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The Crystal Structures of Americium Trichloride Hexahydrate and Berkelium Trichloride Hexahydrate[1]

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Received May 1, 1970

The structure of $AmCl_3 \cdot 6H_2O$ has been studied by single-crystal X-ray diffraction methods. Some 1468 observations were used in a least-squares refinement of the 46 parameters which describe the structure with anisotropic temperature factors. Starting values for the parameters were obtained from the known structure of $GdCl_3 \cdot 6H_2O$ and the final values yielded an agreement index of 0.039. The structure consists of $AmCl_2(OH_2)_6{}^+$ cations and Cl^- anions joined by a network of hydrogen bonds, the existence of which are inferred from the Cl···O···Cl distances and angles found. The monoclinic unit cell of $AmCl_3 \cdot 6H_2O$ has the dimensions $a = 9.702$ (1) Å, $b = 6.567$ (1) Å, $c = 8.009$ (1) Å, and $\beta = 93° 37$ (1)′; the space group is $P2/n$. X-Ray diffraction by a powder sample of $BkCl_3 \cdot 6H_2O$ showed that it is isostructural, and the following unit cell was derived: $a = 9.66$ (1) Å, $b = 6.54$ (1) Å, $c = 7.97$ (1) Å, and $\beta = 93° 46$ (5)′. Unit-cell data are presented for the trichloride heptahydrates of La, Ce, and Pr.

Introduction

The lanthanide trichlorides are known to form heptahydrates for La, Ce, and Pr and hexahydrates for the elements Nd to Lu, and two actinide homologs are known:[3] $PuCl_3 \cdot 6H_2O$ and $AmCl_3 \cdot 6H_2O$. Recently, Brown, Fletcher, and Holah[4] reported on the tribromide hexahydrates of Pr to Lu and U to Am. The crystal structures of $GdCl_3 \cdot 6H_2O$[5] and isostructural $EuCl_3 \cdot 6H_2O$[6] have been determined by single-crystal X-ray diffraction analyses, and all other lanthanide trichloride hexahydrates as well as the above-mentioned tribromide hexahydrates have been shown by powder diffraction[4,7,8] to be isostructural with them. No single-crystal study of an actinide trihalide hexahydrate has been made previously, but the production of gram quantities of ^{243}Am has now made it possible to grow large crystals from solution and analyze them by X-ray diffraction. The availability of microgram quantities of ^{249}Bk has enabled us to study $BkCl_3 \cdot 6H_2O$. Unit-cell dimensions of the heptahydrates mentioned above were determined also.

Experimental Section

Compound Preparation.—The $AmCl_3 \cdot 6H_2O$ was prepared by dissolving about 100 mg of AmO_2 (isotopic composition: mass 243, 99%; mass 241, 1%) in excess 6 *N* HCl and allowing the solution to evaporate in the filtered room air of a glove box. The crystals obtained were well-formed, thick plates of a pale yellow-rose color. Suitable specimens for X-ray diffraction were selected in the glove box and sealed in glass capillary tubes for the purpose of containing the α contamination and maintaining a fixed humidity for these somewhat hygroscopic crystals.

The $BkCl_3 \cdot 6H_2O$ was obtained accidently by slow leakage of room air into a capillary tube containing a sample of anhydrous $BkCl_3$. The preparation of this 2-μg sample was carried out by the single ion-exchange bead technique described by Cunningham and coworkers.[9,10] The ^{249}Bk (half-life 314 days) was self-contaminated by the daughter ^{249}Cf to an extent of 15–20% by the time the powder pattern was recorded. The quantity of Bk available did not make it feasible to grow crystals from solution. Microcrystalline $BkCl_3 \cdot 6H_2O$ is yellow-green, not visibly different from anhydrous $BkCl_3$.[10]

The lanthanide trichloride heptahydrate crystals were grown by evaporation of 6 *N* HCl solutions of the oxides of La, Ce, and Pr. Samples were sealed in glass capillary tubes to maintain the humidity.

Data Collection.—X-Ray precession photographs of a crystal of $AmCl_3 \cdot 6H_2O$ were used to obtain the pattern of systematic absences, $h0l$ for $l = 2n + 1$, and preliminary values for the monoclinic unit-cell dimensions. These data indicated that the structure is isotypic with that of the lanthanide trichloride hexahydrates, for which the space group is $P2/n$, and the number of formula weights in the unit cell is 2; $d_{calcd} = 2.98$ g cm^{-1}.

The crystal used for intensity data collection was a typical tabular specimen with the b axis normal to the tablet face and was mounted in a glass capillary with the a axis approximately along the ϕ axis of the goniometer. Specimen dimensions were 0.36 × 0.08 × 0.28 mm along a, b, and c, respectively. A computer-controlled Picker X-ray diffractometer equipped with a scintillation-counter detector was used with Nb-filtered, Mo Kα radiation to obtain intensity data and measurement of angles for precise determination of the unit-cell dimensions at 23°. Intensities of all nonequivalent reflections up to $2\theta = 60°$ were recorded by a θ–2θ scan technique, with the background counted at the ends of the ∼2° scan and averaged. The takeoff angle was 2°. A reference reflection was measured about once in 1 hr to follow the decreasing diffracting ability of the crystal with time. There was also a continual broadening of the diffraction peaks, requiring a 2θ scan of 2.5° and a receiving aperture of 6 × 6 mm by the end of the data collection. Both the broadening and intensity decrease can be attributed to the self-radiolysis of the compound by radioactive decay of Am. The α particles released can produce defects in the structure and also cause dehydration by local heating as they are absorbed. Similar deterioration of crystals has been observed in organic chelates of Am, but in a study of anhydrous $AmCl_3$ the diffracted intensity remained constant for at least 1 week.[11]

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(1) Research sponsored by the U. S. Atomic Energy Commission under contract with the Union Carbide Corp.

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TABLE I

REFINED PARAMETERS AND THEIR STANDARD ERRORS FOR $AmCl_3 \cdot 6H_2O$

Atom	Site[a]	x	y	z	β_{11}[b]	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Am	2e	$^1/_4$	0.14700 (4)	$^1/_4$	0.00340 (4)	0.00734 (8)	0.00578 (6)	0.0	0.00039 (3)	0.0
Cl(1)	2f	$^3/_4$	0.3789 (4)	$^1/_4$	0.0063 (3)	0.0142 (6)	0.0090 (4)	0.0	0.0005 (2)	0.0
Cl(2)	4g	0.0567 (2)	0.8315 (3)	0.2589 (3)	0.0052 (2)	0.0120 (4)	0.0110 (3)	−0.0020 (2)	0.0012 (2)	−0.0006 (2)
O(1)	4g	0.2816 (6)	0.0445 (9)	0.5468 (6)	0.0084 (6)	0.0168 (14)	0.0074 (7)	−0.0002 (7)	0.0000 (5)	0.0037 (8)
O(2)	4g	0.1462 (6)	0.4265 (9)	0.0854 (7)	0.0085 (6)	0.0124 (11)	0.0102 (8)	0.0034 (7)	0.0009 (5)	0.0016 (8)
O(3)	4g	0.4421 (6)	0.3002 (9)	0.1044 (7)	0.0065 (5)	0.0157 (11)	0.0107 (9)	−0.0026 (7)	0.0028 (5)	−0.0010 (9)

[a] Symmetry site in space group $P2/n$. General positions: $\pm(x, y, z;\ ^1/_2 + x, \bar{y}, ^1/_2 + z)$. [b] Coefficients in the temperature factor $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

TABLE II

STRUCTURE FACTORS FOR $AmCl_3 \cdot 6H_2O$ ON AN ABSOLUTE SCALE[a]

H	OBS	CAL
** H 0 0 **		
2X	200	156-
4	195	166
6	159	148-
8	79	74
10	115	101-
12	58	51
** H 0 1 **		
-13	36	33
-11	74	71-
-9	79	81
-7	58	60-
-5	152	157
-3X	103	96-
-1X	162	159
1X	164	167-
3X	10	18
5	115	125-
7	68	73
9	56	61-
11	63	65
13	29	29-
** H 0 2 **		
-12	53	52-
-10	94	93
-8	67	71-
-6	106	112
-4	166	177-
-2X	107	115
0X	169	199-
2X	104	122
4	123	127-
6	145	155
8	75	81-
10	81	84
12	54	55-
** H 0 3 **		
-13	34	33-
-11	53	53
-9	64	64-
-7	59	63
-5	121	129-
-3	108	110
-1	102	109-
1	126	141
3	102	106-
5	122	131
7	74	80-
9	54	56
11	54	56-
** H 0 4 **		
-12	50	49
-10	79	80-
-8	78	83
-6	124	131-
-4	136	141
-2	116	122-
0	152	163
2	123	131-
4	119	127
6	94	101-
8	57	59
10	66	68-
12	43	48
** H 0 5 **		
-11	55	55-
-9	61	81
-7	62	65-
-5	92	84
-3	46	47-
-1	88	93
1	94	96-
3	62	65
5	82	86-
7	46	49
9	51	52-
11	50	51
** H 0 6 **		
-10	73	72
-8	55	54-
-6	85	88
-4	89	91-
-2	83	84
0	143	147-
2	91	92
4	91	94-
6	87	91
8	54	56-
10	62	63
** H 0 7 **		
-11	48	46
-9	54	53-
-7	39	40
-5	85	87-
-3	61	63
-1	86	87-
1	90	92
3	43	43-
5	68	72
7	46	47-
9	36	38
** H 0 8 **		
-10	60	59-
-8	47	47
-6	64	65-
-4	88	89
-2	57	60-
0	84	87
2	57	60-
4	53	56
6	66	68-
8	39	39
** H 0 9 **		
-7	30	30-
-5	58	57
-3	47	46-
-1	40	41
1	56	58-
3	38	39
5	51	52-
7	40	40
** H 0 10 **		
-6	49	48
-4	62	60-
-2	47	48
0	60	61-
2	57	57
4	49	48-
** H 0 11 **		
-3	34	34
-1	35	36-
1	42	41
** H 1 0 **		
1X	126	116-
2X	78	74-
3	125	119
4	103	96
5	66	65-
6	73	73-
7	78	79
8	34	35
9	58	59-
10	40	38-
11	40	38
12	25	24
13	40	40-
** H 1 1 **		
-13	25	27
-12	45	50-
-11	44	46-
-10	54	54
-9	57	58
-8	95	94-
-7	80	78-
-6	81	78
-5	124	116
-4	157	131-
-3	95	80-
-2X	189	167
-1X	120	105
0X	137	116-
1X	118	110-
2X	173	162
3X	111	91
4	102	93-
5	99	92-
6	99	90
7	48	44
8	97	91-
9	48	46-
10	57	47
11	50	43
12	58	49-
13	32	27-
** H 1 2 **		
-13	41	44-
-12	22	24-
-11	31	33
-10	40	44
-9	54	59-
-8	34	37-
-7	68	73
-6	55	58
-5	49	51-
-4	33	35-
-3	140	143
-2	17	18
-1X	56	63-
0X	62	90-
1X	78	85
2	56	55
3	118	116-
4	69	66-
5	55	52
6	53	52
7	87	87-
8	33	33-
9	55	52
10	50	47
11	36	34-
12	30	28-
13	44	41
** H 1 3 **		
-13	20	22-
-12	45	49
-11	39	43
-10	36	38-
-9	47	51-
-8	86	94
-7	47	50
-6	68	73-
-5	78	84-
-4	100	108
-3	65	69
-2	145	161-
-1	127	133-
0	66	66
1	131	139
2	150	158-
3	77	79-
4	103	106
5	87	90
6	71	73-
7	57	57-
8	86	88
9	53	51
10	39	39-
11	44	43-
12	45	44
** H 1 4 **		
-12	23	24
-11	30	32-
-10	43	46-
-9	47	47
-8	31	33
-7	81	87-
-6	50	53-
-5	42	44
-4	69	73
-3	103	109-
-2	60	64-
-1	76	79
0	91	96
1	57	58-
2	37	39-
3	114	118
4	36	38
5	42	43-
6	49	50-
7	58	60
8	26	28
9	50	50-
10	34	33-
11	27	27
12	22	20
** H 1 5 **		
-12	45	47-
-11	36	39-
-10	34	37
-9	44	46
-8	73	77-
-7	37	39-
-6	72	76
-5	70	75
-4	60	62-
-3	61	65-
-2	113	118
-1	62	64
0	87	70-
1	62	64-
2	97	100
3	40	40
4	85	89-
5	63	66-
6	47	49
7	48	49
8	65	69-
9	36	37-
10	40	40
11	36	34
** H 1 6 **		
-11	32	34
-10	29	29
-9	36	37-
-8	23	24-
-7	60	62
-6	37	38
-5	41	41-
-4	52	52-
-3	64	66
-2	34	34
-1	75	77-
0	50	50-
1	52	54
2	44	45
3	75	76-
4	48	49-
5	43	45
6	48	50
7	45	47-
8	27	28-
9	43	44
10	29	28
** H 1 7 **		
-11	26	27
-10	35	36-
-9	37	38-
-8	53	55
-7	42	43
-6	57	58-
-5	62	62-
-4	61	61
-3	54	54
-2	65	65-
-1	55	57-
0	65	66
1	64	65
2	69	70-
3	54	53-
4	60	63
5	50	52
6	45	46-
7	32	33-
8	49	50
9	25	26
10	32	32-
** H 1 8 **		
-10	28	28-
-9	33	33
-8	28	28
-7	45	44-
-6	34	34-
-5	37	37
-4	32	33
-3	54	56-
-2	24	25-
-1	45	47
0	37	39
1	39	40-
2	31	30-
3	51	51
4	26	27
5	34	34-
6	24	25-
7	37	37
8	15	17
** H 1 9 **		
-8	45	43-
-7	27	26-
-6	41	40
-5	37	35
-4	46	46-
-3	30	30-
-2	57	58
-1	40	40
0	40	39-
1	45	44-
2	54	54
3	32	31
4	46	46-
5	35	35-
6	35	35
7	27	28
** H 1 10 **		
-6	24	22
-5	27	25-
-4	30	28-
-3	42	41
-2	27	26
-1	36	35-
0	36	35-
1	31	30
2	25	25
3	45	42-
4	23	22-
5	25	25
** H 1 11 **		
-3	31	28
-2	45	42-
-1	29	27-
0	31	29
1	29	27
** H 2 0 **		
0X	41	52-
1X	143	145-
2	21	15-
3	212	208
4	38	36-
5	104	108-
6	44	43
7	118	119
8	6	5-
9	80	78-
10	29	27
11	52	51
12	14	12-
13	58	57-
** H 2 1 **		
-13	6	5-
-12	50	51-
-11	18	18
-10	40	41
-9	19	19-
-8	95	96-
-7	11	10
-6	89	89
-5	54	54-
-4	93	91-
-3	6	5
-2	157	142
-1	25	28-
0X	43	52-
1	85	79
2	154	144
3	32	27-
4	114	107-
5	70	65
6	84	59
7	27	25-
8	85	82-
9	18	16
10	47	42
11	32	26-
12	55	48-
13	8	8
** H 2 2 **		
-13	54	54-
-12	10	8
-11	56	55
-10	29	27-
-9	80	76-
-8	21	21
-7	124	120
-6	36	33-
-5	91	86-
-4	56	51
-3	161	140
-2	23	19-
-1	165	146-
0	88	77
1	146	136
2	63	54-
3	189	174-
4	26	23
5	89	82
6	36	34-
7	107	98-
8	5	6
9	93	82
10	22	19-
11	60	54-
12	14	13
** H 2 3 **		
-13	10	10
-12	47	48
-11	21	21-
-10	43	44-
-9	27	27
-8	78	79
-7	21	20-
-6	72	72-
-5	32	31
-4	75	74
-3	25	25-
-2	130	132-
-1	29	29
0	101	103
1	42	42-
2	122	122-
3	11	9
4	89	91
5	19	18-
6	65	65-
7	18	18
8	81	79
9	16	15-
10	48	46-
11	19	20
12	43	42
** H 2 4 **		
-12	12	12-
-11	53	55-
-10	20	19
-9	71	72
-8	10	9-
-7	97	97-
-6	22	17
-5	91	89
-4	19	18-
-3	147	144-
-2	27	26
-1	138	136
0	39	37-
1	116	117-
2	20	19
3	122	120
4	25	25-
5	80	79-
6	30	30
7	88	89
8	21	20-
9	68	67-
10	23	21
11	45	44
12	10	12-
** H 2 5 **		
-12	43	44-
-11	18	18
-10	39	39
-9	12	12-
-8	65	66-
-7	16	16
-6	58	58
-5	38	38-
-4	78	77-
-3	27	27
-2	102	99
-1	49	47-
0	56	54-
1	35	32
2	86	83
3	22	22-
4	70	68-
5	37	37
6	58	59
7	19	19-
8	66	65-
9	15	15
10	35	35
11	14	13-
** H 2 6 **		
-11	40	41
-10	20	21-
-9	54	55-
-8	8	9
-7	80	81
-6	32	33-
-5	69	69-
-4	30	30
-3	112	111
-2	16	14-
-1	89	88-
0	33	32
1	86	83
2	9	7-
3	114	111-
4	20	20
5	70	71
6	21	21-
7	73	74-
8	9	5
9	55	55
10	13	13-
** H 2 7 **		
-10	29	29-
-9	15	15
-8	60	60
-7	10	9-
-6	57	57-
-5	30	30
-4	57	56
-3	5	4-
-2	82	80-
-1	18	13
0	50	48
1	26	24-
2	79	75-
3	10	9
4	64	63
5	26	26-
6	41	39-
7	14	13
8	49	48
9	11	10-
** H 2 8 **		
-9	46	45
-8	9	8-
-7	72	69-
-6	15	14
-5	52	51
-4	23	21-
-3	71	71-
-2	7	6
-1	70	71
0	30	30-
1	60	60-
2	24	22
3	78	76
4	15	16-
5	47	47-
6	22	21
7	49	47
8	7	8-
** H 2 9 **		
-8	43	40-
-7	8	9
-6	43	40
-5	22	20-
-4	35	34-
-3	19	16
-2	58	56
-1	17	16-
0	44	43-
1	26	25
2	51	48
3	11	9-
4	46	45-
5	12	12
6	33	32
** H 2 10 **		
-6	9	8-
-5	45	40-
-4	18	14
-3	57	53
-2	9	9-
-1	60	56-
0	14	12
1	49	46
2	13	9-
3	55	51-
4	7	6
** H 2 11 **		
-1	10	10
** H 3 0 **		
0	123	128-
1	59	57-
2	80	79
3	41	40
4	90	92-
5	15	16-
6	86	87
7	28	28
8	53	53-
9	21	21-
10	69	67
11	17	17
12	41	40-
** H 3 1 **		
-12	26	27-
-11	60	62
-10	29	30
-9	63	64-
-8	35	36-
-7	74	74-
-6	44	44
-5	121	121-
-4	49	48-
-3	95	95
-2	90	85
-1	165	156-
0	63	62-
1	144	140
2	41	39
3	108	106-
4	38	36-
5	137	132
6	38	36
7	80	77-
8	40	38-
9	83	74
10	26	25
11	89	82-
12	21	19-
** H 3 2 **		
-12	39	38
-11	20	20
-10	67	66-
-9	11	11-
-8	48	47
-7	18	18
-6	102	101-
-5	27	25-
-4	102	100
-3	26	26
-2	94	90-
-1	34	33-
0	162	152
1	5	4
2	72	70-
3	21	19-
4	104	100
5	37	34
6	92	87-
7	30	28-
8	50	45
9	24	21
10	71	63-
11	18	15-
12	38	33
** H 3 3 **		
-12	20	19
-11	65	63-
-10	23	23-
-9	71	68
-8	24	23
-7	70	69-
-6	50	49-
-5	134	131
-4	57	55
-3	79	76-
-2	52	50-
-1	128	124
0	59	57
1	125	122-
2	52	50-
3	70	67
4	62	62
5	118	116-
6	50	49-
7	64	64
8	30	28
9	60	58-
10	24	23-
11	59	57
12	20	18
** H 3 4 **		
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-10	63	64
-9	20	19
-8	39	40-
-7	27	26-
-6	74	75
-5	40	40
-4	89	87-
-3	24	22-
-2	48	47
-1	21	20
0	118	118-
1	36	35-
2	69	68
3	27	26
4	77	76-
5	25	25-
6	85	87
7	15	13
8	40	40-
9	10	9-
10	57	55
11	18	15
** H 3 5 **		
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-10	25	25
-9	63	64-
-8	32	32-
-7	52	53
-6	32	32
-5	107	105-
-4	34	33-
-3	83	78
-2	28	25
-1	96	95-
0	42	41-
1	122	121
2	46	45
3	71	71-
4	28	29-
5	93	94
6	29	29
7	59	62-
8	22	23-
9	44	46
10	21	22
** H 3 6 **		
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-10	54	54-
-9	18	17-
-8	46	45
-7	15	15
-6	60	60-
-5	12	11-
-4	82	79
-3	20	20
-2	53	54-
-1	25	24-
0	87	85
1	34	33
2	63	62-
3	22	22-
4	52	52
5	16	16
6	58	60-
7	21	21-
8	35	34
9	16	15
10	41	40-
** H 3 7 **		
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-9	53	51
-8	25	24
-7	47	46-
-6	26	25-
-5	74	71
-4	35	32
-3	61	61-
-2	43	43-
-1	67	68
0	37	36
1	77	76-
2	31	32-
3	54	53
4	24	24
5	66	65-
6	25	24-
7	50	49
8	24	22
9	40	40-
** H 3 8 **		
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-8	34	32-
-7	15	15-
-6	49	47
-5	17	16
-4	57	54-
-3	21	19-
-2	46	45
-1	17	17
0	65	64-
1	10	9-
2	47	47
3	12	12
4	50	47-
5	14	13-
6	49	49
7	12	14
** H 3 9 **		
-7	41	38
-6	23	21
-5	63	59-
-4	24	21-
-3	49	47
-2	21	21
-1	56	56-
0	20	20-
1	59	58
2	19	21
3	42	40-
4	25	24-
5	50	49
6	21	20
** H 3 10 **		
-5	18	16-
-4	45	41
-3	11	12
-2	31	28-
-1	14	13-
0	47	44
1	17	15
2	31	31-
3	18	15-
** H 4 0 **		
0	118	121-
1	42	43
2	95	96
3	74	74-
4	93	94-
5	9	8
6	83	85
7	50	51-
8	66	64-
9	41	39
10	62	60
11	20	18-
12	40	39-
** H 4 1 **		
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-11	41	41
-10	16	16-
-9	47	48-
-8	49	49
-7	49	49
-6	26	27-
-5	71	73-
-4	38	38
-3	64	65
-2	100	101-
-1	89	90-
0	35	36
1	84	83
2	75	74-
3	69	68-
4	58	57
5	81	79
6	36	34-
7	55	55-
8	69	63
9	55	50
10	26	23-
11	43	39-
12	26	24
** H 4 2 **		
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-11	22	22-
-10	60	60-
-9	28	27
-8	59	58
-7	55	54-
-6	86	86-
-5	36	35
-4	101	100
-3	85	84-
-2	102	100-
-1	72	71
0	124	123
1	32	31-
2	93	90-
3	74	71
4	101	98
5	35	34-
6	85	82-
7	47	43
8	62	57
9	40	35-
10	62	55-
11	15	13
** H 4 3 **		
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-10	21	20
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-7	49	47-
-6	47	46
-5	79	78
-4	48	47-
-3	62	59-
-2	64	62
-1	75	73
0	23	23-
1	76	72-
2	49	46
3	57	55
4	53	51-
5	73	70-
6	33	31
7	45	43
8	42	40-
9	45	42-
10	20	19
11	38	37
** H 4 4 **		
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-9	27	27-
-8	53	53-
-7	42	42
-6	72	72
-5	28	26-
-4	84	85-
-3	46	46
-2	73	71
-1	33	32-
0	99	100-
1	41	41
2	80	80
3	64	64-
4	79	81-
5	32	34
6	70	72
7	37	38-
8	49	49-
9	30	29
10	50	51
** H 4 5 **		
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-10	18	18-
-9	43	43-
-8	45	45
-7	40	41
-6	32	31-
-5	61	62-
-4	38	40
-3	52	54
-2	56	56-
-1	64	63-
0	38	39
1	72	73
2	66	67-
3	50	51-
4	40	42
5	56	58
6	26	25-
7	38	41-
8	35	36
9	35	37
10	16	18-
** H 4 6 **		
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-3	51	51-
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2	61	65-
3	45	45
4	56	58
5	12	12-
6	54	57-
7	27	28
8	39	41
9	24	25-
** H 4 7 **		
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-7	35	34-
-6	20	20
-5	48	46
-4	22	22-
-3	43	43-
-2	49	50
-1	46	47
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1	48	48-
2	34	36
3	38	37
4	30	30-
5	44	43-
6	17	19
7	34	34
8	34	34-
** H 4 8 **		
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-7	31	30
-6	47	45
-5	19	16-
-4	55	53-
-3	38	38
-2	51	50
-1	35	35-
0	59	58-
1	19	19
2	48	50
3	41	36-
4	50	49-
5	23	22
6	45	45
** H 4 9 **		
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-4	24	22
-3	35	34
-2	38	36-
-1	38	37-
0	19	19
1	39	38
2	26	24-
3	33	31-
4	28	27
5	34	33
** H 4 10 **		
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0	43	40
1	14	14-
** H 5 0 **		
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1	66	68
2	3	1
3	94	95-
4	5	5-
5	58	60
6	6	2-
7	75	75-
8	5	4-
9	51	51
10	0	3-
11	39	38-
** H 5 1 **		
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-9	12	13-
-8	75	76
-7	14	14
-6	73	74-
-5	11	12-
-4	81	83
-3	23	23
-2	108	108-
-1	5	3-
0	81	81
1	22	22
2	124	123-
3	29	28-
4	99	98
5	9	9
6	79	75-
7	16	15-
8	84	78
9	10	9
10	47	44-
11	9	5-
** H 5 2 **		
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-10	6	4
-9	53	54
-8	14	13
-7	78	77-
-6	9	8
-5	60	60
-4	0	2
-3	97	96-
-2	6	4-
-1	88	86
0	12	11-
1	89	87-
2	21	20-
3	104	103
4	8	5-
5	54	53-
6	13	7
7	68	64
8	8	8
9	53	49-
10	0	3
11	39	35
** H 5 3 **		
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-10	46	46
-9	15	14
-8	77	76-
-7	18	18-
-6	69	68
-5	8	4-
-4	76	75-
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-2	108	105
-1	16	13
0	76	75-
1	17	13-
2	100	97
3	28	26
4	74	73-
5	3	4
6	60	56
7	18	15
8	74	68-
9	18	12-
10	44	42
** H 5 4 **		
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-9	45	44-
-8	10	10-
-7	81	80
-6	10	8-
-5	45	45-
-4	7	7
-3	82	81
-2	20	18
-1	74	74-
0	16	15
1	64	64
2	5	3
3	78	80-
4	6	1-
5	49	51
6	8	8-
7	61	62-
8	12	12-
9	50	50
10	5	2-
** H 5 5 **		
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-8	59	61
-7	16	16
-6	61	62-
-5	7	2-
-4	70	72
-3	21	21
-2	90	97-
-1	18	19-
0	65	69
1	1	1-
2	79	83-
3	21	23-
4	69	72
5	9	9
6	54	57-
7	10	10-
8	58	59
9	9	11
** H 5 6 **		
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-7	59	59-
-6	5	4-
-5	49	49
-4	0	2-
-3	67	69-
-2	8	8-
-1	55	58
0	5	8-
1	45	48-
2	9	1
3	62	64
4	7	8
5	41	42-
6	5	4
7	44	46
8	8	3
** H 5 7 **		
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-6	52	51
-5	11	8
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-3	9	9-
-2	59	64
-1	10	8
0	46	47-
1	5	6-
2	61	63
3	16	14
4	55	56-
5	11	8-
6	42	43
7	9	4
** H 5 8 **		
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-2	6	1
-1	48	49-
0	7	2
1	45	46
2	4	4
3	55	55-
4	8	2-
5	35	36
** H 5 9 **		
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-1	8	8-
0	41	43
1	12	7
2	47	47-
** H 6 0 **		
0	71	74
1	61	63
2	38	39-
3	49	49-
4	59	59
5	55	55
6	58	58-
7	44	44-
8	26	25
9	36	37
10	40	40-
** H 6 1 **		
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-8	24	26
-7	37	38-
-6	34	34-
-5	73	73
-4	42	43
-3	38	38-
-2	40	40-
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1	72	71-
2	47	46-
3	46	45
4	40	38
5	71	69-
6	47	45-
7	36	35
8	32	31
9	35	33-
10	30	28-
** H 6 2 **		
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-9	37	37
-8	32	31-
-7	45	45-
-6	54	53
-5	60	58
-4	54	54-
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1	69	68-
2	49	49
3	57	54
4	52	49-
5	52	50-
6	54	49
7	52	48
8	28	26-
9	34	31-
** H 6 3 **		
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-8	33	33-
-7	33	32
-6	37	36
-5	60	58-
-4	41	40-
-3	40	39
-2	39	40
-1	62	61-
0	34	33-
1	69	67
2	41	39
3	37	36-
4	32	31-
5	58	55
6	36	34
7	36	35-
8	29	28-
9	36	34
** H 6 4 **		
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-7	43	43
-6	43	41-
-5	44	42-
-4	51	50
-3	48	48
-2	42	41-
-1	50	50-
0	66	67
1	51	50
2	36	37-
3	54	55-
4	42	42
5	45	46
6	46	47-
7	42	42-
8	26	27
** H 6 5 **		
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-5	53	55
-4	30	31
-3	39	41-
-2	37	40-
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1	47	50-
2	37	39-
3	29	30
4	35	37
5	49	51-
6	28	29-
7	30	32
8	22	24
** H 6 6 **		
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-3	38	40-
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0	45	49-
1	40	43-
2	30	31
3	35	37
4	35	37-
5	33	35-
6	38	40
** H 6 7 **		
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-4	25	26-
-3	26	28
-2	24	24
-1	33	35-
0	27	30-
1	43	45
2	23	25
3	27	29-
4	23	23-
5	38	40
** H 6 8 **		
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-3	33	34
-2	20	23-
-1	33	34-
0	38	41
1	37	38
2	29	31-
3	30	31-
** H 7 0 **		
0	72	76
1	17	17-
2	51	53-
3	30	30
4	54	55
5	9	7-
6	50	50-
7	18	17
8	39	40
** H 7 1 **		
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-5	64	65
-4	10	8-
-3	64	64-
-2	15	15
-1	74	74
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1	75	75-
2	15	15
3	57	55
4	0	2-
5	68	66-
6	0	3-
7	54	51
8	11	11-
** H 7 2 **		
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-7	16	17
-6	49	48
-5	4	2-
-4	65	65-
-3	24	25
-2	57	57
-1	10	8-
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1	10	11
2	53	52
3	27	26-
4	55	53-
5	3	2
6	58	54
7	20	19-
8	41	38-
** H 7 3 **		
-8	15	14
-7	47	47
-6	0	2
-5	65	63-
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-3	62	61
-2	20	19-
-1	60	59-
0	7	6-
1	63	62
2	19	18-
3	60	57-
4	10	8
5	62	60
6	2	1
7	47	46-
** H 7 4 **		
-7	21	21-
-6	48	47-
-5	0	3
-4	51	50
-3	27	27-
-2	46	47-
-1	18	16
0	54	55
1	9	7-
2	51	52-
3	25	25
4	51	54
5	0	4-
6	43	44-
7	14	12
** H 7 5 **		
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-5	51	53
-4	4	2
-3	46	48-
-2	13	14
-1	55	59
0	3	8
1	56	59-
2	10	6
3	49	52
4	10	7-
5	48	49-
6	0	3-
** H 7 6 **		
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-3	15	15
-2	38	41
-1	12	13-
0	47	52-
1	9	7
2	37	39
3	20	21-
4	37	39-
** H 7 7 **		
-2	13	12-
-1	44	47-
0	1	2
1	43	46
2	11	9-
** H 8 0 **		
0	15	16
1	49	49-
2	33	33-
3	54	54
4	29	29
5	35	35-
6	20	20-
** H 8 1 **		
-6	37	37
-5	11	11
-4	44	46-
-3	25	23-
-2	57	58
-1	17	16
0	41	42-
1	18	15-
2	55	54
3	32	31
4	41	41-
5	16	14-
6	39	38
** H 8 2 **		
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-5	33	32-
-4	24	23-
-3	53	53
-2	35	34
-1	42	42-
0	26	26-
1	43	41
2	35	34
3	52	50-
4	31	30-
5	34	34
6	15	16
** H 8 3 **		
-6	37	36-
-5	12	10-
-4	42	42
-3	20	19
-2	56	55-
-1	22	19-
0	36	37
1	18	16
2	55	54-
3	20	20-
4	44	44
5	14	10
** H 8 4 **		
-5	33	32
-4	23	23
-3	48	48-
-2	31	30-
-1	40	41
0	25	23
1	38	38-
2	26	26-
3	47	49
4	20	23
** H 8 5 **		
-3	22	23-
-2	41	45
-1	17	16
0	30	33-
1	11	14-
2	39	42
** H 9 0 **		
0	32	33-
1	39	38-
2	17	16
3	39	39
** H 9 1 **		
-2	41	41
-1	18	18-
0	40	41-
1	21	20
2	43	42
** H 9 2 **		
-1	38	38-
0	30	30
1	37	38

[a] The column headings have the following meanings: H, running index; OBS, observed structure amplitude; CAL, calculated structure factor followed by sign. Values marked with X were omitted from the refinement.

The X-ray powder diffraction pattern of $BkCl_3 \cdot 6H_2O$ was obtained with a 114.6-mm diameter Norelco camera and Ni-filtered, Cu Kα X-rays at 23°. The diffraction rings were somewhat spotty in nature because the particle size of the sample was not controlled. This limited our ability to estimate intensities visually in this case, but the sample was not thick enough to cause difficulties in measuring line position because of absorption. The film was measured on a Norelco film reader with a traveling cross hair and steel vernier scale; a correction for film shrinkage was judged to be negligible by measurement of the distance between fiducial dots previously calibrated for the camera.

Crystals of the trichloride heptahydrates were photographed by X-ray precession techniques using Polaroid films; this has been shown by Swink and Carpenter[12] to be a reliable method of obtaining unit-cell dimensions.

Calculations.—Absorption corrections for $AmCl_3 \cdot 6H_2O$ were

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[12] L. N. Swink and G. B. Carpenter, *Acta Crystallogr.*, **22**, 602 (1967).

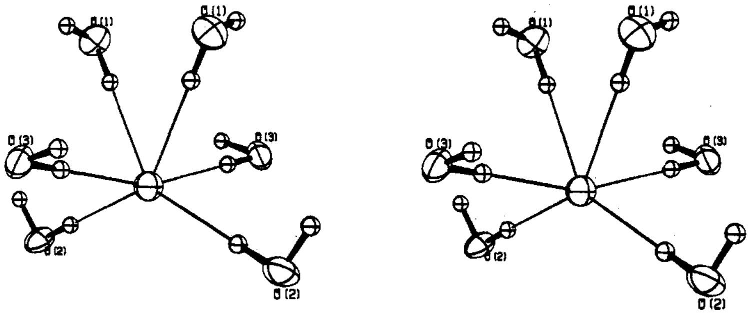

Figure 1.—Stereoscopic drawing of the octahedron of water molecules around the Cl(1) ion.

calculated with the ORABS program.[13] An estimate of the mass absorption coefficient of Am of 60 cm^2 g^{-1} was obtained by extrapolation of the measured values of U and Pu.[14] From this, a linear absorption coefficient for $AmCl_3 \cdot 6H_2O$ of 103.8 cm^{-1} was derived, and the calculated transmission factors ranged from 0.15 to 0.42. Intensities were normalized by interpolation between measurements of the standard reflections (which varied by 1–2% in addition to the general decline noted earlier), corrected for absorption, and reduced to squared structure factors.

Because the unit-cell and space-group data indicated that $AmCl_3 \cdot 6H_2O$ is isotypic with the previously reported $GdCl_3 \cdot 6H_2O$[5] and $EuCl_3 \cdot 6H_2O$ structures,[6] no structure determination was required; parameter refinement by the method of least squares was carried out directly using the parameters of $GdCl_3 \cdot 6H_2O$ as the starting point. For these computations a modification of the full-matrix computer program by Busing, Martin, and Levy[15] was used. The function minimized was $\Sigma w(s^2F_c^2 - F_o^2)^2$, where F_c and F_o are calculated and observed structure factors, s is a scale factor, and $w = 1/\sigma^2(F^2)$. The variance of an observation, $\sigma^2(F^2)$, was estimated from counting statistics, modified, as described previously,[16] to include a contribution of 5% of the intensity for possible systematic errors. Reflections measured to be less than the averaged background were set equal to zero. In the calculation of structure factors scattering factors for O, Cl$^-$, and Am^{3+} were taken from Cromer and Waber,[17] and the scattering of the heavy atom was corrected for the real and imaginary components of anomalous dispersion by use of Cromer's tables.[18] Anisotropic temperature factors were applied to all atoms. After convergence, hydrogen atoms were added to the model at appropriate distances along interatomic vectors and further refinement was attempted. No significant improvement in agreement occurred, and some of the hydrogen atom coordinates failed to converge to stable values. Difference Fourier maps also failed to reveal the hydrogen sites, so these atoms were omitted from the final calculations. Some 22 low-angle reflections were omitted in the final cycles of refinement because they were judged to be in error due to rapidly changing backgrounds in this region of measurement.

For the 1468 reflections used to refine the 46 parameters and one scale factor, the agreement indices were $R_F = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.039$, $R_{F^2} = \Sigma|F_o^2 - F_c^2|/\Sigma F_o^2 = 0.076$, and $R_{wF^2} = \Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^4 = 0.096$, and the standard deviation of an observation of unit weight was 1.67.

The theoretical powder pattern of $BkCl_3 \cdot 6H_2O$ was calculated from the structural parameters of $AmCl_3 \cdot 6H_2O$ using a program by Smith.[19] Refinement, by least-squares methods, of the unit-cell dimensions of both substances was carried out on the PDP-8 computer used to control the diffractometer.[20] Reference 20 also describes the techniques and programs for crystal centering, scanning of reflections, measuring backgrounds, and preliminary data processing.

Results and Discussion

$AmCl_3 \cdot 6H_2O$.—The monoclinic unit cell of this substance has the following dimensions: $a = 9.702$ (1) Å, $b = 6.567$ (1) Å, $c = 8.009$ (1) Å, and $\beta = 93°\ 37$ (1)$'$, with least-squares standard errors in parentheses. Atomic coordinates and thermal parameters obtained from the refinement are given in Table I. Structure factors calculated using these parameters along with the observed structure amplitudes are listed in Table II. Interatomic distances and their standard errors are listed in Table III for all the contacts made by each kind of atom with other atoms of the structure. The Am, O(1), O(2), and O(3) are each eight-coordinated, Cl(2) is seven-coordinated, and Cl(1) is six-coordinated.

TABLE III

INTERATOMIC DISTANCES IN $AmCl_3 \cdot 6H_2O$

Multiplicity	Atoms	Distance (σ), Å	Multiplicity	Atoms	Distance (σ), Å
2	Am–O(2)	2.440 (6)	1	Cl(2)–O(1)	3.302 (6)
2	Am–O(1)	2.471 (5)	1	Cl(2)–O(1)	3.377 (7)
2	Am–O(3)	2.474 (5)	1	O(1)–O(2)	2.828 (8)
2	Am–Cl(2)	2.799 (2)	1	O(1)–O(3)	2.944 (9)
2	Cl(1)–O(3)	3.180 (6)	1	O(1)–O(1)	3.35 (1)
2	Cl(1)–O(2)	3.194 (6)	1	O(2)–O(3)	2.804 (8)
2	Cl(1)–O(1)	3.246 (6)	1	O(2)–O(3)	2.983 (8)
1	Cl(2)–O(2)	3.148 (6)	1	O(2)–O(2)	3.22 (1)
1	Cl(2)–O(3)	3.167 (6)	1	O(2)–O(2)	3.22 (1)
1	Cl(2)–O(1)	3.178 (6)	1	O(3)–O(3)	3.34 (1)
1	Cl(2)–O(3)	3.267 (7)			

No qualitative differences were found between the structure of $AmCl_3 \cdot 6H_2O$ and that previously reported for $GdCl_3 \cdot 6H_2O$[5] and $EuCl_3 \cdot 6H_2O$;[6] hence this is another example of analogous behavior of actinide and lanthanide ions having comparable radii. The basic units of the structure are $AmCl_2(OH_2)_6{}^+$ cations and Cl$^-$ anions. The latter are octahedrally coordinated by water molecules, probably through hydrogen bonds, as seen in Figure 1.[21] The complex cation and adjoining Cl ions to which the water molecules are believed to

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[13] D. J. Wehe, W. R. Busing, and H. A. Levy, "ORABS, a Fortran Program for Calculating Single-Crystal Absorption Corrections," Report ORNL-TM-229, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1962.

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[17] D. T. Cromer and J. T. Waber, *ibid.*, **18**, 104 (1965).

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[19] D. K. Smith, "A Fortran Program for Calculating X-Ray Powder Diffraction Patterns," Report UCRL 7196, Lawrence Radiation Laboratory, Livermore, Calif., 1963.

[20] W. R. Busing, R. D. Ellison, H. A. Levy, S. P. King, and R. T. Roseberry, "The Oak Ridge Computer-Controlled X-Ray Diffractometer," ORNL-4143, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1968.

[21] Hydrogen atoms are included in Figures 1 and 2 along the O · · · Cl vectors at 0.96 Å from the O; although this is expected to be their approximate positions, these have not been determined directly.

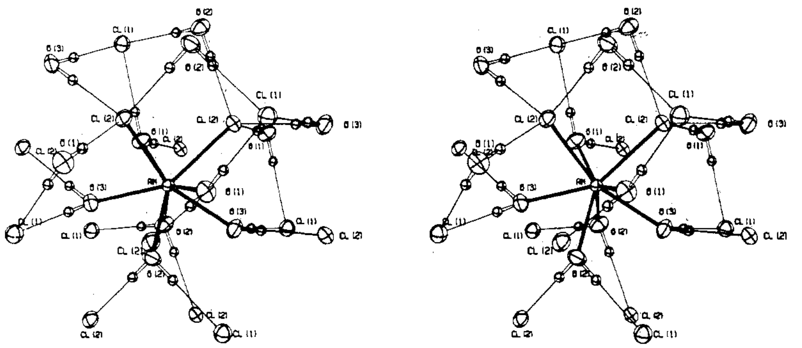

Figure 2.—Stereoscopic view of the $AmCl_2(OH_2)_6^+$ ion and its neighbors.

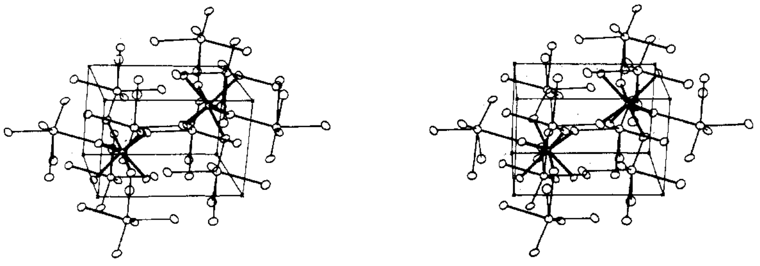

Figure 3. Stereoscopic view of one unit cell of the structure of $AmCl_3 \cdot 6H_2O$ and some of its environs.

be hydrogen bonded are illustrated in Figure 2. The three-dimensional network of ions joined by hydrogen bridges is presented in Figure 3, which shows the contents of one unit cell and some of its environs.

Each Cl(2), which is bonded directly to the Am, has three water oxygen atoms, O(2), O(3), and O(1), at distances of 3.148 (6), 3.167 (6), and 3.178 (6) Å, respectively. It is reasonable to assume that each of these oxygen atoms is hydrogen bonded to Cl(2) and to nearby Cl(1) atoms which are at distances of 3.194 (6), 3.180 (6), and 3.246 (6) Å from O(2), O(3), and O(1), respectively. The Cl(2)–O(n)–Cl(1) angles are 94.2 (2), 98.3 (2), and 89.5 (2)° for $n = 1$, 2, and 3. Since the normal H–O–H angle is about 105°, these Cl···O···Cl angles provide further evidence of hydrogen bonds in this structure. The somewhat long Cl···O distances and the deviations of the Cl···O···Cl angles from 105° suggest that the bonds are not very strong.

Ellipsoids representing the 50% probability contour of thermal motion, drawn by the ORTEP program,[22] are shown for all the atoms in Figures 1 and 2. The Am and Cl(1) atoms move nearly isotropically, as is expected from their rather symmetrical surroundings of atoms bonded equally strongly, while the motions of the O and Cl(2) atoms are anisotropic, having somewhat greater amplitudes perpendicular to their bonds to Am than along them.

$BkCl_3 \cdot 6H_2O$.—From the X-ray powder pattern it was seen that this substance is isostructural with $AmCl_3 \cdot 6H_2O$; hence the pattern could be indexed, and from ten resolved lines the unit-cell dimensions were determined. They are $a = 9.655$ (3) Å, $b = 6.534$ (2)

TABLE IV

POWDER PATTERN OF $BkCl_3 \cdot 6H_2O$

hkℓ	$I_{obs.}$[a]	$d_{obs.}$, Å	$d_{calc.}$, Å	$I_{calc.}$
010	m	6.52	6.54	42
$10\bar{1}$	m	6.34	6.34	100
101	ms	5.93	5.94	96
110	s	5.40	5.41	77
011	m	5.03	5.05	66
200	s	4.81	4.82	54
$11\bar{1}$	m	4.53	4.55	43
111	ms	4.40	4.40	44
002	mw	3.979	3.976	54
[b] 210	m	3.880	3.878	16
[b] $\bar{2}11$	ms	3.559	3.561	63
[b] 211	s	3.414	3.415	60
$20\bar{2}$ }	vw	3.150	3.171 }	12
112			3.149	13
[b] 120	m	3.094	3.095	36
$30\bar{1}$ }	vw	3.035	3.049 }	8
021			3.023	5
202	vw	2.965	2.972	12
[b] 310	m	2.882	2.883	21
$31\bar{1}$	vw	2.764	2.763	9
311	w	2.655	2.661	10
[b] $22\bar{1}$	m	2.589	2.590	23
[b] 221	m	2.530	2.532	23
$12\bar{2}$	w	2.467	2.468	22
122 }			2.418 }	18
$11\bar{3}$ }	ms	2.410	2.416 }	17
400			2.409	13
320	s	2.285	2.291	36
$22\bar{2}$ }	w	2.270	2.276 }	1
312			2.271	20
$41\bar{1}$	vw	2.222	2.210	14
030	vw	2.193	2.179	7
411 }	vw	2.144	2.140 }	7
213			2.136	18
$13\bar{1}$	mw	2.061	2.061	16
004 }			1.988 }	8
230 }	mw	1.977	1.985 }	4
303			1.982	4
[b] 322	ms	1.945	1.946	17
[b] 032	ms	1.910	1.911	13
[b] $41\bar{3}$	m	1.775	1.775	5

[a] Visually estimated intensities: s = strong, ms = medium strong, m = medium, mw = medium weak, w = weak, vw = very weak.

[b] Values used in least-squares refinement of unit cell.

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(22) C. K. Johnson, "ORTEP, a Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations," Report ORNL-3794R, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

Å, c = 7.967 (5) Å, and β = 93° 47 (1)′, with least-squares standard errors in parentheses. An approximate correction for the ^{249}Cf content of the sample was made by assuming a linear change in all lattice dimensions with ionic radius, proceeding from Am to Cf; the slightly larger, corrected dimensions of the $BkCl_3 \cdot 6H_2O$ cell are a = 9.658 Å, b = 6.536 Å, c = 7.969 Å, and β = 93° 46′. The indexed powder pattern, calculated from this cell, is listed along with the observed lines in Table IV. That there is only rough qualitative agreement between observed and calculated intensities is probably due to the preferred orientation and limited number of crystals in the powder sample.

Other Hydrates.—Our values for the dimensions of the reduced[23] triclinic unit cells of the trichloride heptahydrates of La, Ce, and Pr are listed in Table V along with the reduced[23] cells derived from the cells of Iveronova, *et al.*,[7] for comparison. The reduced cells should be in agreement, but are not. We believe that there is an error in the published values of Iveronova, *et al.*, because their cell volumes are too small when compared with the expected values obtained by extrapolation of the hexahydrate cell volumes plus the volume of one additional H_2O per molecule.

TABLE V

TRICLINIC UNIT-CELL DIMENSIONS[a] OF LANTHANIDE TRICHLORIDE HEPTAHYDRATES

Compound	a, Å	b, Å	c, Å	α, deg	β, deg	γ, deg	V, Å^3
$LaCl_3 \cdot 7H_2O$	10.44	8.18	8.03	91.8	104.1	122.1	552.6
$LaCl_3 \cdot 7H_2O$	(10.1	8.1	10.2	98.8	117.9	121.4	534)[b]
$CeCl_3 \cdot 7H_2O$	10.43	8.20	7.98	91.9	104.0	122.1	549.6
$PrCl_3 \cdot 7H_2O$	10.29	8.22	7.90	91.9	105.1	121.3	540.3
$PrCl_3 \cdot 7H_2O$	(10.1	8.2	10.1	98.9	117.2	122.3	536)[b]

[a] Our values have an estimated error of ±0.5%. [b] Values in parentheses are derived from Iveronova, *et al.*[7]

Since the trichlorides of these three largest lanthanide ions form heptahydrates and the trichlorides of Nd–Lu form hexahydrates, it is of interest to compare the actinide trichloride compounds with corresponding radii. With respect to size of the trivalent ion, $AmCl_3 \cdot 6H_2O$ and $BkCl_3 \cdot 6H_2O$ are bracketed by the known $NdCl_3 \cdot 6H_2O$ and $EuCl_3 \cdot 6H_2O$. Using only ionic size as a criterion, one would expect that if uranium and neptunium trichlorides form stable hydrates they should contain seven water molecules, whereas all the trichlorides of actinides heavier than Pu should form hexahydrates. Nevertheless, tribromide hexahydrates with the structure discussed here are known for U, Np, and Pu, but the slightly larger Br ions may be effective in preventing a seventh water molecule from entering the complex cation.

Acknowledgments.—We thank R. D. Baybarz for the separation and purification of the Bk and C. E. Higgins for growing the crystals of $AmCl_3 \cdot 6H_2O$.

[23] The reduction was carried out by the method of Delaunay as described in the "International Tables for X-Ray Crystallography," Vol. I, Kynoch Press, Birmingham, England, 1952, p 530.

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Crystal Structure of Bis(triethylammonium) Octadecahydroicosaborate. The Structure of Octadecahydroicosaborate(2−)

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Received December 29, 1969

The centrosymmetric $B_{20}H_{18}^{2-}$ ion, octadecahydroicosaborate(2−), consists of two B_{10} units, very similar to that in $B_{10}H_{10}^{2-}$, linked by B···B interaction among one pair of adjacent apical and equatorial B atoms from each B_{10} unit. These interactions are interpreted as involving two localized three-center BBB bonds among these four B atoms. Location of all H atoms indicates that no hydrogen bridges are present. The triethylammonium ion has approximately C_3 symmetry. Twofold positional disorder exists in all ions in the monoclinic crystal, which has symmetry $P2_1/a$, and two formula weights per unit cell having parameters a = 17.52 (2), b = 7.60 (1), c = 11.72 (1) Å, and β = 109.7 (2)°. The value of $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ is 0.10 for the 1874 X-ray diffraction maxima.

The polyhedral $B_{10}H_{10}^{2-}$ ion, first prepared and recognized by Lipscomb, Pitochelli, and Hawthorne,[1] was proposed[2] to dimerize by formation of two hydrogen bridges to form the $B_{20}H_{18}^{2-}$. Subsequently, $B_{20}H_{18}^{2-}$ was prepared,[3] but the nuclear magnetic resonance study by Pilling, Hawthorne, and Pier[4] indicated linkage of the B_{10} units by two three-center BBB bonds rather than through bridge hydrogens. The boron framework was confirmed in Babcock's X-ray study[5] of $[(CH_3)_3S]_2B_{20}H_{18}$, but hydrogen atoms were not located and the B–B distances have not been published. Thus, the possibility of additional bonding through unsymmetri-

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