diazonium fluoroborate in ~100 ml of DMF was added dropwise. Gas evolution proceeded rapidly and stopped at 2.1 l. Another portion of $C_6H_5N_2BF_4$ dissolved in DMF was added dropwise until the total volume of gas evolved was 2.5 l. The reaction mixture was poured into 800 ml of water and $MgSO_4$ was added to facilitate filtration of the mixture. The red solid was purified by chromatography on alumina eluting with methylene chloride. The orange band was collected and stripped of solvent. The residue was triturated with hexane and filtered, yielding 30.2 g (64.3%) of solid. Recrystallization from toluene gave red crystals, mp 225–226° dec. Properties of the product and of other MN═NAr compounds are listed in Table III.

Contribution from the Lawrence Radiation Laboratory and Department of Chemistry,
University of California, Berkeley, California 94720
and the Savannah River Laboratory, E. I. du Pont de Nemours and Company, Aiken, South Carolina 29801

The Crystal and Molecular Structure of the Heptacoordinate Complex Tris(diphenylpropanedionato)aquoholmium, $Ho(C_6H_5COCHCOC_6H_5)_3 \cdot H_2O$[1]

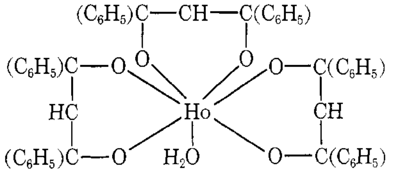
By Allan Zalkin, David H. Templeton, and David G. Karraker

Received January 27, 1969

$Ho(C_6H_5COCHCOC_6H_5)_3 \cdot H_2O$, tris(diphenylpropanedionato)aquoholmium, crystallizes in space group R3 with one molecule per rhombohedral cell. The dimensions of the triply primitive hexagonal cell are $a = 22.713$ (12) and $c = 6.334$ (4) Å. The measured density is 1.48 g/cm³; the calculated X-ray density is 1.50 g/cm³. The structure was determined from measurements with Cu Kα radiation of 1365 reflections which included 120 Friedel pairs. It was refined to $R = 0.031$ with anisotropic temperature factors on all but the hydrogen atoms. The holmium atom, on the threefold axis, has a sevenfold coordination. The six oxygen atoms of the diphenylpropanedionato ligands are at distances 2.275 (4) and 2.305 (7) Å from holmium at the corners of a substantially distorted octahedron. The water molecule, also on the threefold axis, is 2.39 (2) Å from holmium and caps the above octahedron; its hydrogen atoms are presumed to be in disordered positions. The molecule as a whole has the appearance of a three-bladed propeller, each blade of which consists of a planar six-membered ring consisting of a holmium, two oxygen, and three carbon atoms; the planes of the two phenyl groups are twisted by 10 and 19° to the above plane.

Introduction

Holmium in the trivalent state, like other rare earths, reacts with dibenzoylmethane[2] to form a complex with the structure



The water molecule is known to be firmly bound, and Hoard[3] in 1961 suggested this heptacoordinate structure for some similar complexes. The structure determination was undertaken in order to ascertain the coordination geometry about the holmium atom and to investigate the nature of the hydration of the water molecule in this material. Recently the structures of tris(1-phenyl-1,3-butanedionato)aquoyttrium,[4] ytterbium acetylacetonate monohydrate,[5] and tris (acetylacetonato)aquoytterbium–hemibenzene[6] were reported which also have sevenfold coordination.

Experimental Section

$Ho(C_6H_5COCHCOC_6H_5)_3 \cdot H_2O$ was prepared by adding an aqueous solution of holmium trichloride to a refluxing mixture of dibenzoylmethane, acetone, and potassium hydroxide. Yellow crystals of the compound crystallized from the filtered and cooled solution. This procedure is from preparation no. 17 as described by Melby, *et al.*[2]

A crystal fragment of dimensions 0.15 × 0.15 × 0.10 mm was cleaved from a needle and glued to a Pyrex fiber. A preliminary set of Weissenberg films had shown the crystal to be rhombohedral with the fiber parallel to the hexagonal c axis. Cell dimensions were measured using a GE XRD-5 X-ray diffraction apparatus equipped with a quarter-circle Eulerian cradle and a molybdenum anode tube ($\lambda_{K\alpha_1}$ 0.70926 Å, $\lambda_{K\alpha_2}$ 0.71354 Å, and $\lambda_{K\beta}$ 0.632253 Å). Cell dimensions were determined from a series of 2θ measurements along the hexagonal $00l$ and $h00$ directions using peaks where Kβ, Kα_1, and Kα_2 X-rays were resolved. All measurements were made at room temperature, *i.e.* ~23°.

The crystal was transferred to a quarter-circle type of automatic GE XRD-5 diffractometer. The intensity measurements were made using a Cu anode X-ray tube set at a takeoff angle of 2° and operated at 35 kV and 20 mA. The diffracted beam was filtered by an Ni foil just ahead of the scintillation counter which was equipped with a pulse height analyzer. The θ–2θ scanning technique was used; scanning began 0.6° in 2θ lower than the Kα_1 position and continued to 0.6° beyond the Kα_2 position at a rate of 1°/min. Backgrounds were taken at 0.4° ahead of and beyond the scan limits. The net intensity I was then calculated to be $I = C - (B_1 + B_2)(T_c/2T_b)$. C is the total counts in the scan time T_c. B_1 and B_2 are the two stationary backgrounds which were each counted for $T_b = 10$ sec. The data included 1366 independent reflections. Fourteen intensities, of which two were actually recorded as zero, were less than their estimated standard deviations. One reflection, $(24,-12,3)$, was deleted from the data set due to a recording error by the automatic diffractometer, *i.e.*, a background was

---

(1) Research performed under the auspices of the U. S. Atomic Energy Commission.

(2) L. R. Melby, N. J. Rose, E. Abramson, and J. C. Caris, *J. Am. Chem. Soc.*, **86**, 5117 (1964).

(3) J. L. Hoard, G. S. Smith, and M. Lind, "Advances in the Chemistry of the Coordination Compounds," Proceedings of the Sixth International Conference on Coordination Chemistry, S. Kirschner, Ed., The Macmillan Co., New York, N. Y., 1961, p 296.

(4) F. A. Cotton and P. Legzdins, *Inorg. Chem.*, **7**, 1777 (1968).

(5) J. A. Cunningham, D. E. Sands, W. F. Wagner, and M. F. Richardson, *ibid.*, **8**, 22 (1969).

(6) E. D. Watkins, II, J. A. Cunningham, T. Phillips, II, D. E. Sands, and W. F. Wagner, *ibid.*, **8**, 29 (1969).

erroneously recorded as zero. As the diffractometer was no longer functioning at the time of this discovery, we did not remeasure this datum. The final results are based on 1365 independent reflections. In this data set there are 120 Friedel pairs, *i.e.*, $h,k,0$ and $h + k,\bar{h},0$ sets. The data were corrected for Lorentz and polarization effects. No correction was made for absorption. The absorption parameter is 44 cm^{-1} which we estimate causes the absorption factor to vary by a factor 1.25 between the most extreme cases.

The standard deviations of the intensities and the structure factors were estimated as follows. The standard deviation of I is

$$\sigma(I) = [C + (T_c/2T_b)^2(B_1 + B_2) + (qI)^2]^{1/2}$$

where q (taken here as 0.05) is an arbitrary fraction of the intensity which has been included to account for such nonrandom errors in the data as absorption, extinction, and the lack of stability of the electronic circuits of the instrument. The standard deviation of the structure factor, by the method of finite differences, is taken as $\sigma(F) = F_o - [F_o^2 - s\sigma(I)/Lp]^{1/2}$, where s is the scaling factor in the equation $F_o = (sI/Lp)^{1/2}$. For the reflections where $I \leqq \sigma(I)$, $\sigma(F) = [s\sigma(I)/Lp]^{1/2}$. L and p are the Lorentz and polarization corrections.

Our unpublished full-matrix least-squares program minimizes the function R_2^2, where $R_2^2 = \Sigma w(\Delta F)^2/\Sigma wF_o^2$, F_o and F_c are the observed and calculated structure factors, and ΔF is the difference of their magnitudes. The weighting factor w is $1/\sigma^2(F)$. The program accommodates both the real and imaginary parts of the dispersion correction.

Scattering factors for neutral holmium, carbon, oxygen, and hydrogen atoms were used.[7,8] The real and imaginary dispersion corrections for holmium are −14.02 and 3.52 electrons, respectively.[9] The anisotropic temperature factor has the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$. B_{ij} values (in square ångströms) are reported for the thermal parameters: $B_{ij} = 4\beta_{ij}/a_i^*a_j^*$, where a_i^* is the ith reciprocal cell length.

Results

Unit Cell and Space Group.—The space group is R3 and contains a single formula unit in the rhombohedral unit cell. The cell dimensions of the triply primitive hexagonal cell at 23° are $a_h = 22.713 \pm 0.012$ and $c_h = 6.334 \pm 0.004$ Å; the errors are subjective estimates. The observed density from flotation in a mixture of ethylene dichloride and carbon tetrachloride at 25° is 1.48 $\mathrm{g/cm^3}$, and the calculated X-ray density is 1.501 ± 0.005 $\mathrm{g/cm^3}$. All of the atomic positions mentioned throughout this paper are based on the above triply primitive hexagonal cell.

Determination of the Structure.—The holmium atom is in the special position (hexagonal) $0,0,z$. As the space group is polar, the origin was arbitrarily placed at the Ho atom, *i.e.*, $z = 0.0$. A Fourier summation phased by Ho, all signs positive, was calculated, but because the holmium structure is centric this function shows two images of the structure. Some of the largest peaks were selected and their positions were refined by least squares using isotropic temperature factors. Atoms were rejected if their thermal parameters became excessively large. This procedure resulted in a structure of eight atoms in addition to holmium which refined to $R = 0.25$, where $R = \Sigma|\Delta F|/\Sigma|F_o|$. A second Fourier summation was calculated using structure factors phased on the above results, and the same procedure was continued. A total of 15 atoms were deduced and the structure at this point was refined to an R value of 0.14. Unfortunately the structure did not make chemical sense, and a third Fourier based on these latest results was calculated. We arrived at the final trial structure by shifting some atoms to their inverse positions such that the resulting molecule made chemical sense. This final trial structure refined to an R value of 0.10.

The above calculations were performed with the 120 Friedel pairs averaged together according to the Laue symmetry. A least-squares refinement of the structure with the unaveraged data, but without the imaginary anomalous dispersion term included for holmium, yielded an R value of 0.099; the inclusion of this term reduced R to 0.097. Anisotropic temperature factors on holmium further reduced R to 0.065, and the application of anisotropic temperature factors to the carbon and oxygen atoms lowered R to 0.036.

A difference Fourier was calculated to search for hydrogen atoms. A peak of significant electron density, 0.16–0.34 electron, was found near every location where a hydrogen atom was calculated to be. There were many spurious peaks in the map as well which had electron densities comparable to the hydrogen peaks. An attempt to refine the hydrogens on the carbon atoms was partially successful, but it was finally decided to put each hydrogen atom at a calculated location 1.0 Å from its associated carbon atom. Isotropic temperature factors were assigned that gave each hydrogen atom the comparable average mean-square displacement of the carbon atom to which it was bonded. The hydrogen atoms associated with the water molecule must be disordered, *i.e.*, the water oxygen atom is on a threefold axis, and their locations are not indicated on the Fourier maps.

The concluding refinement of the structure resulted in a final R factor of 0.031 using all 1365 data. The weighted R factor, R_2, was 0.039. The standard deviation of an observation of unit weight was 1.14. The maximum shift of any parameter in the last cycle was less than 2% of its standard deviation. The list of observed and calculated structure factors is shown in Table I, the final positional parameters in Table II, the anisotropic thermal parameters in Table III, and the assumed isotropic thermal parameters for hydrogen in Table IV.

A refinement of the inverse structure resulted in an increase in both R and R_2 to 0.046 and 0.062, respectively. This is confirmation that the absolute configuration of the molecule in this particular crystal is correct. We have not made a correlation of the absolute configuration with respect to morphology or any other physical property.

Description of Structure.—The basic unit in this structure is a unimolecular species that consists of all of the atoms in the chemical formula. A projection

---

(7) D. T. Cromer and J. T. Waber, *Acta Cryst.*, **18**, 104 (1965).

(8) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, **42**, 3178 (1964).

(9) D. T. Cromer, *Acta Cryst.*, **18**, 17 (1965).

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS IN TRIS(DIPHENYLPROPANEDIONATO)AQUOHOLMIUM [FCA(0,0,0) = 3834]

K	FOB	FCA
H,L= 0, 0		
3	317	310
6	133	138
9	342	351
12	300	297
15	148	144
18	108	108
21	29	32
24	30	29
H,L= 0, 1		
2	435	445
5	129	106
8	49	58
11	324	327
14	93	99
17	78	77
20	44	44
23	36	33
H,L= 0, 2		
1	296	289
4	338	326
7	273	278
10	231	226
13	114	118
16	53	56
19	77	79
22	37	39
H,L= 0, 3		
0	449	442
3	222	243
6	243	254
9	222	225
12	179	178
15	59	60
18	70	66
21	47	50
H,L= 0, 4		
2	211	210
5	167	162
8	205	213
11	217	219
14	130	126
17	39	45
20	35	31
H,L= 0, 5		
1	162	167
4	140	151
7	147	137
10	63	65
13	101	102
16	34	35
H,L= 0, 6		
0	75	76
3	86	82
6	99	107
9	70	72
12	49	52
15	43	45
H,L= 0, 7		
2	48	50
5	62	64
8	72	74
H,L= 1, 0		
1	647	686
4	858	863
7	290	294
10	304	301
13	217	218
16	127	126
19	42	42
22	29	29
H,L= 1, 1		
0	457	490
3	726	751
6	102	103
9	369	383
12	145	148
15	103	103
18	59	54
21	50	50
H,L= 1, 2		
2	252	261
5	261	272
8	250	256
11	190	189
14	47	45
17	72	72
20	65	68
23	32	34
H,L= 1, 3		
1	434	428
4	218	223
7	224	226
10	220	220
13	71	71
16	57	53
19	63	66
H,L= 1, 4		
0	241	244
3	143	137
6	132	129
9	198	199
12	132	131
15	85	83
18	38	40
H,L= 1, 5		
2	165	171
5	191	189
8	139	140
11	100	98
14	57	51
17	38	39
H,L= 1, 6		
1	57	57
4	91	93
7	92	93
10	58	53
13	40	40
H,L= 1, 7		
0	74	77
3	38	42
6	60	65
9	36	38
H,L= 2, 0		
-1	675	689
2	513	505
5	565	581
8	339	344
11	342	335
14	142	143
17	118	114
20	24	16
23	33	33
H,L= 2, 1		
1	831	871
4	632	642
7	94	100
10	286	277
13	163	158
16	57	51
19	73	73
22	10	10
H,L= 2, 2		
0	327	323
3	294	288
6	238	236
9	198	203
12	156	156
15	40	42
18	83	82
21	39	42
H,L= 2, 3		
-1	306	298
2	352	349
5	232	241
8	216	222
11	100	100
14	88	89
17	75	69
20	53	49
H,L= 2, 4		
1	250	251
4	130	134
7	182	175
10	96	99
13	107	108
16	55	56
19	37	41
H,L= 2, 5		
0	152	154
3	181	180
6	220	222
9	91	90
12	85	85
15	46	46
H,L= 2, 6		
-1	77	80
2	83	82
5	108	104
8	65	65
11	27	37
14	25	31
H,L= 2, 7		
1	59	65
4	45	52
7	45	45
H,L= 3, 0		
0	278	276
3	368	362
6	397	416
9	380	375
12	178	185
15	87	90
18	56	55
21	28	30
H,L= 3, 1		
-1	362	365
2	792	843
5	473	469
8	461	441
11	129	133
14	80	81
17	64	65
20	32	35
H,L= 3, 2		
-2	322	327
1	371	378
4	462	462
7	162	159
10	178	167
13	115	117
16	117	114
19	35	33
H,L= 3, 3		
0	468	463
3	246	251
6	228	235
9	169	165
12	115	114
15	90	94
18	50	52
H,L= 3, 4		
-1	236	236
2	224	229
5	180	181
8	48	51
11	123	125
14	70	64
17	49	49
H,L= 3, 5		
-2	159	153
1	173	177
4	223	222
7	109	105
10	111	109
13	75	74
16	54	54
H,L= 3, 6		
0	102	106
3	115	123
6	77	77
9	42	43
12	29	37
H,L= 3, 7		
-1	63	59
2	46	45
5	50	53
8	19	26
H,L= 4, 0		
-2	482	465
1	223	228
4	339	353
7	364	371
10	315	320
13	86	90
16	104	98
19	46	44
22	33	33
H,L= 4, 1		
-3	402	413
0	162	158
3	375	380
6	577	582
9	288	285
12	101	102
15	55	51
18	58	61
21	21	20
H,L= 4, 2		
-1	337	329
2	549	533
5	517	513
8	70	80
11	69	71
14	58	100
17	69	68
20	36	37
H,L= 4, 3		
-2	301	292
1	212	215
4	288	289
7	182	194
10	64	67
13	157	158
16	56	57
19	29	30
H,L= 4, 4		
-3	191	179
0	249	248
3	205	198
6	104	108
9	123	121
12	83	80
15	58	58
18	35	36
H,L= 4, 5		
-1	163	159
2	230	227
5	122	124
8	74	80
11	100	99
14	47	52
H,L= 4, 6		
-2	125	125
1	128	124
4	103	104
7	57	59
10	48	45
13	51	50
H,L= 4, 7		
-3	54	57
0	60	56
3	51	51
6	35	36
H,L= 5, 0		
-4	160	173
-1	858	840
2	488	484
5	221	223
8	366	356
11	159	163
14	89	91
17	66	66
20	40	43
H,L= 5, 1		
-2	362	331
1	473	479
4	344	344
7	208	205
10	174	178
13	91	88
16	69	68
19	51	52
H,L= 5, 2		
-3	193	190
0	414	437
3	612	605
6	353	339
9	139	145
12	50	49
15	55	54
18	55	55
H,L= 5, 3		
-4	337	331
-1	340	326
2	179	200
5	238	230
8	150	148
11	120	129
14	79	78
17	59	61
H,L= 5, 4		
-2	192	193
1	167	165
4	176	178
7	79	80
10	121	123
13	53	51
16	54	55
H,L= 5, 5		
-3	121	125
0	180	184
3	173	173
6	97	94
9	56	58
12	71	67
15	46	47
H,L= 5, 6		
-4	115	111
-1	139	137
2	126	125
5	91	96
8	45	41
11	48	46
H,L= 5, 7		
-2	67	70
1	47	46
4	37	38
7	42	41
H,L= 6, 0		
-3	412	389
0	125	135
3	298	307
6	324	324
9	224	223
12	157	156
15	44	45
18	67	68
H,L= 6, 1		
-4	277	281
-1	203	209
2	536	536
5	162	170
8	201	204
11	135	135
14	100	97
17	66	66
20	35	34
H,L= 6, 2		
-5	213	202
-2	398	390
1	457	447
4	348	363
7	259	247
10	132	125
13	46	46
16	55	56
19	20	22
H,L= 6, 3		
-3	312	316
0	326	326
3	185	188
6	156	168
9	180	176
12	75	77
15	69	66
18	37	40
H,L= 6, 4		
-4	123	120
-1	191	186
2	185	185
5	88	91
8	158	150
11	68	67
14	48	48
17	27	28
H,L= 6, 5		
-5	82	84
-2	156	146
1	193	199
4	116	110
7	65	68
10	78	79
13	58	57
H,L= 6, 6		
-3	137	131
0	104	103
3	111	112
6	58	54
9	43	43
H,L= 6, 7		
-4	86	93
-1	65	68
2	32	35
5	47	47
H,L= 7, 0		
-5	487	475
-2	566	569
1	267	275
4	191	191
7	272	266
10	230	235
13	64	57
16	84	80
19	24	31
H,L= 7, 1		
-6	316	313
-3	351	352
0	339	351
3	305	300
6	167	177
9	132	135
12	96	98
15	90	89
18	48	47
H,L= 7, 2		
-4	214	216
-1	489	468
2	115	117
5	267	259
8	164	168
11	137	135
14	63	63
17	28	28
H,L= 7, 3		
-5	314	311
-2	359	353
1	238	241
4	203	194
7	142	147
10	151	148
13	37	31
16	32	29
H,L= 7, 4		
-6	188	194
-3	139	131
0	174	172
3	92	91
6	121	120
9	120	130
12	41	41
15	29	28
H,L= 7, 5		
-4	119	125
-1	158	153
2	120	116
5	118	116
8	86	86
11	69	68
14	24	22
H,L= 7, 6		
-5	99	104
-2	118	119
1	83	77
4	75	78
7	54	55
10	44	44
H,L= 7, 7		
-6	88	85
-3	76	76
0	41	45
3	39	42
H,L= 8, 0		
-7	273	274
-4	346	348
-1	283	282
2	245	249
5	223	225
8	184	190
11	113	104
14	84	87
17	53	53
H,L= 8, 1		
-5	173	173
-2	312	304
1	350	352
4	229	235
7	169	169
10	89	90
13	115	115
16	39	47
19	30	31
H,L= 8, 2		
-6	192	207
-3	520	497
0	135	141
3	228	242
6	155	149
9	109	108
12	119	121
15	86	82
18	16	16
H,L= 8, 3		
-7	276	270
-4	302	295
-1	347	333
2	210	216
5	105	115
8	234	229
11	45	43
14	34	35
17	24	25
H,L= 8, 4		
-5	162	159
-2	145	139
1	130	126
4	116	120
7	96	90
10	90	89
11	35	30
H,L= 8, 5		
-6	114	109
-3	157	153
0	79	80
3	154	159
6	94	94
9	70	69
12	43	45
H,L= 8, 6		
-7	90	93
-4	119	115
-1	31	34
2	86	92
5	79	75
8	53	54
H,L= 8, 7		
-5	86	85
-2	52	48
1	37	40
4	43	46
H,L= 9, 0		
-6	306	309
-3	432	436
0	356	359
3	252	264
6	189	193
9	127	123
12	75	74
15	74	73
18	23	24
H,L= 9, 1		
-7	150	149
-4	216	208
-1	359	355
2	158	156
5	125	125
8	118	119
11	106	110
14	89	85
17	25	28
H,L= 9, 2		
-8	168	162
-5	262	265
-2	405	398
1	140	136
4	165	161
7	107	111
10	137	135
13	84	88
16	38	42
H,L= 9, 3		
-6	254	248
-3	308	301
0	169	171
3	128	123
6	184	192
9	101	102
12	59	58
15	35	31
H,L= 9, 4		
-7	196	189
-4	145	142
-1	139	138
2	129	123
5	80	76
8	94	98
11	62	56
14	27	25
H,L= 9, 5		
-8	101	101
-5	168	162
-2	61	59
1	162	158
4	109	109
7	59	58
10	49	52
H,L= 9, 6		
-6	114	112
-3	58	58
0	75	77
3	78	77
6	61	63
9	44	42
H,L= 9, 7		
-7	70	69
-4	48	47
-1	40	39
2	43	46
H,L= 10, 0		
-8	234	237
-5	224	222
-2	347	344
1	286	275
4	126	129
7	114	113
10	68	65
13	68	70
16	43	41
H,L= 10, 1		
-9	237	231
-6	187	193
-3	337	324
0	221	225
3	194	185
6	145	142
9	70	71
12	82	88
15	47	49
H,L= 10, 2		
-7	245	246
-4	336	328
-1	174	177
2	163	166
5	124	123
8	148	146
11	89	88
14	54	56
H,L= 10, 3		
-8	232	243
-5	269	259
-2	197	192
1	166	166
4	81	84
7	191	185
10	94	95
13	38	41
H,L= 10, 4		
-9	239	226
-6	122	119
-3	133	130
0	133	132
3	85	90
6	35	28
9	68	70
12	43	41
H,L= 10, 5		
-7	101	105
-4	120	124
-1	112	110
2	112	111
5	68	69
8	48	50
11	46	47
H,L= 10, 6		
-8	83	83
-5	73	68
-2	89	87
1	57	58
4	74	72
7	49	53
H,L= 10, 7		
-9	50	55
-6	45	48
-3	39	37
0	42	41
H,L= 11, 0		
-10	269	259
-7	176	176
-4	359	359
-1	323	313
2	145	143
5	145	147
8	69	74
11	81	73
14	39	44
H,L= 11, 1		
-8	224	217
-5	340	340
-2	222	226
1	166	162
4	171	169
7	93	104
10	70	72
13	68	64
16	23	28
H,L= 11, 2		
-9	326	315
-6	236	238
-3	159	164
0	221	217
3	177	175
6	137	129
9	128	130
12	41	41
15	42	40
H,L= 11, 3		
-10	199	202
-7	240	241
-4	111	116
-1	177	170
2	133	131
5	118	118
8	118	117
11	54	54
14	39	39
H,L= 11, 4		
-8	78	70
-5	118	111
-2	140	136
1	122	117
4	54	53
7	84	83
10	39	38
13	16	17
H,L= 11, 5		
-9	111	109
-6	100	95
-3	90	90
0	122	127
3	66	66
6	55	54
9	39	38
H,L= 11, 6		
-10	63	65
-7	79	78
-4	98	93
-1	62	58
2	62	65
5	46	45
H,L= 11, 7		
-8	36	42
-5	48	47
-2	37	40
H,L= 12, 0		
-9	252	262
-6	305	301
-3	375	364
0	312	304
3	130	131
6	102	109
9	74	79
12	52	57
15	26	25
H,L= 12, 1		
-10	109	116
-7	159	162
-4	355	352
-1	201	198
2	155	162
5	61	61
8	108	103
11	55	59
14	37	38
H,L= 12, 2		
-11	219	218
-8	277	285
-5	140	142
-2	190	184
1	185	188
4	88	78
7	146	150
10	38	38
13	37	36
H,L= 12, 3		
-9	272	265
-6	141	140
-3	139	137
0	177	176
3	144	142
6	118	121
9	62	60
12	51	50
H,L= 12, 4		
-10	171	165
-7	139	134
-4	103	99
-1	114	106
2	105	106
5	67	66
8	42	46
11	24	29
H,L= 12, 5		
-11	123	128
-8	84	81
-5	88	90
-2	92	92
1	84	80
4	57	53
7	22	25
H,L= 12, 6		
-9	82	83
-6	55	59
-3	72	69
0	74	74
3	49	48
H,L= 12, 7		
-7	39	42
-4	31	37
H,L= 13, 0		
-11	126	126
-8	217	218
-5	377	358
-2	329	322
1	226	225
4	115	111
7	107	108
10	67	63
13	36	32
H,L= 13, 1		
-12	226	226
-9	82	89
-6	263	265
-3	147	153
0	150	147
3	69	68
6	68	71
9	61	61
12	51	53
H,L= 13, 2		
-10	238	242
-7	183	179
-4	246	235
-1	167	169
2	89	87
5	191	182
8	58	54
11	34	31
14	30	32
H,L= 13, 3		
-11	243	237
-8	160	153
-5	180	179
-2	125	126
1	165	163
4	88	90
7	93	89
10	34	38
H,L= 13, 4		
-12	104	105
-9	52	51
-6	110	114
-3	90	87
0	121	121
3	101	96
6	42	38
9	44	41
H,L= 13, 5		
-10	118	116
-7	83	81
-4	77	79
-1	94	95
2	63	61
5	30	29
8	34	32
H,L= 13, 6		
-11	79	79
-8	58	55
-5	49	51
-2	53	49
1	54	54
4	31	31
H,L= 14, 0		
-13	217	220
-10	137	136
-7	286	274
-4	323	322
-1	210	210
2	132	131
5	181	180
8	99	93
11	54	53
14	19	22
H,L= 14, 1		
-11	181	177
-8	216	210
-5	224	223
-2	155	148
1	97	95
4	39	38
7	51	50
10	26	30
13	19	25
H,L= 14, 2		
-12	165	167
-9	218	216
-6	134	135
-3	116	116
0	105	110
3	149	144
6	80	88
9	37	40
12	34	30
H,L= 14, 3		
-13	180	184
-10	144	142
-7	174	171
-4	168	161
-1	148	143
2	98	99
5	128	123
8	41	35
11	23	26
H,L= 14, 4		
-11	85	90
-8	106	105
-5	96	95
-2	110	112
1	101	100
4	68	69
7	44	43
10	30	32
H,L= 14, 5		
-12	95	96
-9	79	77
-6	100	98
-3	70	69
0	67	66
3	50	49
6	37	33
H,L= 14, 6		
-13	47	51
-10	55	59
-7	70	68
-4	16	28
-1	41	41
2	38	40
H,L= 15, 0		
-12	123	127
-9	189	190
-6	243	237
-3	172	176
0	158	149
3	137	137
6	101	100
9	81	81
12	33	29
H,L= 15, 1		
-13	175	168
-10	196	198
-7	178	177
-4	107	107
-1	150	146
2	174	171
5	88	86
8	26	31
11	21	21
H,L= 15, 2		
-14	102	108
-11	179	173
-8	142	141
-5	112	111
-2	82	75
1	75	75
4	128	131
7	38	33
10	28	26
H,L= 15, 3		
-12	217	221
-9	93	93
-6	177	169
-3	163	167
0	116	113
3	71	70
6	62	64
9	42	41
H,L= 15, 4		
-13	62	58
-10	82	72
-7	109	111
-4	100	98
-1	99	97
2	73	72
5	35	37
8	34	34
H,L= 15, 5		
-14	54	60
-11	83	81
-8	83	85
-5	87	87
-2	69	68
1	66	64
4	36	36
H,L= 15, 6		
-12	51	48
-9	83	83
-6	9	19
-3	37	34
0	35	34
H,L= 16, 0		
-14	134	135
-11	141	143
-8	208	208
-5	163	164
-2	134	137
1	121	121
4	99	99
7	81	79
10	48	52
H,L= 16, 1		
-15	72	80
-12	206	206
-9	194	190
-6	156	150
-3	121	121
0	171	178
3	138	131
6	112	114
9	20	19
H,L= 16, 2		
-13	138	144
-10	174	171
-7	118	116
-4	87	87
-1	74	74
2	80	74
5	57	56
8	28	23
11	14	15
H,L= 16, 3		
-14	133	139
-11	68	62
-8	125	120
-5	146	140
-2	108	112
1	102	98
4	64	59
7	48	40
H,L= 16, 4		
-15	81	80
-12	111	107
-9	94	89
-6	90	87
-3	121	122
0	72	72
3	34	34
6	26	30
H,L= 16, 5		
-13	52	55
-10	88	85
-7	78	74
-4	57	57
-1	79	79
2	23	33
H,L= 16, 6		
-14	11	23
-11	70	73
-8	45	42
-5	32	40
-2	33	35
H,L= 17, 0		
-16	135	131
-13	109	108
-10	114	112
-7	236	238
-4	92	91
-1	124	123
2	100	101
5	53	53
8	46	46
H,L= 17, 1		
-14	126	126
-11	147	145
-8	126	125
-5	105	105
-2	130	127
1	119	118
4	138	136
7	42	42
10	23	23
H,L= 17, 2		
-15	82	84
-12	137	135
-9	104	105
-6	88	92
-3	116	116
0	102	99
3	61	65
6	34	28
9	13	13
H,L= 17, 3		
-16	86	83
-13	117	111
-10	101	103
-7	81	85
-4	81	80
-1	101	103
2	47	53
5	49	49
8	27	28
H,L= 17, 4		
-14	46	45
-11	75	73
-8	99	102
-5	81	83
-2	86	82
1	40	42
4	22	27
H,L= 17, 5		
-15	36	28
-12	72	75
-9	71	75
-6	70	68
-3	77	77
0	41	37
3	35	34
H,L= 17, 6		
-13	39	36
-10	50	51
-7	38	37
-4	39	39
H,L= 18, 0		
-15	143	144
-12	134	135
-9	120	120
-6	151	150
-3	90	91
0	113	112
3	72	68
6	41	42
9	36	38
H,L= 18, 1		
-16	88	90
-13	140	145
-10	113	104
-7	125	126
-4	74	72
-1	67	68
2	120	116
5	55	49
8	20	23
H,L= 18, 2		
-17	51	56
-14	160	162
-11	117	120
-8	96	93
-5	99	100
-2	148	149
1	84	85
4	64	67
7	20	21
H,L= 18, 3		
-15	62	57
-12	52	53
-9	74	75
-6	56	54
-3	67	73
0	64	59
3	36	33
6	20	23
H,L= 18, 4		
-16	31	37
-13	74	76
-10	60	59
-7	60	61
-4	88	89
-1	50	45
2	44	37
5	38	36
H,L= 18, 5		
-17	19	30
-14	52	56
-11	76	78
-8	57	57
-5	53	56
-2	43	50
1	35	36
H,L= 18, 6		
-9	36	38
H,L= 19, 0		
-17	98	98
-14	187	182
-11	95	97
-8	107	102
-5	88	92
-2	121	118
1	39	33
4	42	53
7	34	37
H,L= 19, 1		
-18	54	56
-15	155	152
-12	85	85
-9	42	42
-6	128	123
-3	134	135
0	46	44
3	56	51
6	24	23
H,L= 19, 2		
-16	98	100
-13	111	107
-10	61	61
-7	67	71
-4	144	143
-1	82	85
2	68	66
5	22	28
H,L= 19, 3		
-17	87	90
-14	73	74
-11	52	54
-8	82	86
-5	77	75
-2	59	59
1	24	28
4	28	27
H,L= 19, 4		
-18	33	25
-15	41	44
-12	38	37
-9	57	54
-6	54	56
-3	52	52
0	30	31
3	30	32
H,L= 19, 5		
-16	37	36
-13	59	62
-10	65	64
-7	52	48
-4	43	47
-1	40	41
H,L= 20, 0		
-19	26	24
-16	87	94
-13	117	118
-10	75	71
-7	52	50
-4	102	96
-1	48	47
2	58	56
5	30	35
H,L= 20, 1		
-17	79	77
-14	131	130
-11	86	91
-8	31	23
-5	128	126
-2	62	64
1	62	62
4	23	25
H,L= 20, 2		
-18	57	56
-15	131	132
-12	54	53
-9	63	62
-6	108	113
-3	93	92
0	44	41
3	47	51
H,L= 20, 3		
-19	72	76
-16	46	46
-13	51	55
-10	81	83
-7	77	82
-4	80	86
-1	38	33
2	16	17
H,L= 20, 4		
-17	38	38
-14	40	43
-11	26	23
-8	46	45
-5	55	51
-2	27	28
1	13	13
H,L= 20, 5		
-15	35	36
-12	56	58
-9	30	32
-6	47	44
-3	39	41
H,L= 21, 0		
-18	59	62
-15	98	95
-12	82	85
-9	71	72
-6	54	52
-3	46	50
0	39	40
3	59	56
H,L= 21, 1		
-19	34	39
-16	69	69
-13	103	103
-10	91	90
-7	68	65
-4	67	69
-1	63	63
2	39	37
5	23	23
H,L= 21, 2		
-20	68	66
-17	69	68
-14	67	64
-11	86	83
-8	73	75
-5	56	58
-2	62	64
1	29	21
4	24	28
H,L= 21, 3		
-18	50	52
-15	77	76
-12	51	46
-9	83	79
-6	93	95
-3	47	51
0	29	30
H,L= 21, 4		
-19	32	29
-16	30	38
-13	47	46
-10	37	34
-7	31	28
-4	40	39
-1	0	12
H,L= 21, 5		
-14	49	45
-11	32	31
-8	36	36
H,L= 22, 0		
-20	50	48
-17	53	54
-14	89	91
-11	63	59
-8	77	80
-5	66	69
-2	18	21
1	59	60
4	18	20
H,L= 22, 1		
-21	42	43
-18	38	38
-15	72	79
-12	57	61
-9	57	57
-6	47	48
-3	47	46
0	42	44
3	14	21
H,L= 22, 2		
-19	52	58
-16	68	69
-13	48	46
-10	83	79
-7	62	59
-4	58	57
-1	39	35
2	31	31
H,L= 22, 3		
-20	42	42
-17	49	53
-14	54	55
-11	82	86
-8	69	70
-5	54	58
-2	39	41
H,L= 22, 4		
-18	27	28
-15	31	37
-12	39	41
-9	43	43
-6	21	23
-3	28	22
H,L= 23, 0		
-22	53	58
-19	41	51
-16	77	74
-13	71	68
-10	55	61
-7	75	77
-4	36	39
-1	33	35
2	34	36
H,L= 23, 1		
-20	19	14
-17	42	54
-14	42	42
-11	61	58
-8	55	58
-5	50	49
-2	17	20
1	38	39
H,L= 23, 2		
-21	34	38
-18	42	46
-15	39	40
-12	79	78
-9	80	82
-6	28	29
-3	18	35
0	28	29
H,L= 23, 3		
-19	37	33
-16	44	43
-13	39	43
-10	61	61
-7	56	56
-4	18	21
H,L= 23, 4		
-17	31	31
-14	54	54
-11	46	47
-8	14	21
-5	24	30
H,L= 24, 0		
-21	49	57
-18	37	46
-15	75	75
-12	45	55
-9	76	72
-6	64	66
-3	41	35
0	30	27
H,L= 24, 1		
-22	40	40
-19	33	31
-16	57	57
-13	35	37
-10	42	44
-7	35	34
-4	48	46
-1	21	29
H,L= 24, 2		
-23	15	20
-20	28	32
-17	17	27
-14	10	21
-11	61	66
-8	39	43
-5	25	30
-2	18	21
H,L= 24, 3		
-18	27	33
-15	48	52
-6	30	29
H,L= 24, 4		
-13	51	50
-10	24	31
H,L= 25, 0		
-23	34	37
-20	39	40
-17	48	48
-14	50	51
-11	43	45
-8	52	52
-5	41	42
-2	36	36
H,L= 25, 1		
-21	15	11
-18	48	50
-15	29	28
-12	62	65
-9	28	17
-6	35	38
-3	28	29
H,L= 25, 2		
-19	8	15
-16	29	29
-13	59	60
-10	29	29
-7	43	42
-4	18	28
H,L= 25, 3		
-17	28	30
-14	36	40
-11	37	35
-8	26	29
H,L= 26, 0		
-22	20	20
-19	36	42
-16	39	46
-13	33	36
-10	40	43
-7	29	33
-4	32	34
H,L= 26, 1		
-20	34	39
-17	43	38
-14	44	42
-11	35	42
-8	30	32
-5	28	29
H,L= 26, 2		
-18	5	14
-15	18	15
-12	24	26
-9	47	49
H,L= 27, 0		
-18	35	38
-15	24	26
-12	31	27
-9	0	22
H,L= 27, 1		
-19	39	38
-16	22	21
-13	38	40
-10	35	28
H,L= 27, 2		
-14	18	21
-11	27	27
H,L= 28, 0		
-14	23	26

of the molecule down the threefold axis is shown in Figure 1. The holmium atom, on the threefold axis, is surrounded by a distorted octahedron of six oxygen atoms from the organic ligands and a seventh oxygen from the water. The water oxygen is also on the threefold axis and caps the expanded triangular face made up of three O(2) oxygen atoms; The O(2)···O(2) distance is 3.78 Å as opposed to an O(1)···O(1) distance of 3.03 Å in the opposing face. A list of some interatomic distances and their standard deviations is shown in Table V. The angles about holmium and water are given in Table VI.

The water molecule is at a distance of 2.39 Å from the holmium atom. It has six oxygen atoms as nearest neighbors; three O(2) atoms 2.80 Å distant which are in the same coordination complex about the holmium, and three O(1) atoms at a 3.00-Å distance that are in the adjacent coordination complex. With threefold symmetry inherent in the water position, it must be concluded that the water hydrogen atoms are disordered. A study of the geometry to the adjacent three O(1) atoms at 3.00 Å does not suggest any orderly hydrogen-bond schemes for the water, *i.e.*, the O(1)–H_2O–O(1) angles are 61° and are much too tight to be hydrogen-bond type angles.

The over-all complex resembles a right-handed three-bladed propeller, this being the absolute configuration of this particular crystal. The phenyl rings are twisted

TABLE II

POSITIONAL PARAMETERS[a]

Atoms	x	y	z
Ho	0.0000[b]	0.0000[b]	0.0000[c]
H_2O	0.0000[b]	0.0000[b]	0.3771 (8)
O(1)	0.0670 (2)	−0.0170 (2)	−0.2373 (6)
O(2)	0.1090 (2)	0.0724 (2)	0.1015 (6)
C(1)	0.1305 (2)	0.0073 (2)	−0.2610 (7)
C(2)	0.1801 (2)	0.0583 (3)	−0.1378 (9)
C(3)	0.1681 (3)	0.0892 (2)	0.0330 (8)
C(4)	0.1517 (2)	−0.0224 (2)	−0.4359 (8)
C(5)	0.2166 (4)	−0.0029 (7)	−0.4883 (22)
C(6)	0.2336 (4)	−0.0287 (7)	−0.6544 (24)
C(7)	0.1887 (4)	−0.0758 (4)	−0.7747 (15)
C(8)	0.1244 (4)	−0.0916 (5)	−0.7455 (17)
C(9)	0.1071 (3)	−0.0640 (4)	−0.5786 (14)
C(10)	0.2268 (3)	0.1450 (2)	0.1486 (9)
C(11)	0.2183 (4)	0.1619 (3)	0.3491 (10)
C(12)	0.2722 (5)	0.2152 (4)	0.4598 (13)
C(13)	0.3360 (5)	0.2511 (4)	0.3601 (17)
C(14)	0.3450 (4)	0.2339 (3)	0.1718 (16)
C(15)	0.2918 (3)	0.1817 (3)	0.0587 (12)
H(2)[d]	0.2300	0.0740	−0.1740
H(5)	0.2520	0.0310	−0.3920
H(6)	0.2820	−0.0140	−0.6870
H(7)	0.2020	−0.0960	−0.8920
H(8)	0.0900	−0.1250	−0.8460
H(9)	0.0590	−0.0770	−0.5530
H(11)	0.1720	0.1360	0.4150
H(12)	0.2650	0.2270	0.6060
H(13)	0.3750	0.2890	0.4340
H(14)	0.3910	0.2600	0.1080
H(15)	0.2990	0.1700	−0.0870

[a] Standard deviations appear in parentheses. [b] Special position parameters fixed by symmetry. [c] Polar space group origin fixed at $z = 0.0$ for Ho. [d] Hydrogen atoms fixed relative to carbon. Each hydrogen atom is numbered with the same number as the carbon atom to which it is bonded.

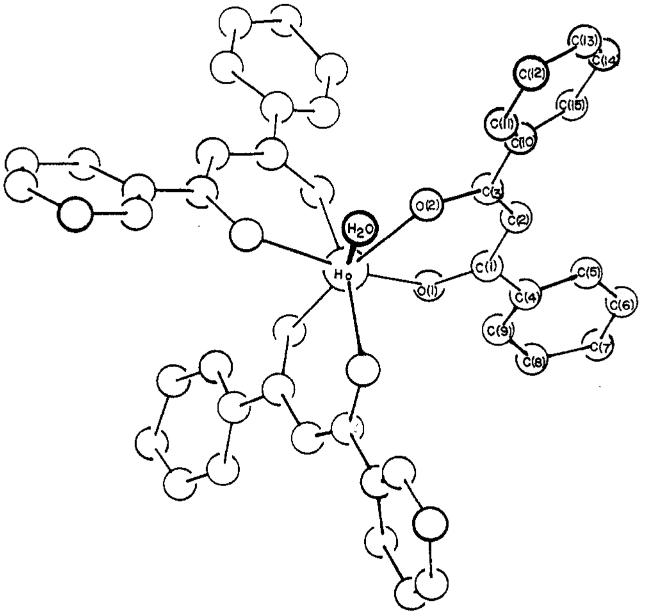


Figure 1.—The molecular unit of tris(diphenylpropanedionato)-aquoholmium projected down the threefold axis. The water molecule is directly above Ho but has been displaced slightly in this drawing to show the structure better.

TABLE III

ANISOTROPIC THERMAL PARAMETERS (Å^2)[a,b]

Atoms	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ho	5.11 (2)	5.11 (2)	3.38 (2)	2.56 (1)	0.00	0.00
H_2O	7.15 (20)	7.15 (20)	2.82 (21)	3.57 (10)	0.00	0.00
O(1)	4.54 (13)	5.58 (14)	4.49 (14)	2.42 (11)	−0.41 (11)	−1.42 (12)
O(2)	5.65 (16)	5.74 (15)	4.44 (15)	2.75 (13)	−0.87 (13)	−1.37 (12)
C(1)	4.60 (18)	4.57 (17)	4.17 (17)	2.49 (15)	−0.55 (14)	−0.61 (14)
C(2)	4.49 (19)	5.36 (21)	5.52 (24)	1.98 (17)	−0.52 (17)	−1.35 (18)
C(3)	5.41 (21)	4.45 (18)	4.31 (17)	2.39 (16)	−1.25 (16)	−0.80 (15)
C(4)	4.31 (18)	4.29 (18)	5.52 (22)	2.30 (15)	−0.39 (16)	−0.97 (17)
C(5)	4.70 (28)	16.29 (86)	17.14 (100)	3.33 (40)	−1.32 (40)	−11.96 (86)
C(6)	5.12 (30)	16.16 (86)	17.74 (107)	3.35 (41)	0.64 (44)	−10.74 (85)
C(7)	7.11 (33)	7.91 (36)	9.71 (47)	3.93 (29)	0.83 (32)	−3.27 (35)
C(8)	7.74 (38)	12.17 (57)	10.47 (57)	5.61 (39)	−1.86 (36)	−6.70 (50)
C(9)	5.79 (28)	10.89 (47)	9.17 (46)	4.48 (31)	−1.33 (28)	−5.32 (41)
C(10)	6.24 (25)	4.17 (18)	5.36 (22)	2.54 (18)	−1.72 (19)	−0.83 (17)
C(11)	8.04 (34)	7.04 (30)	5.68 (27)	4.84 (28)	−2.57 (26)	−2.30 (24)
C(12)	11.49 (57)	8.67 (42)	7.50 (38)	7.05 (46)	−4.06 (41)	−4.11 (35)
C(13)	8.74 (45)	5.83 (29)	11.20 (58)	3.58 (30)	−5.17 (45)	−3.19 (35)
C(14)	7.15 (34)	5.61 (27)	10.47 (53)	1.75 (25)	−3.69 (36)	−1.35 (32)
C(15)	6.55 (29)	5.30 (24)	7.08 (33)	1.61 (21)	−2.47 (26)	−0.59 (23)

[a] Standard deviations appear in parentheses. [b] The symmetry of the special position of Ho and H_2O restrains their thermal parameters as follows: $2B_{12} = B_{22} = B_{11}$; $B_{13} = B_{23} = 0$.

from the plane of the propanedionato portion of the complex. The phenyl group consisting of atoms C(4) through C(9) is twisted ~10° from the plane made up of atoms O(1), C(1), and C(2); the phenyl group consisting of atoms C(10) through C(15) is twisted ~19° from the plane defined by O(2), C(3), and C(2). This twisting is discernible in the stereographic picture shown in Figure 2. Figure 3 shows the geometry in one blade of the molecule. A least-squares plane calculated through the six atoms O(1), C(1), C(2), C(3), O(2), and Ho shows that the deviations from this plane are, in the order as listed above, −0.01, 0.02, 0.0, −0.02, 0.02, and −0.01 Å, respectively.

There is sizable thermal motion in the two phenyl rings. This is apparent from the thermal parameters, and the stereographic view of the molecule in Figure 2 depicts the nature of the thermal motion. The phenyl ring consisting of atoms C(4) through C(9) appears

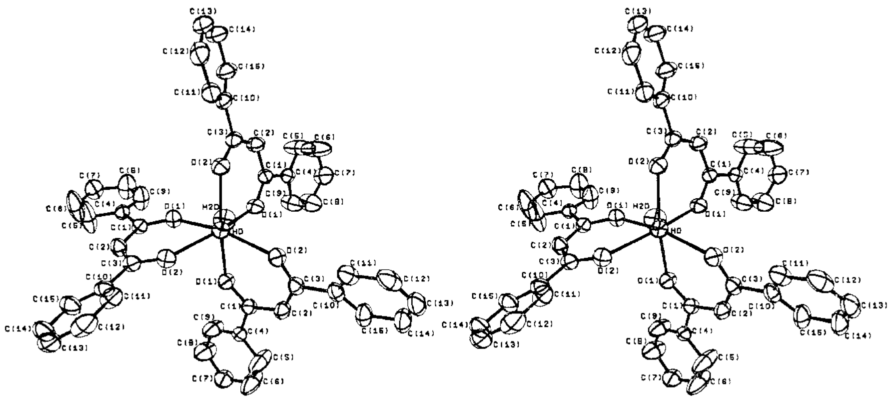


Figure 2.—A stereographic pair drawing of the molecule of tris(diphenylpropanedionato)aquoholmium.

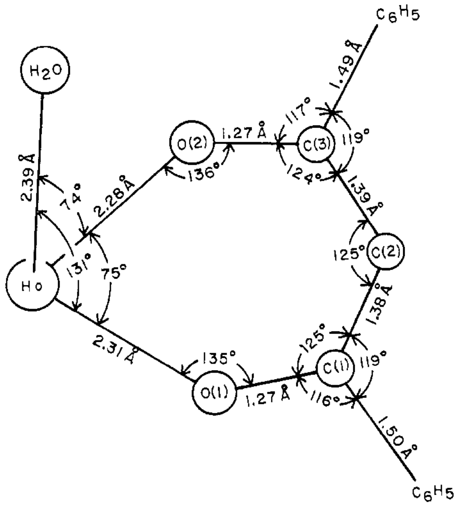


Figure 3.—Some distances and angles of the ligands about holmium. The estimated standard deviation of the angles is about 0.5° and of the distances is about 0.02 Å.

TABLE IV

ISOTROPIC THERMAL PARAMETERS (Å^2)[a]

Atoms	B	Atoms	B	Atoms	B
H(2)	5.30	H(8)	9.90	H(13)	8.60
H(5)	13.60	H(9)	8.50	H(14)	8.40
H(6)	13.70	H(11)	6.40	H(15)	6.90
H(7)	8.20	H(12)	8.30		

[a] These estimated values were not allowed to refine in the least-squares calculations.

TABLE V

INTERATOMIC DISTANCES IN TRIS(DIPHENYLPROPANEDIONATO)AQUOHOLMIUM[a]

Atoms	Dist, Å	Atoms	Dist, Å
Ho–3O(2)	2.275 (4)	O(2)–C(1)	2.90 (1)
Ho–3O(1)	2.305 (7)	C(1)–C(2)	1.38 (1)
Ho–H_2O	2.39 (2)	C(1)–C(4)	1.50 (1)
H_2O–3O(2)	2.80 (1)	C(2)–C(3)	1.39 (1)
H_2O–3O(1)	3.00 (1)	C(3)–C(10)	1.49 (1)
O(1)–C(1)	1.27 (1)	C(4)–C(9)	1.33 (1)
O(1)–C(4)	2.35 (1)	C(4)–C(5)	1.35 (1)
O(1)–C(2)	2.35 (1)	C(5)–C(6)	1.35 (1)
O(1)–C(9)	2.76 (1)	C(6)–C(7)	1.29 (2)
O(1)–O(2)	2.77 (1)	C(7)–C(8)	1.33 (1)
O(1)–C(3)	2.91 (1)	C(8)–C(9)	1.38 (1)
O(1)–2O(1)	3.03 (1)	C(10)–C(11)	1.37 (1)
O(1)–O(2)	3.10 (1)	C(10)–C(15)	1.40 (1)
O(2)–C(3)	1.27 (1)	C(11)–C(12)	1.41 (1)
O(2)–C(10)	2.36 (1)	C(12)–C(13)	1.41 (1)
O(2)–C(2)	2.35 (1)	C(13)–C(14)	1.30 (2)
O(2)–C(11)	2.78 (1)	C(14)–C(15)	1.40 (1)

[a] Standard deviations appear in parentheses.

TABLE VI

HOLMIUM- AND WATER-CENTERED ANGLES[a]

Atoms	Angle, deg	Atoms	Angle, deg
H_2O–Ho–O(1)	130.7 (2)	Ho–H_2O–O(1)	144.4 (2)
H_2O–Ho–O(2)	73.6 (2)	Ho–H_2O–O(2)	51.4 (2)
O(1)–Ho–O(1)	82.1 (3)	O(1)–H_2O–O(1)	60.5 (3)
O(1)–Ho–O(2)	74.6 (3)	O(1)–H_2O–O(2)	163.3 (2)
O(1)–Ho–O(2)	154.7 (3)	O(1)–H_2O–O(2)	109.9 (3)
O(1)–Ho–O(2)	85.1 (2)	O(2)–H_2O–O(2)	85.1 (3)
O(2)–Ho–O(2)	112.4 (2)	O(1)–H_2O–O(2)	103.1 (3)

[a] Standard deviations in parentheses.

to be oscillating in and out of its plane about an axis passing through C(4) and C(7). The other phenyl ring, C(10) through C(11), seems to be vibrating in the plane of the phenyl ring itself. It is not surprising that the bond lengths in these rings are shorter than expected. No correction has been made for this thermal contraction effect in the C–C bond lengths of the phenyl groups, as the benzene geometry is well established and is not a significant part of this structure determination.

These results are in excellent agreement with those reported by Cotton and Legzdins[4] for $Y(C_6H_5COCHCOCH_3)_3 \cdot H_2O$. In the yttrium compound the complex has no symmetry and all three organic ligands are independently determined. Other than the distances involving the heavy atoms, there are no significant differences in the bond distances and angles in the comparable portions of the two structures. The Ho–H_2O distance is 0.05 Å longer than the Y–H_2O distance, and the ligand Ho–O distances are both 0.02 Å longer than the comparable Y–O distances.

$Yb(C_5H_7O_2)_3 \cdot H_2O$[5] and $Yb(C_5H_7O_2)_3 \cdot H_2O \cdot 0.5C_6H_6$[6] form sevenfold coordination complexes similar to our Ho complex. All of the ligand oxygen to Ho distances are just slightly longer than the comparable distances to Yb. The authors of the Yb complexes[5,6] described the heptacoordination as capped distorted trigonal prisms, but stated that the "description is also possible in terms of the capped octahedron. . . . the capping ligand in this case would be the carbonyl oxygen atoms rather than a water molecule. . . ."