THE AMERICAN MINERALOGIST, VOL. 44, SEPTEMBER–OCTOBER, 1959

AN *X*-RAY STUDY OF UMOHOITE*

SAMUEL R. KAMHI
Columbia University, New York, N.Y.

ABSTRACT

Precession photographs of umohoite from Marysvale, Utah, indicate a primitive orthorhombic unit cell with dimensions $a_o = 6.32$ Å: $b_o = 7.48$ Å: $c_o = 12.4$ and 14.5–17.0 Å. The range of c_o with humidity and with temperature is established. *X*-ray powder data for umohoite with $c_o = 12.4$ Å are listed, and indices have been assigned on the basis of the cell dimensions given. Powder data are also furnished for material with $c_o = 14.5$ Å.

INTRODUCTION

A new occurrence of the hydrous uranyl molybdate, umohoite, has recently been described (Coleman and Appleman, 1957). In addition, work is now in progress in the mineralogical laboratory at Columbia University by Miss P. K. Hamilton on hydrous uranium-molybdenum minerals which includes another occurrence of umohoite. This has led the author to re-examine, by means of *x*-rays, some properties of the original umohoite as described by Brophy and Kerr (1953), and Kerr, et al. (1957).

The author wishes to express his gratitude to Professor Paul F. Kerr for initiating this study, supplying the mineral specimens examined, and critically reading the manuscript. Miss P. K. Hamilton and Dr. William A. Bassett have kindly offered suggestions during the course of the work.

SINGLE CRYSTAL DATA

A small rectangular plate of umohoite approximately .12×.09×.005 mm. was cut under the microscope from a larger specimen. A precession photograph of the [001] zone gave sharp reflections using MoK_α radiation. Figure 1 is a precession photograph of this zone taken with CuK_α, which is a more reproducible photograph, and demonstrates the pseudo-hexagonal character of the reflections. Because of the excessively long (75 hours) exposures required by molybdenum radiation, MoK_α was only employed for the zero level of the [001] zone. CuK_α was utilized for upper levels of this zone and for the [010] zone.

Initial photographs of the [010] zone involved two sets of reflections corresponding to two values of c_o, 12.4 and 14.5 Å. Both sets showed diffuse streaking parallel to the *c*-axis. The *hkO* reflections were independent of the value of c_o. At the time the photographs were taken it was found that the 12.4 Å spacing could be maintained throughout the umohoite by circulating warm air (40° C.) about the specimen during the time inter-

* This work was made possible by the U. S. Atomic Energy Commission, through the office of Dr. Daniel R. Miller, Division of Research.

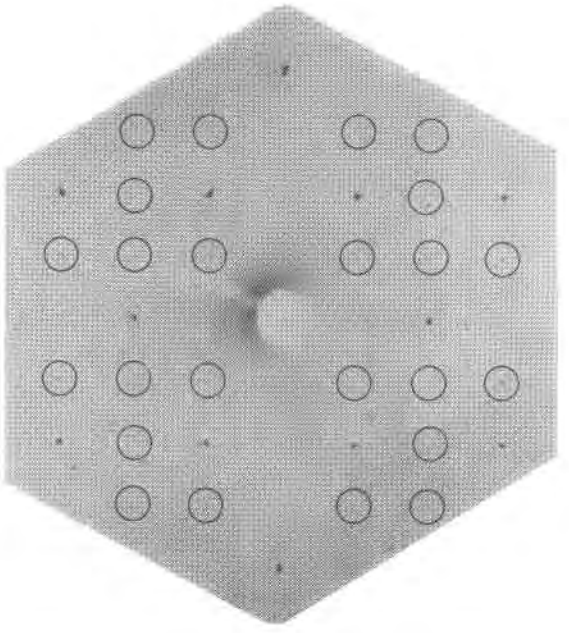

FIG. 1. Umohoite: zero level, c-axis precession photograph, b* vertical, a* horizontal; CuK_α, 4 hours. Weak reflections are circled.

val of the exposure. All single crystal data given refer to the crystal at this spacing.

The reflections observed indicate the Laue symmetry group $n\,mm$. There is no evidence that the orthorhombic symmetry is altered by the increase of c_o from 12.4 to 14.5 Å. Systematic absences observed are Okl with k odd. The space group is probably one of the following: $Pb2_1m(C_{2v}^{\ 2})$, $Pbm2(C_{2v}^{\ 4})$ or $Pbmm(D_{2h}^{\ 5})$.

Unit cell dimensions obtained from single crystal films and diffractometer runs of powdered material give

$a_0 = 6.32 \pm .03$ Å.
$b_0 = 7.48 \pm .03$ Å.
$c_0 = 12.4, 14.5 \pm .1$ Å (single crystal).
$14.5\text{–}17.0 \pm .1$ Å (diffractometer).

Study of the powdered material reveals a continuous variation of c_o between 14.5 and 17.0 Å, depending upon humidity conditions. The variable c-dimension was noted in the original description of umohoite and considered a function of water content. However, a dimension as small as 12.4 Å has not previously been reported in a specimen at room temperature.

Powder Data

Umohoite is fragile and crystals are apt to be destroyed by ordinary grinding. A small quantity of material was effectively powdered by placing it in an electric blender, adding about 100 cc. of distilled water, and allowing the machine to operate at top speed for a few minutes. Diffractometer runs of material oriented for 00*l* reflections were made on successive days and the temperature and humidity of the laboratory were measured by means of a sling psychrometer. At temperatures of about 30° C. and a relative humidity above 31 per cent, the 001 reflection was ob-

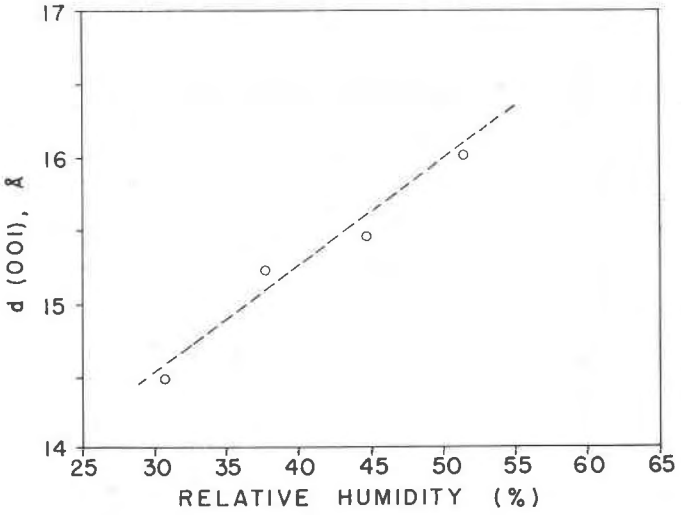

Fig. 2. Variation of (001) with relative humidity.

served to be poorly formed and to vary regularly with small variations in relative humidity (Fig. 2). Dry air circulated about the specimen caused the spacing to drop to 14.5 Å, with relatively well-defined peaks. Moist air circulated in a similar manner caused the spacing to reach 17.0 Å with equally well-formed peaks.

The existence of a 12.4 Å spacing in the case of the single crystal photographs is possibly caused by the combined effects of the more intense beam at the spot focus of the *x*-ray tube and the adsorptive effect of some clay samples stored in the room containing the single crystal *x*-ray apparatus.

Some powdered umohoite was deposited on the Pt-Rh sample holder of the high temperature diffractometer (Bassett and Lapham, 1957) and the 001 reflection observed in the course of heating the material to 425° C.

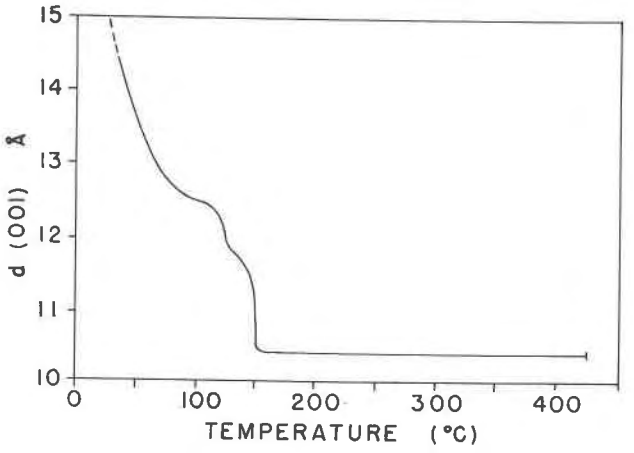

FIG. 3. Variation of (001) with temperature.

(Fig. 3). At 425° C. an unidentified yellow compound was formed. A second specimen was prepared in a similar manner and heated to 350° C. with the result that the 001 reflection yielded a value of 10.6 Å. The material changed in color from the normal pale green of powdered umohoite to a bluish-black. Exposure to steam for several days restored the original pale green color and brought the spacing up to 12.7 Å. No further changes were observed.

A diffractometer run was made of randomly oriented powder under conditions of low humidity which yielded $c_o = 14.5$ Å. Observed line spacings and intensities are listed in Table 1. Table 2 lists diffractometer data obtained from an unoriented powder which had been kept over-

TABLE 1. DIFFRACTOMETER DATA FOR UMOHOITE ($c_0 = 14.5$ Å). Cu RADIATION, Ni FILTER

d (Å)	I	d (Å)	I
14.3	1	2.38	1
7.21	10	2.07	2
4.82	4	2.03	2
3.63	2	1.92	1
3.20	5	1.87	1
3.15	4	1.84	1
3.06	4	1.81	1
3.02	3	1.78	1
2.90	4	1.61	2
2.53	1	1.59	1

TABLE 2. DIFFRACTOMETER DATA FOR UMOHOITE (c_0=12.4 Å). Cu RADIATION, Ni FILTER

hkl	d_{calc} (Å)	d_{obs} (Å)	I	d_{obs} (Å)	I
001	12.4	12.2	3	1.95	1
002	6.20	6.18	3	1.87	2
102	4.42	4.40	1	1.83	1
003	4.13	4.13	10	1.79	1
021	3.58	3.57	1	1.78	1
120	3.22	3.20	5	1.75	1
200	3.16	3.15	4	1.73	1
121	3.12	3.11	2	1.71	1
004	3.10			1.61	1
201	3.07	3.06	1	1.55	1
122	2.86	2.85	1	1.50	1
023	2.78	2.76	1		
123	2.53	2.54	2		
203	2.51	2.50	1		
220	2.42				
024	2.39	2.40	1		
124	2.23	2.22	1		
204	2.21				
115	2.21				
132	2.17	2.14	1		
214	2.12				
301	2.08				
223	2.08				
006	2.07	2.07	3		
025	2.07				

night in an oven at 50° C. It was found to have a c-dimension of 12.4 Å and has been assigned indices on the basis of the orthorhombic cell determined from single crystal photographs.

IDENTIFICATION OF UMOHOITE

A Patterson projection on (001) indicated an approximately hexagonal arrangement of uranium atoms. The lack of coincidence of the 120 and 200 reflections indicates the deviation of the uranium atoms from a truly hexagonal arrangement. These reflections are strong because of in-phasal scattering of uranium atoms. The fact that they are independent of the value of c_o makes them especially useful in the identification of umohoite in unoriented specimens, particularly since these reflections persist even through the usual grinding procedures. If specimens are oriented, consideration of the diffractometer curves reproduced by Kerr, et al. (1957) will be most useful if it is understood that these represent the approximate limits of the range of c_o under normal conditions.

SUMMARY

Unit cell dimensions and powder diffraction data have been obtained for umohoite from the type locality. Umohoite is observed to have a *c*-dimension which varies directly with normal variations in atmospheric humidity, and inversely with increasing temperature. Ordinarily, c_o may be expected to lie somewhere betwee 14.5 and 17.0 Å.

REFERENCES

BASSETT, W. A., AND LAPHAM, D. M. (1957), A thermal increment diffractometer: *Am. Mineral.*, **42,** 548–555.

BROPHY, G. P. AND KERR, P. F. (1953), Hydrous uranium molybdate in Marysvale ore: *Annual Report for June 30, 1952 to April 1, 1953, U. S. Atomic Energy Comm. RME-3046, pp. 26–44,* issued by the U. S. Atomic Energy Comm. Tech. Inf. Service Extension, Oak Ridge, Tenn.

COLEMAN, R. G. AND APPLEMAN, D. E. (1957), Umohoite from the Lucky Mc Mine, Wyoming: *Am. Mineral.*, **42,** 657–660.

KERR, P. F., BROPHY, G. P., DAHI, H. M., GREEN, J. AND WOOLARD, L. E. (1957), Marysvale, Utah, Uranium Area: *Geol. Soc. Amer. Special Paper 64.*

Manuscript received November 11, 1958