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STRONTIAN META-AUTUNITE FROM THE DAYBREAK MINE, MT. SPOKANE, WASHINGTON[1]

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ABSTRACT

Chemical analysis, *x*-ray, and optical data of meta-autunite from Daybreak Mine, near Mt. Spokane, Wash., are compared with previous data on meta-autunite. It is shown that Mt. Spokane meta-autunite represents a very pure material with properties close to the theoretical with the exception of the strontium content. According to the analysis the composition of Mt. Spokane meta-autunite corresponds with the formula proposed by Beintema (1938)—$Ca(UO_2)_2(PO_4)_2 \cdot 6\frac{1}{2}H_2O$.

INTRODUCTION

In 1955 Mr. W. Oke, Curator in Mineralogy at California Institute of Technology, drew my attention to an unusually beautiful and well-crystallized sample of yellowish green and dark green meta-autunite from Mt. Spokane, Washington, that appeared very similar to torbernite. In 1957 Prof. J. Butler, Metallurgist at the Nevada Mining Analytical Laboratory, kindly supplied me with adequate amounts of the mentioned meta-autunite. This work was done on these samples which have been stored for over a year in a dry cellar room with a steady temperature of about 65° F. in the Mackay School of Mines Building. When this manuscript was completed, the author learned that Mr. G. W. Leo, U. S. Geological Survey, Menlo Park, California, was working on the same material and had already published an abstract in connection with the Fifty-Fourth Annual Meeting of the Geological Society of America, Cordilleran Section, Eugene, Oregon (1958). Before sending the manuscript, the author was able to meet Mr. Leo and to discuss both investigations. It was then discovered that the material on which both authors have worked is from the same locality. The scope of each work is different, so that they can be regarded as supplementing each other with very little overlapping information. Mr. Leo's work deals mainly with the fully hydrated autunite and with the variation in composition and physical properties of the zoned mineral; it is concerned also with considerations regarding the probable origin of the deposits. The present paper deals only with the stable, dark-green meta-autunite from Daybreak Mine. It was mutually agreed then to publish each paper separately.

According to our knowledge, combined *x*-ray, optical, and chemical data on natural meta-autunite, obtained from the same material, are not listed in the literature. Berzelius (1824) gives an analysis of Autun material that is the closest in composition to meta-autunite. Fairchild

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[1] Publication authorized by the Director, Nevada Bureau of Mines.

(1929) gives an analysis of synthetic meta-autunite practically identical with the theoretical composition of meta-autunite with $6\frac{1}{2}$ H_2O. No optical data for a distinct meta-autunite species, nor a complete chemical analysis of such natural species is available. This situation and the excellent material obtained have stimulated this study.

META-AUTUNITE AND AUTUNITE

The stable variety of autunite at room conditions is called meta-autunite (I) (was called first metakalkuranit by Rinne, 1901), is tetragonal, and contains, according to Beintema (1938), $6\frac{1}{2}$ to $2\frac{1}{2}H_2O$. The formula is written $Ca(UO_2)_2(PO_4)_2 \cdot 2\text{–}6H_2O$ by Donnay and Donnay (1955). Brichard and Brasseur (1958) have recently restudied the autunite problem and arrived at results very similar to those of Donnay and Donnay. They state, on the basis of dehydration experiments on autunite at temperatures up to 250° C., that the stable variety of "natural autunite" contains $6H_2O$ and is identical with Beintema's meta-autunite I. This is in slight disagreement with Beintema's dehydration studies at different vapor tensions (1938, p. 159) which show that the stable meta-autunite variety contains $6\frac{1}{2}H_2O$ at temperatures 15°, 38°, and 57° C., and vapor tensions 0.8–2, 10–20, and 30–90 mm. Hg respectively. Beintema's and Brichard's and Brasseur's (1958) experiments show that meta-autunite I is easily transformed to autunite $Ca(UO_2)_2(PO_4)_2 \cdot 10\frac{1}{2}H_2O$ when stored in humid atmosphere or treated with water at room temperature.

The fully hydrated autunite is supposed to contain theoretically $12H_2O$ (21.92% H_2O). No natural material so far analyzed has contained so much water. Work by Beintema (1938) and Nakanishi (1951) indicates that a stable variety of autunite at high humidity values and in water exists and contains $10\frac{1}{2}$ (Beintema)—10 (Nakanishi) H_2O. Analyses of natural autunite are in good agreement with these results (Church, 1875; Henrich, 1922; Hodge Smith, 1926). Frondel, Riska and Frondel (1956) write the formula with $8\text{–}12H_2O$, Fiorentini Potenza (1958) uses $12H_2O$ for the unit cell, but does not analyze the water content of the Biella autunite. Strunz (1957), based on Beintema's data (1938), writes 10(12-10) H_2O for autunite, emphasizing therefore $10H_2O$ for the formula. The reason for so many different data is obviously the reversible transformation meta-autunite$\rightleftharpoons$autunite and the instability of autunite under laboratory conditions.

META-AUTUNITE FROM MT. SPOKANE

The Daybreak Mine is located about 30 miles NE of Spokane in Sec. 11, R. 44 E., T. 28 N., in Spokane Co., Washington. The described meta-autunite occurs in shear zones in the Loon Lake quartz monzonite. The sample came from the oxidized zone, about 10 feet below the surface (Donald L. Hetland, private communication). No other uranium minerals have so far been discovered here (G. W. Leo, private communication). Norman (1957) gives a short description of the mine with photographs of the shear contact, a typical meta-autunite sample, and the mine. Thurlow (1955) describes another autunite occurrence NW of Spokane on the Indian reservation at the contact of the Weaver's Deer Trail argillite and the Loon Lake granite. Here also uranophane and uraninite associated with pyrite have been found.

The sample consisted of coarse aggregates of tabular slightly deformed dark green crystals usually several mm. in diameter, standing on their edges upon compact crusts of dark green meta-autunite. Central parts of the crystals were colored dark bluish green and some edges a yellowish green with traces of canary-yellow material that under magnification appeared to represent areas crushed probably when the sample was taken. The change in color is apparently due to minor cracks and therefore presence of air along the (001) cleavage planes. The greenish yellow color on edges seemed to become more intense in a few days on disturbed edges and surfaces of the green flakes used for specific gravity determination by immersion in toluene and carbon tetrachloride with the Berman balance. The color of the fresh green meta-autunite resembles that of torbernite very closely. Specific gravity: 3.48. During specific gravity determinations with Berman balance, it was noted here that longer immersion gave higher values, e.g., 3.28 (immediate)—3.35, 3.40 (2 hrs.) —3.48 (>3 hrs.) in toluene, and 3.35, 3.40 (1 hr.)—3.48 (>2 hrs.) in carbon tetrachloride. This indicates that it takes that long for the liquids to penetrate and fill the minor cracks in the micaceous mineral. It is obvious that this is possible only to a certain extent, so that the true value must be slightly higher. Beintema (1938) gives the specific gravity for $Ca(UO_2)_2(PO_4)_2 \cdot 6\frac{1}{2}H_2O$ as 3.569 (calc.); his meta-autunite had a value >3.33. The fluorescence under short-wave ultraviolet light, of parts of the dark green material is a weak yellowish green. The yellowish green material and parts of the green material fluoresce a bright yellowish green, see Iimori and Iwase (1938). Refractive indices were determined using gelatin-coated slides on a universal stage. To obtain a correct value for the *c*-direction (ϵ) an oil immersion objective was used and no cover glass was applied, so as not to disturb the grains standing on their edges. The upper hemisphere was left off, and immersion liquids were applied directly between the objective and the slide. (Any corrosive liquids must be avoided and the objective cleaned carefully immediately after the work is completed.) Sodium light was used. The range of values obtained for the green mineral are $(\epsilon) \pm 0.003$ and $(\omega) \pm 0.002$. The refractive indices of this meta-autunite are higher than previously reported for autunites, corresponding well with George's data (1949).

Refractive indices for greenish yellow material were not measured separately, but appear to be close to the lower limits of the range given in Table 1, or slightly lower for the more yellow material.

2V was measured on the universal stage on large flakes on gelatin-coated slides. Examined in polarized light the flakes appeared homogeneous. Under crossed nicols over 60 per cent of the area of the flakes appears in extinction. Very low birefringence can be detected in the other parts. These optically different areas resemble the quadrille structure of microcline. In some of these flakes straight completely isotropic lamellae occur. 2V was measured in several spots which had the highest birefringence (light gray). The highest value in about 20 measurements was 20°. Typical series of measurements were: 2V = 18°, 12°, 16°, 13°, 16°, 15°, 13°, 15°, 18°, 20°, 16°, 15°. Dichroic with $\omega > \epsilon$, ω—grayish green, ϵ—faint yellowish green.

TABLE 1. OPTICAL PROPERTIES OF META-AUTUNITE AND AUTUNITE

	Meta-autunite Mt. Spokane	Autunite Autun[1]	Autunite Maryland[2]	Meixner[3]	Autunites—Meta-autunite[4]
$\epsilon(\alpha) = c$	1.584 ± 0.003	1.553	1.555		1.585–1.600
(β)		1.575	1.575	1.58–1.59	1.595–1.610
$\omega(\gamma)$	1.607 ± 0.002	1.577	1.578	1.59–1.60	1.595–1.613
2V	0°–20° (anomalous) also uniaxial in part				
Birefringence	0.023	0.024	0.023		
Sign	—	—	—		

[1] Michel-Levy and Lacroix (1888).
[2] Shannon (1926).
[3] Meixner (1940).
[4] George, D'Arcy (1949).

Powder diagrams were recorded with a Norelco wide-range goniometer. A nickel filter was used (CuK—1.5418 Å). In Table 2 the *x*-ray data so obtained are compared with the data for natural meta-autunite given by Brichard and Brasseur (1958) and the data for meta-autunite from Sabugal, Portugal, by Frondel, etc. (1956). The *d*-values and intensities were obtained with the wide-range goniometer at a speed of $\frac{1}{4}° = 1$ min. with coll. $1° - 0.006''$. Intensity is measured by estimating the peak curve areas. Differences in intensities as compared to those given by Brichard (1958) and Frondel (1956) are due to the micaceous nature of the mineral. The powder samples for the goniometer scanning were prepared by pressing the mineral powder slightly in the sample holder form with a glass plate and removing the excess by sliding the glass plate in a diagonal direction until a smooth surface was obtained. This way better peaks and intensities were obtained, but the preferred orienta-

TABLE 2. *X*-RAY DATA FOR META-AUTUNITE FROM MT. SPOKANE, WASH., COMPARED WITH DATA ON META-AUTUNITE I BY BRICHARD (1958) AND FRONDEL (1956)

Meta-Autunite, Mt. Spokane					Meta-Autunite I, Brichard				Meta-Autunite Sabugal, Portugal Frondel	
d meas.	*d* calc.	I meas.	*hkl*[1]	*HKL*[2]	Synthetic *d* meas.	I	*hkl*[3]	Natural *d* meas.	*d* meas.	I
					10.15 Aut.					
8.47	8.47	100	001	001	8.465	s	001	8.52	8.51	10
5.37	5.38	44	101	221	5.39	s	221	5.41	5.39	7
4.93	4.93	21	110	040	4.92	s	040	4.95	4.96	5
	4.26		111	041	4.42	vf	240*		4.28	6
4.23	4.24	69	002	002	4.25	m	002	4.26		
3.61	3.62	85	102	222	3.64	m	222	3.64	3.63	8
3.48	3.49	31	200	440	3.48	s	440	3.51	3.50	9
					3.30 Aut.					
3.22	3.22	23	201	441					3.24	8
	3.21		112	042	3.23	s	042	3.24		
2.93	2.93	15	211	261	2.93	m	621	2.94	2.94	4
---	2.82		003	003						
	2.69		202	442	2.70	vf	213*	2.705	2.68	1
2.61	2.62	35	103	223				2.62	2.61	3
2.51	2.51	6	212	262	2.52	f	323*	2.52	2.51	2
2.47	2.47	4	220	080	2.47	w	080	2.48	2.47	2
2.45	2.45	4	113	043						
2.37	2.37	8	221	081	2.38	w	423*	2.38	2.38	3
2.24	2.24	5	301	661	2.24	w	523*	2.25	2.25	2
2.21	2.21	10	310	480	2.21	m	480	2.215	2.21	3
	2.19		203	443						
	2.13		311	481	2.14	w	613*	2.14	2.14	3
	2.13		222	082						
2.11	2.12	71	004	004						
	2.09		213	263	2.10	f	623	2.10	2.10	3
2.04	2.04	9	302	662	2.05	w	633*	2.05	2.04	3
2.02	2.03	14	104	224						
	1.956		312	482						
1.943	1.946	14	114	044					1.941	2
	1.885		321	2.10.1	1.89	f	653*	1.89	1.893	1
	1.857		223	083				1.85		
1.807b	1.810	8	204	444						
	1.794		303	663				1.795	1.802	2
1.758b	1.759	6	322	2.10.2	1.77	f	10.2.2	1.77	1.757b	3
	1.752		214	264	1.75	f	544*	1.75		
1.745	1.743	9	400	880						

vs—very strong; s—strong; m—medium; w—weak; f—faint; vf—very faint; b—broad.

[1] Small cell, $a_0=6.972$, c_0 8.47.

[2] Large cell, $a_0=19.72$, c_0 8.47; by the transformation $\bar{2}20/220/001$.

[3] Where Brichard and Brassuer's indexing does not agree with ours the *hkl* indices are marked with an asterisk.

tion of the mineral particles could not be avoided, which is clearly demonstrated by the intense 001 peak.

In Table 2 measured and calculated *d* values and all possible planes in the smaller cell ($a_0=6.97$ Å, $c_0=8.47$ Å), with the space group *P4/nmm*, are given for spacings greater than 1.743. Through transformation $\bar{2}20/220/001$ corresponding indices of the larger cell are given in column *HKL*.

Unit cell dimensions of the Mt. Spokane meta-autunite and $c_0:a_0$ ratios, obtained from the wide-range goniometer pattern measurements, are in close agreement with those measured by Beintema (1938). If Beintema's data are converted to true Å we obtain: $a_0=6.994$, and $c_0=8.437$.

TABLE 3. UNIT CELL DIMENSIONS OF META-AUTUNITE

Meta-autunite Mt. Spokane (Volborth)	Meta-autunite I (Beintema)	Meta-autunite I (Donnay & Donnay)	"Autunite naturelle" (Brichard & Brasseur)
$a_0=6.972$ Å	6.994	7.01	6.97
($a_0'=19.72$ Å)		(19.82 Å±0.3%)	(19.72 Å±0.3%)
$c_0=8.47$ Å	8.437	8.49 Å±0.3%	8.50 Å±0.3%
$c_0/a_0=1.211$	1.206	1.211	1.211
($c_0/a_0'=0.4295$)		(0.4282)	(0.4309)
Sp.gr.$_{\text{meas.}}$ 3.48	3.33	3.48	
Sp.gr.$_{\text{calc.}}$ 3.586 ($6\frac{1}{2}H_2O$)	3.569 ($6\frac{1}{2}H_2O$)		
Sp.gr.$_{\text{calc.}}$ 3.542 ($6H_2O$)		3.50 ($6H_2O$)	
Z=1 (8)	1	(8)	

Since Brichard and Brasseur (1958) and Donnay and Donnay (1955) have recently used a larger cell, a_0 is also transformed in Table 3 according to $a_0'=a_0\cdot 2\sqrt{2}$ for comparison. The data for the larger cell are given in parentheses.

Since the meta-autunite from Daybreak Mine is zoned with colors ranging from yellow to green and dark green, nearly black, an attempt was made to distinguish between the yellow and green material. Optical properties of both mineral varieties proved to be very similar. In a mixture of grains these varieties cannot be distinguished in a satisfactorily manner. In thin flakes the green material shows only a slightly stronger dichroism and higher refractive indices.

Dr. A. Pabst kindly offered to run precession and Weissenberg patterns on small green and yellow meta-autunite crystals for comparison. His data are practically identical, indicating that both the green and the yellow phases are the equivalent of Beintema's meta-autunite I. See Table 4.

Pabst found no evidence for the 19.76 Å a_0 since all the reflections on the *hk*0 and *hkl* precession patterns could be accounted for by taking a cell with an a_0 of 6.985.

For chemical analysis dark blue-green, thick, compact flakes were selected. The yellowish edges were avoided as much as it was possible in hand separation. The color of this selected material was practically identical with the color of the fresh meta-autunite from the same locality that was coated with plastic immediately after sampling. Since the uncoated, undisturbed material does not change its color even after being stored for over a year and does not show any visible alteration which could be due to possible dehydration, it would seem probable that its composition also does not change, and it can be therefore assumed that the analyzed material represents the natural meta-autunite as found in the fault gouge at Mt. Spokane, or is very close to it in its composition and physical properties. According to Norman (p. 665–666, 1957), the primary mineral at Daybreak Mine is meta-autunite.

TABLE 4. UNIT CELL DIMENSIONS OF GREEN AND YELLOW META-AUTUNITE (PABST)

	Small green xl. Å	Small yellow xl. Å
a_0 (precession)	6.985±0.010	6.99±0.01
a_0 (Weissenberg)	6.98 ±0.02	6.96±0.02
c_0 (precession)	8.44 ±0.02	8.45±0.02
c_0 (Weissenberg)	8.45 ±0.02	8.44±0.03

Results of the chemical analysis are practically identical with the theoretical data for meta-autunite with a formula $Ca(UO_2)_2(PO_4)_2 \cdot 6\frac{1}{2}H_2O$ as is shown in Table 5.

Qualitative spectrographic analysis by Mr. Harold Vincent (Bausch and Lomb quartz spectrograph) shows the following elements present in traces: Al, Fe, Mg, Pb, V. Not detected: As, Bi, Cu, Mn, Sb, Sn, Tl.

Pereira-Forjaz (1917) found traces of Cu, Al, V, Fe, Pb, Mn, As, Sn, Bi, Mg, and Tl in autunite from Nellas, Portugal.

This analysis was carried out using the method proposed by the author (1954, 1958). The water determination was done according to the Penfield method (1894). The results indicate $6\frac{1}{2}H_2O$ molecules for the stable natural meta-autunite which is in good agreement with Beintema's data (p. 159, 1928). Unit cell dimensions and specific gravity determinations (Table 3) indicate, however, that $6H_2O$ molecules are perhaps more likely. The difficulty of the specific gravity determination (see above), and the high molecular weight of the mineral makes it difficult to determine the water content of the meta-autunite on the basis of physical data. It is possible that the original material contained a minor amount of an autunite with more water. Brichard and Brasseur (1958) conclude on the basis of dehydration experiments conducted up to 250° C. that meta-autunite contains only $6H_2O$ molecules. Recent experiments in this laboratory indicate that the last water in phosphates escapes at temperatures as high as 900–1000° C.

TABLE 5. STRONTIAN META-AUTUNITE, MT. SPOKANE, WASHINGTON
Anal., A. Volborth

	Per cent	Atomic ratios to $18\frac{1}{2}$ oxygens	$Ca(UO_2)_2(PO_4)_2\cdot 6\frac{1}{2}H_2O$ M=887.27
CaO	5.16		6.32
SrO[1]	1.38		
Na_2O	0.22	1.06	
K_2O	0.33		
UO_3	63.92	1.98	64.48
P_2O_5	15.54		16.00
SiO_2	0.39	2.00	
H_2O	13.36	6.57	13.20
Rem.[2]	0.14		
	100.44		100.00
	Sp. gr. 3.48		

[1] SrO was determined by Mr. H. Vincent flame-spectrophotometrically (Beckman Model DU) using the standard addition method described by Chow and Thompson (1955). Mr. G. W. Leo drew my attention to the fact that this mineral contains Sr.

[2] $Fe_2O_3+Al_2O_3=0.03\%$; insol.$=0.11\%$; $MgO=0.00\%$; $BaO=0.00\%$; Cu—no blue coloration in NH_4OH filtrate. Values for K_2O and Na_2O have to be regarded as slightly too high because of the accumulation of traces of these elements during the analysis from the reagents and glassware used. See Volborth, 1954, 1958.

It is obvious that much of the natural autunite material as found today is actually meta-autunite. The question of whether the natural material is autunite or meta-autunite seems to be of minor importance considering the fast reversible change between these minerals. It merely depends on whether the mineral is found in humid or dry environments. Beintema's study (p. 159, 1938) on water content indicates that at higher temperatures and higher humidity values, the stability of meta-autunite increases. To tell whether meta-autunite is actually the stable phase under hydrothermal conditions, a study in a pressurized bomb is needed.

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