Hydrothermal Investigation of the Systems Y_2O_3-H_2O-Na_2O, Y_2O_3-D_2O-Na_2O, Y_2O_3-H_2O, and Y_2O_3-H_2O-NH_3. The Crystal Structure of $Y(OH)_3$

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Hydrothermal methods have been used in the preparation of a hexagonal modification of yttrium trihydroxide, $Y(OH)_3$, and yttrium trideuteroxide, $Y(OD)_3$. The structure was investigated by three-dimensional single-crystal X-ray analysis. The deuterium position was obtained from neutron diffraction powder patterns. The space group is $P6_3/m$, (No. 176) with $a = 6.241$ Å, $c = 3.539$ Å. There are two formula units in the cell. Yttrium trihydroxide is isostructural with the rare earth trihydroxides. Atomic coordinates, temperature factors and other relevant crystallographic data are reported.

Three new yttrium hydroxide phases have been prepared by hydrothermal methods. The compounds are characterised by chemical analysis, X-ray powder patterns, differential thermal analysis, and infra-red spectra.

Earlier X-ray studies [1,2] have shown yttrium trihydroxide, $Y(OH)_3$, to have the uranium trichloride structure.[3] The same structure is characteristic of the rare earth trihydroxides. Atoji and Williams [4] showed by a neutron diffraction investigation that $La(OD)_3$ has the uranium trichloride structure reported by Zachariasen.[3] Structure Reports [5] gives a list of twenty-two compounds including uranium trichloride and lanthanum trihydroxide, all isostructural with yttrium trihydroxide. In a hydrothermal investigation of the formation ranges of yttrium trihydroxide, yttrium oxide hydroxide, and yttrium oxide, it was observed by Shafer and Roy [6] that the powder pattern of the yttrium hydroxide obtained by them was different from the powder pattern of lanthanum trihydroxide, and it was claimed by Roy and McKinstry,[7] that yttrium trihydroxide did not have the uranium trichloride structure.

Since the structure of yttrium trihydroxide was not settled, it was decided to reinvestigate the structure by a three-dimensional single-crystal X-ray analysis.

EXPERIMENTAL

Chemistry. Hydrothermal syntheses were performed in pressure bombs lined with pure silver or pure gold. The balanced pressure technique was used. The experimental conditions used for the systems $Y_2O_3-H_2O-Na_2O$ and $Y_2O_3-D_2O-Na_2O$ are given in Table 1. Freshly precipitated $Y(OD)_3$ was treated with a NaOD solution in 99.7 % D_2O. The NaOD solution was prepared by dissolving sodium in D_2O. All manipulations with compounds containing deuterium were performed in a glove box under dry oxygen-free nitrogen.

Table 1. Experimental conditions for hydrothermal preparation of $Y(OH)_3$ and $Y(OD)_3$. Freshly precipitated yttrium hydroxide is prepared from yttrium nitrate solution and 1 M sodium hydroxide.

Expt. No.	Max. temp. °C	Pressure atm.	Time h	Base conc. M	Initial condition	Result
1	300	85	60	15 NaOH	$Y(OH)_3$	$Y(OH)_3$
2	250	35	25	0.1 NaOD	$Y(OD)_3$	$Y(OD)_3$

Yttrium was determined in $Y(OH_3)$ and in $Y(OD)_3$ by EDTA titration.[8] (Found: Y 63.6. Calc. for $Y(OH)_3$: Y 63.5. Found: Y 64.9. Calc. for $Y(OD)_3$: Y 62.2).

The new phases could be prepared when yttrium hydroxide was precipitated from yttrium nitrate solutions with 4 M ammonia solutions. The precipitate was washed with water by filtration until the filtrate had pH = 7, and was then heated with water or solutions of ammonia in pressure bombs lined with pure silver or pure gold. The experimental conditions for the systems $Y_2O_3-H_2O$ and $Y_2O_3-H_2O-NH_3$ are given in Table 2.

Yttrium was determined in the new phases by EDTA titration.[8] (Found: Y 57.3 (Phase I); Y 58.5 (Phase II); Y 61.8 (Phase III). Calc. for $Y(OH)_3$: Y 63.5).

The sodium content in $Y(OH)_3$ and in III was determined by using a flame photometer which was calibrated with solutions of sodium chloride. (Found: Na 0.11 ± 0.04 in $Y(OH)_3$; Na 0.02 ± 0.04 in III).

X-Ray technique, hexagonal Y(OH)₃. The powder pattern of $Y(OH)_3$ was obtained with a Guinier camera using $CuK\alpha_1$ radiation, $\lambda = 1.54051$ Å, and sodium chloride as standard, $a_{NaCl} = 5.6389$ Å. The unit cell parameters were determined from the powder pattern, which was similar to the powder pattern of holmium trihydroxide.[9]

A needle shaped single crystal of $Y(OH)_3$ with dimensions 1 mm × 0.01 mm × 0.01 mm was investigated by Weissenberg methods. Integrated Weissenberg photographs were taken of $(h\bar{k}0)$, $(hk1)$, and $(hk2)$, using the multiple film technique, with Ni-filtered Cu-radiation. 55 independent reflections were measured photometrically. No absorption correction was applied.

Another single crystal of $Y(OH)_3$ with dimensions 0.2 mm × 0.04 mm × 0.04 mm was investigated by Weissenberg methods. The needle axis proved to be [001]. A total of 119 independent reflections were collected with an automatic diffractometer using Mo radiation and balanced filter techniques with a scintillation counter. Each reflection is symmetry related to five others. Each layer line was measured twice so that each reflection used in the structure refinement is an average of 12 measurements. No absorption correction was applied. The two sets of data were treated separately.

Table 2. Experimental conditions for hydrothermal preparation of crystalline phases.

Expt. No.	Max.temp. °C	Pressure atm.	Time h	NH_3 conc. M	Initial condition	Result
1	250	40	72	4	$Y(OH)_3$	$Y(OH)_3$ (hexagonal)
2	250	40	60	1	»	I
3	230	28	54	1	»	II
4	260	55	54	0	»	III
5	250	40	50	1	»	II
6	250	40	72	1	»	I, II and III
7	215	500	52	1	»	II
8	230	28	55	1	»	II
9	235	30	55	1	»	II
10	250	40	60	0	»	III
11	270	55	50	0	»	III and II
12	260	50	72	0	»	III and II
13	260	55	52	0	»	III and I
14	260	50	77	0	»	III and II
15	260	55	52	0	»	III and I

X-Ray technique, new phases. The powder patterns of the new phases were obtained with a Guinier-de Wolff camera using $CuK\alpha_1$ radiation, $\lambda = 1.54051$ Å. Sodium chloride was used as internal standard, $a_{NaCl} = 5.6389$ Å. Phases I and III gave sharp powder patterns, whereas phase II gave broad lines in the powder pattern. The pattern of phase I is given in Table 3, and the powder patterns of phases II and III are given in Table 4. A single crystal of phase I was investigated by Weissenberg and precession methods.
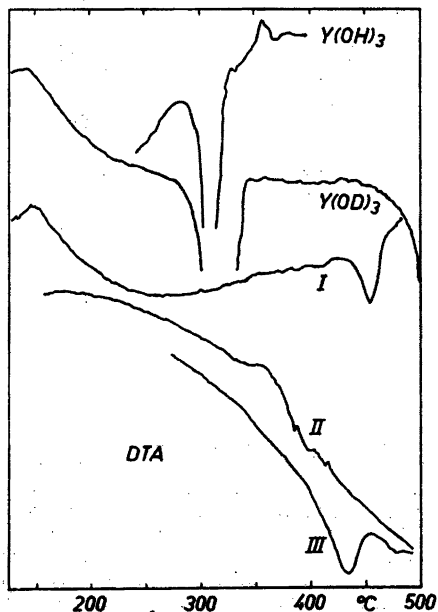

Fig. 1. DTA diagrams of $Y(OH)_3$, $Y(OD)_3$, and the phases I, II, and III.

Table 3. X-Ray powder pattern of phase I.

$a = 6.25$ Å, $b = 6.01$ Å, $c = 15.40$ Å, $\beta = 97.5°$.

d_{obs}	d_{calc}	$h\,k\,l$	I_o
6.213	6.197	1 0 0	s
4.330	4.314	1 1 0	m
3.821	3.817	0 0 4	vw
3.457	3.458	$\bar{1}$ 0 4	w
3.230	3.222	0 1 4	m
3.019	3.011	$\bar{2}$ 0 2	m
3.004	3.005	0 2 0	m
2.805	2.796	0 2 2	w
2.763	2.754	2 1 0	m
2.738	2.738	1 1 4	m
2.716	2.722	0 1 5	w
2.580	2.576	$\bar{2}$ 0 4	w
2.373	2.367	$\bar{2}$ 1 4	w
2.271	2.268	$\bar{1}$ 2 4	m
2.163	2.163	$\bar{2}$ 2 1	w
2.155	2.157	2 2 0	w
2.123	2.120	2 1 4	vw
2.068	2.066	3 0 0	vw
1.959	1.956	$\bar{2}$ 2 4	m
1.926	1.925	$\bar{3}$ 0 4	w
	1.924	2 2 3	
1.909	1.906	1 3 0	w
1.893	1.895	$\bar{1}$ 0 8	w
1.812	1.809	$\bar{1}$ 3 3	w
	1.809	2 2 4	
1.778	1.774	0 3 4	m
1.738	1.733	$\bar{1}$ 3 4	w
1.728	1.729	$\bar{1}$ 0 8	vw
1.689	1.691	2 2 5	w
1.685	1.685	$\bar{2}$ 3 1	vw
1.661	1.659	2 3 1	vw
1.624	1.621	$\bar{3}$ 2 4	w
1.612	1.611	0 2 8	w
1.604	1.603	$\bar{1}$ 2 8	w
1.585	1.589	1 3 5	vw
	1.581	2 3 4	
1.520	1.519	1 2 8	vw
	1.520	$\bar{2}$ 3 5	

s strong; m medium; w weak; vw very weak.

Weissenberg photographs were taken of ($hk0$) and ($hk4$), and precession photographs were taken of ($h0l$), ($h1l$), ($0kl$), and ($1kl$). From the film data the space group $P2_1/c$ (No. 14) and the unit cell parameters $a = 6.25$ Å, $b = 6.01$ Å, $c = 15.40$ Å, $\beta = 97.5°$ were obtained. The powder pattern of phase I was indexed using this unit cell (Table 3).

Neutron diffraction technique, $Y(OD)_3$. Neutron diffractograms of $Y(OD)_3$ were obtained by a neutron diffractometer at the Swedish Research Councils' Laboratory, Studsvik, using neutrons with a wavelength of 1.07 Å. The sample was contained in an aluminium tube of 20 mm diameter. The intensities were obtained from the recorded

Table 4. X-Ray powder patterns of phases II and III.

Phase II		Phase III		Powder pattern from Ref. 7	
d_{obs}	I_o	d_{obs}	I_o	d_{obs}	I/I_o
8.65	w	8.25	m	8.4	10
5.837	s	8.03	m	8.12	100
5.697	s	5.752	w	5.82	5
4.238	m	4.684	w	4.69	20
4.136	w	4.577	w	4.57	15
				4.41	10
3.408	w	4.094	m	4.09	70
3.025	w	4.007	m		
				3.677	65
2.975	vs	3.470	w		
2.878	m	3.256	m		
2.840	s	3.117	w		
2.459	m	3.083	w		
2.353	w	3.060	s	3.07	70
2.277	s	3.005	m		
2.158	w	2.970	s	3.00	45
2.115	m	2.909	s		
2.096	m	2.851	w	2.857	5
2.068	w	2.729	w	2.730	20
1.926	s	2.669	w	2.660	20
1.891	s	2.458	w		
1.840	w	2.406	w		
1.753	w	2.344	w		
1.744	m	2.330	m	2.344	15
1.736	m	2.256	w		
1.678	w	2.161	m	2.217	3
1.662	w	2.123	w	2.117	3
1.608	w	2.058	w	2.04	5
1.509	w	2.046	vw		
1.498	w	2.034	vw		
1.484	w	2.024	vw		
		1.999	vw		
		1.976	vw		
		1.952	m	1.997	20
		1.885	w		
		1.870	w	1.866	15
		1.809	s	1.824	7
		1.765	s	1.781	5
				1.758	5
		1.731	w		
		1.710	m		
		1.676	w	1.672	5
		1.654	vw		
		1.647	vw		
		1.635	vw	1.634	7
		1.602	vw	1.602	10
		1.558	m	1.561	10
		1.550	w	1.528	10
		1.507	w		
		1.497	vw		
		1.430	vw		
		1.412	vw		
		1.341	vw		

diffractograms by measuring the areas under the peaks. Seven consecutive powder patterns were recorded, and the intensities used in the structure calculation were obtained as an average of the seven measurements. Intensities of 18 peaks are given in Table 5. Due to overlapping only eight of the peaks corresponded to single reflections. Of these eight peaks the (111) reflection is overlapped by the (111) reflection from the aluminium container. The (200) reflection from aluminium overlaps the combined (210), (120) reflection. Six of the seven peaks were used in the structure factor calculations. The (221) reflection was excluded because the height of the peak above the background was less than half of the background intensity.

Physical measurements. Differential thermal analysis of $Y(OH)_3$, $Y(OD)_3$, and the phases I, II, and III were obtained on a Du Pont 900 Differential Thermal Analyzer.

Table 5. Observed and calculated intensities of the neutron diffraction powder pattern of $Y(OD)_3$. $R = \sum |I_o - kI_c|/\sum I_o = 5.7 \%$, (the reflections (111) and (210) are excluded).

$2\theta_c{}^\circ$	$h\,k\,l$	I_o	I_c	
22.8	2 0 0	51	53	
26.4	1 1 1	113 *	74	
28.8	2 0 1	356	348	
30.4	2 1 0 ⎱	59 *	39 ⎱	45
	1 2 0 ⎰		6 ⎰	
34.6	3 0 0	10	9	
35.2	0 0 2 ⎫		93 ⎫	
35.2	2 1 1 ⎬	107	8 ⎬	102
	1 2 1 ⎭		1 ⎭	
38.9	3 0 1	21	21	
40.1	2 2 0 ⎱	23	31 ⎱	32
40.7	1 1 2 ⎰		1 ⎰	
42.4	2 0 2	26	29	
44.0	2 2 1	5	1	
45.6	1 3 1 ⎱	6	5 ⎱	5
	3 1 1 ⎰		0 ⎰	
46.7	4 0 0 ⎫		28 ⎫	
47.2	2 1 2 ⎬	65	32 ⎬	65
	1 2 2 ⎭		5 ⎭	
50.2	3 0 2 ⎧		9 ⎫	
50.2	4 0 1 ⎪	35	1 ⎪	32
51.1	2 3 0 ⎨		1 ⎬	
	3 2 0 ⎩		21 ⎭	
53.9	1 4 0 ⎧		2 ⎫	
	4 1 0 ⎪		8 ⎪	
54.1	2 3 1 ⎬	99	53 ⎬	97
	3 2 1 ⎪		4 ⎪	
54.4	2 2 2 ⎭		32 ⎭	
55.3	1 0 3 ⎱		0 ⎫	
55.8	1 3 2 ⎬	11	0 ⎬	6
	3 1 2 ⎭		6 ⎭	
57.1	1 4 1 ⎱	9	4 ⎱	8
	4 1 1 ⎰		4 ⎰	
58.0	1 1 3	14	14	
59.3	2 0 3 ⎫		75 ⎫	
59.3	5 0 0 ⎬	113	15 ⎬	123
59.8	4 0 2 ⎭		33 ⎭	

* Not corrected for the contributions from the (111) and (200) reflections from the aluminium container.

A heating rate of 10°C/min was used. Fig. 1 shows the diagrams obtained. The infra-red spectra were obtained over the frequency range 400 to 4000 cm⁻¹ with a Perkin-Elmer Model 521 spectrophotometer. The pellet technique was used with mixtures of 4 mg of sample and 200 mg of CsI. The infra-red spectra of I, II, and III are shown in Fig. 2.

CRYSTAL DATA AND STRUCTURE REFINEMENT

The crystal data obtained demonstrated that $Y(OH)_3$ is hexagonal with space group $P6_3/m$ (No. 176). The unit cell contains two formula units. The axes are $a = 6.241 \pm 0.005$ Å, $c = 3.539 \pm 0.005$ Å. Density 3.88 g/cm³. The absorption coefficient μ is 346 cm⁻¹ for $CuK\alpha$ radiation, and 249 cm⁻¹ for $MoK\alpha$ radiation. In $Y(OD)_3$ μ is 0.015 cm⁻¹ for neutrons, $(\lambda = 1.08$ Å).
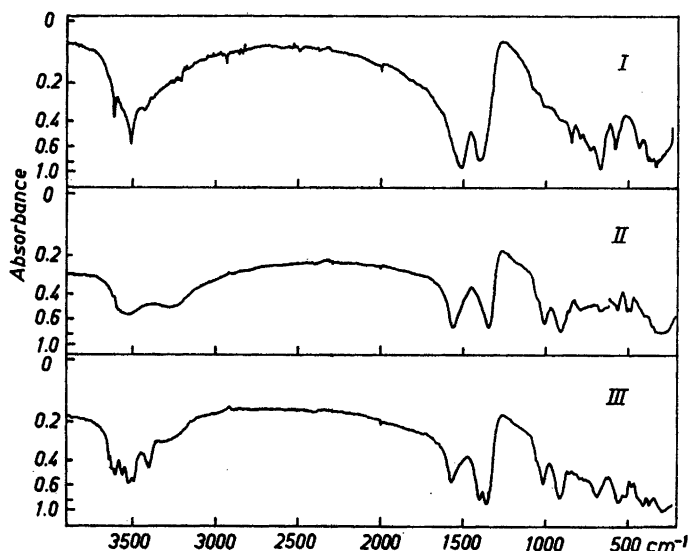

Fig. 2. Infra-red spectra of the phases I, II, and III.

Yttrium trihydroxide is isostructural with uranium trichloride.[3] Using the data obtained with $CuK\alpha$ radiation an R-value of 17.8 % was obtained on inserting the Y atoms only in the structure factor calculation, using the coordinates $(\frac{1}{3}, \frac{2}{3}, \frac{1}{4})$. An R-value of 9.9 % was obtained on inserting Y and O atoms in the structure factor calculation using the coordinates for chlorine obtained from uranium trichloride. Coordinates and temperature factors were refined by the method of Bhuiya and Stanley.[10] The structure factors were calculated using the atomic scattering factor for oxygen from Vol. III of the *International Tables of X-ray Crystallography*. Cromer's [11] values were used for yttrium. The interpolation formula of Bassi [12] was employed. The program used was written by Danielsen.[13] The refinement yielded an R-value of 8.1 %.

Table 6. Atomic coordinates and temperature factors.

Weissenberg data, 55 reflections, $R = 8.1$ %, Crystal 1.

Atom	x	σx	y	σy	z	B (Å²)	σB (Å²)
Y	0.3333		0.6667		0.25	0.24	0.07
O	0.398	0.004	0.314	0.004	0.25	1.1	0.5

Diffractometer data, 119 reflections, isotropic refinement, $R = 2.3$ %, Crystal 2.

Atom	x	σx	y	σy	z	B (Å²)	σB (Å²)
Y	0.3333		0.6667		0.25	0.64	0.01
O	0.3957	0.0007	0.3112	0.0007	0.25	0.75	0.06

Diffractometer data, 119 reflections, anisotropic refinement, $R = 2.2$ %, Crystal 2.

Atom	x	σx	y	σy	z
Y	0.3333		0.6667		0.25
O	0.3957	0.0008	0.3115	0.0007	0.25

	b_{11}	σb_{11}	b_{22}	σb_{22}	b_{33}	σb_{33}	b_{12}	σb_{12}
Y	0.0057	0.0002	0.0057	0.0002	0.0123	0.0006	0.0057	0.0002
O	0.0072	0.0012	0.0063	0.0012	0.0167	0.0029	0.0079	0.0022

Neutron diffractometer data, 6 reflections, $R = 2.0$ %, Powder.

Atom	x	σx	y	σy	z	B (Å²)
Y	0.3333		0.6667		0.25	0.64
O	0.3957		0.3112		0.25	0.75
D	0.279	0.004	0.142	0.004	0.25	0.9

The diffractometer data were treated separately. An R-value of 6.3 % was obtained on inserting Y and O atoms in the structure factor calculation using the coordinates obtained from the Weissenberg data. The isotropic refinement gave an R-value of 2.3 %. A difference Fourier-synthesis gave the possible hydrogen coordinates (0.27, 0.12, 0.25), corresponding to an O—H distance of 1 Å. Coordinates and anisotropic temperature factor parameters for the Y and O atoms were refined by the method of least-squares using

Table 7. Observed and calculated structure factors ($\times$ 10) for yttrium trihydroxide. Diffractometer data; $R = 2.1$ %.

h	k	l	Fo	Fc
0	1	0	793	755
0	2	0	635	619
0	3	0	1211	1220
0	4	0	581	598
0	5	0	437	443
0	6	0	537	545
0	7	0	265	269
1	1	0	944	963
1	2	0	97	115
1	3	0	409	414
1	4	0	760	769
1	5	0	131	134
1	6	0	319	316
1	7	0	432	436
2	1	0	771	757
2	2	0	818	836
2	3	0	544	547
2	4	0	274	286
2	5	0	605	601
3	1	0	595	590
3	2	0	239	242
3	3	0	631	652
3	4	0	373	384
3	5	0	295	306
4	1	0	883	896
4	2	0	205	199
4	3	0	355	363
4	4	0	602	637
5	1	0	408	395
5	2	0	504	499
5	3	0	236	271
6	1	0	218	234
6	3	0	376	357
7	1	0	583	586
0	1	1	845	862
0	2	1	1202	1214
0	4	1	453	461
0	5	1	618	618
0	7	1	351	359

h	k	l	Fo	Fc
1	1	1	482	434
1	2	1	864	858
1	3	1	701	699
1	5	1	553	552
1	6	1	559	563
2	1	1	1062	1065
2	3	1	911	915
2	4	1	605	597
3	1	1	554	561
3	2	1	651	654
3	4	1	398	389
3	5	1	352	333
4	2	1	597	600
4	3	1	520	506
4	5	1	338	328
5	1	1	681	672
5	2	1	161	136
5	3	1	472	472
6	1	1	434	425
6	2	1	449	413
0	1	2	539	531
0	2	2	466	462
0	3	2	952	955
0	4	2	490	482
0	5	2	373	376
0	6	2	488	481
0	7	2	252	239
1	1	2	781	779
1	2	2	171	162
1	3	2	348	351
1	4	2	661	661
1	5	2	156	128
1	6	2	268	278
1	7	2	371	387
2	1	2	573	560
2	2	2	701	701
2	3	2	453	454
2	4	2	272	253
2	5	2	544	529
3	1	2	483	473

h	k	l	Fo	Fc
3	2	2	233	226
3	3	2	565	570
3	4	2	343	334
4	1	2	748	757
4	2	2	189	183
4	3	2	308	316
5	1	2	353	341
5	2	2	450	443
5	3	2	242	240
6	1	2	214	208
0	1	3	580	589
0	2	3	743	738
0	4	3	370	375
0	5	3	484	473
1	1	3	188	184
1	2	3	597	598
1	3	3	515	517
1	5	3	407	428
1	6	3	429	440
2	1	3	698	698
2	3	3	656	655
2	4	3	465	458
3	1	3	442	440
3	2	3	494	493
3	4	3	272	310
4	2	3	463	459
5	1	3	536	513
6	1	3	365	335
0	1	4	286	318
0	2	4	282	287
0	3	4	609	603
1	1	4	521	516
1	2	4	156	147
1	3	4	233	238
1	4	4	438	452
2	1	4	336	337
2	2	4	475	471
3	1	4	292	303
4	0	4	288	310
4	4	4	428	401

a full-matrix least-squares program written by Gantzel, Sparks, Long and Trueblood.[14] The IBM 7090 computer at N.E.U.C.C. was used. The anisotropic refinement gave an R-value of 2.2 %. Table 6 gives atomic coordinates and temperature factor coefficients with their standard deviations. No anisotropic vibration of the atoms was observed.

In the structure factor calculation of the neutron data only the x and y parameters of the deuterium atom were varied. The deuterium atom was inserted at (0.27, 0.12, 0.25), and the temperature factor was chosen as 0.9. The coordinates and temperature factors used for the yttrium atom and for the oxygen atom are the values from the isotropic refinement of the diffractometer data (Table 6). Only the structure factors of the (200), (201), (300),

(301), (202), and (113) reflections were used in the refinement of the deuterium atom coordinates. The atomic scattering amplitudes for yttrium,[15] oxygen, and deuterium [16] were 0.788, 0.577, and 0.65 (cm $\times$ 10^{-12}), respectively. The method of refinement and the program employed were as those used for the X-ray data.[13] The deuterium coordinates after the refinement were (0.279, 0.145, 0.25), (Table 6). The coordinates from the neutron diffractometer data of Table 6 were used in the calculation of the intensities of the peaks. Table 5 gives observed and calculated intensities. The agreement is as good as can be expected from neutron powder data.

Table 8. Interatomic distances l in Å and bond angles v with standard deviations.

| | Diffractometer data | |
	l	σl
Y_1-O_1	2.437	0.003
Y_1-O_2	2.403	0.003
$O_1-O_{1'}$	3.539	0.005
O_1-O_2	2.865	0.006
O_1-O_3	2.704	0.004
O_2-O_3	2.817	0.004
	Neutron data	
D_1-O_1	0.94	0.02
D_1-D_2	2.33	0.03

Angles about the oxygen atom (degrees)

	v	σv
$Y_1-O_1-D_1$	129.6	1.9
$Y_1-O_1-Y_2$	112.1	0.1
$Y_2-O_1-Y_{2'}$	94.8	0.2
$Y_2-O_1-D_1$	101.2	1.2

Structure factors calculated with the hydrogen atom included with coordinates (0.279, 0.145, 0.25) and $B = 0.9$ gave an R-value of 2.1 % with the diffractometer data. The observed and calculated structure factors for these data are given in Table 7. Table 8 gives interatomic distances and bond angles with their standard deviations.

DISCUSSION

The present hydrothermal investigation shows that new compounds different from the previously known compounds $Y(OH)_3$, $YOOH$, and Y_2O_3 can be prepared at temperatures and pressures within the reported formation range of yttrium trihydroxide.[6] The yttrium trihydroxide prepared by Shafer and Roy,[6] and described by Roy and McKinstry [7] is probably identical with III from the present investigation, (see Table 4). Compound III has probably a composition which deviates from $Y(OH)_3$. I, II, and III can be prepared when sodium ions are excluded in the precipitation of the yttrium ions, and in
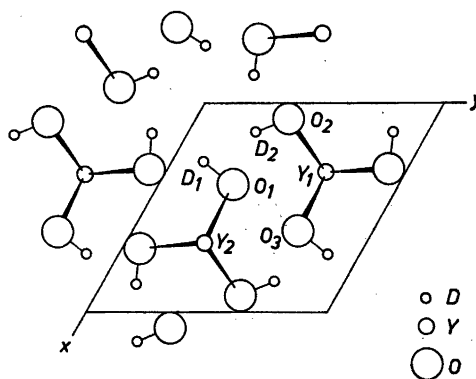
Fig. 3. Projection of the yttrium trihy-
droxide structure on (001).

the hydrothermal solution. If sodium ions are present, $Y(OH)_3$ can be formed; the yttrium trihydroxide so prepared has a measurable content of sodium.

The structure of $Y(OH)_3$ corresponds to the structure of UCl_3,[3] and $La(OD)_3$.[4] An O—O distance of 3.72 Å reported in Ref. 1 is calculated as 3.53 Å. The agreement between distances calculated from the diffractometer data and from the Weissenberg data is good but some of the distances deviate more than three standard deviations from those reported by Schubert and Seitz.[1] With the deuterium atom coordinates (0.279, 0.142, 0.25) an O—D distance of 0.94 Å with a standard deviation of 0.03 Å and a D—D distance of 2.33 Å with a standard deviation of 0.04 Å are obtained. A short O—D distance is characteristic of structures without hydrogen bonds. Thus the O—H distance in $Ca(OH)_2$, a non-hydrogen bonded structure, is 0.936 $\pm$ 0.003 Å.[17] The D—D distance is approximately twice the van der Waals radius of a hydrogen atom (1.2 Å).[18] Fig. 3 is a projection of the structure on (001). The yttrium atom is coordinated with nine oxygen atoms. There are two significantly different Y—O distances, one set with a distance of 2.437 Å and standard deviation of 0.003 Å, and one set with a distance of 2.403 Å and standard deviation of 0.003 Å. There are three sets of short O—O distances within the coordination poly-hedron, with distances 2.865 Å, 2.704 Å, and 2.817 Å, and standard deviations 0.006 Å, 0.004 Å, and 0.004 Å, respectively. These short distances cannot be interpreted in terms of hydrogen bonds and the structure is not hydrogen bonded. The deuterium (hydrogen) atoms form a helix in the structure around the origin in the [001] direction. The infra-red spectrum also con-firms that the OH-group does not participate in hydrogen-bonding.

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