

## Preparation and X-Ray Diffraction Studies of Curium Hydrides<sup>1</sup>

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Curium hydrides were prepared by reaction of curium-248 metal with hydrogen and characterized by X-ray powder diffraction. Several of the syntheses resulted in a hexagonal compound with average lattice parameters of  $a_0 = 0.3769(8)$  nm and  $c_0 = 0.6732(12)$  nm. These products are considered to be  $\text{CmH}_{3.8}$  by analogy with the behavior of lanthanide-hydrogen and lighter actinide-hydrogen systems. Face-centered cubic products with an average lattice parameter of  $a_0 = 0.5322(4)$  nm were obtained from other curium hydride preparations. This parameter is slightly smaller than that reported previously for cubic curium dihydride,  $\text{CmH}_{2+x}$  (B. M. Bansal and D. Damien, *Inorg. Nucl. Chem. Lett.* **6**, 603, 1970). The present results established a continuation of typical heavy trivalent lanthanide-like behavior of the transuranium actinide-hydrogen systems through curium. © 1985 Academic Press, Inc.

### 1. Introduction

Only a face-centered cubic (fcc) hydride of curium has been reported previously (1). This phase was taken to be the dihydride,  $\text{CmH}_{2+x}$ , by analogy with isostructural phases of other actinide-hydrogen systems. The lattice parameter of  $a_0 = 0.5344(5)$  nm obtained in that study is similar to those of the preceding fcc actinide dihydrides,  $\text{NpH}_2$  ( $a_0 = 0.5343$  nm) (2),  $\text{PuH}_2$  ( $a_0 = 0.5360$  nm) (3), and  $\text{AmH}_2$  ( $a_0 = 0.5344$  nm) (4). This small variance in lattice parameters for these transuranium dihydrides is in contrast to the regular con-

traction observed between the parameters for the fcc lanthanide dihydrides with increasing atomic number (5). The unique behavior of the light trans thorium actinide metals (through Pu) and their interstitial compounds (hydrides, carbides, etc.) can be attributed to the effects of participation of  $5f$  electrons in bonding (6). Beyond plutonium in the actinide series, the  $5f$  electrons are primarily localized and properties similar to those of the lanthanide elements are generally observed for these elements, although differences between the transplutonium and lanthanide series are known (6). One aim of this study was to establish curium-hydrogen phase behavior for comparison with that of lanthanide and other actinide hydrides.

The lanthanide elements beyond promethium (excluding europium and ytterbium) and the transuranium elements nep-

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tunium, plutonium, and americium have each been shown to form a trihydride phase of hexagonal symmetry in addition to their fcc dihydrides. In contrast to the other lanthanide metals which are trivalent, europium and ytterbium are divalent metals and form alkaline earth-like (7), orthorhombic dihydrides. Whereas other compounds of europium and ytterbium exhibit stoichiometries and structures indicative of trivalent lanthanide elements, their hydrides thus reflect the stability of the divalent electronic state. Similarly, the hydriding properties of the transplutonium actinide elements may reflect the trend toward increasing stability of the divalent state across that series (8).

If the hydriding behavior characteristic of the trivalent lanthanides prevails beyond americium in the actinide series, then curium might be expected to exhibit similar hydriding behavior to its electronic homolog, gadolinium, and form both a fcc dihydride and a hexagonal trihydride phase. Reported here are the preparation and characterization by X-ray powder diffraction of both fcc  $\text{CmH}_{2+x}$  and hexagonal  $\text{CmH}_{3.6}$ .

## 2. Experimental

### 2.1. Materials

The curium metal used to prepare the hydrides was a mixture of mainly the curium-246 (3%) and curium-248 (97%) isotopes and will be referred to as curium-248. Curium-248 has the lowest specific radioactivity of curium isotopes available for such solid state studies. The curium-248 was a daughter product of californium-252 produced at the High Flux Isotope Reactor and Transuranium Processing Facility at the Oak Ridge National Laboratory. After final separation/purification of the curium by chromatography (9), two curium metal preparations were made by lithium vapor

reduction of milligram quantities of  $\text{CmF}_4$  at elevated temperature (10); during the procedure, the LiF and excess Li metal were vaporized away from the product.

Although the limited quantity of curium metal available precluded determination of impurities such as oxygen and nitrogen, its physical appearance and malleability suggested an absence of major anionic contamination. Spark source mass spectrographic analyses indicated a total metallic impurity level of  $\leq 150$  ppm by weight in each of the two curium metal preparations. X-Ray analyses established that both preparations were double hexagonal close-packed (dhcp) curium metal with lattice parameters in agreement with those given by Stevenson and Peterson (11).

For most of the hydride syntheses, ultrahigh-purity hydrogen gas (cylinder, 99.999%) was used although other sources were tried in a few cases. For some of the preparations, a mixture of 4%  $\text{H}_2$ /96% Ar by volume (cylinder, 99.95% net) provided a reduced hydrogen partial pressure for the syntheses.

### 2.2. Hydride Preparation

Individual curium metal samples of 10–100  $\mu\text{g}$  were placed into fused silica capillaries in a helium atmosphere gloved box. These capillaries were subsequently attached to a vacuum/gas manifold without exposing the curium to air. In some cases, the capillaries were preheated to  $\sim 600^\circ\text{C}$  under high vacuum before inserting the curium metal to remove adsorbed impurities (oxygen, water, etc.) from the capillary walls. Following degassing under high vacuum ( $1.5 \times 10^{-4}$  Pa) and at least two flushings with hydrogen at room temperature, the curium samples were hydrided under  $1 \times 10^5$  Pa  $\text{H}_2$  (or 4%  $\text{H}_2$ /96% Ar,  $1 \times 10^5$  Pa total pressure) at temperatures from 200 to  $650^\circ\text{C}$  for 1 to 70 hr. Reaction was visually evidenced by formation of several fragments of grey hydride product from the cu-

rium metal. In addition, several hydride products were annealed under hydrogen atmospheres in the capillaries at 100 to 250°C for up to 100 hr in attempts to improve the crystallinity of the material.

### 2.3. X-Ray Diffraction

The capillaries containing the products under  $5 \times 10^4$  Pa  $H_2$  (or  $H_2/Ar$ ) were flame-sealed. The resulting capillaries were loaded into 114.6-mm-diameter Debye-Scherrer powder cameras. Niobium-filtered  $MoK\alpha$  radiation from a microfocus X-ray generator was used to obtain the powder diffraction patterns. Even those products which had been annealed *in situ* for extended periods of time exhibited rather diffuse diffraction lines, with the higher angle lines being too broad to measure accurately. Due to the absence of high-angle lines with the  $MoK\alpha$  radiation, shrinkage factors could not be obtained for the Kodak DEF-392 film. Experience with other samples has shown that film shrinkage is less than 0.1%.

Refined lattice parameters were obtained with the LCR-2 program (12) applying Nelson-Riley corrections to the indexed lines. Uncertainties reported here represent estimates of the precision of the diffraction results based upon both the standard deviation within each diffraction data set and that between data obtained for different samples. Systematic effects such as deviations of the hydride products from stoichiometry have not been considered in these uncertainty assignments.

## 3. Results and Discussion

In developing the preparative procedure employed for the curium-hydrogen system, its efficacy was confirmed using lanthanides as stand-ins. The fcc dihydrides of neodymium, samarium, and dysprosium and the hexagonal trihydride of dysprosium were prepared and all exhibited lattice

parameters within 0.15% of their literature values (13-15). In addition, a blank run and subsequent annealings and X-ray analyses were performed with curium using helium instead of hydrogen to assure that no appreciable reaction of the metal occurred in an inert atmosphere at the temperatures of the hydride syntheses.

### 3.1. Curium Trihydride

A hexagonal curium hydride was obtained in eight products, although in two of these cases there were an insufficient number of diffraction lines to calculate precise lattice parameters. In most of the X-ray diffraction patterns there were a few diffuse and generally weak additional lines not assignable to the hexagonal phase. In some patterns these additional lines suggested the presence of a fcc  $CmH_{2+x}$  phase while in most they indicated the presence of a poorly crystalline body-centered cubic (bcc) form of  $Cm_2O_3$  (16). The work of Muromura *et al.* (3) on plutonium hydrides suggests that the presence of such an oxide phase probably does not crystallographically affect the hydride to an extent discernable with these experiments. It is not expected that a superstructure due to ordering of hydrogen atoms would be detected with this X-ray diffraction technique, which is sensitive primarily to the heavier atom positions.

From the six best hexagonal curium hydride preparations, the following average lattice parameters were derived:  $a_0 = 0.3769(8)$  nm,  $c_0 = 0.6732(12)$  nm. These average values were obtained from trihydride products derived from each of the two curium metal preparations. The  $c/a$  ratio of 1.786 for these parameters differs significantly from the close-packed value of 1.633, but is in good agreement with the  $1.795 \pm 0.015$  observed for the isostructural lanthanide (5), neptunium (2), plutonium (17), and americium (4) trihydrides. Although no direct analytical measure of

TABLE I  
OBSERVED AND CALCULATED POWDER DIFFRACTION  
LINES FOR HEXAGONAL CURIUM TRIHYDRIDE  
AND FACE-CENTERED CUBIC CURIUM DIHYDRIDE

h	k	l	Observed			Calculated <sup>c</sup>	
			Intensity <sup>a</sup>	2 $\theta^b$	d (nm)	2 $\theta^b$	d (nm)
CmH <sub>3-<math>\delta</math></sub> <sup>d</sup>							
0	0	2	3	12.11	0.3369	12.12	0.3363
1	0	0	3	12.56	0.3249	12.51	0.3257
1	0	1	10	13.91	0.2935	13.91	0.2931
1	0	2	2	17.51	0.2335	17.46	0.2340
1	1	0	5	21.71	0.1887	21.77	0.1880
1	0	3	2	22.21	0.1845	22.17	0.1847
0	0	4	<1	24.26	0.1691	24.38	0.1682
1	1	2	4	25.06	0.1638	24.99	0.1641
2	0	1	3	25.96	0.1582	25.93	0.1583
1	0	4	<1	27.41	0.1500	27.49	0.1494
2	0	2	<1	27.96	0.1471	28.04	0.1466
2	0	3	1	31.26	0.1319	31.27	0.1318
1	1	4	2	33.06	0.1249	32.91	0.1254
2	1	1	3	34.11	0.1212	34.11	0.1211
2	1	2	<1	35.71	0.1159	35.78	0.1156
2	1	3	2	38.41	0.1080	38.43	0.1079
3	0	2	2	40.21	0.1034	40.21	0.1033
1	1	6	<1	43.31	0.0963	43.28	0.0963
2	1	5	2	46.06	0.0908	46.04	0.0908
CmH <sub>2+x</sub> <sup>f</sup>							
1	1	1	10	13.28	0.3074	13.28	0.3070
2	0	0	5	15.34	0.2663	15.35	0.2659
2	2	0	5	21.76	0.1883	21.78	0.1880
3	1	1	6	25.61	0.1604	25.59	0.1603
2	2	2	3	26.74	0.1537	26.75	0.1535
4	0	0	1	31.02	0.1329	30.99	0.1329
3	3	1	4	33.84	0.1221	33.85	0.1220
4	2	0	4	34.74	0.1190	34.76	0.1189
4	2	2	3	38.22	0.1086	38.20	0.1085
3	3	3	3	40.62	0.1024	40.61	0.1023
5	1	1					
4	4	0	<1	44.40	0.0941	44.39	0.0940
5	3	1 <sup>e</sup>	2	46.48	0.0901	46.55	0.0899
6	0	0 <sup>e</sup>	1	47.29	0.0886	47.25	0.0886
4	4	2 <sup>e</sup>					
6	2	0 <sup>e</sup>	<1	49.92	0.0842	49.97	0.0841
6	4	2 <sup>e</sup>	<1	59.96	0.0711	59.97	0.0711
7	3	1 <sup>e</sup>	<1	61.72	0.0693	61.73	0.0692
5	5	3 <sup>e</sup>					

<sup>a</sup> Intensities estimated visually; 10 = strongest.

<sup>b</sup> 2 $\theta$  for MoK $\alpha$  radiation ( $\lambda\alpha = 0.07108$  nm).

<sup>c</sup> Calculated using Nelson-Riley extrapolation function.

<sup>d</sup> Curium trihydride of undetermined stoichiometry, hexagonal lattice,  $a_0 = 0.3763$  nm,  $c_0 = 0.6731$  nm.

<sup>e</sup> Broad and/or weak line assigned lower weighting for lattice parameter refinement due to difficulty in accurately measuring its position.

<sup>f</sup> Curium dihydride of undetermined stoichiometry, fcc,  $a_0 = 0.5321$  nm.

product stoichiometries was made, these X-ray data strongly support the conclusion that the products were hexagonal CmH<sub>3- $\delta$</sub> .

The "3- $\delta$ " is used to indicate the possibility of trihydride substoichiometry. Based upon the behavior of the samarium (18) and plutonium (19) trihydride systems, it is expected that  $\delta$  for CmH<sub>3- $\delta$</sub>  will probably lie in the range:  $0 < \delta < 0.2$ . In Table I are given the observed and calculated X-ray diffraction lines for a particular curium hydride product which exhibited hexagonal lattice parameters ( $a_0 = 0.3763$  nm,  $c_0 = 0.6731$  nm) similar to those selected here as the best for CmH<sub>3- $\delta$</sub> .

### 3.2. Curium Dihydride

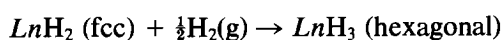
Three hydride products were prepared which exhibited a sufficient number of diffraction lines to calculate precise fcc lattice parameters. These products are considered to be CmH<sub>2+x</sub> based on comparison with isostructural hydride phases of lanthanides and other actinides. A second phase present in two of these products was identified as the bcc form of Cm<sub>2</sub>O<sub>3</sub> (16) from its diffraction pattern. Residual dhcp curium metal (11) was present in the third preparation. Within the assigned uncertainty, all three dihydride samples gave the same fcc lattice constant of 0.5322(4) nm. The agreement between the three values suggests that the oxygen activity indicated by the presence of the sesquioxide phase in the two products did not affect the hydride unit cell volume within the precision of this work. This observation is consistent with the results of structural studies on PuH<sub>2+x</sub>/PuO<sub>2</sub> two-phase systems (3). Observed and calculated diffraction lines for a particular fcc CmH<sub>2+x</sub> product which exhibited a lattice parameter ( $a_0 = 0.5321$  nm) similar to the average value are reported in Table I.

The fcc lanthanide and actinide dihydrides exist over wide composition ranges with  $x$  in the formula MH<sub>2+x</sub> ranging from approximately -0.2 up to 1 for the light lanthanides through neodymium. For lanthanide dihydride phases with lattice parameters similar to that of curium dihydride

(e.g.,  $\text{GdH}_{2+x}$ ),  $x$  is limited to approximately  $-0.2 < x < 0.3$  (5). As the actual hydrogen contents of neither the curium dihydrides prepared in this work nor those of the previous study (1) were measured, the 0.4% difference between the two lattice parameters (0.5322 here vs 0.5344 nm from (1)) may be at least partially attributable to differences in stoichiometry. Based on the established behavior of the lanthanide hydrides, a contraction of the  $\text{CmH}_{2+x}$  lattice with increasing  $x$  would be expected. However, the presence of residual curium metal in one of the products obtained here implies that the dihydrides were probably of a composition at or near the lower phase boundary (i.e.,  $x$  less than or close to 0 and  $a_0$  close to its maximum value). Another contribution to the observed difference in lattice parameters may relate to the use of the curium-244 rather than the curium-248 isotope in the earlier work. Curium-244 ( $t_{1/2} = 18$  years) has a higher specific alpha activity than the much longer-lived curium-248 ( $t_{1/2} = 3 \times 10^5$  years) used in this work. Lattice expansions of a few tenths of a percent due to self-radiation damage have been reported for compounds containing alpha emitters (16, 20) and such an effect could be expected with curium-244 hydrides.

### 3.3. Comparison with Other Hydrides

The  $\text{NdH}_{2+x}$  system with  $a_0 = 0.547$  nm (when  $x = 0$ ) (13) is the most contracted lanthanide dihydride phase to accommodate hydrogen atoms in its octahedral interstices up to  $x = 1$  (i.e., to  $\text{NdH}_3$ ) while retaining its fcc structure. The transneodymium trivalent lanthanides, with the possible exception of promethium (whose hydrides have not been well characterized) exhibit a phase separation between a hyperstoichiometric fcc dihydride and a hexagonal trihydride (5). The reaction



(where  $\text{Ln} =$  lanthanides which form both a

fcc dihydride and a hexagonal trihydride) invariably involves an increase in lattice volume per metal atom, suggesting that the metal lattices expand to accommodate additional interstitial hydrogen.

Table II presents crystallographic parameters for hydrides of selected lanthanides and transuranium actinides together with the calculated volume difference (expansion) per metal atom between the fcc dihydrides and hexagonal trihydrides. The volume change is given for the stoichiometric hydrides except for curium where the hydride compositions were not actually measured. A continuation of typical heavy trivalent lanthanide/transuranium actinide behavior through curium is evident from the data in Table II. Since all of the presently known transuranium dihydrides exhibit fcc lattice parameters comparable to or smaller than that of  $\text{SmH}_2$ , it is not surprising that a fcc  $\text{AnH}_{2+x}$  - hexagonal  $\text{AnH}_{3-\delta}$  phase separation occurs in each of these systems. This general behavior might be expected to continue across the actinide series as long as these  $5f$  elements behave as their trivalent lanthanide counterparts.

Although gadolinium is the electronic homolog of curium, the ionic radius of  $\text{Cm(III)}$  is closer to that of  $\text{Sm(III)}$  (7). Due to the bonding similarities among the trivalent lanthanide and transplutonium actinide elements, subtle differences in behavior are largely attributable to size effects. While the uncertainties in exact curium hydride stoichiometries may preclude quantitative comparison, the range of lattice parameters is not expected to be large enough to invalidate qualitative comparisons with lanthanide and other actinide hydrides. (The fcc lattice parameter of structurally similar samarium dihydride varies by less than 0.5% over its stoichiometry range (18) while the parameters of hexagonal  $\text{SmH}_{3-\delta}$  each change by 0.2% or less as  $\delta$  varies from 0 to 0.18 (18, 15).) As seen in Table II, the lattice parameters of both fcc  $\text{CmH}_{2+x}$

TABLE II  
CRYSTALLOGRAPHIC DATA FOR SELECTED LANTHANIDE AND ACTINIDE HYDRIDES

<i>M</i>	fcc $MH_2$	Hexagonal $MH_3$			$\Delta V \times 10^{3a}$ ( $nm^3$ )	References
	$a_0$ (nm)	$a_0$ (nm)	$c_0$ (nm)	$c/a$		
Lanthanides						
Nd <sup>b</sup>	0.5469	—	—	—	—	13
Sm	0.5363	0.3782	0.6779	1.792	3.42	14, 15
Gd	0.5303	0.373	0.671	1.80	3.14	21
Dy	0.5201	0.3671	0.6615	1.802	3.43	15
Actinides						
Np	0.5343	0.3771	0.6713	1.780	3.20	2
Pu	0.5360	0.3779	0.6771	1.792	3.37	3, 17
Am	0.5344	0.3764	0.6763	1.797	3.34	4
Cm <sup>c</sup>	0.5322	0.3769	0.6732	1.786	3.72	This study

<sup>a</sup> Change in lattice volume per metal atom for the reaction:  $MH_2$  (fcc) +  $\frac{1}{2}H_2(g) \rightarrow MH_3$  (hex.).

<sup>b</sup> Hexagonal form of  $NdH_3$  is unknown; the fcc phase extends up to essentially  $NdH_3$  with an extrapolated decrease in  $a_0$  to 0.542 nm.

<sup>c</sup> Dihydride and trihydride of unknown stoichiometry.

and hexagonal  $CmH_{3.8}$  are intermediate between those of the corresponding compounds of samarium and gadolinium. There is only a very small contraction in the fcc dihydride lattice upon moving from Am to Cm, and only a  $\sim 0.2\%$  decrease in the trihydride cell volumes ( $0.08298 \text{ nm}^3$  for  $AmH_3$  vs  $0.08282 \text{ nm}^3$  for  $CmH_{3.8}$  although their hexagonal  $c/a$  ratios differ by 0.6%). This behavior is consistent with that of other curium compounds (as well as curium metal) which tend to exhibit a smaller actinide contraction effect than do the corresponding compounds of other transplutonium actinides (22, 23). Studies of additional transplutonium hydride systems are necessary to clearly establish the details of the actinide contraction trend in these compounds and to determine how far into the transuranium actinide series behavior is trivalent lanthanide-like for these particular systems.

#### 4. Conclusion

The studies of curium hydrides reported

here have shown that a hexagonal curium hydride exists. This phase was assigned a composition corresponding to curium trihydride based on comparisons of its X-ray diffraction pattern with those of known lanthanide and actinide trihydrides.

In addition, a fcc curium dihydride was prepared. The lattice parameters of the cubic products are similar to that previously reported for a curium dihydride prepared with the more radioactive isotope, curium-244 (1). The difference of 0.4% between lattice parameters may reflect only the lattice expansion resulting from the higher specific radioactivity of the curium-244 isotope. In both this work and the earlier work (1), the exact stoichiometries of the products were not determined. In view of the uncertainties in stoichiometry, and the possibility of self-radiation damage in the curium-244 dihydrides, the deviation noted between these fcc lattice parameters is not excessive and the present results can even be considered a confirmation of the earlier result.

Further work on curium hydrides should establish the exact composition of the ma-

terials studied and determine the stoichiometry ranges over which the fcc and hexagonal structures are stable.

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