

tained, interpretation has been incorrect. A further test is that the same sites with the same occupancies should be found in different centrosymmetric projections of any one adduct.

Similar procedures could be used for other centrosymmetric plane groups. The statement that in an AB correlation map an A site is at the centre of a group of vector peaks representing the B sites, and *vice versa*, is valid wherever there is a centre of symmetry; but the multiplicity and orientation of the group of peaks for any one site depend on the plane group symmetry. For instance, for the one centrosymmetric projection of monoclinic protein crystals (symmetry $p2$), there would be, around each A site, pairs of diametrically opposite peaks representing B sites, and the diameters would be in different radial directions, not parallel to any axis; and in plane group $p4$ (for the c projection of some tetragonal crystals), the groups are squares with their sides inclined to the cell axes. A peak at the origin is always an indication of a common site, and the superposition procedures, for Patterson maps on a correlation map or for different correlation maps on each other, are the same as those given for the orthorhombic example.

There are obviously limitations to the complexity of the structures that might be solved by these methods. The number of sites, multiplicity of equivalent positions, range of occupancies and the effective resolution of the data employed are all involved:

(i) For the twofold multiplicity of a monoclinic

centric projection, it should be possible to deal with a larger number of sites than for the fourfold orthorhombic multiplicity.

(ii) It will probably be possible to locate only the sites of relatively high occupancy; sites of low occupancy would have to be detected by established methods based on electron density maps phased by the sites of high occupancy.

I wish to acknowledge the financial support of the Medical Research Council which has made this work possible. I also wish to thank my colleagues Dr P. C. Moews, Dr M. E. Baumber and Mrs E. M. Kestelman for X-ray diffraction data and computer calculations used in the application of the methods to the problems of rennin adducts.

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The Crystal Structure of UZn_{12} *

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(Received 21 September 1970)

The crystal structure of the most zinc-rich compound in the uranium-zinc system is hexagonal with space group symmetry $P6/mmm$. Crystals formed by cooling a 95 wt. % zinc alloy have a composition range of $UZn(10.4 - 11.0) \pm 0.1$ as determined by microprobe analysis; lattice parameters are $a = 8.950 \pm 0.001$ and $c = 8.902 \pm 0.002$ Å. The structure is based on an ideal UZn_{12} stoichiometry, but substitution between pairs of zinc atoms and uranium atoms leads to lower concentrations of zinc, $UZn_{10.4}$ being found for the crystal studied. The reported equivalence of the $SmZn_{12}$ and UZn_{12} structures based on powder work is confirmed. They differ only in the extent of substitution at different lattice sites.

Introduction

Two intermetallic compounds are known to exist in the uranium-zinc system. U_2Zn_{17} is reported to have both a rhombohedral and a hexagonal form. The status of the structural work has been summarized by Johnson, Smith & Wood (1968).

The existence of a second compound richer in zinc than U_2Zn_{17} has been shown by a series of investigations by researchers at the Argonne National Laboratory (Martin & Wach, 1960, 1962a & 1962b; Veleckis & Goetzinger, 1960; Argonne National Laboratory, 1965). They report a hexagonal compound with a composition range of $UZn_{9.36}$ to $UZn_{11.47}$. Veleckis, Schablaske & Tani (1966), using power methods, found that this compound, designated UZn_{12} , is isostructural with the high-temperature form of $SmZn_{12}$. A detailed

* Work was performed in the Ames Laboratory of the U.S. Atomic Energy Commission. Contribution No. 2849.

single-crystal determination of the high-temperature form of SmZn_{12} by Mason, Harsha & Chiotti (1970) has shown that substitution between pairs of zinc atoms and samarium atoms results in a structure with an approximate composition of SmZn_{11} . The purpose of this work is to determine the structure of the zinc-rich uranium-zinc compound.

Experimental procedure

The materials used were reactor grade uranium of 99.9% purity and slab zinc of 99.99+ % purity from the Bunker Hill Co. A 95 wt. % zinc alloy was sealed under argon in a tantalum crucible which in turn was enclosed in stainless steel. The alloy was equilibrated at 900°C and slowly cooled to precipitate the zinc-rich compound. The formed crystallites were isolated by selectively dissolving the zinc matrix with diluted nitric acid. Because of the difficulty in obtaining homogeneous bulk samples, density measurements and chemical analyses were not made.

Two samples were examined by an electron microprobe: a uranium-zinc diffusion couple and a portion of the 95 wt. % zinc alloy. The $\text{UZn}_{8.5}$ compound was used as a reference standard in order to minimize the absorption and fluorescence effects on the measurements of the unknown compound. The measured compositions varied from $\text{UZn}_{10.4}$ to $\text{UZn}_{11.0}$ for crystals in both samples, with an indicated precision in the zinc-to-uranium ratio of ± 0.1 . The total accuracy of the measurements, however, is not known.

Lattice parameters were determined from Debye-Scherrer powder patterns of crystallites from the 95 wt. % alloy, using nickel-filtered copper radiation ($\lambda = 1.5418 \text{ \AA}$). A least-squares procedure, utilizing the Nelson-Riley extrapolation function, gave lattice parameters of $a = 8.950 \pm 0.001$ and $c = 8.902 \pm 0.002 \text{ \AA}$.

All single crystals examined by oscillation and Weissenberg methods had the same Laue symmetry, $6/mmm$. The observed Weissenberg photographs were essentially identical in spot position and intensity with the corresponding SmZn_{12} films. No systematic extinctions were observed. The possible space groups are $P\bar{6}m2$,

$P6mm$, $P622$, $P\bar{6}2m$ and $P6/mmm$. Relative intensity data were obtained by the 2θ scan technique using a General Electric single-crystal orienter equipped with a scintillation counter. Zirconium-filtered $\text{Mo } K\alpha$ radiation ($\lambda = 0.7107 \text{ \AA}$) was used. All reflections of the type hkl with $h \geq k$ were examined in the range $0-70^\circ$, 2θ . The φ dependence was measured at $\chi = 90^\circ$.

The crystal used was basically a prism of height 0.20 mm and maximum thickness 0.09 mm. Several edges were cleaved, making the crystal rather irregular. The measured φ dependence was used to determine a φ -dependent absorption correction for all reflections. Reflections were considered observed if $(I - \text{background}) > 2.5(I + \text{background})^{1/2}$. Of the 599 possible reflections, 345 were observed. Atomic scattering factors were taken from Cromer & Waber (1965). Anomalous dispersion terms were taken from Cromer (1965). The full-matrix least-squares program *ORFLS* of Busing, Martin & Levy (1962) was used in the refinement. All R indices given are for $\sum |F_o - KF_c| / \sum |F_o|$ where K is a scale factor and only observed reflections are included.

Structure determination

The initial trial model was based on the centric space group $P6/mmm$ with the SmZn_{12} parameters as input. This gave an R index of 0.16, indicating the essential correctness of the model. The strong low-angle reflections showed extinction effects and the strongest were excluded from the initial refinements. A series of difference Fourier analyses and least-squares refinements indicated a substitution of uranium atoms for pairs of zinc atoms. The uranium atoms occupy the $\frac{1}{3}, \frac{2}{3}, 0$ positions 24% of the time, with a corresponding decrease to 76% of the zinc pair occupancy of the $\frac{1}{3}, \frac{2}{3}, 0.14$ positions. The criterion used to obtain the degree of substitution about any position was the minimization of the difference Fourier. The R index based on this defect model and using anisotropic temperature factors was 0.055. The R index including the strong low-angle reflections was 0.074. Extinction corrections of the form $F_o(\text{corrected}) = F_o(1 + \beta I_o)$ (Zachariasen, 1963), where β is the extinction parameter and I_o is the measured

Table 1. Least-squares parameters for UZn_{12} (space group $P6/mmm$)

Values in parentheses are standard deviations in the rightmost digits.

Wyckoff notation	x	y	z	β_{11}^* $\times 10^4$	β_{22} $\times 10^4$	β_{33} $\times 10^4$	β_{12} $\times 10^4$	β_{13} $\times 10^4$	β_{23} $\times 10^4$	Occupancy
Zn(1)	12 (o)	0.1683 (2)	2x	0.2390 (4)	33 (2)	51 (3)	51 (4)	1 (1)	$2\beta_{13}$	100
Zn(2)	6 (j)	0.3550 (6)	0.0	0.0	102 (7)	281 (18)	42 (7)	—	—	100
Zn(3)	6 (k)	0.2953 (5)	0.0	$\frac{1}{2}$	38 (4)	19 (5)	36 (6)	—	—	100
Zn(4)	6 (i)	$\frac{1}{2}$	0.0	0.2753 (6)	44 (4)	54 (7)	60 (5)	—	—	100
Zn(5)	2 (e)	0.0	0.0	0.3537 (7)	37 (6)	β_{11}	13 (6)	—	—	100
Zn(6)	4 (h)	$\frac{1}{2}$	$\frac{2}{3}$	0.1472 (10)	72 (8)	β_{11}	56 (9)	—	—	76
Zn(7)†	4 (h)	$\frac{1}{2}$	$\frac{2}{3}$	0.3492 (135)	71	β_{11}	64	—	—	5
U(1)	1 (a)	0.0	0.0	0.0	40 (3)	β_{11}	28 (4)	—	—	100
U(2)	2 (d)	$\frac{1}{2}$	$\frac{2}{3}$	$\frac{1}{2}$	18 (2)	β_{11}	24 (3)	—	—	95
U(3)	2 (c)	$\frac{1}{2}$	$\frac{2}{3}$	0.0	40 (8)	β_{11}	42 (10)	—	—	24

* The anisotropic temperature-factor relation used is $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

† Temperature terms held constant during refinement cycle.

intensity, were applied to the data. A value of $\beta=1.0 \times 10^{-4}$ gave the lowest R index (0.050) after two refinement cycles using all observed reflections.

Table 2. Observed and calculated structure factors

H	K	L	F _o	F _c	H	K	L	F _o	F _c	H	K	L	F _o	F _c	H	K	L	F _o	F _c						
0	0	1	0	-23	6	0	2	43	-40	2	1	4	34	-18	3	3	6	339	324						
2	0	0	-18	7	0	2	35	42	3	1	4	0	-4	4	3	6	0	7	9	1	9	84	89		
3	0	0	447	452	8	0	2	25	9	4	1	4	54	-49	5	3	6	0	-7	2	2	9	0	-5	
4	0	0	0	36	9	0	2	167	178	5	1	4	39	-33	6	3	6	198	199	3	2	9	136	126	
5	0	0	0	7	10	0	2	0	-26	6	1	4	23	-17	7	3	6	0	-17	7	3	9	0	6	
6	0	0	640	623	11	0	2	0	2	7	1	4	30	25	8	3	6	0	10	0	2	0	0	-21	
7	0	0	0	34	1	1	2	202	208	8	1	4	27	-29	9	3	6	109	109	6	2	9	32	33	
8	0	0	0	9	2	1	2	44	-35	9	1	4	28	-26	4	4	6	36	22	7	2	9	77	76	
9	0	0	97	106	3	1	2	0	-2	10	1	4	45	43	5	4	6	26	-22	8	2	9	0	-46	
10	0	0	0	30	4	1	2	201	202	2	2	4	319	305	6	4	6	0	23	3	3	9	0	-22	
11	0	0	38	38	5	1	2	53	-44	3	2	4	44	-37	7	4	6	168	171	4	3	9	35	38	
12	0	0	182	196	6	1	2	0	-4	4	2	4	58	-48	8	4	6	0	-5	5	3	9	99	98	
13	1	0	96	81	7	1	2	172	174	5	2	4	43	-39	5	5	6	120	121	6	3	9	0	-17	
14	1	0	0	7	8	1	2	44	-44	6	2	4	39	-37	6	4	6	0	-17	7	3	9	35	-19	
15	1	0	0	0	9	1	2	0	-17	7	2	4	0	-17	7	2	4	0	-21	4	4	9	0	-14	
16	1	0	103	106	10	1	2	126	134	8	2	4	181	182	6	6	6	0	18	5	4	9	89	84	
17	1	0	24	-24	11	1	2	0	-29	9	2	4	32	-33	7	6	6	0	6	6	4	9	0	-23	
18	1	0	0	0	12	2	2	305	-312	10	2	4	38	-41	0	0	7	0	-27	7	4	9	0	-32	
19	1	0	112	122	3	2	2	28	-23	3	3	4	136	126	1	0	7	32	-29	5	5	9	0	-14	
20	1	0	0	-19	4	2	2	32	-21	4	3	4	22	25	2	0	7	167	167	6	5	9	78	80	
21	1	0	0	-17	5	2	2	172	170	5	3	4	0	-16	3	0	7	0	-18	0	0	10	97	101	
22	1	0	98	105	6	3	2	30	-25	4	3	5	50	-52	6	5	6	52	51	1	0	9	0	-25	
23	1	0	0	-14	7	2	2	0	-46	7	3	4	0	16	5	0	7	24	35	2	0	10	29	34	
24	2	0	636	630	8	2	2	75	-79	8	3	4	0	-2	6	0	7	0	-25	3	0	10	174	172	
25	2	0	33	-30	9	2	2	0	-20	9	3	4	41	33	7	0	7	65	-66	4	0	10	33	33	
26	2	0	0	-10	10	2	2	0	-10	10	1	4	0	12	0	0	7	82	82	5	0	10	0	0	
27	2	0	0	83	78	11	2	2	100	102	4	4	4	283	275	9	0	7	-13	6	0	10	45	41	
28	2	0	27	-23	12	3	2	385	367	5	4	4	48	-44	10	0	7	0	0	12	7	0	10	2	
29	2	0	0	36	13	3	2	0	6	6	4	4	0	-17	1	1	7	0	-15	8	0	10	15	0	
30	2	0	290	295	14	3	2	0	-13	7	5	4	49	-53	2	1	7	0	9	1	1	10	32	38	
31	2	0	0	-26	15	3	2	212	211	8	4	4	36	-40	3	1	7	38	-40	2	1	10	32	38	
32	2	0	0	-27	16	3	2	0	17	9	4	4	0	-5	4	1	7	0	-19	3	1	10	0	-20	
33	2	0	50	50	17	3	2	0	10	5	5	4	0	-10	5	1	7	35	35	4	1	10	46	53	
34	3	0	310	297	18	3	2	96	107	6	5	4	0	-5	6	1	7	0	-19	5	1	10	34	-37	
35	3	0	47	44	19	3	2	0	-2	7	5	5	5	-33	6	5	6	0	-27	7	0	10	36	-35	
36	3	0	0	-4	20	4	2	103	-101	8	5	4	0	9	8	1	7	41	35	7	1	10	52	62	
37	3	0	153	147	21	4	2	30	-29	6	6	4	160	165	9	1	7	0	6	8	1	10	0	-35	
38	3	0	31	32	22	4	2	0	-5	7	6	4	0	-7	10	1	7	0	-28	2	2	10	52	-49	
39	3	0	0	0	23	4	2	183	183	9	6	4	0	-30	1	0	8	25	24	3	2	10	36	-35	
40	3	0	87	85	24	4	2	0	-24	1	0	5	111	118	3	2	7	47	42	4	2	10	0	8	
41	3	0	29	24	25	4	2	0	-24	11	0	5	61	53	4	2	7	118	114	5	2	10	48	50	
42	3	0	487	464	26	4	2	116	112	2	0	5	29	30	5	2	7	0	-16	6	2	10	0	0	
43	3	0	40	-37	27	4	2	0	-13	11	0	5	20	-26	6	2	7	130	127	7	0	10	36	-35	
44	3	0	0	6	28	4	2	0	-29	4	0	5	91	-94	7	2	7	0	8	3	3	10	131	128	
45	3	0	121	121	29	4	2	91	91	5	0	5	164	165	8	2	7	0	-25	4	3	10	0	-12	
46	3	0	30	-24	30	4	2	0	-16	6	0	5	20	-20	9	2	7	45	36	5	3	10	0	-22	
47	3	0	0	0	31	4	2	0	-25	7	1	5	20	-26	6	3	7	0	18	6	4	10	74	80	
48	3	0	74	75	32	4	2	0	5	8	0	5	0	27	3	3	7	66	-64	7	3	10	0	0	
49	3	0	0	7	33	4	2	0	4	10	0	5	44	-46	5	3	7	40	39	4	4	10	0	10	
50	3	0	0	-25	34	4	2	132	131	9	0	5	0	-16	6	3	7	0	-15	5	4	10	0	-31	
51	3	0	50	46	35	4	2	0	-25	10	1	5	25	-22	7	7	7	57	-52	6	4	10	0	13	
52	3	0	0	-9	36	4	2	0	128	131	11	1	5	61	66	3	7	0	20	5	0	10	35	38	
53	3	0	262	258	37	4	2	47	45	2	1	5	161	156	9	3	7	0	-15	0	0	11	0	-38	
54	3	0	0	2	38	4	2	0	319	328	3	1	5	79	77	4	4	7	0	-35	1	0	11	43	44
55	3	0	0	0	39	4	2	0	-13	4	1	5	36	-35	5	4	7	25	29	2	0	11	154	153	
56	3	0	118	122	40	4	2	0	36	-43	5	1	5	147	147	6	5	7	0	-46	3	1	11	0	-10
57	3	0	0	-23	41	4	2	0	116	124	6	1	5	61	60	7	4	7	0	-34	4	0	11	81	84
58	3	0	50	49	42	4	2	0	168	173	7	1	5	24	-28	8	4	7	71	71	5	0	11	63	65
59	3	0	89	29	43	4	2	0	-24	8	1	5	108	109	5	5	7	0	-16	6	0	11	0	-28	
60	3	0	0	-18	44	4	2	0	11	9	1	5	5	-34	6	5	7	0	29	8	0	11	82	97	
61	3	0	80	-86	45	4	2	0	154	159	10	1	5	28	-28	7	5	7	34	21	8	0	11	82	97
62	3	0	126	130	46	4	2	0	-15	2	2	5	23	-24	6	6	7	0	-19	1	1	11	0	-6	
63	3	0	0	-18	47	4	2	0	32	38	3	2	5	175	169	0	8	463	473	2	1	11	58	59	
64	3	0	54	54	48	4	2	0	-25	1	1	5	54	-57	1	0	8	25	24	3	1	11	26	31	
65	3	0	0	-14	49	4	2	0	70	69	5	2	5	0	-22	0	8	46	-48	4	0	11	0	-8	
66	3	0	0	14	50	4	2	0	173	171	6	2	5	50	51	3	0	8	236	234	5	1	11	57	64
67	3	0	42	40	51	4	2	0	82	81	7	2	5	87	88	4	0	8	0	-14	6	1	11	30	29
68	3	0	0	0	52	4	2	0	31	-34	8	2	5	8	0	4	1	8	0	11	1	11	0	-12	
69	3	0	0	-16	53	4	2	0	159	160	9	2	5	95	90	6	0	8	269	276	2	1	11	0	-12
70	3	0	25	-18	54	4	2	0	63	63	10	2	5	0	-18	7	0	8	0	29	3	2	11	78	73
71	3	0	102	105	55	4	2	0	25	-35	3	3	5	0	21	8	0	8	0	-16	4	2	11	116	115
72	3	0	0	0	56	4	2	0	13	13	4	3	0	25	9	8	1								

Table 3 (cont.)

	Type neighbor	Number of neighbors	Distance Å	
Zn(1)	Zn(1)	6	3.329	
	Zn(4)	6	3.563	
	Zn(4)	2	2.592	
	Zn(1)	2	2.610	
	Zn(2)	2	2.660	
	Zn(6)	1	2.686	
	Zn(3)	2	2.694	
	Zn(7)	1	2.738	
	Zn(5)	1	2.803	
	U(3)	1	3.329	
Zn(2)	U(1)	1	3.368	
	U(2)	1	3.458	
	Zn(2)	1	2.596	
	Zn(1)	4	2.660	
	Zn(4)	2	2.775	
	U(3)	2	2.892	
	Zn(6)	4	3.176	
	Zn(2)	2	3.178	
	U(1)	1	3.178	
	Zn(3)	2	2.644	
Zn(3)	Zn(1)	4	2.694	
	Zn(4)	2	2.714	
	Zn(5)	2	2.948	
	U(2)	2	3.168	
	Zn(7)	4	3.444	
	Zn(3)	1	3.665	
	Zn(4)	Zn(1)	4	2.592
		Zn(7)	2	2.665
		Zn(3)	2	2.714
		Zn(2)	2	2.775
Zn(6)		2	2.825	
U(2)		2	3.269	
U(3)		2	3.563	
Zn(5)		Zn(5)	1	2.606
		Zn(3)	6	2.948
		Zn(1)	6	2.803
	U(1)	1	3.151	
	Zn(6)	Zn(6)	1	2.623
		Zn(1)	3	2.687
		Zn(4)	3	2.825
		U(2)	1	3.142
		Zn(2)	6	3.176
		Zn(7)	Zn(4)	3
Zn(7)			1	2.701
Zn(1)			3	2.738
U(3)			1	3.104
Zn(3)			6	3.444

U(3)-Zn(6), U(2)-Zn(7) and Zn(6)-Zn(7) pairs do not occur simultaneously.

Microprobe results on crystallites in the 95 wt. % zinc alloy indicate a composition range of $UZ_{10.4}$ to $UZ_{11.0}$. Lattice parameters obtained from powdered crystallites extracted from the same alloy indicate a composition of $UZ_{11.0}$. This composition is based of

the assumption of a linear relation between the composition limits reported by Veleckis & Goetzinger (1960) and on the lattice parameters reported by workers at the Argonne National Laboratory (1965). The observed composition range indicates a solidus curve, for the zinc-rich alloy, which varies with temperature.

There was no evidence of a low-temperature modification of the UZ_{12} compound corresponding to the tetragonal $SmZn_{12}$ form. All examined crystals were hexagonal. However, no special effort was made to prepare a low-temperature form; therefore, such a structure cannot be ruled out.

The assistance of R. W. Buttry in the experimental portions of the work and of F. C. Laabs for the microprobe analysis is gratefully acknowledged.

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