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UMOHOITE FROM CAMERON, ARIZONA

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ABSTRACT

Studies indicate that the hydrous uranium-molybdate, umohoite, is a more widely distributed mineral than has been previously known. The original crystalline material was found at Marysvale, Utah. Similar crystalline material has since been reported by Coleman and Appleman from the Gas Hills district, Wyoming. Recently, a new occurrence in a fine-grained form has been discovered at Cameron, Arizona. A similar fine aggregate has been recognized in recently collected material from Marysvale. The identification of fine-grained umohoite has also been confirmed from an undisclosed locality in the U.S.S.R.

The change in lattice dimensions under *x*-ray bombardment or variable conditions of humidity and temperature has constituted a problem in the *x*-ray diffraction study of this mineral. In order to overcome this difficulty, the adsorption of the large ethylene glycol molecule into the water positions has been utilized to stabilize the umohoite structure. This may be accomplished without destruction of the structure.

Three coexistent structural modifications are recognized in umohoite. These are designated Modes 1, 2 and 3. The modes range in intensity of development as shown by *x*-ray diffraction. In fine-grained umohoite (Cameron, fine Marysvale and U.S.S.R.) Modes 2 and 3 are better developed than Mode 1. In coarser Marysvale umohoite Mode 1 is more prominent than Modes 2 and 3. The coexistence in the same mineral of three systematic sequences of lattice variation (or modes), each exhibiting a range in crystallinity in response to physical conditions, is an unusual mineralogical feature.

INTRODUCTION

Umohoite was first discovered (Brophy and Kerr, 1953) in the Freedom No. 2 mine at Marysvale, Utah, where it occurs in distinct bluish black plate-like crystals. Subsequently, Coleman and Appleman (1957) reported a second occurrence of crystalline umohoite aggregates at the Lucky Mc mine in the Gas Hills, Wyoming.

During the investigation of uranium mineralization and associated alteration at Cameron, Arizona, radioactive blue-black sooty masses and carbonaceous trash replacements at the Alyce Tolino mine were obtained through the courtesy of Mr. Page Blakemore, Jr., geologist for the Cameron Uranium Company. After a few days' exposure to the atmosphere, a freshly broken surface of this material develops a bright blue water soluble molybdenum efflorescence which is mainly ilsemannite. Such areas have been found to contain umohoite, although the aggregate differs in megascopic appearance from previously described crystals.

The three U.S. localities, as well as an occurrence in the U.S.S.R., indicate that this unusual hydrous uranium-molybdate may be more widespread than was considered likely at the time of the original dis-

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* Deceased. See note on page 1260.

covery. Knowledge of the existence of several other localities on the Colorado Plateau where secondary molybdenum is associated with uranium suggests the possibility that further study may disclose an even more widespread distribution of umohoite.

In the examination of the fine-grained molybdenum-uranium aggregate from Cameron, it was first found necessary to re-examine reference umohoite from Marysvale. A major difficulty recognized since the original investigation of umohoite by *x*-ray diffraction has been its tendency to produce shifting reflections as the mineral loses or gains water during *x*-ray bombardment. Kamhi (1959) in a restudy of individual Marysvale crystals carried on in this laboratory has shown that the lattice dimension normal to the flat flakes varies directly with ordinary variations in atmospheric humidity, and inversely with increasing temperature. In studying the Cameron material, it has been found necessary to re-examine this unusual behavior of the Marysvale umohoite. The study has also led to the investigation of umohoite shown in the U.S.S.R. exhibit at the first Geneva Conference on the Peaceful Uses of Atomic Energy (1955) and made available by Dr. A. P. Vinogradov of the Vernadsky Institute of Geochemistry and Analytical Chemistry, Moscow.

TECHNIQUE

In the examination of umohoite, it has been found desirable to stabilize the structure both during *x*-ray bombardment and under varying conditions of temperature and humidity. The analogy between the layered structure of umohoite, as well as the range in states of hydration, with the well-known shifts in basal spacing observed for the clay mineral montmorillonite, suggested the use of ethylene glycol to establish lattice stability. Umohoite crystals were chopped in distilled water in the Waring Blendor, sedimented onto a glass slide and then placed in an ethylene glycol atmosphere for two days in a closed container at room temperature. The slide was then transferred directly to the Norelco *x*-ray diffractometer (Fig. 1A).

No fundamental change in umohoite appears to accompany the adsorption of ethylene glycol. No shift in reflections as shown in Table 1 and Figure 1B appeared, nor were new lines produced. The only difference noted in the unglycolated and glycolated spacings lies in the intensity of reflections. After *x*-ray bombardment a gradual return takes place to the unglycolated intensities (Fig. 1C) which would not be the condition if a change in structure had occurred. After glycolation of the umohoite, changes under *x*-ray bombardment and variations with temperature and humidity disappear. The presence of a large organic molecule in the water positions seems to support the surrounding layers

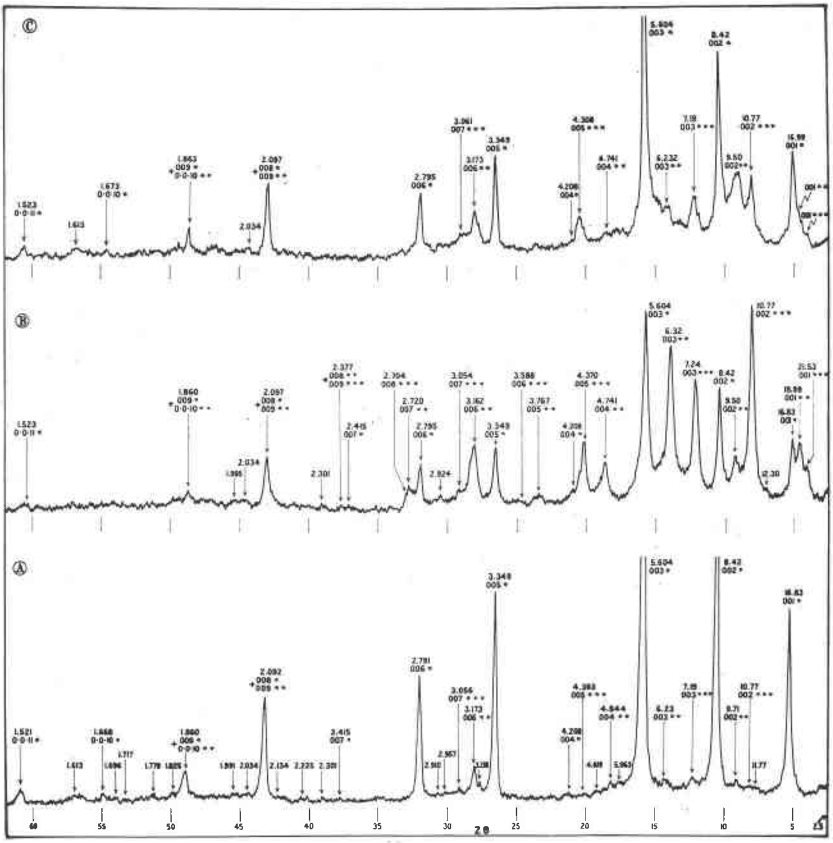

FIG. 1. *X*-ray diffractometer patterns of oriented, coarsely crystalline umohoite, Marysvale, Utah.

(A) Unglycolated material shows more intense Mode 1 reflections (=*) in contrast to Modes 2 and 3 (=** and =***). General *hkl* reflections are shown without asterisks.

(*B*) Glycolated material shows the stabilized structure. Modes 2 and 3 are increased in intensity while Mode 1 is decreased. Reflections 21.53 Å and 18.99 Å represent increased intensities which merely cause a slight asymmetry in pattern A.

(C) The same sample following *x*-ray bombardment. The pattern returns toward type A. Mode 1 shows stronger reflections than Modes 2 and 3. Asymmetry returns to peak 16.99 Å which corresponds to 16.83 Å of A.

and inhibit their shift or collapse with dehydration. It seems possible that the glycolated condition is in effect equivalent to a maximum state of hydration.

Study of the glycolated crystalline umohoite *x*-ray diffraction pattern (Fig. 1) shows that there are three distinct first order (001) reflections in which values are respectively: (1) 16.83 Å, (2) 18.99 Å and (3) 21.53 Å. Each stands at the head of a successive sequence of about 12 higher order reflections all present in the same pattern. These groups are designated Modes 1, 2 and 3. Table 2 compares the calculated interplanar spacings with observed diffractometer values for each of the three modes. The intensities of each mode (Table 2) are determined independently and are based on the intensity of the strongest reflection for each mode.

It is noted that except for the weak 4.2, 2.4 and 1.668 Å spacings in unglycolated crystalline umohoite, the Mode 1 (Fig. 1) reflections before glycolation are stronger than those given by Modes 2 and 3. The adsorption of the organic molecule appears systematically to strengthen the intensity of Mode 2 and 3 reflections (Fig. 1), and to decrease that of

TABLE 1. *X*-RAY DIFFRACTION POWDER DATA FOR COARSELY CRYSTALLINE UMOHOITE, MARYSVALE, UTAH

Cu radiation, Ni filter λ=1.5418 Å

(no glycol)		(glycolated)		(no glycol)		(glycolated)	
*d*Å	I	*d*Å	I	*d*Å	I	*d*Å	I
		21.53	15	2.957	2		
		18.99	29	2.910	2	2.924	5
16.83	68	16.83	31	2.791	45	2.795	22
11.77	2	12.30	5			2.720	13
10.77	2	10.77***	100			2.704	9
9.71	4	9.50	13	2.415	2	2.415	3
8.42	100	8.42	53			2.377	3
7.19	5	7.24	57	2.301	2	2.301	2
6.23	4	6.32**	66	2.225	2		
5.604*	100	5.604	83	2.134	3		
5.063	2			2.092	37	2.097	26
4.844	3	4.741	19	2.034	2	2.034	4
4.619	3			1.991	2	1.995	4
4.393	2	4.370	26	1.860	10	1.860	7
4.208	3	4.208	4	1.825	3		
		3.767	5	1.779	3		
		3.588	3	1.717	2		
3.349	74	3.349	31	1.696	2		
3.218	3			1.668	3		
3.173	10	3.162	29	1.613	3		
3.056	3	3.054	5	1.521	6	1.523	4

* Strong Mode 1.

** Strong Mode 2.

*** Strong Mode 3.

TABLE 2. *X*-RAY DIFFRACTION POWDER DATA FOR MODES RECOGNIZED IN GLYCOLATED COARSELY CRYSTALLINE UMOHOITE, MARYSVALE, UTAH

Cu radiation, Ni filter λ = 1.5418 Å

	Mode 3		Mode 2		Mode 1	
hkl	*d*Å obs. (calc.)	I	*d*Å obs. (calc.)	I	*d*Å obs. (calc.)	I
001	21.53 (21.57)	18	18.99 (18.92)	44	16.83 (16.80)	37
002	10.77 (10.79)	100	9.50 (9.46)	20	8.42 (8.40)	63
003	7.24 (7.19)	57	6.32 (6.31)	100	5.604 (5.60)	100
004	— (5.39)		4.471 (4.73)	29	4.208 (4.20)	5
005	4.37 (4.31)	26	3.767 (3.78)	9	3.349 (3.36)	37
006	3.588 (3.60)	3	3.162 (3.15)	42	2.795 (2.80)	26
007	3.054 (3.08)	9	2.720 (2.70)	20	2.415 (2.40)	4
008	2.704 (2.70)	9	2.377 (2.377)	4	2.097 (2.10)	33
009	2.377 (2.40)	4	2.097 (2.10)	40	1.860 (1.37)	9
0·0·10	— (2.16)		1.860 (1.89)	11	— (1.68)	
0·0·11	— (1.96)		— (1.72)		1.523 (1.53)	5
0·0·12	— (1.80)		— (1.58)		— (1.40)	

Mode 1. The latter, however, remains the strongest. After bombardment of glycolated Marysvale umohoite (Fig. 1) Mode 1 becomes more intense and Modes 2 and 3 decrease in intensity. Although the 18.9 Å and 21.5 Å glycolated spacings (Fig. 1) of Modes 2 and 3 (Table 1) appear to be new, a definite asymmetry (Fig. 1) toward the lower angles is present in both the unglycolated and glycolated bombarded 16.8 Å reflection. Since the (002) reflections at 10.7 and 9.5 Å are much more intense, it seems likely that before glycolation these weaker (001) reflections are only strong enough to produce asymmetry. Likewise, the 3.7 Å and 3.5 Å Mode 2 and 3 reflections are weak in the glycolated material and do not appear at all in the unglycolated specimen. The 1.860 Å line is believed to be principally a Mode 1 (009) reflection, although this is also the spacing for the Mode 2 (0·0·10) 1.90 Å reflection. After glycolation this 1.860 Å peak decreases in intensity; after bombardment it becomes stronger. This behavior is characteristic of Mode 1 reflections; so the peak is assigned to this mode. Likewise, the 2.092 Å reflection decreases in intensity with glycolation, and increases after bombardment; so it is believed to be mainly Mode 1 (008), although it overlaps the (009) Mode 2 spacing.

FINE-GRAINED CAMERON UMOHOITE

Microscopic Observation

Thin-section study shows that the Cameron material is blue-black, opaque and contains no pleochroic blue-green umohoite crystals as reported either by Brophy and Kerr (1953) or Coleman and Appleman (1957). Scattered grains of angular quartz are associated with prominent bands of carbonate and pyrite. A few fragments of an unidentified secondary uranium mineral occur with the umohoite. Polished surfaces of this black fine-grained umohoite are isotropic. The apparent lack of double refraction is attributed to the extremely finely divided state. Associated with the umohoite are small cubes of cobalt-rich pyrite which enclose marcasite.

X-ray Diffraction

The identification of fine-grained Cameron umohoite depends largely on *x*-ray diffraction data. The material was purified by stirring in cool, distilled water in order to remove ilsemannite and any other water soluble impurities. After the water soluble fraction was decanted, more cool, distilled water was added to the residue, after which it was stirred briefly and allowed to settle for a few minutes. Umohoite (Sp. Gr. 4.55–4.93) settles from suspension after pyrite (Sp. Gr. 4.95–5.10) and before quartz (Sp. Gr. 2.653–2.660). However, no fine-grained umohoite fraction was separated which was entirely free from quartz. Umohoite was next sedimented onto a glass slide so that a preferred orientation developed (Brophy and Kerr, 1953). The slide was mounted directly in a Norelco *x*-ray diffractometer.

X-ray diffraction investigation shows that the fine-grained Cameron umohoite develops a preferred orientation and responds to ethylene glycol as do the coarse Marysvale umohoite crystals. Unoriented samples of both forms of umohoite give weak, diffuse patterns which improve moderately after orientation. Thus the Cameron particles which are estimated to be a few microns in diameter develop a preferred orientation which is similar to that of the coarse Marysvale crystals which are tenths of a millimeter in diameter. The adsorption of ethylene glycol stabilizes the Cameron umohoite lattice in the same way that it does the Marysvale. Therefore the *x*-ray diffraction pattern shows a consistent increase of Mode 2 and 3 intensities (Fig. 2). After bombardment by *x*-rays, the

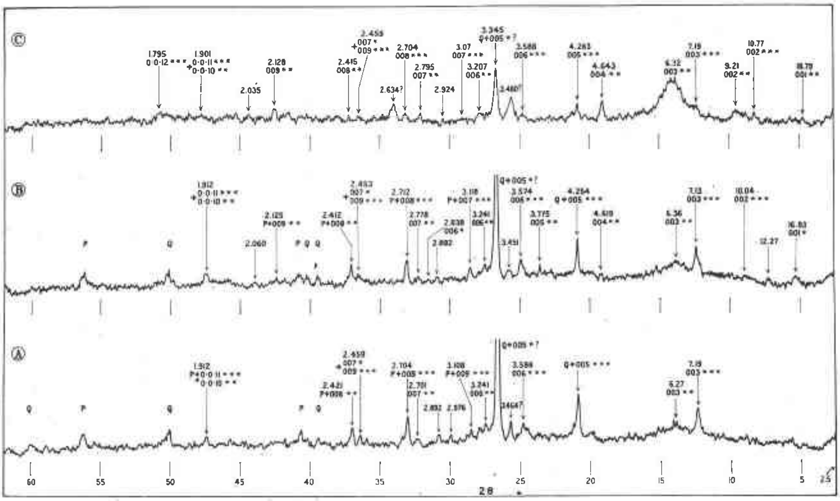

FIG. 2. *X*-ray diffractometer patterns of oriented fine-grained umohoite, Cameron, Arizona.

(A) Unglycolated material shows characteristic Mode 2 and Mode 3 reflections. However, these reflections are weaker than those of the coarsely crystalline umohoite (Fig. 1). Mode 1 reflections are weak or missing. Quartz (Q) and pyrite (P) mask several umohoite 00*l* reflections.

(B) Ethylene glycol adsorption strengthens coexistent Modes 2 and 3. The strong 7.13 Å reflection shows that in this specimen Mode 3 is more prominent than Mode 2.

(C) In this glycolated specimen the intensity of the 6.32 Å reflection shows that Mode 2 is stronger than Mode 3.

intensity of these modes decreases to resemble that of the unglycolated specimen which also is similar to the behavior of the coarse crystals. Although the Cameron diffraction patterns (Fig. 2) do not approach the definition and intensity of those of the Marysvale crystals (Fig. 1), they are similar.

The *x*-ray diffraction pattern of the Cameron umohoite (Table 3) is characterized by prominent Mode 2 and 3 reflections and weak or missing Mode 1 reflections. This differs from crystalline Marysvale umohoite which has strong Mode 1 reflections, and relatively weaker Mode 2 and 3 reflections. In one Cameron specimen (Fig. 2C) Mode 2 is stronger than coexistent Mode 3 (Table 3); whereas in another, Mode 3 is dominant (Fig. 2B). In the stronger Mode 2 pattern it is interesting to note that the 18.79 Å (001) Mode 2 reflection appears only after glycolation as in the diffraction pattern of the coarsely crystalline Marysvale umohoite. The intensities of the Cameron Mode 2 and 3 reflections also have the same relation to each other as those in the Marysvale crystals. However, an unexplained exception occurs in the Cameron strong Mode 3 pattern (Table 3, Fig. 2B) since the 7.1 Å (003) reflection is considerably stronger than the 10 Å (002) reflection, whereas in the Marysvale crystals (Table 1, Fig. 1) the 10 Å spacing is stronger.

In the Cameron material (Fig. 2B) in which Mode 3 is prominent, Mode 1 is fairly well developed. The following reflections from this mode appear: (001) 16.83, (006) 2.838 and (007) 2.453 Å. Quartz may mask the strong umohoite Mode 1 3.3 Å line and pyrite the umohoite Mode 1 2. 7Å reflection. However, the strongest Mode 1 reflection in the coarsely crystalline umohoite at 5.6 Å is missing in the Cameron specimens. At present there is no direct evidence for the degree to which Modes 1, 2 and 3 may be developed. However, further investigation may reveal the relationship of the oxidation state, hydration and uranium-molybdenum ratio to the intensities of modes in umohoite.

X-ray Spectrograph

X-ray spectrographic analyses confirm the presence of uranium and molybdenum. These elements are accompanied in decreasing order by: silica, sulfur, iron, cobalt, nickel, arsenic and thallium. The presence of thallium as an associated impurity is interesting, but the site of its occurrence is unknown. It is not localized to any extent in the accompanying pyrite. The 3.4 Å and 2.6 Å (Fig. 2) unidentified *x*-ray diffraction reflections in the Cameron specimens may be caused by a mineral which contains some of these accompanying elements. It is worthy of note that no evidence for the presence of molybdenite or molybdite was found during the investigation.

TABLE 3. *X*-RAY DIFFRACTION POWDER DATA FOR FINE-GRAINED GLYCOLATED UMOHOITE

hkl	Mode	Cameron, Arizona				Marysvale, Utah		U.S.S.R.	
		*d*Å	I	*d*Å	I	*d*Å	I	*d*Å	I
001	(3)							19.62	100
	(2)			18.79	20	18.99**	86	18.79	100
	(1)	16.83	50			16.67*	100	17.01	86
002	(3)	10.04	40	10.77	33	10.77	14	9.93	43
	(2)			9.21	20				
	(1)							8.34	57
003	(3)	7.13***	100	7.19	33	7.13***	43	7.36	43
	(2)	6.36	40	6.32**	100	6.02	71	6.34**	57
	(1)					5.604	29	5.607*	43
004	(3)								
	(2)	4.619	30	4.643	47	4.692	43		
	(1)								
005	(3)	4.254^{+Q}	100	4.263^{+Q}	40	4.263^{+Q}	100	4.350***	57
	(2)	3.775	50			3.675	43	3.767	43
	(1)	3.342^{+Q}	100	3.345^{+Q}	100	3.349^{+Q}	100	3.349^{+Q}	100
006	(3)	3.574	60	3.588	20	3.588	14	3.631	57
	(2)	3.241	40	3.207	20	3.184	14	3.184	100
	(1)	2.838	20						
007	(3)	3.118^{+p}	50	3.07	13	3.104	29	3.051	43
	(2)	2.778	30	2.795	27			2.769	43
	(1)	2.453	30	2.459	20				
008	(3)	2.712^{+p}	80	2.704	20	2.712	14		
	(2)	2.412^{+p}	80	2.415	13	2.453^{+Q}	100	2.453^{+Q}	100
	(1)								
009	(3)	2.453	30	2.459	20				
	(2)	2.125	40	2.128	27	2.123^{+Q}	86	2.127^{+Q}	100
	(1)								
0·0·10	(3)								
	(2)	1.912^{+p}	40	1.901	13			1.862	29
	(1)								
0·0·11	(3)	1.912^{+p}	40	1.901	13			1.862	29
	(2)								
	(1)								
0·0·12	(3)			1.795	20			1.795	43
	(2)								
	(1)								

p pyrite.
Q quartz.
* Strong Mode 1.
** Strong Mode 2.
*** Strong Mode 3.

ADDITIONAL FINE-GRAINED UMOHOITE LOCALITIES

Marysvale, Utah

Recently, fine-grained blue-black umohoite masses have been found at Marysvale, Utah. The occurrence is significant as it establishes the presence of this form of umohoite at the type locality. The nature of its geologic relation to coarsely crystalline umohoite requires further study.

The intensity and definition of the *x*-ray diffraction pattern of this material are similar to that of the Cameron umohoite. An oriented, glycolated specimen (Fig. 3, Table 3) shows Mode 1, 2 and 3 reflections. Mode 2 is somewhat better developed than Mode 3. Since pyrite is not detected by *x*-ray diffraction in this fine-grained umohoite, the Mode 3 3.10 Å (007) and 2.7 Å (008) umohoite reflections are not obscured. This confirms the assignment of these spacings in the pyrite contaminated Cameron material (Table 3, Fig. 2) to umohoite masked by pyrite. It is interesting to note that as in the Cameron umohoite the Mode 1 16.67 Å (001) reflection is conspicuous, although several higher orders are missing.

X-ray spectrographic analyses show that uranium and molybdenum are accompanied by iron, thallium and arsenic as contamination. Although thallium and arsenic impurities are not reported by Brophy and Kerr (1953) in the coarsely crystalline Marysvale umohoite, they are associated with the fine-grained umohoite from Marysvale and

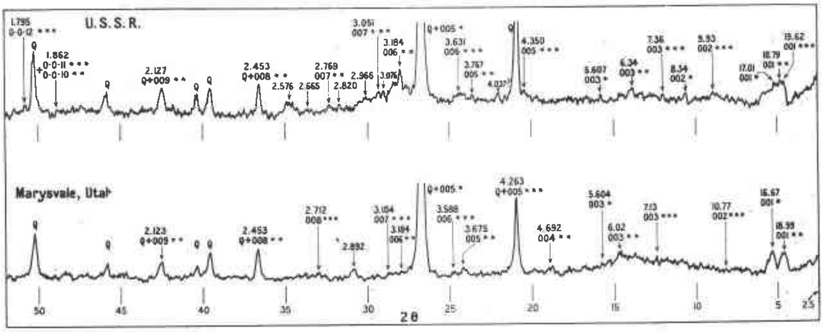

FIG. 3. *X*-ray diffractometer powder patterns of oriented and glycolated, fine-grained umohoite from Marysvale, Utah and the U.S.S.R.

Marysvale material shows the characteristic coexistent modes which are identified in the coarse crystals (Fig. 1). Mode 2 is somewhat better developed than Mode 3. Note that as in one Cameron specimen (Fig. 2B) the 16.67 Å reflection is conspicuous, although many Mode 1 higher orders are missing.

The U.S.S.R. specimen has prominent coexistent Mode 1, 2 and 3 reflections. Therefore this material more closely resembles the coarsely crystalline Marysvale umohoite (1B) than the fine-grained specimens from Cameron and Marysvale.

Cameron. The association of thallium and arsenic as impurities at both localities may have some genetic significance.

U.S.S.R.

A specimen of fine-grained umohoite from an unknown locality in the U.S.S.R. was sent to the Mineralogical Laboratory, Columbia University, by A. P. Vinogradov of the Vernadsky Institute of Geochemistry and Analytical Chemistry, Moscow. The specimen is very interesting since it comes from a distant locality and suggests that umohoite may be rather widespread. The fine-grained black umohoite matrix includes granules of quartz and unidentified vitreous dark brown material. A soft yellow mineral occurs as blebs which are scattered throughout the specimen and as a coating along one edge.

X-ray spectrographic analyses confirm the presence of uranium and molybdenum in this material. Thallium and arsenic were not detected in this fine-grained umohoite. Iron, vanadium and lead are the principal accompanying impurities.

The *x*-ray diffraction pattern (Fig. 3, Table 3) of the oriented, glycolated black matrix is similar to that of the fine-grained umohoite from Cameron and Marysvale. Vinogradov (written communication) suggested that this specimen contained more than one form of umohoite. An interesting feature of the diffraction pattern (Table 3, Fig. 3) is that it shows prominent Mode 1 reflections. Coexistent Modes 2 and 3 also are well developed so that this umohoite is the most similar of the three fine-grained umohoites to the coarsely crystalline umohoite from Marysvale. Its tendency to orient and respond to ethylene glycol also is similar. Thus after glycolation Mode 2 and 3 reflections are strengthened whereas Mode 1 reflections are weakened. This is particularly evident in the glycolated (001) reflections since Modes 2 and 3 are present, whereas before glycolation they appear only as a diffuse asymmetric reflection. In addition to quartz, reflections from an unidentified impurity are present; however, they do not interfere with the identification of umohoite.

CONCLUSIONS

Umohoite adsorbs ethylene glycol at room temperature and pressure. This stablizes the structure without a shift of interplanar spacings or the appearance of new reflections. Once stability is established, there are no lattice dimension changes with varying conditions of temperature and humidity. The presence of the large organic molecule in the water positions seems to support the surrounding layers and inhibit their shift or collapse. It is possible that the glycolated condition is equivalent to a maximum hydration state.

The presence of three coexistent structural modifications, Modes 1, 2 and 3, in umohoite is established. *X*-ray diffraction study shows that each mode exhibits an orderly sequence of reflections, and all three groups appear in the same diffraction pattern. With the adsorption of ethylene glycol there is a systematic increase in the intensities of Modes 2 and 3, along with a decrease of Mode 1 intensities. This response to ethylene glycol is a characteristic property of umohoite which is useful in identification, and should provide assistance in future structural studies.

A fine-grained form of umohoite has been recognized. It gives a weak and somewhat diffuse *x*-ray diffraction pattern, but the same three modes are present as in the coarse crystals. Modes 2 and 3 tend to be somewhat more prominent than Mode 1 in the fine-grained form, whereas Mode 1 is stronger in the coarse crystals. The relative intensities of Modes 2 and 3 may vary, even among specimens from the same locality.

Coarse umohoite crystals from Marysvale, Utah (Brophy and Kerr, 1953) and the Gas Hills area, Wyoming (Coleman and Appleman, 1957) have been described. The occurrence at Cameron, Arizona, of fine-grained umohoite establishes a third locality in the United States for this hydrous uranium-molybdate. The identity of an interesting specimen of fine-grained umohoite from the U.S.S.R. is confirmed. Thus, umohoite is more widespread in occurrence than was known previously, and the fine-grained form may be considerably more abundant than is now known.

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Miss Peggy-Kay Hamilton, Research Associate in Mineralogy, Columbia University, passed away on September 19, following an operation. Miss Hamilton was a Fellow of the Mineralogical Society of America.