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COCONINOITE, A NEW URANIUM MINERAL FROM UTAH AND ARIZONA[1]

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ABSTRACT

Coconinoite, named for Coconino County, Arizona, is a new uranium mineral with the probable formula

$$Fe_2Al_2(UO_2)_2(PO_4)_4(SO_4)(OH)_2 \cdot 20\ H_2O.$$

It has been found in the oxidized zone of uranium deposits in Utah and Arizona and occurrences have been documented from Wyoming, New Hampshire, and from two unknown localities. Coconinoite is light creamy yellow, nonfluorescent, and soft. It forms aggregates of microcrystalline lathlike to platy grains which have a maximum observed length of 20 microns. Measured specific gravity is 2.70 and specific gravity calculated from the Gladstone and Dale rule is 2.68. It is biaxial (−) with $\alpha = 1.550 \pm 0.005$, $\beta = 1.588 \pm 0.005$, $\gamma = 1.590 \pm 0.005$, 2V about 40°; the pleochroism is α = colorless, $\beta = \gamma$ = pale yellow. Although the mineral is too fine grained for single-crystal *x*-ray study, monoclinic symmetry is suggested by extinction angles. The d-spacings of the strongest lines are 11.05 Å (VVS), 5.52(S), 3.28(M), 3.67(M) and 5.61(M). The formula is based on microchemical analyses of samples from Utah and Arizona. The sample from the Blackwater No. 4 Mine, Apache County, Arizona, contained (in weight per cent) $Fe_2O_3 = 9.7$, $Al_2O_3 = 6.6$, $UO_3 = 34.9$, $P_2O_5 = 18.3$, $SO_3 = 5.4$, $H_2O- = 18.2$, $H_2O+ = 5.8$, acid insol. = 0.5, $CaO = 0.1$, $CO_2 = <0.1$, $Na_2O = <0.1$, Total = 99.5. The sample from the Jomac Mine, White Canyon, San Juan County, Utah, contained (in weight per cent) $Fe_2O_3 = 10.3$, $Al_2O_3 = 6.5$, $UO_3 = 34.2$, $P_2O_5 = 17.7$, $SO_3 = 6.1$, $H_2O- = 18.8$, $H_2O+ = 4.6$, acid insol. = 1.0, $CaO = <0.1$, $CO_2 = 0.7$, $Na_2O = 0.1$, Total = 100.0.

Differential thermal analysis study of coconinoite shows a large endothermic peak between 150° and 250° C. which corresponds to the water loss and a smaller endothermic peak near 800° C. which corresponds to the loss of SO_2.

INTRODUCTION AND ACKNOWLEDGMENTS

Coconinoite was first collected in 1953 by A. F. Trites, Jr., U. S. Geological Survey, during his study of the uranium deposits of the White Canyon area, San Juan County, Utah (Trites and Hadd, 1958). Two samples from the Jomac mine, studied by Trites, A. D. Weeks and D. R.

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[1] Publication authorized by the Director, U. S. Geological Survey.

Ross, were considered a new mineral on the basis of the x-ray diffraction powder pattern and the combination of Fe, Al, U and P found by spectrographic analyses. However, the pattern was very complex, and the material was too fine grained for single crystal study.

Fortunately, many additional samples, from a total of eight different locations, were submitted to the U. S. Geological Survey for identification. D. R. Ross noted coconinoite in a sample from an unspecified locality in New Hampshire, and in two samples from unknown localities (submitted by the public). R. P. Marquiss identified it in samples from two mines in Arizona and from an unspecified locality in Wyoming. Samples large enough for further investigation, from new localities in Arizona, were studied by E. J. Young.

Thanks are gratefully extended to A. F. Trites, Jr., and R. G. Petersen, U. S. Geological Survey, and to R. A. Laverty, E. B. Gross and S. R. Austin, Atomic Energy Comm., who submitted samples; to D. R. Ross and R. P. Marquiss, who took x-ray diffraction patterns, and to George Ashby for his effort to index the complex powder patterns. Malcolm Ross and Mary E. Mrose critically reviewed the manuscript.

The preliminary investigation of this mineral was part of a program of geologic and mineralogic studies made by the U. S. Geological Survey on behalf of the Raw Materials Division of the Atomic Energy Commission.

The new mineral is named coconinoite for Coconino County, Arizona, where samples were obtained from the Sun Valley mine and the Huskon No. 7 mine. The name has been approved by the New Minerals and Mineral Names Committee, I.M.A.

OCCURRENCE

Coconinoite occurs in the oxidized zone of sandstone-type uranium deposits in Triassic strata at four mines in the Colorado Plateau.

Coconinoite was first found in two samples from the Jomac mine (T. 34 S., R. 14 E., Salt Lake principal meridian) in a uranium deposit about 13 miles northeast of the town of White Canyon, San Juan County, Utah. It was in sandy siltstone just above the contact between the Moenkopi Formation (Early and Middle(?) Triassic) and the Shinarump Member of the Chinle Formation (Late Triassic). Trites and Hadd (1958, Fig. 24 and p. 178–9) illustrated the occurrence and referred to the mineral as "unidentified massive yellow uranium mineral." It is associated with gypsum, jarosite, limonite, quartz, clay and coalified wood. Sample T-J2-53 has crusts of coconinoite on a layer of gypsum coating buff-colored clay. These samples were used for the preliminary study by Weeks and Ross and for a microchemical analysis by Meyrowitz.

Coconinoite from the Sun Valley mine, Coconino County, Arizona (sample EVC-6) was collected by R. G. Petersen (1957) and studied by Young. This occurrence was also at the contact between the Moenkopi Formation and the Shinarump Member of the Chinle Formation. The coconinoite is in seams 1 mm or less thick, predominantly along the bedding planes of a light-colored, arkosic sandstone that is fine grained, poorly sorted, and thinly bedded. Gypsum occurs as visible crystals with the coconinoite and it is present on joint surfaces. This sample (EVC-6) was used for study of optical properties and *x*-ray patterns before and after dehydration.

Two other localities in Arizona which were sampled are in similar geologic settings. Sample 1715, from the Blackwater No. 4 mine in Apache County, Arizona, yielded sufficient material for a thermogravimetric study and differential thermal analysis as well as a microchemical analysis. A sample from the Huskon No. 7 mine near Cameron, Coconino County, Arizona, was identified by its *x*-ray diffraction powder pattern.

No geologic data are available for the other four samples submitted for identification—two from unknown localities, one from Wyoming, and one from New Hampshire, which is certainly in a different geologic environment from that of the Triassic deposits on the Colorado Plateau.

The association of coconinoite with gypsum, jarosite, limonite and clay indicates an environment of oxidizing pyritic, low-valent uranium ore, such as found in various places in White Canyon. Coconinoite is more likely to occur in uranium deposits which contain little or no vanadium or copper.

PHYSICAL PROPERTIES

Coconinoite is light creamy yellow and consists of aggregates of microcrystalline grains. Only in sample EVC-6 from the Sun Valley mine were there some lathlike grains as large as 6 by 20 microns. The mineral is soft, although its hardness cannot be measured accurately. The porosity of the microcrystalline aggregates precludes an accurate specific gravity measurement by the Berman balance or pycnometer. An approximate measurement was made by diluting bromoform with dimethylformamide in a 10-ml graduated cylinder until disaggregated mineral grains were suspended just beneath the surface of the liquid. The tube was stoppered and let stand overnight to insure complete wetting of the sample. The specific gravity of the liquid (and sample) was 2.70. This agrees satisfactorily with a value of 2.68 calculated from the chemical analysis and the mean index of refraction according to the rule of Gladstone and Dale. Coconinoite is nonfluorescent in both short wave (2537 Å) and long wave (3660 Å) ultraviolet light. It is also nonfluorescent after dehydration (see hydration study).

OPTICAL PROPERTIES

Most of the samples were too fine grained for detailed optical study. In sample EVC-6 from the Sun Valley mine, the lathlike grains are more amenable for study than the equant grains, but all are so small that a larger-than-normal error is given for the indices of refraction. The α index, 1.550 ± 0.005, is a good approximation for the minimum index, and γ, 1.590 ± 0.005, is approximately the maximum index. The β index is 1.586 ± 0.005. As shown in Fig. 1, Y is inclined at a small angle to the longest dimension of the grains and the optic plane is transverse to the laths. This orientation explains why grains with positive elongation show parallel extinction, and grains with negative elongation have inclined extinction. By means of the universal stage, 2V was measured on grains mounted in glyptal at room temperature. The measurements averaged about 40°, and ranged from 28° to 43°. Calculation of β using a 2V of 40° gives 1.588, which is in good agreement with the measured 1.586.

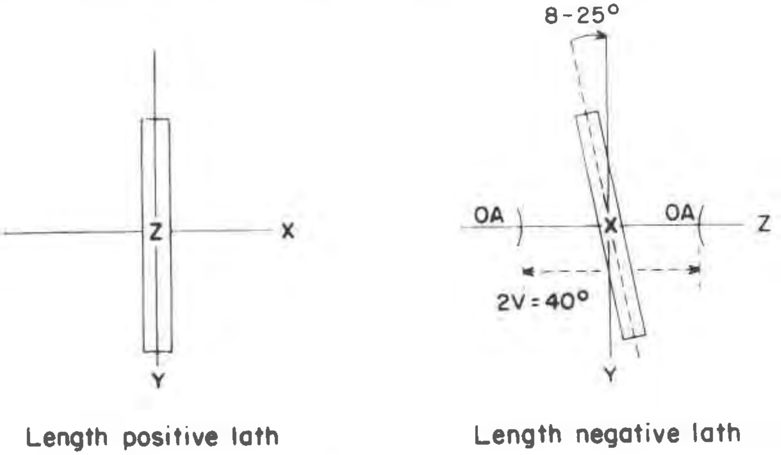

FIG. 1. Optic orientation of coconinoite from the Sun Valley Mine, Arizona (sample EVC-6).

Aroclor, which requires heating, was used in the first attempt to mount the grains for the universal stage. It was suspected that the heating had caused dehydration and changes in the indices of refraction and 2V. The optical properties of both the natural hydrated mineral and the heat-treated, dehydrated mineral are summarized in Table 1. The dehydrated sample was held in an oven at 135° C. for $2\frac{1}{2}$ hours. This dehydration is reversible, as shown by the return of the optical properties of naturally occurring coconinoite when the mineral is rehydrated. Further

TABLE 1. OPTICAL PROPERTIES OF COCONINOITE (SAMPLE EVC-6) FROM THE SUN VALLEY MINE

Hydrated or natural state	Dehydrated at 135° C. for $2\frac{1}{2}$ hours
Biaxial (−)	*Biaxial* (−)
$\alpha = 1.550 \pm .005$	$\alpha = 1.605 \pm .005$
$\beta = 1.586 \pm .005$	$\beta = 1.630$ (calc.)
1.588 (calc.)	
$\gamma = 1.590 \pm .005$	$\gamma = 1.645 \pm .005$
2V between 28° and 43°	$2V = 75° \pm 5°$ (U-stage)
average 40° (U-stage)	Birefringence$= 0.040 \pm .010$.
Birefringence$= 0.040 \pm 0.10$.	
Y = length of laths with positive elongation.	
$Y \wedge$ length of laths with negative elongation = 8° to 25°.	
Pleochroism: α = colorless, β = pale yellow, γ = pale yellow.	

heating at 174° C. for $2\frac{1}{2}$ hours caused no change in the optical properties of the sample at 135° C., but the *x*-ray pattern changed as noted under *x*-ray diffraction data.

THERMAL PROPERTIES

The weight loss of coconinoite upon heating was determined by means of a Chevenard thermogravimetric balance. Sample 1715 from the Blackwater No. 4 mine was checked with an *x*-ray diffraction powder pattern and no impurity could be detected. A 514-mg portion of the sample was heated from 22° to 982° C. It lost 23.7 per cent of its weight between room temperature and 450° C.; most of this loss occurred between 100° and 300° C. (Fig. 2). This loss represents the water content and agrees closely with the water determination in the chemical analyses (Table 2). An additional 5.3 per cent loss of weight between 600° and 850° C. represents loss of SO_2, which corresponds reasonably well with the amount calculated from the SO_3 determinations in the chemical analyses.

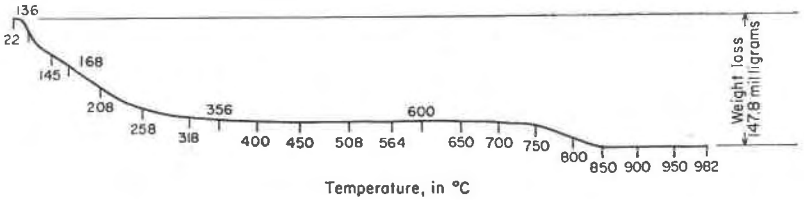

FIG. 2. Weight loss curve of coconinoite from the Blackwater No. 4 Mine, Apache County, Arizona, as a result of progressive heating. Sample weight 514 mg; weight loss 147.8 mg or 28.7 per cent.

TABLE 2. MICROCHEMICAL ANALYSES OF COCONINOITE
[Ratios calculated on basis of $UO_3 = 1.00$. Analyst, Robert Meyrowitz]

Sample No.	T-J2-53			1715			Footnote
Location	Jomac Mine, White Canyon San Juan County, Utah			Blackwater No. 4 mine Apache County, Arizona			
Constituent	Per Cent	Ratio	Proportion	Per Cent	Ratio	Proportion	
Fe_2O_3	10.3	0.54	1	9.7	0.50	1	d, i
Al_2O_3	6.5	0.53	1	6.6	0.50	1	f, i
UO_3	34.2	1.00	2	34.9	1.00	2	c, i
P_2O_5	17.7	1.04	2	18.3	1.06	2	e, i
SO_3	6.1	0.64	1	5.4	.55	1	j
H_2O	23.4	10.86	22	24.0	10.92	22	k
Acid insoluble.	1.0	—	—	0.5	—	—	b, i
CaO	<0.1	—	—	0.1	—	—	g, i
CO_2	0.7	—	—	<0.1	—	—	k
Na_2O	0.1	—	—	<0.1	—	—	h, i
Total	100.0	—	—	99.5	—	—	—
H_2O-	18.8	—	—	18.2	—	—	a, i

[a] Dried to constant weight at 110±5° C.

[b] The acid insoluble was determined by treating the sample with (1+3) acid (HCl for No. T-J2-53 and HNO_3 for No. 1715) in a weighed Emich microbeaker and sintered glass filterstick. The residue was filtered, washed with water, and dried to constant weight at 110±5° C.

[c] UO_3 was determined spectrophotometrically by the ammonium thiocyanate procedure in acetone-water medium after separation of the uranium by extraction with ethylacetate from an aluminum nitrate solution.

[d] Fe_2O_3 was determined spectrophotometrically by the o-phenanthroline procedure. The valence state of the iron was determined by dissolving the mineral in (1+3) H_2SO_4 and titrating the resulting solution with approximately 0.03 N standard $KMnO_4$. The sample sizes for the valence determination were approximately 7 mg for No. T-J2-53 and approximately 32 mg for No. 1715.

[e] P_2O_5 was determined spectrophotometrically by the molybdovanado-phosphoric acid procedure. A standard curve was made using known P_2O_5 solutions containing approximately the same concentration of uranium present in the aliquot of the sample solution taken for the determination of P_2O_5.

[f] Al_2O_3 was determined spectrophotometrically using Alizarin Red S with calcium. A standard curve was made using known Al_2O_3 solutions containing approximately the same concentrations of iron and uranium present in the aliquot of the sample solution taken for the determination of Al_2O_3. Hydroxylamine hydrochloride was used to reduce the ferric iron and then the ferrous iron was complexed by the addition of potassium ferricyanide.

[g] CaO was determined by flame photometry (wave length = 554 mμ). The solution

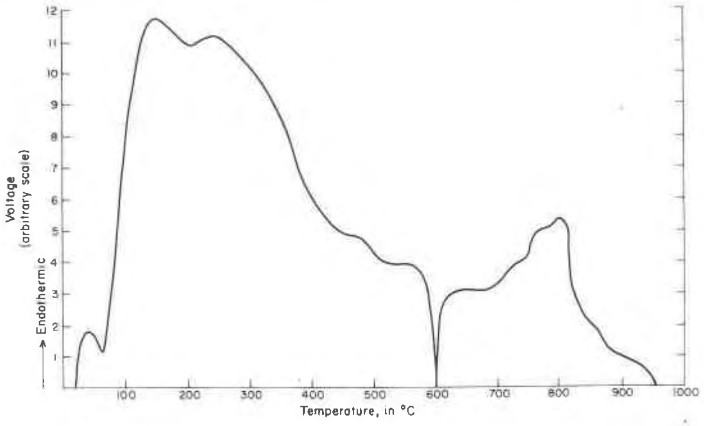

FIG. 3. Differential thermal analysis curve of coconinoite from Blackwater No. 4 Mine, Apache County, Arizona (sample 1715).

A differential thermal analysis was made on another portion of sample 1715 by means of an Eberhard portable instrument. The results are shown on Fig. 3 by a large endothermic peak, with a double top, between 150° and 250° C., which corresponds to the loss of the large amount of water, and by the smaller endothermic peak near 800° C., which corresponds to the loss of SO_2.

CHEMICAL COMPOSITION

Chemical analyses. The methods employed in the chemical analyses were indicated by qualitative and semiquantitative spectrographic analyses of

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was compared to standard calcium solutions containing approximately the same concentrations of uranium, phosphorus, and iron present in the solution analyzed.

[h] Na_2O was determined by flame photometry (wave length = 589 mμ). The solution was compared to standard calcium solutions containing approximately the same concentrations of uranium, phosphorus, and iron present in the solution analyzed.

[i] Separate aliquots of the filtrate from the acid insoluble determination were used for the UO_3, Fe_2O_3, P_2O_5, Al_2O_3, CaO and Na_2O determinations. The sample size for the above six determinations and the H_2O (−) and acid insoluble determinations was approximately 30 mg.

[j] SO_3 was determined gravimetrically as $BaSO_4$. Qualitative spectrographic analysis of the $BaSO_4$ precipitate showed that barium was the only major constituent present and that there were no minor constituents. The sample size was approximately 22 mg.

[k] H_2O and CO_2 were determined by use of a modified microcombustion train of the type used for the determination of carbon and hydrogen in organic compounds. The sample was decomposed by ignition at 900° C. in a stream of oxygen. The sample size was approximately 20 mg.

the samples (Table 3) by Katherine V. Hazel, U. S. Geological Survey. Approximately 100 mg of sample T-J2-53 (Jomac mine) and approximately 300 mg of sample 1715 (Blackwater No. 4 mine) were available for chemical analysis (Table 3). Both samples were analyzed by essentially similar methods.

One portion of each sample was used for the total water and carbon dioxide determinations, another for $H_2O(-)$, acid insoluble, Fe_2O_3, Al_2O_3,

TABLE 3. SPECTROGRAPHIC ANALYSES (IN PER CENT) OF COCONINOITE.
[M = major constituent, greater than 10 %; 0 = looked for but not found; asterisk (*) = not looked for]

Element	Sample No. and locality		
	T-J2-53[1] Jomac mine	1715[2,3] Blackwater mine	EVC 6[3,4] Sun Valley mine
U	M	M	M
P	1–5	7.0	M
Fe	0.05–1	7.0	5–10
Al	0.1–0.5	7.0	2–5
Si	—	0.15	*
Ca	—	0.015	<0.1
Na	0.05–0.1	0.0007	0
Bi	—	0	<0.02
Cu	—	0	0.01–0.02
As	—	0.015	0
Sc	—	0	0.01–0.02
Mg	—	0.0003	<0.01
Ba	—	0.0015	0.002–0.005
Cr	—	0	0.001–0.002
K	—	0.003	0
B	—	0.003	0
Be	—	0.00003	0
Mn	—	0.0015	*

[1] Qualitative spectrographic analysis by Katherine V. Hazel, U. S. Geological Survey. Dashes (—) indicate that the element was either absent or its concentration was below the limits of detection of the procedure (Stich, 1953, p. 7). Other elements were looked for but not found. Because purified material was scarce, this analysis was performed on a 1-mg sample mostly for detection of major constituents. This sample was also chemically analyzed.

[2] Semiquantitative analysis by Katherine V. Hazel. This sample was also chemically analyzed.

[3] Figures are reported to the nearest number in the series 7, 3, 1.5, 0.7, 0.3, 0.15, etc. These numbers represent midpoints of group data on a geometric scale. Comparisons of this type of semiquantitative results with data obtained by quantitative methods, either chemical or spectrographic, show that the assigned group includes the quantitative value about 60 % of the time. (Waring and Annell, 1953.)

[4] Semiquantitative analysis by Nancy M. Conklin, U. S. Geological Survey.

UO_3, P_2O_5, CaO and Na_2O determinations, and a third portion for the SO_3 determination. Microprocedures and semimicroprocedures were used throughout.

Carbon dioxide and total water were determined by use of a modified microcombustion train of the type used for the determination of carbon and hydrogen in organic compounds. The sample was decomposed by ignition at 900° C. in a stream of oxygen.

The second portion was dried to constant weight at $110° \pm 5°$ C. and then boiled with (1+3) acid (HCl for sample T-J2-53 and HNO_3 for sample 1715). An Emich microbeaker and sintered glass filterstick were used to separate the insoluble residue, which was washed with water and then dried to constant weight at 110° C. The insoluble residue in sample 1715 was found to be quartz.

Aliquots of the filtrate from the acid insoluble determination were used for the Fe_2O_3, Al_2O_3, UO_3, P_2O_5, CaO, and Na_2O determinations.

A test of the homogeneity of coconinoite from the Blackwater No. 4 mine, Apache County, Arizona, was attempted by Cynthia W. Mead, of the U. S. Geological Survey, using an electron microprobe. The attempt was not very successful because a polished surface on the soft powdery mineral could not be obtained and this prevented the maintenance of a stable beam current on the sample. Nevertheless, by scanning an area 0.2 mm on a side, Fe and probably small amounts of U, Al, and P were detected. S was not detected, but, because of instrumental difficulties, its presence is not precluded.

CALCULATION OF FORMULA

The molecular proportions calculated from the chemical analyses are given in Table 4. The probable chemical formula derived from these analyses is

$$Fe_2Al_2(UO_2)_2(PO_4)_4(SO_4)(OH)_2 \cdot 20\ H_2O.$$

The formula weight is approximately 1600 and the measured specific gravity is 2.7. Inasmuch as the unit cell has not been determined, the formula cannot be verified. The complex *x*-ray diffraction powder pattern suggests a complex structure. The presence of some (SO_4) with dominant (PO_4) suggests a compensating substitution of S^{6+} for P^{5+} to balance another atomic substitution, but the iron appears to be Fe^{3+} and not Fe^{2+}. The substitution of SO_4^{2-} for PO_4^{3-} apparently occurs in the beudantite mineral group.

RADIOACTIVE EQUILIBRIUM

The equivalent uranium contents of three occurrences of coconinoite were measured with a micro alpha counter. The counter, which is

TABLE 4. EQUIVALENT AND CHEMICAL URANIUM CONTENTS OF COCONINOITE (IN PER CENT)

Grain	Sample No. and Location			
	1715 Blackwater No. 4 mine, Apache Co., Arizona	EVC-6 Sun Valley mine, Coconino Co., Arizona	T-J1-53 Jomac mine, San Juan Co., Utah	T-J2-53 Jomac mine, San Juan Co., Utah
	Equivalent uranium			
1	7.6	11.4	19	23
2	7.5	11	28	32
3	7.5	9.5	23	25.5
Average	7.5	11	23	27
	Chemical uranium[1]			
	29.1	—	—	28.3

[1] Analyst, Robert Meyrowitz, U. S. Geological Survey.

The preceding data indicate that coconinoite from the Jomac mine, Utah, is at or near equilibrium and, therefore, probably formed at least 200,000 years ago. The two samples from Arizona are decidedly out of equilibrium, and, therefore, probably formed more recently than 200,000 years ago.

equipped with a photomultiplier tube activated by alpha particles striking microscopic ZnS crystals causing them to emit light, makes possible the measurement of radioactivity of small grains (one-half of a square milimeter). These values, believed to be correct within at least 25 per cent of the measured value, are given in Table 4 for three grains of each sample. Chemical uranium content is also given for the samples from the Jomac mine, San Juan County, Utah, and from the Blackwater No. 4 mine, Apache County, Arizona.

X-RAY DIFFRACTION DATA

The *x*-ray diffraction powder pattern of coconinoite is complex with more than 80 lines (Table 5). No single-crystal study is possible on the fine-grained material. The first samples, which occurred with gypsum, limonite, jarosite and clay at the Jomac mine, were carefully checked to see if the *x*-ray pattern of coconinoite showed any contamination, and none was found. The *x*-ray powder pattern of vauxite, $Fe\,Al_2\,(PO_4)_2(OH)_2 \cdot 7H_2O$, shows some similarity to that of coconinoite, possibly implying a similar structure, but close inspection of the two powder patterns forces us to the conclusion that vauxite is not present in coconinoite as an impurity.

Table 5 gives the measured d-spacings of three samples, including the

TABLE 5. MEASURED d-SPACINGS OF X-RAY DIFFRACTION POWDER PATTERNS OF COCONINOITE

Sun Valley Mine (EVC-6)[1]		Jomac Mine (T-J1-53) Analyzed[2]		Blackwater No. 4 Mine (1715) Analyzed[3]			
Fe/Mn=1.9373 Å film		Cu/Ni=1.5418 Å diff. chart		Fe/Mn=1.9373 Å film		Cr/V=2.29092 Å film	
d(Å)	I	d(Å)	I	d(Å)	I	d(Å)	I
		12.33[4]	8	12.08	vvf	12.12	vvf
11.05	vvs	11.12	100	11.06	vvs	11.03	vvs
		9.20	3	9.15	vvf	9.11	vvf
8.62	vf	8.66	8	8.62	f	8.55	f
8.30	vf	8.40	3	8.30	f	8.31	f
7.62	vvf	7.66	8	7.67	vf	7.66	vf
		7.28	3				
6.52	vvf			6.63	vvf	6.61	vvf
		6.24	2	6.19	vvf	6.20	vvf
5.90	vf	5.91	3	5.98	vf	5.963	vf
				5.80	f	5.859	vf
5.61	m	5.64	18	5.61	w	5.602	w
5.52	s	5.56	42	5.52	vs	5.521	s
4.99	f	5.02	9	5.00	w	5.111	f
						5.092	
4.57	f	4.59	14	4.57	m	4.567	w
—		4.497	3	4.48	vvf	4.462	vf
4.29	f	4.31	10	4.31	w	4.293	f
		4.21	2	4.22	vvf	4.201	vf
		4.17	4	4.14	vvf	4.137	vf
4.05	f (b)	4.05	7	4.07	vf	4.063	vf
				4.03	vf	4.015	vf
		3.965	3	3.96	f	3.952	f
3.91	f (b)	3.91	6	3.89	f	3.890	f
3.79	vf	3.82	5	3.82	f	3.809	f
3.74	f	3.77	7	3.76	w	3.747	f
3.67	m	3.71	12	3.69	m	3.687	m
				3.62	vf	3.610	vf
				3.52	vvf	3.505	vvf
		3.46	2	3.45	vvf	3.440	vvf
3.38	f	3.39	5	3.39	w	3.382	f
		3.36(?)					
3.28	m	3.30	22	3.29	s	3.286	s
		3.26(?)					
3.16	f	3.18	6	3.17	f	3.178	f
3.09	vf	3.12	3 (b)	3.11	vf	3.115	f
3.02	f	3.04		3.04	m	3.038	m
2.98	f	3.00	5	3.00	f	3.000	w
2.91	vvf			2.94	vvf	2.944	vf
				2.92	vvf		
				2.88	vvf	2.887	vf
				2.87	vvf	2.864	vf
2.83	f	2.84	7	2.83	w	2.836	w
2.79	vf	2.80	4	2.80	f	2.800	f
2.77	vf			2.76	vf	2.768	vf
2.66	f (b)	2.67	7	2.67	w	2.668	w
2.55	vf	2.56	3 (b)	2.57	f	2.565	vf
						2.527	vvf

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[1] Film D 3449, corrected for film shrinkage, measured by R. P. Marquiss.

[2] Chart 1570 of analyzed sample, measured by A. D. Weeks.

[3] Film W-13867 (Fe/Mn) and W-13851 (Cr/V) of spindle 11950, analyzed sample, measured by A. D. Weeks.

[4] Possibly a β line.

TABLE 5—(*continued*)

Sun Valley Mine (EVC-6)[1]		Jomac Mine (T-J1-53) Analyzed[2]		Blackwater No. 4 Mine (1715) Analyzed[2]			
Fe/Mn = 1.9373 Å fiilm		Cu/Ni = 1.5418 Å diff. chart		Fe/Min = 1.9373 Å fiilm		Cr/V = 2.29092 Å film	
d(Å)	I	d(Å)	I	d(Å)	I	d(Å)	I
2.49	vf	2.50	3	2.50	f	2.500	vf
				2.43	vf	2.426	vvf
2.41	vvf (b)	2.40	2	2.40	vf	2.403	vvf
2.34	vf	—		2.35	vf	2.350	f (b)
				2.31	vvf	2.314	vvf
				2.29	vvf	2.289	vvf
2.23	vf (b)	2.24	3	2.23	f	2.233	f (b)
2.20	vvf			2.20	vf	2.199	vf (b)
2.15	vvf	2.16 (d)	3	2.15	vf	2.157	vf
2.13	vvf (b)	2.13	2	2.12	vf	2.135	vvf
						2.122	vf
		2.10	2				
2.07	vf (b)	2.08 (d)	4	2.08	f (b)	2.078	f (b)
2.05	vvf	2.05	2	2.06	vf	2.055	vf
2.02	vf (b)	2.02	2	2.02	vf	2.031	vvf
						2.017	vf
1.986	vvf			1.990	vvf	1.9872	vvf
		{1.969–					
1.961	f	1.962	vb	1.964	f	1.9653	f (b)
1.946	vvf			1.942	vvf	1.9418	vf
				1.910	vvf	1.9045	vf
1.875	vf			1.882	vvf	1.8816	vf
1.862	vvf					1.8647	vvf
						1.8502	vvf
1.820	vf (b)			1.825	f (b)	1.8251	vf
1.798	vvf (b)	1.800	2	1.791	vvf	1.7989	vvf
						1.7876	vvf
1.760	vf (b)			1.758	vf	1.7602	vvf
				1.739	vvf	1.7399	vvf
1.726	vvf			1.723	vf	1.7244	vvf
1.697	vvf			1.694	vvf	1.6931	vvf
1.678	vvf			1.677	vvf	1.6764	vvf
1.668	vvf			1.665	vf	1.6641	vvf
1.650	vf			1.648	f	1.6460	vf
1.617	vvf (b)					1.6314	vvf
1.588	vf (b)			1.585	vf (b)	1.5884	vf
1.549	vf (b)	1.556	2	1.553	vf (b)	1.5487	vf (b)
1.521	vf (b)			1.517	vf (b)	1.5201	vf (b)
1.513	vf (b)	1.507	2			1.5098	vvf
1.484	vvf			1.486	vf (b)	1.4824	vvf
						1.4750	vvf
1.468	vvf			1.463	vvf	1.4626	vvf
1.446	vvf					1.4467	vvf
1.416	vvf			1.416	vvf	1.4150	vvf
1.378	vvf (b)			1.380	vf	1.3784	vvf
1.356	vvf						
1.342	vvf (b)			1.340	vvf	1.3413	vvf (b)
1.294	vvf (b)			1.294	vvf		
1.271	vvf (b)			1.270	vvf		
1.180	vf (b)			1.183	vvf (b)		
1.150	vvf (b)						
1.136	vvf (b)			1.108	vvf (b)		
1.098	vf (b)			1.098	vvf (b)		
1.040	vvf (b)						

TABLE 6. X-RAY DIFFRACTION DATA FOR HEAT-TREATED, DEHYDRATED COCONINOITE

Coconinoite, sample EVC-6, from Sun Valley mine					
Dehydrated for 2½ hours at 135° C.—film D3455[1]		Dehydrated at 2½ hours at 174° C.—film D3454		Dehydrated for 3 hours at 215° C.—film D3467[1]	
d(obs.)	I	d(obs.)	I	d(obs.)	I
8.49	vvs	8.30	vf	8.18	m
7.57	w	7.28	s	6.96	s
6.03	f	5.99	s	4.26	f
5.17	vf(b)	3.47	w	3.99	w
4.49	vf(b)	3.34	w	3.34	vs
4.46	vf(b)	3.00	s	2.85	vf
4.24	f(b)	2.81	s	2.69	vf
4.03	f	2.12	vf(b)	2.51	f
3.65	vvf(b)	1.843	w	1.923	vvf
3.29	w	1.695	vvf	1.818	vvf
3.16	w	1.672	vvf	1.543[1]	vvf
3.04	vvf	—	—	—	—
2.85	vf(b)	—	—	—	—
2.47[1]	vvf(b)	—	—	—	—

[1] Several more extremely faint lines too faint to be measured accurately.

two chemically analyzed samples. Efforts to index the patterns were unsuccessful. The crystal system is possibly monoclinic, as suggested by the optical properties. All samples were fully hydrated until heat-treated in the laboratory.

Table 6 shows the d-spacings of heat-treated, dehydrated coconinoite. Specimens of sample EVC-6, from the Sun Valley mine, were heated for 2½ to 3 hours at 135° C., 175° C., and 215° C. The exposure was begun within an hour after the sample had cooled enough for the *x*-ray spindles to be prepared. A test was also made to determine if the diffraction pattern of a sample stored over water for 24 hours is different from that of a natural-state sample. No difference was observed and it is concluded that natural coconinoite is stable in the fully hydrated state.

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