to 1.2 wt.% by EMP. The level of Pb determined by EMP is greater than that obtained by LAM–ICP–MS. On the other hand, K is higher by LAM–ICP–MS than by EMP.

FIG. 2. Photograph showing a closer view of the variable alteration of uraninite to mineral "A" and related secondary uranium minerals (Palermo no. 1). HUMM# 132005. The width of the field of view is 6 mm.

TABLE 2. POWDER DIFFRACTION DATA FOR MINERAL "A" FROM THE PALERMO AND RUGGLES MINES, GRAFTON COUNTY, NH.

Mineral "A" Ruggles mine Frondel (1956)		Mineral "A" Palermo mine (this paper)		Mineral "A" Palermo mine Powder diffractometer			$UO_{2.86}$·$1.5H_2O$ ICDD 23-1461		β-UO_2·$2H_2O$ ICDD 30-1403	
d_{obs}	I/I_o	d_{obs}	I/I_o	I/I_o	d_{obs}	d_{calc}	I/I_o	d_{obs}	I/I_o	d_{obs}
				4	7.87	7.38*			10	7.80
				19	7.40	7.38	100	7.53		
6.37	7	6.35	6	49	6.37	6.37			10	6.37
							5	5.92	2	5.91
							5	5.32		
				25	5.09	5.10	2	5.07		
									3	4.97
		4.70	1	3	4.80	4.72*				
									1	4.49
							5	4.26	10	4.26
							5	4.02	1	4.00
									3	3.98
		3.90	1	9	3.95	3.956				
				15	3.71	3.70	30	3.76	4	3.75
3.49	10	3.50	10	100	3.528	3.527	100	3.56	4	3.55
									10	3.49
				49	3.433	3.431			4	3.42
				15	3.376	3.380	50	3.32	7	3.36
									7	3.27
				81	3.241	3.239	100	3.22	1	3.21
3.18	7b	3.20	8b	97	3.192	3.197	55	3.17	6	3.16
									1	3.09
3.02	5	3.04	6b						6	3.05
									2	2.97
				6	2.989	2.986	5	2.96		
		2.90	3b	26	2.948	2.927*	20	2.92	7	2.91
				10	2.863	2.862	5	2.84	1	2.87
				1	2.814	2.809			3	2.80
							5	2.66	3	2.68
							5	2.645	5	2.64
2.61	3b	2.60	2	61	2.609	2.607	15	2.587	3	2.61
				15	2.551	2.550	5	2.513	2	2.54
				31	2.485	2.485	5	2.49	2	2.48
									4	2.44
				4	2.388	2.390	5	2.38	2	2.40
							10	2.36	3	2.33
				5	2.313	2.313			3	2.32
									2	2.30
							2	2.27		
							5	2.26	2	2.25
				12	2.206	2.206	5	2.22	2	2.22
							5	2.17	3	2.17
							5	2.16	1	2.16
				5	2.143	2.141			3	2.15
2.09	1	2.10	3	22	2.103	2.104	10	2.13	3	2.13
				20	2.053	2.053	10	2.05	4	2.03
				11	2.028	2.023	5	2.04		
2.00	2	2.00	2	31	1.994	1.994	30	2.00	3	2.01
				10	1.979	1.979	30	1.97	3	1.963
				7	1.951	1.954				
									3	1.945
1.92	3b	1.93	2				5	1.935	2	1.91
		1.87	1	5	1.881	1.881	10	1.88		
				2	1.823	1.821	10	1.82		
1.81	1	1.80	2							
				3	1.797	1.797				
				11	1.778	1.776	45	1.779		
							10	1.770		
1.75	3	1.75	3	46	1.748	1.748				
							35	1.732		
				13	1.717	1.716	10	1.723		

TABLE 2. — Continued

								1.5	1.66
								0.5	1.62
								1.5	1.61
				4	1.593	1.593			
								0.5	1.585
1.57	1							0.5	1.580
1.52	1	1.51	2	31	1.525	1.525			
								0.5	1.564
				7	1.484	1.486			
				12	1.438	1.438			
				1	1.403	1.403			
				7	1.370	1.370			
				2	1.329	1.329			
				7	1.307	1.308			
				2	1.295	1.295			

Note: Powder data collected using a Siemens D-500 instrument, 0.02° step increment, 20 second count times per increment, 45 kV, 35 mA, CuKα_1 (1.54059Å) radiation, NBS 940 silicon used as an internal standard. Data from Frondel (1956) are from Debye-Scherrer 114.6 mm films and data in column 2 are from 114.6 mm diameter Gandolfi films.

A convergent, five-cycle refinement for mineral "A" yields a schoepite-type orthorhombic cell with: *a* 14.110(3)Å *b* 16.831(4)Å *c* 14.766(9)Å. Lines marked with an asterisk were rejected in the refinement; all others were accepted. However, no extinction conditions were used for the refinement.

A possible interpretation based on available data is that we have a mixture of $UO_{2.86}\cdot 1.5H_2O$, β-$UO_2\cdot 2H_2O$ and other structural derivatives of the schoepite-type unit cell, which is supported by the chemistry which is that of schoepite and its structural derivatives.

The only minerals that are closely related chemically and structurally to mineral "A" are members of the schoepite family: schoepite, $[(UO_2)_8O_2(OH)_{12}](H_2O)_{12}$; metaschoepite, $UO_3\cdot 1$–$2H_2O$; dehydrated schoepite, $8[(UO_2O_{0.25})(OH)_{1.5}]$. In addition, at least two synthetic uranyl oxide hydrates [$UO_{2.86}\cdot 1.5H_2O$ (formerly known as "paraschoepite") and $UO_2(OH)_2$] bear some similarity to mineral "A". Crystal-chemical studies of the various hydrates of schoepite and other uranyl oxide–hydroxide hydrates were done by Christ & Clark (1960).

Infrared spectra (KBr pellet technique) of mineral "A" from 200 to 4,000 wavenumbers show the presence of both (OH) and H_2O. Additional unidentified vibrations are present at 2925 (triplet), 650, 620 and 465 wavenumbers (Fig. 3).

A determination of specific gravity was made on five grains of mineral "A", aggregating 7.5 mg, using a Berman microbalance. An average of three determinations gave 5.45(2). A calculated density for schoepite (fully hydrated) using the data in Finch *et al.* (1996a,

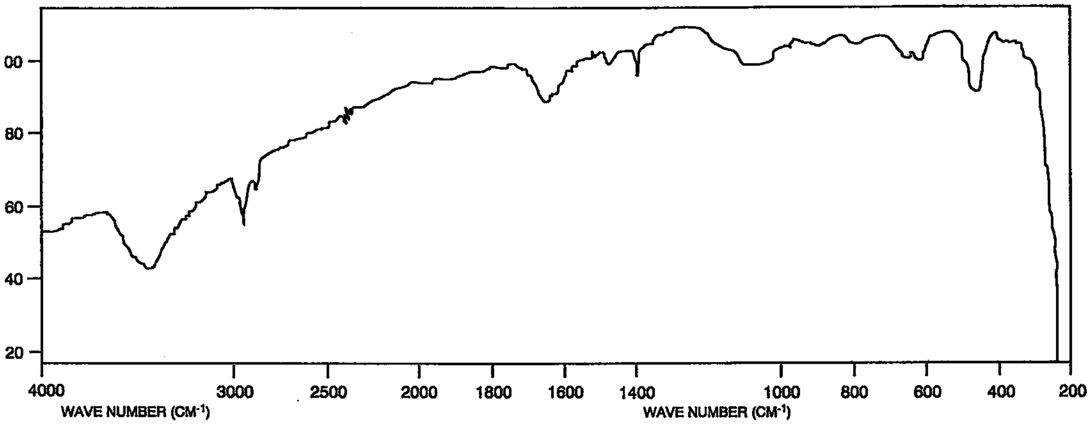

FIG. 3. Transmission infrared spectrum for mineral "A" from the Palermo mine.

1997) is 4.88, which is significantly less than the 5.45 determined for mineral "A".

X-ray-diffraction studies were done for mineral "A" using 114.7-mm diameter Gandolfi cameras, and Cu$K\alpha$ radiation. A powder pattern using NBS 640a silicon as an internal standard was made of mineral "A" on a Siemens D–500 diffractometer. All of the X-ray data indicate that there is a very close resemblance between the patterns for mineral "A" and the schoepite family of minerals, as well as several synthetic compounds. The X-ray data for synthetic $UO_{2.86}{\cdot}1.5H_2O$ and $UO_2(OH)_2$ most closely match those for mineral "A" (Table 2). Because of the extremely fine grain-size, no single-crystal X-ray studies are possible.

A unit cell for a schoepite-type mineral or other uranyl oxide–hydroxide hydrate was sought using the iterative zone-axis method of Visser (1969), but was not successful, even though the mineral is not metamict and gives sharp to relatively sharp reflections. An orthorhombic, schoepite-type cell, using no extinction conditions, was found (Table 2). However, at least five reflections are not indexable on the orthorhombic cell chosen. Calculated X-ray powder-diffraction data for schoepite agree well with data reported for natural schoepite (Finch *et al.* 1996b). The X-ray powder pattern for the sample of lemon-yellow "schoepite" from Shinkolobwe, Zaire is substantially different from that of mineral "A", and most closely matches that reported for synthetic metaschoepite (ICDD #18–1436).

In addition, a Rietveld refinement of the mineral was attempted through the courtesy of Dr. Brian C. Chakoumakos, Oak Ridge National Laboratories, Tennessee. Unfortunately, the high content of hydrogen, which produced a low peak-to-background ratio, prevented acquisition of usable data.

To check the reported water content of schoepite, and its type (*i.e.*, + or –), TGA analysis of 4.8 mg of yellow needles of a mineral labeled "schoepite" (now metaschoepite because of dehydration) from Shinkolobwe showed a rapid loss of 5 wt.% H_2O by 100°C, and then a slower loss of 6.8 wt.% H_2O by 1000°C. The additional loss occurs in two steps: 5 wt.% H_2O by 420°C, and the final 1.8 wt.% by 1000°C. The loss in the second step is due to the conversion of UO_3 to U_3O_8 (1.86 wt.% O loss). The total weight-loss, 11.8 wt.% H_2O, is also close to that (11.2 wt.%) required by the formula $UO_3{\cdot}2H_2O$ (metaschoepite). Some H_2O is readily released (by 100°C), but the remaining water is more tightly bound in the structure. Much, if not all, of the remaining water is likely present as hydroxyl.

Both mineral "A" and "schoepite" from Shinkolobwe show similar weight-loss and first derivative (rate) curves. The dominant losses are at 100°C, with secondary events at 400°C and 600°C. The chemistry of the "schoepite" was also checked by a LAM–ICP–MS analysis, which showed essentially pure $UO_3{\cdot}2H_2O$, with 0.25 wt.% PbO. Minor amounts of Na_2O (0.31 wt.%) and K_2O (0.69 wt.%) were also found. The very low lead content is indicative of a young age for the schoepite, as opposed to the primary uraninite, which contains 9.1 wt.% PbO and has a calculated age of 1,040 million years.

After heating (to 1000°C), the "schoepite" (metaschoepite) from Zaire and mineral "A" both give identical X-ray-diffraction patterns. The resulting black powder is the high-temperature rhombohedral form (ICDD file no. 8–244) of U_3O_8. Heating studies at 350° and 500°C indicate the existence of additional, as yet unidentified, phases intermediate between mineral "A" and U_3O_8.

STUDIES OF URANINITE

Because a significant quantity of potassium was detected by EMP and LAM–ICP–MS methods in the Ruggles mine sample of mineral "A", we decided also to analyze four samples of uraninite, all from granitic pegmatites. Sample provenance and results are given in Table 3. The Swamp Quarry uraninite is somewhat unusual in that it shows no signs of secondary alteration. The mineral is thorium-bearing (10.7 wt.% ThO_2) and contains 3.19 wt.% REE_2O_3.

The lead in uraninite from the Swamp Quarry (2.5 wt.% PbO), Palermo No. 1 (4.6 wt.% PbO) and Ruggles (5.9 wt.% PbO) pegmatites is all radiogenic. The $^{207}Pb/^{206}Pb$ ratio for the Swamp Quarry uraninite is 0.0526, which gives an age of 312 Ma. The $^{207}Pb/^{206}Pb$ ratio for the Palermo No.1 pegmatite is 0.0533, which gives an age of 342 Ma. For the Ruggles pegmatite, the $^{207}Pb/^{206}Pb$ ratio is 0.0530, which gives an age of 329 Ma. A high concentration of K was detected (by both EMP and LAM–ICP–MS) in the uraninite from both pegmatites (Table 3). Likewise, Finch & Ewing (1992) reported the presence of K from uraninite from Shinkolobwe. However, its role in the uraninite structure remains unknown.

TABLE 3. RESULTS OF LAM-ICP-MS ANALYSES OF URANITITE

Wt. % oxide	Swamp Quarry Topsham, ME	Ruggles N. Grafton, NH		Palermo #1 North Groton, NH	Shinkolobwe, Zaire
UO_3	83.0	89.4	90.06$^+$	91.3	87.5
ThO_2	10.7	0.73	0.43	0.35	0.003
PbO (total)	2.5	5.9	3.63	4.6	9.1
Na_2O	0.03	0.50	—	0.27	1.0
K_2O	0.01	2.24	—	2.55	<0.01
CaO	0.10	0.57	0.81	0.18	1.6
MnO	0.01	0.17	0.09	0.11	0.02
Fe_2O_3	0.06	<0.01	0.66	0.23	0.02
Y_2O_3	1.68	0.05	—	<0.01	0.066
REE_2O_3	1.51	0.21			
ZrO_2	<0.01	0.07	—	0.15	0.02
Mo_2O_3	<0.01				0.02
SrO	<0.01	0.08	—	0.01	—
SiO_2	0.2	0.47	0.80	0.46	—
Al_2O_3	0.05	0.15	0.44	0.07	—
MgO	<0.01	0.50	0.17	0.27	—
Totals	99.85	101.04	97.09*	100.55	99.35

Notes: * Shaub (1938) also includes 0.16 wt. % P_2O_5, H_2O (total): 0.73 + 1.74, insoluble residue 0.54; $^+$ reported as U_3O_8.

CONCLUSIONS

In view of the current state of knowledge of the pressure–temperature conditions of crystallization in granitic pegmatites, it is quite reasonable to have a hydroxyl-bearing uranium oxide be the first mineral to form from oxidation of primary uraninite. The calculated ages for the primary uraninite and surrounding mineral "A", at the Ruggles mine are the same (329 Ma), and all of the lead in both minerals is radiogenic. This favors a high-temperature hydrothermal origin for mineral "A". Completely hydrous minerals (including rutherfordine, soddyite, uranophane-β, phosphuranylite, and meta-autunite-I, with molecular water only, surround mineral "A", and were formed subsequent to it (Korzeb *et al.* 1997). A structure refinement of schoepite has now been completed (Finch *et al.* 1996a), and the phase transformations and crystallographic relations among schoepite, metaschoepite and dehydrated schoepite (Finch *et al.* 1997) have been clarified.

"Mineral A" is rare and does not occur in many pegmatite localities world-wide. The $f(O_2)$ seems to be an important factor in explaining its formation, because unoxidized and unaltered uraninite is found in pegmatites containing columbite–tantalite-group minerals, triphyllite or magnetite [*e.g.*, Topsham (Maine), the Spruce Pine district (North Carolina), Branchville and Portland (Connecticut), and Moss (Norway)]. If the $f(O_2)$ is sufficiently high, then any uraninite also present is partially or completely altered to minerals containing U^{6+}. On the basis of all of the above, it seems clear that mineral "A" was formed as a product of hydrothermal alteration of uraninite very shortly after crystallization of the uraninite within the host pegmatites.

Mineral "A" is not a single mineral, but is a mixture of at least two different uranyl oxide–hydroxide hydrates (a member or members of the schoepite group and other minerals currently known only as synthetic compounds) and perhaps other U-bearing minerals as well. This is indicated by the X-ray-diffraction data, the IR and TGA data, specific gravity and the chemical data.

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