CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF KENTUCKY, LEXINGTON, KENTUCKY 40506

The Crystal and Molecular Structure of Yttrium Acetylacetonate Trihydrate[1]

BY JAMES A. CUNNINGHAM, DONALD E. SANDS, AND WILLIAM F. WAGNER

Received September 15, 1966

The crystal structure of $Y(C_5H_7O_2)_3 \cdot 3H_2O$ has been determined from three-dimensional X-ray diffraction data. Four molecules crystallize in a monoclinic unit cell of symmetry $P2_1/n$ and dimensions $a = 11.24$, $b = 22.20$, $c = 8.44$ A; $\beta = 100.5°$. The yttrium ion is eight-coordinate, being bonded to three acetylacetonate rings and two water molecules. The third water associated with each molecule takes part in a chain of hydrogen bonds to link the molecules in pairs. The coordination polyhedron about the yttrium ion is a distorted square antiprism.

Introduction

The trisacetylacetonates of the lanthanides and yttrium form metastable trihydrates which dehydrate to stable monohydrates.[2] Single crystals of the trihydrates are readily prepared, but the monohydrates are not easily crystallized. It is also possible to prepare such crystalline compounds as $Y(C_5H_7O_2)_3H_2O \cdot 2CH_3OH$. This study of the trihydrate of the yttrium chelate was undertaken in order to obtain a structural interpretation of the chemical data. The yttrium compound was chosen because yttrium is the lightest of the M(III) elements exhibiting two hydrated forms.

Experimental Section

Yttrium acetylacetonate, prepared by the method of Stites, McCarty, and Quill,[3] was recrystallized from a 60% ethanol–water mixture to yield the trihydrated form. The needlelike crystals were washed twice with water and were then air dried.

With the aid of a polarizing microscope a suitable crystal, with dimensions 0.27 × 0.05 × 0.01 mm, was chosen and sealed in a thin-walled glass capillary. The monoclinic unit cell dimensions, obtained from oscillation, Weissenberg, and precession photographs using Cu Kα radiation (λ 1.5418 A), are $a = 11.24 \pm 0.03$, $b = 22.20 \pm 0.06$, $c = 8.44 \pm 0.02$ A; $\beta = 100.5 \pm 0.3°$. The density measured by flotation is 1.37 g cm^{-3}; the calculated value for four molecules per unit cell is 1.38 g cm^{-3}. The systematic absence of $h0l$ reflections for $h + l$ odd and $0k0$ reflections for k odd suggests space group $P2_1/n$.

Multiple-film equiinclination Weissenberg photographs were obtained for the $0kl$ through $8kl$ levels using nickel-filtered Cu Kα radiation, and the intensities were estimated by visual comparison with calibrated intensity strips made with the same crystal. The intensities within each multiple-film packet were brought to the level of the most intense film by appropriate scale factors. Corrections for spot extension and contraction were applied,[4] and an average intensity for each independent reflection was obtained. After correction for the velocity and Lorentz-polarization factors, scale factors for the various levels were obtained by comparison with data from an $h0l$ precession photograph. No corrections were made for absorption of extinction. A total of 2582 independent intensities were recorded, of which 786 were unobserved.

Structure Determination and Refinement

Trial yttrium positions were deduced from the three-dimensional Patterson function. Further interpretation of the Patterson was not attempted owing to extensive crowding and overlapping of the peaks. The yttrium ions occupy the $P2_1/n$ general positions 4(e): $\pm(x, y, z; \, ^1/_2 + x, \, ^1/_2 - y, \, ^1/_2 + z)$ with $x = 0.26$, $y = 0.02$, and $z = 0.12$.

The contributions of Y^{3+} to the observed structure factors were computed, and a Fourier synthesis was based on the calculated signs and observed magnitudes of the 1518 terms for which $|F_c| \geq 0.25|F_o|$. This Fourier synthesis had five prominent peaks at approximately the right distance from the yttrium ion to be oxygen atoms, but two of these were only 1.47 A apart. It was subsequently shown that the next to the strongest of these five peaks was extraneous, while the other four corresponded to oxygen atoms. The persistent appearance of this spurious peak hindered the determination of the structure, and three additional Fourier syntheses were required before all of the carbon atoms could be located.

The atomic parameters and the nine scale factors were refined by the method of least squares using an IBM 7040 adaptation of the program of Busing, Martin, and Levy.[5] The atomic form factors included were taken from Cromer, Larson, and Waber[6] for Y^{3+} and from the compilation of Ibers[7] for oxygen and carbon. The contributions to the least-squares sums were weighted by $1/\sigma^2$, in which σ for each reflection was given by $0.1\ F/n^{1/2}$, where n is the number of times that reflection was observed. Unobserved data were omitted from the refinements. Individual isotropic temperature factors were assigned to each atom and were included in the refinements. No attempt was made to locate hydrogen atoms. The final value of the residual, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, was 0.120 for the 1796 observed data. The mean positional parameter shift during the last least-squares cycle was 0.0009.

A final Fourier synthesis still showed the large extraneous peak near one of the oxygen atoms. This peak was flanked by two highly negative regions. A difference map, based on $F_o - F_c$, showed neither the spurious

---

[1] Work supported by U. S. Atomic Energy Commission Contract No. AT(40-1)2124. We also thank the University of Kentucky Computing Center.

[2] G. W. Pope, J. F. Steinbach, and W. F. Wagner, *J. Inorg. Nucl. Chem.*, **20**, 304 (1961).

[3] J. G. Stites, C. N. McCarty, and L. L. Quill, *J. Am. Chem. Soc.*, **70**, 3142 (1948).

[4] D. C. Phillips, *Acta Cryst.*, **7**, 746 (1954).

[5] W. R. Busing, K. O. Martin, and H. A. Levy, "ORFLS, a Fortran Crystallographic Least-Squares Program," ORNL-TM-305, 1962.

[6] D. T. Cromer, A. C. Larson, and J. T. Waber, *Acta Cryst.*, **17**, 1044 (1964).

[7] "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 202.

TABLE I

OBSERVED AND CALCULATED STRUCTURES FACTORS FOR OBSERVED DATA, EACH MULTIPLIED BY 10

K	L	FO	FC
	H= 0		
4	0	196	-224
6	0	339	321
8	0	276	246
10	0	248	-261
12	0	307	-295
14	0	322	-263
16	0	188	-185
18	0	320	-277
20	0	336	-250
2	1	317	-338
3	1	267	240
4	1	299	-333
5	1	503	497
6	1	332	-357
7	1	91	97
8	1	175	-185
9	1	173	-165
10	1	91	-86
11	1	238	-223
12	1	182	-173
13	1	221	-242
14	1	185	-154
15	1	232	-229
16	1	117	-106
17	1	139	-152
19	1	262	-254
20	1	86	93
21	1	130	-112
22	1	75	82
0	2	67	60
2	2	194	-195
3	2	107	89
4	2	206	150
5	2	265	-276
6	2	216	221
7	2	536	-681
8	2	170	183
9	2	469	-505
10	2	80	105
11	2	388	-372
12	2	83	86
13	2	304	-297
15	2	172	-172
17	2	52	-68
19	2	62	87
20	2	113	-107
21	2	143	135
23	2	98	94
1	3	616	-629
2	3	304	-314
3	3	381	-410
4	3	334	-314
5	3	343	-293
6	3	211	-203
7	3	236	-231
8	3	163	-149
9	3	71	71
10	3	301	-318
11	3	134	146
12	3	357	-332
13	3	161	169
14	3	82	-85
15	3	114	106
17	3	180	165
19	3	113	107
20	3	141	129
21	3	64	68
22	3	134	140
2	4	491	-604
4	4	305	-299
5	4	48	-53
6	4	143	-155
7	4	103	-83
8	4	58	66
11	4	86	-73
13	4	111	-128
14	4	128	140
16	4	202	195
18	4	243	245
20	4	237	205
22	4	107	119
1	5	348	-375
2	5	103	97
3	5	364	-352
4	5	219	219
5	5	282	-255
7	5	82	-70
8	5	131	124
10	5	93	80
12	5	174	163
13	5	184	153
14	5	193	180
15	5	202	176
18	5	89	67
17	5	165	153
19	5	146	148
21	5	136	125
22	5	42	-45
0	6	205	-193
2	6	175	-168
3	6	104	84
4	6	123	-105
5	6	208	216
6	6	106	-111
7	6	222	224
9	6	222	193
11	6	261	229
12	6	54	47
13	6	206	201
14	6	54	78
5	6	7	87
19	6	88	-81
1	7	145	141
3	7	54	55
6	7	268	225
6	7	239	199
10	7	123	129
11	7	54	-69
14	7	104	107
16	7	46	64
19	7	115	-89
20	7	48	-44
0	8	308	266
2	8	263	233
4	8	191	171
12	8	66	-84
14	8	94	-93
16	8	104	-88
19	8	56	-20
1	9	253	227
3	9	182	164
5	9	80	80
13	9	86	-83
2	10	35	50
3	10	43	-56
4	10	54	57
5	10	56	-61
7	10	101	-91
9	10	86	-87
	H= 1		
2	0	392	-430
3	0	44	-44
4	0	266	-258
5	0	198	199
6	0	640	-654
7	0	282	239
8	0	581	-586
9	0	182	142
10	0	489	-450
11	0	150	156
12	0	359	-313
14	0	208	-210
15	0	111	117
16	0	74	-93
20	0	176	149
22	0	199	192
1	1	176	-166
2	1	565	-547
3	1	270	255
4	1	376	-305
5	1	222	-146
6	1	112	-109
7	1	431	-473
8	1	211	167
9	1	334	-319
10	1	339	318
11	1	196	-212
12	1	303	325
13	1	229	-235
14	1	276	277
15	1	115	-106
16	1	181	165
17	1	67	-73
18	1	166	166
19	1	58	74
20	1	219	190
21	1	197	187
22	1	100	100
23	1	123	115
25	1	130	112
1	-1	129	-136
2	-1	322	361
3	-1	256	-219
4	-1	303	-315
5	-1	701	-597
6	-1	179	-164
7	-1	242	-297
8	-1	129	-107
9	-1	460	-391
10	-1	85	-92
11	-1	527	-480
12	-1	183	-203
13	-1	236	-232
14	-1	165	-151
15	-1	51	71
16	-1	92	-94
17	-1	95	108
18	-1	206	-196
19	-1	127	158
20	-1	123	-139
21	-1	218	196
3	2	324	235
4	2	187	-167
5	2	70	-80
6	2	118	134
7	2	345	-363
8	2	66	-47
9	2	83	98
10	2	158	-151
11	2	280	302
12	2	47	-62
13	2	331	322
15	2	259	293
17	2	253	270
18	2	107	-106
19	2	293	264
21	2	172	140
1	-2	691	689
2	-2	78	71
3	-2	510	451
4	-2	131	109
5	-2	386	366
8	-2	156	-182
9	-2	236	-170
10	-2	382	-282
11	-2	74	-81
13	-2	312	-249
15	-2	259	-287
16	-2	94	95
17	-2	233	-243
18	-2	81	88
19	-2	220	-188
20	-2	83	56
21	-2	118	-95
0	3	597	-661
1	3	219	-233
2	3	189	-182
3	3	89	-72
4	3	65	84
5	3	88	71
7	3	190	193
9	3	256	260
11	3	297	325
13	3	221	226
14	3	103	112
15	3	144	137
16	3	86	104
18	3	206	200
20	3	195	189
0	-3	250	288
1	-3	71	54
2	-3	160	179
3	-3	315	321
4	-3	76	79
5	-3	450	430
7	-3	495	469
8	-3	151	-146
9	-3	111	110
10	-3	160	-212
11	-3	181	185
12	-3	265	-289
13	-3	215	213
14	-3	265	-249
16	-3	97	-100
20	-3	241	-235
21	-3	57	-64
22	-3	152	-144
23	-3	89	-75
1	4	42	46
2	4	133	122
4	4	311	335
5	4	173	-190
6	4	397	394
8	4	322	344
10	4	226	216
11	4	57	-74
12	4	208	232
14	4	169	173
16	4	142	93
20	4	148	-161
22	4	161	-141
24	4	178	-137
1	-4	181	177
2	-4	206	199
4	-4	483	440
5	-4	138	121
6	-4	566	613
7	-4	111	121
8	-4	528	498
9	-4	87	-67
10	-4	296	253
12	-4	276	294
14	-4	151	148
15	-4	99	-105
17	-4	83	-63
18	-4	101	-75
20	-4	126	-94
21	-4	94	-84
22	-4	139	-132
24	-4	148	-136
0	5	208	212
1	5	88	59
2	5	201	215
3	5	244	248
4	5	156	151
5	5	286	291
6	5	99	87
7	5	317	331
9	5	256	285
10	5	99	-102
11	5	245	284
13	5	124	126
14	5	60	-62
16	5	158	-164
18	5	126	-143
19	5	86	-82
20	5	52	-79
21	5	112	-101
22	5	92	-77
23	5	90	-73
0	-5	142	-156
1	-5	90	-91
2	-5	156	-136
4	-5	157	-136
5	-5	220	203
6	-5	84	-71
7	-5	378	344
9	-5	211	203
11	-5	201	221
13	-5	266	255
15	-5	131	115
17	-5	82	-83
19	-5	168	-142
20	-5	53	62
21	-5	86	-99
23	-5	100	-96
1	6	191	214
2	6	88	107
3	6	167	160
4	6	105	115
5	6	103	104
6	6	99	91
11	6	161	-156
13	6	176	-184
15	6	137	-143
17	6	127	-131
19	6	156	-134
21	6	136	-108
1	-6	179	-182
3	-6	177	-200
4	-6	53	-51
5	-6	119	-93
10	-6	127	114
12	-6	108	90
13	-6	142	150
15	-6	197	183
17	-6	183	167
19	-6	140	123
21	-6	84	60
0	7	216	249
2	7	218	235
4	7	162	165
5	7	66	-94
7	7	119	-107
8	7	84	-86
9	7	152	-151
11	7	87	-101
14	7	93	-97
16	7	115	-102
18	7	107	-89
19	7	37	54
20	7	81	-74
0	-7	184	-192
1	-7	81	-107
2	-7	235	-221
3	-7	129	-112
4	-7	172	-176
9	-7	102	-84
10	-7	83	57
12	-7	128	117
13	-7	126	-100
14	-7	165	156
16	-7	196	147
18	-7	141	104
20	-7	101	86
2	8	58	-70
4	8	179	-157
6	8	178	-183
8	8	137	-127
12	8	111	-92
14	8	75	-70
3	-8	143	-128
4	-8	101	-90
6	-8	200	-159
8	-8	205	-151
10	-8	175	-124
12	-8	106	-83
13	-8	88	70
14	-8	85	-70
15	-8	104	77
16	-8	44	-40
17	-8	40	39
0	9	174	-127
2	9	71	-72
3	9	50	-52
12	9	104	73
13	9	69	-56
14	9	41	41
1	10	117	-111
3	10	83	-80
5	10	52	-48
6	10	36	-37
	H= 2		
2	0	481	-466
3	0	166	177
4	0	486	-508
5	0	236	205
6	0	287	-323
7	0	180	143
8	0	131	-119
9	0	542	499
10	0	136	135
11	0	355	267
12	0	302	236
14	0	368	309
16	0	256	265
17	0	112	-80
18	0	262	246
19	0	97	-82
20	0	245	212
22	0	108	123
1	1	441	-477
2	1	357	-380
3	1	42	-59
4	1	403	287
5	1	91	-117
6	1	602	567
7	1	107	-102
8	1	459	424
9	1	122	-123
10	1	561	557
11	1	188	190
12	1	487	469
13	1	397	363
14	1	284	278
15	1	304	292
17	1	193	206
19	1	221	218
20	1	53	-61
22	1	125	-104
24	1	125	-114
26	1	101	-93
1	-1	431	-505
2	-1	154	-146
3	-1	379	-393
4	-1	47	38
5	-1	425	-462
6	-1	109	-100
7	-1	381	-379
9	-1	159	-140
10	-1	148	99
11	-1	242	253
12	-1	180	-124
13	-1	243	223
15	-1	152	138
17	-1	93	93
19	-1	230	198
22	-1	149	131
24	-1	130	123
26	-1	100	85
0	2	368	-396
2	2	108	104
3	2	51	-26
4	2	156	145
5	2	178	166
7	2	325	352
9	2	423	444
10	2	85	64
11	2	412	412
12	2	47	44
13	2	315	297
15	2	102	94
0	-2	647	-543
1	-2	99	-91
2	-2	66	-100
3	-2	216	-189
4	-2	83	49
5	-2	380	-384
6	-2	79	-55
7	-2	832	-751
8	-2	142	-132
9	-2	460	-471
10	-2	153	116
11	-2	381	-383
12	-2	48	60
13	-2	369	-365
15	-2	82	-88
17	-2	78	93
19	-2	128	100
21	-2	136	106
23	-2	131	109
1	3	520	569
2	3	240	267
3	3	384	444
4	3	272	236
5	3	195	182
6	3	365	396
7	3	254	257
8	3	321	286
9	3	133	141
10	3	127	105
11	3	77	-71
12	3	65	70
13	3	220	-213
15	3	198	-180
17	3	152	-138
18	3	52	49
19	3	52	-72
20	3	72	-65
21	3	120	-101
22	3	81	-90
24	3	69	-66
1	-3	189	-160
2	-3	251	-208
3	-3	95	105
4	-3	367	-446
5	-3	217	183
6	-3	641	-593
8	-3	629	-522
9	-3	110	-124
10	-3	343	-335
12	-3	390	-355
14	-3	181	-177
15	-3	191	-200
16	-3	51	65
17	-3	238	-247
18	-3	161	137
19	-3	82	-91
20	-3	81	85
22	-3	90	88
24	-3	102	91
0	4	383	437
1	4	370	397
2	4	380	420
3	4	227	261
4	4	266	243
5	4	168	-182
6	4	194	180
7	4	44	-45
8	4	45	-67
10	4	191	-193
11	4	84	-89
12	4	174	-190
13	4	130	-130
14	4	161	-178
15	4	96	-105
16	4	223	-212
18	4	188	-172
20	4	115	-110
21	4	47	-36
22	4	91	-91
0	-4	499	475
2	-4	599	558
4	4	378	358
6	-4	210	189
7	-4	367	-312
9	-4	397	-362
10	-4	43	-65
11	-4	165	-167
12	-4	200	-199
13	-4	48	-75
14	-4	191	-203
15	-4	50	-86
16	-4	192	-183
18	-4	168	-158
20	-4	129	-120
22	-4	123	-107
24	-4	66	-46
1	5	274	245
2	5	103	134
3	5	183	199
4	5	195	-198
5	5	176	173
6	5	307	-325
7	5	110	125
8	5	289	-302
10	5	236	-252
11	5	78	-70
12	5	170	-160
13	5	139	-163
14	5	128	-117
15	5	113	-126
17	5	111	-123
18	5	78	69
19	5	112	-98
1	-5	198	206
2	-5	159	171
3	-5	484	452
4	-5	176	207
5	-5	324	294
6	-5	217	197
7	-5	44	55
8	-5	78	71
9	-5	103	-72
11	-5	152	-146
13	-5	212	-241
15	-5	208	-212
17	-5	177	-162
19	-5	57	-90
21	-5	99	-93
0	6	51	-82
3	6	183	-177
5	6	252	-254
7	6	292	-266
8	6	92	-105
9	6	311	-304
11	6	213	-252
13	6	113	-123
19	6	86	94
0	-6	198	159
1	-6	46	-35
2	-6	206	246
4	-6	149	163
5	-6	236	236
7	-6	281	267
8	-6	49	59
9	-6	254	233
10	-6	51	73
11	-6	198	183
13	-6	116	127
14	-6	109	-99
1	7	127	-146
2	7	93	-71
3	7	116	-111
4	7	147	-157
5	7	101	-91
6	7	169	-164
8	7	121	-130
10	7	100	-105
11	7	72	74
12	7	81	-89
14	7	50	-70
15	7	54	68
17	7	47	60
3	-7	152	-59
4	-7	118	106
5	-7	72	-79
6	-7	175	161
8	-7	224	208
10	-7	232	203
12	-7	164	135
14	-7	147	123
15	-7	106	89
0	8	213	-194
2	8	191	-166
4	8	124	-135
6	8	70	-72
12	8	93	73
14	8	90	73
0	-8	152	-168
2	-8	178	-173
4	-8	132	-131
5	-8	119	88
7	-8	155	135
9	-8	164	149
11	-8	82	87
12	-8	79	97
13	-8	44	78
14	-8	146	130
15	-8	56	70
16	-8	132	117
1	9	96	-94
3	9	85	-88
4	9	42	51
5	9	71	-75
6	9	40	57
8	9	91	90
1	-9	156	-135
3	-9	149	-135
5	-9	91	-91
7	-9	76	-77
0	-10	82	-73
2	-10	81	-79
4	-10	50	-57
	H= 3		
1	0	178	186
2	0	404	413
4	0	214	239
5	0	291	-253
6	0	340	396
7	0	34	42
8	0	443	427
9	0	65	46
10	0	347	290
11	0	274	-229
12	0	312	234
13	0	67	-81
14	0	206	206
15	0	45	-60
17	0	61	-93
19	0	152	-142
21	0	156	-146
22	0	153	-123
23	0	74	-77
24	0	163	-141
26	0	99	-109
0	1	432	546
1	1	156	186
2	1	465	582
4	1	450	532
5	1	181	134
6	1	273	260
7	1	295	295
8	1	299	239
9	1	310	304
10	1	133	-136
11	1	246	229
12	1	259	-249
13	1	136	113
14	1	223	-195
15	1	68	-53
16	1	271	-263
18	1	247	-269
19	1	99	83
20	1	188	-191
22	1	112	-100
23	1	48	-46
1	-1	183	154
2	-1	242	-176
3	-1	162	153
4	-1	204	-252
7	-1	353	376
9	-1	526	511
10	-1	98	-43
11	-1	377	425
13	-1	319	296
14	-1	127	105
15	-1	63	67
16	-1	178	157
18	-1	73	76
21	-1	115	-113
23	-1	134	-122
25	-1	128	-105
1	2	461	574
2	2	118	136
3	2	375	434
5	2	273	328
6	2	95	108
7	2	312	271
10	2	174	-131
11	2	403	-384
12	2	81	-68
13	2	420	-394
14	2	87	-77
15	2	387	-355
17	2	268	-254
18	2	118	111
19	2	157	-148
20	2	50	66
21	2	49	-73
1	-2	209	-121
2	-2	73	64
3	-2	347	-374
4	-2	186	-121
5	-2	535	-578
7	-2	609	-591
8	-2	274	223
9	-2	159	-155
10	-2	192	135
11	-2	207	146
13	-2	323	252
15	-2	235	232
17	-2	295	263
19	-2	259	194
20	-2	123	-125
21	-2	139	127
22	-2	70	-84
23	-2	48	57
0	3	347	425
1	3	233	230
2	3	338	417
4	3	273	402
5	3	281	-316
6	3	162	156
7	3	290	-270
8	3	77	120
9	3	242	-224
11	3	237	-237
12	3	155	-143
13	3	162	-163
14	3	171	-156
15	3	52	-68
16	3	74	-76
17	3	53	57
18	3	74	-80
20	3	58	-57
0	-3	289	-370
1	-3	192	167
2	-3	541	-628
3	-3	112	63
4	-3	491	-479
5	-3	172	-164
6	-3	183	-174
7	-3	319	-278
9	-3	152	-127
10	-3	54	47
11	-3	168	-141
12	-3	278	250
13	-3	177	-145
14	-3	255	299
16	-3	289	258
17	-3	73	86
18	-3	238	212
20	-3	209	169
22	-3	111	102
1	4	161	-172
3	4	197	-227
4	4	261	-275
6	4	371	-386
7	4	103	111
8	4	286	-325
9	4	97	120
10	4	207	-190
11	4	54	66
12	4	168	-147
14	4	95	-93
1	-4	478	-485
2	-4	127	-84
3	-4	255	-268
4	-4	439	-429
5	-4	86	48
6	-4	378	-450
7	-4	168	108
8	-4	387	-295
9	-4	182	144
10	-4	261	-224
11	-4	204	170
12	-4	297	-268
13	-4	228	220
14	-4	205	-151
15	-4	208	167
17	-4	128	125
19	-4	72	74
21	-4	67	70
22	-4	62	71
24	-4	100	96
0	5	346	-308
2	5	354	-344
3	5	116	-130
4	5	160	-163
5	5	113	-130
7	5	176	-192
9	5	209	-210
10	5	94	122
11	5	205	-206
12	5	206	211
13	5	79	-109
14	5	199	189
16	5	200	174
18	5	83	100
20	5	64	69
22	5	59	74
3	-5	139	-136
4	-5	113	93
5	-5	237	-202
6	-5	42	59
7	-5	402	-329
9	-5	356	-301
11	-5	204	-170
13	-5	108	-90
1	6	216	-177
2	6	123	-136
3	6	296	-296
5	6	221	-204
7	6	139	-105
11	6	74	74
13	6	154	136
15	6	187	186
17	6	187	175
19	6	88	81
21	6	66	70
1	-6	290	243
3	-6	425	377
5	-6	335	274
6	-6	133	-118
7	-6	135	119
8	-6	239	-200
10	-6	71	-78
11	-6	115	-114
13	-6	217	-175
15	-6	231	-195
17	-6	133	-123
19	-6	50	-54
0	7	54	-39
2	7	139	-133
4	7	169	-165
5	7	131	148
6	7	97	-97
7	7	133	125
9	7	101	116
11	7	69	83
13	7	46	70
14	7	44	54
16	7	102	91
18	7	49	81
0	-7	333	274
2	-7	306	260
4	-7	213	191
6	-7	51	77
9	-7	148	107
10	-7	124	-93
11	-7	87	73
12	-7	181	-157
14	-7	184	-165
16	-7	196	-173
18	-7	132	-114
2	8	50	57
6	8	97	109
8	8	133	130
10	8	174	164
12	8	106	96
14	8	48	58
1	-8	193	144
3	-8	143	119
4	-8	149	133
6	-8	145	122
8	-8	170	142
10	-8	183	158
0	9	69	80
2	9	39	51
4	9	38	35
6	9	39	43
7	9	37	68
8	9	69	24
10	9	81	-33
0	-9	47	-48
3	-9	122	95
5	-9	90	94
7	-9	123	101
9	-9	198	167
11	-9	152	107
1	-10	63	-60
3	-10	61	-63
4	-10	60	59
5	-10	59	-57
6	-10	73	56
7	-10	76	-60
8	-10	71	65
	H= 4		
1	0	203	-244
2	0	93	82
3	0	244	-276
4	0	53	56
5	0	322	-275
6	0	136	86
7	0	192	-200
8	0	51	-10
9	0	253	-203
10	0	331	-304
11	0	86	-80
12	0	287	-252
13	0	158	-124
14	0	179	-166
15	0	142	-130
16	0	245	-228
18	0	257	-244
20	0	160	-162
21	0	80	89
22	0	85	-55
2	1	418	-440
5	1	223	255
7	1	225	176
8	1	494	-429
10	1	421	-410
11	1	147	-153
12	1	221	-219
13	1	209	-173
14	1	154	-139
15	1	209	-204
17	1	70	-64
2	-1	42	84
5	-1	208	186
7	-1	284	204
8	-1	217	138
9	-1	253	210
11	-1	163	-122
12	-1	105	85
13	-1	265	-220
15	-1	216	-194
17	-1	221	-206
18	-1	96	-72
19	-1	315	-251
21	-1	167	-156
0	2	270	-280
1	2	194	-252
2	2	298	-321
3	2	311	-332
4	2	106	-97
5	2	262	-285
6	2	52	-69
7	2	373	-334
8	2	137	-132
9	2	475	-468
11	2	257	-272
12	2	63	84
13	2	125	-125
1	-2	72	-37
2	-2	214	131
8	-2	215	142
9	-2	448	340
11	-2	379	340
12	-2	196	-140
13	-2	193	197
14	-2	132	-135
16	-2	145	-148
17	-2	95	-96
18	-2	170	-162
20	-2	110	-110
21	-2	83	-115
23	-2	159	-148
25	-2	132	-123
1	3	248	-307
3	3	313	-383
4	3	112	-109
5	3	125	-116
6	3	164	-166
8	3	159	-162
9	3	68	78
10	3	135	-158
11	3	230	228
13	3	174	195
15	3	204	182
17	3	215	227
19	3	150	114
21	3	62	73
22	3	100	86
2	-3	310	264
3	-3	177	128
7	-3	176	-153
10	-3	436	337
12	-3	312	260
14	-3	148	162
16	-3	109	121
0	4	209	-227
2	4	246	-297
3	4	88	120
4	4	155	-161
5	4	144	173
6	4	65	-80
7	4	115	142
9	4	67	51
10	4	88	114
12	4	168	184
14	4	154	154
16	4	234	233
18	4	225	187
20	4	63	86
0	-4	43	-16
1	-4	206	156
2	-4	242	-191
3	-4	251	231
4	-4	272	-216
5	-4	106	103
6	-4	110	-89
7	-4	302	285
9	-4	179	134
12	-4	88	72
13	-4	64	86
14	-4	229	192
15	-4	122	125
16	-4	284	246
18	-4	166	158
20	-4	94	101
21	-4	103	-85
22	-4	96	80
23	-4	75	-79
1	5	69	82
3	5	98	-108
4	5	196	199
6	5	202	221
8	5	175	178
10	5	137	131
12	5	114	130
13	5	114	127
14	5	133	113
15	5	140	131
17	5	71	102
1	-5	345	-313
3	-5	322	-261
5	-5	190	-152
9	-5	62	80
10	-5	63	-69
11	-5	112	108
13	-5	179	168
15	-5	287	248
17	-5	266	230
19	-5	166	126
0	6	165	193
2	6	104	113
5	6	210	225
7	6	223	233
9	6	208	190
11	6	204	168
13	6	147	143
0	-6	272	-212
2	-6	247	-192
3	-6	151	-104
4	-6	108	-112
5	-6	215	-173
7	-6	192	-134
9	-6	320	-264
11	-6	186	-162
12	-6	119	97
14	-6	150	138
16	-6	93	107
1	7	103	124
3	7	73	110
4	7	102	125
6	7	78	106
8	7	87	109
10	7	87	101
13	7	65	-66
15	7	90	-102
2	-7	147	-143
4	-7	240	-233
6	-7	291	-223
8	-7	311	-225
10	-7	266	-204
0	8	65	68
2	8	112	119
4	8	132	130
0	-8	153	121
2	-8	137	125
3	-8	153	-127
5	-8	254	-196
7	-8	189	-144
9	-8	130	-113
10	-8	64	-59
11	-8	62	-84
12	-8	60	-86
13	-8	58	-67
18	-8	35	-59
1	-9	108	103
3	-9	88	99
0	-10	49	72
1	-10	84	27
2	-10	83	76
3	-10	47	82
	H= 5		
1	0	441	-550
2	0	368	-403
3	0	201	-173
4	0	619	-560
6	0	375	-396
8	0	269	-245
9	0	122	-85
10	0	292	-235
11	0	56	71
12	0	165	-140
15	0	88	75
17	0	111	114
19	0	131	121
20	0	67	69
22	0	103	101
24	0	87	86
0	1	442	-541
1	1	59	83
2	1	393	-417
3	1	88	80
4	1	312	-322
6	1	193	-171
8	1	224	-196
9	1	123	-90
10	1	67	76
11	1	152	-157
12	1	244	223
13	1	61	-91
14	1	153	157
16	1	171	155
18	1	234	229
20	1	220	176
22	1	40	45
2	-1	268	278
3	-1	604	-610
4	-1	101	85
5	-1	630	-534
6	-1	214	155
7	-1	445	-409
9	-1	399	-385
11	-1	323	-294
13	-1	175	-162
15	-1	65	-90
17	-1	51	-72
20	-1	42	-36
21	-1	126	122
23	-1	112	115
25	-1	84	90
1	2	411	-430
2	2	156	173
3	2	293	-307
4	2	311	298
6	2	368	351
7	2	299	-284
8	2	265	299
9	2	57	-60
10	2	172	186
11	2	179	184
12	2	152	156
13	2	158	127
14	2	90	89
15	2	193	184
17	2	225	218
19	2	203	184
21	2	101	86
1	-2	325	370
2	-2	291	-231
3	-2	428	373
5	-2	314	313
6	-2	183	-155
7	-2	115	108
8	-2	411	-381
9	-2	132	-143
10	-2	257	-231
12	-2	89	-82
13	-2	171	-136
14	-2	108	-112
15	-2	267	-244
16	-2	126	-122
17	-2	272	-240
18	-2	42	-40
19	-2	128	-121
20	-2	71	80
21	-2	72	-70
0	3	314	-325
2	3	215	-207
3	3	316	305
4	3	108	-133
5	3	328	343
7	3	241	257
8	3	35	-59
9	3	236	227
11	3	207	211
13	3	129	140
15	3	99	90
16	3	130	114
18	3	104	98
0	-3	641	611
1	-3	95	-59
2	-3	625	561
4	-3	448	427
6	-3	409	391
8	-3	54	63
9	-3	78	87
10	-3	33	-10
12	-3	86	-82
13	-3	38	-42
14	-3	257	-237
16	-3	289	-272
18	-3	235	-209
20	-3	144	-135
22	-3	65	-64
1	4	137	123
2	4	177	196
3	4	123	151
4	4	215	232
6	4	140	131
8	4	211	185
10	4	218	207
12	4	146	146
14	4	140	141
16	4	100	108
17	4	60	-66
19	4	68	-80
1	-4	251	302
2	-4	176	117
3	-4	137	172
4	-4	267	223
5	-4	123	121
6	-4	135	130
7	-4	32	19
8	-4	191	146
10	-4	210	177
12	-4	55	83
13	-4	147	-149
14	-4	41	60
15	-4	187	-198
16	-4	116	94
17	-4	143	-136
19	-4	98	-108
21	-4	96	-97
22	-4	89	-76
0	5	218	211
2	5	220	260
4	5	153	161
6	5	79	80
7	5	65	55
9	5	112	108
11	5	67	81
12	5	46	-87
14	5	113	-117
16	5	182	-183
18	5	169	-152
0	-5	199	-154
2	-5	33	-44
3	-5	202	180
5	-5	287	278
6	-5	43	67
9	-5	232	188
10	-5	88	82
11	-5	170	171
13	-5	215	191
15	-5	182	166
17	-5	41	62
1	6	201	212
3	6	237	250
5	6	175	155
7	6	112	108
11	6	43	-67
12	6	86	-75
13	6	82	-81
14	6	92	-58
1	-6	283	-229
2	-6	93	89
3	-6	286	-237
4	-6	231	182
5	-6	160	-142
6	-6	310	242
7	-6	80	-75
8	-6	195	178
10	-6	125	124
12	-6	176	143
13	-6	73	68
14	-6	67	74
15	-6	169	139
17	-6	157	141
19	-6	99	99
20	-6	81	-61
21	-6	54	45
0	7	62	36
2	7	124	127
3	7	62	-72
4	7	97	100
5	7	148	-135
7	7	177	-161
9	7	146	-124
11	7	81	-75
13	7	66	-72
14	7	30	-43
15	7	52	-56
16	7	61	-62
0	-7	310	-251
2	-7	296	-230
3	-7	41	62
4	-7	231	-221
5	-7	156	126
6	-7	137	-113
7	-7	137	117
12	-7	41	62
13	-7	40	46
14	-7	137	129
16	-7	170	145
18	-7	131	111
1	8	37	-63
2	8	82	-70
4	8	143	-141
8	8	96	-109
10	8	79	-90
12	8	58	-72
1	-8	136	-140
2	-8	83	-78
3	-8	177	-161
4	-8	148	-122
5	-8	148	-133
6	-8	41	-51
8	-8	57	-66
10	-8	133	-119
12	-8	36	-49
13	-8	34	57
15	-8	113	95
0	9	95	-117
2	9	82	-86
0	-9	76	-69
3	-9	149	-137
5	-9	151	-133
7	-9	131	-110
9	-9	140	-135
11	-9	111	-95
1	-10	58	56
3	-10	94	87
4	-10	91	-88
5	-10	62	44
6	-10	87	-88
	H= 6		
1	0	261	262
2	0	71	-128
3	0	253	269
4	0	277	-147
5	0	164	177
6	0	254	-213
7	0	243	206
9	0	293	275
10	0	162	148
11	0	93	84
14	0	78	64
16	0	248	162
18	0	289	199
20	0	218	149
1	1	103	-69
2	1	203	192
3	1	106	89
4	1	318	277
6	1	331	305
7	1	140	-107
8	1	148	269
9	1	61	-58
10	1	330	306
12	1	179	149
2	-1	114	-107
3	-1	260	-254
4	-1	86	-126
5	-1	217	-185
6	-1	66	-79
8	-1	115	-129
9	-1	147	128
10	-1	55	-32
11	-1	139	132
13	-1	327	271
15	-1	375	319
17	-1	128	232
19	-1	270	179
1	2	158	171
2	2	85	105
3	2	173	279
4	2	178	161
5	2	224	226
6	2	187	198
7	2	107	99
8	2	92	111
9	2	263	239
11	2	315	255
12	2	110	-135
13	2	196	161
14	2	202	-159
15	2	84	62
16	2	207	-161
18	2	166	-129
19	2	81	-53
20	2	77	-71
21	2	146	-101
0	-2	576	-367
1	-2	209	-90
2	-2	268	-284
3	-2	427	-306
4	-2	374	-300
5	-2	276	-250
6	-2	271	-206
7	-2	312	-234
9	-2	245	-159
10	-2	95	80
11	-2	240	-195
12	-2	250	183
13	-2	152	-123
14	-2	407	342
16	-2	320	251
18	-2	134	131
1	3	62	229
3	3	142	158
5	3	95	91
13	3	205	-167
15	3	224	-172
17	3	311	-235
19	3	268	-210
1	-3	126	89
2	-3	144	-70
4	-3	325	-256
6	-3	465	-402
8	-3	346	-318
10	-3	149	-155
12	-3	217	-193
13	-3	184	141
14	-3	163	-95
0	-4	396	266
2	-4	365	262
3	-4	197	-134
4	-4	233	154
5	-4	268	-171
7	-4	289	-230
8	-4	190	-136
9	-4	87	-96
10	-4	89	-100
11	-4	158	-129
12	-4	93	-103
13	-4	95	-91
14	-4	165	-134
16	-4	212	-120
18	-4	128	-101
1	-5	388	254
3	-5	422	293
5	-5	376	256
13	-5	191	-159
15	-5	327	-238
17	-5	217	-163
19	-5	177	-124
21	-5	162	-102
4	-6	227	183
5	-6	209	137
6	-6	188	135
7	-6	134	87
9	-6	165	117
11	-6	165	115
12	-6	94	-51
13	-6	131	82
14	-6	275	-196
16	-6	233	-180
20	-6	135	-95
8	-7	133	164
10	-7	276	196
12	-7	152	131
14	-7	81	77
15	-7	77	-55
	H= 7		
1	0	298	273
2	0	49	104
3	0	172	274
4	0	187	151
5	0	343	319
7	0	157	146
8	0	208	215
10	0	207	182
12	0	249	229
13	0	141	-108
14	0	145	121
15	0	188	-154
17	0	131	-94
19	0	124	-88
20	0	105	-69
22	0	109	-93
1	1	84	78
2	1	182	151
3	1	78	55
4	1	221	205
5	1	51	-69
6	1	158	146
8	1	74	75
3	-1	128	104
4	-1	82	70
5	-1	260	258
6	-1	69	55
7	-1	391	369
8	-1	185	143
9	-1	327	325
11	-1	314	300
12	-1	175	-127
13	-1	190	148
14	-1	151	-110
1	2	262	272
2	2	180	-132
3	2	120	76
4	2	140	-143
5	2	186	151
6	2	125	-126
7	2	150	139
8	2	225	-209
10	2	307	-288
11	2	144	-110
12	2	205	-187
13	2	86	-49
14	2	51	-70
15	2	72	-72
17	2	100	-109
19	2	114	-127
3	-2	105	-182
4	-2	162	197
5	-2	157	-126
6	-2	300	299
8	-2	354	330
9	-2	84	63
10	-2	314	293
12	-2	254	235
13	-2	183	168
14	-2	61	100
15	-2	191	139
17	-2	229	92
19	-2	99	87
21	-2	74	72
0	3	149	133
3	3	200	-180
5	3	336	-301
7	3	334	-281
8	3	117	-82
9	3	286	-270
10	3	121	-132
11	3	301	-241
13	3	87	-120
2	4	151	-116
3	4	161	-126
4	4	229	-178
5	4	193	-160
6	4	193	-165
8	4	150	-172
10	4	172	-195
12	4	176	-123
14	4	70	-46
15	4	68	63
17	4	98	78
0	5	200	-190
2	5	217	-194
4	5	202	-166
6	5	122	-139
1	6	112	-152
3	6	122	-123
4	6	50	68
5	6	70	-77
6	6	98	109
8	6	67	85
10	6	78	65
11	6	43	45
12	6	92	84
13	6	78	62
	H= 8		
3	0	99	-104
4	0	288	246
5	0	155	-136
6	0	122	116
7	0	132	-128
9	0	197	-202
11	0	159	-140
12	0	69	-42
13	0	94	-94
14	0	134	-128
15	0	98	-101
16	0	116	-90
17	0	55	-38
18	0	86	-71
19	0	37	42
20	0	49	-49
21	0	79	67
2	-1	101	-96
3	-1	279	287
4	-1	54	-56
5	-1	177	204
6	-1	57	75
7	-1	35	30
8	-1	82	-48
10	-1	186	-194
11	-1	90	-82
13	-1	226	-205
15	-1	229	-217
17	-1	175	-180
19	-1	151	-139
21	-1	80	-86
2	1	58	-64
4	1	116	-119
5	1	56	48
6	1	160	-185
7	1	29	23
8	1	221	-223
9	1	33	-39
10	1	296	-307
12	1	202	-204
0	-2	179	200
1	-2	51	-53
2	-2	197	220
4	-2	217	254
6	-2	249	250
7	-2	145	171
8	-2	70	51
9	-2	62	70
10	-2	75	-93
11	-2	179	147
12	-2	176	-155
13	-2	187	170
14	-2	185	-180
16	-2	175	-169
18	-2	142	-131
20	-2	98	-93
22	-2	91	-62
0	2	55	-127
1	2	83	-40
2	2	117	-116
3	2	40	-37
4	2	142	-126
5	2	137	-149
6	2	116	-96
7	2	197	-210
8	2	35	-41
9	2	211	-198
10	2	53	63
11	2	123	-155
12	2	128	128
13	2	56	-86
14	2	163	162
16	2	132	151
18	2	87	97
6	-3	183	169
8	-3	267	300
9	-3	77	-87
10	-3	202	189
12	-3	189	198
14	-3	92	100
1	3	197	-221
2	3	48	-48
3	3	265	-260
5	3	200	-203
8	3	54	42
9	3	78	55
10	3	39	25
11	3	105	95
15	3	138	131
17	3	127	118
0	-4	100	-104
1	-4	177	141
2	-4	185	-143
3	-4	274	260
4	-4	243	-225
5	-4	194	179
6	-4	94	-91
7	-4	263	229
8	-4	62	43
9	-4	207	202
11	-4	169	178
13	-4	105	115
14	-4	83	84
16	-4	48	64
18	-4	56	55
19	-4	36	-48
20	-4	33	39
21	-4	67	-66
0	4	242	-265
2	4	168	-165
3	4	138	124
4	4	76	-86
5	4	191	168
6	4	75	-94
7	4	158	145
8	4	40	-48
9	4	105	109
11	4	110	96
12	4	66	90
14	4	61	69
16	4	65	69
1	-5	308	-286
3	-5	224	-222
4	-5	103	97
5	-5	181	-174
6	-5	116	100
8	-5	89	94
11	-5	47	63
13	-5	114	115
15	-5	131	134
17	-5	116	107
19	-5	110	97
2	5	56	44
4	5	123	132
6	5	137	148
8	5	128	136
10	5	140	152
12	5	133	146
14	5	54	52
0	-6	96	-131
1	-6	95	-70
2	-6	188	-170
3	-6	56	-56
4	-6	253	-227
6	-6	142	-115
9	-6	94	-92
10	-6	46	59
11	-6	81	-91
12	-6	92	98
14	-6	144	133
16	-6	115	112
18	-6	107	98
3	6	64	11
4	6	63	74
5	6	62	49
6	6	100	57
9	6	82	83
11	6	88	86
1	-7	85	-78
2	-7	69	-67
3	-7	44	-39
4	-7	66	-54
6	-7	156	-137
8	-7	185	-194
10	-7	160	-162
12	-7	118	-94
13	-7	37	48
14	-7	38	-41
15	-7	54	65
0	-8	66	40
2	-8	56	53
3	-8	65	-77
5	-8	92	-73
7	-8	110	-96
9	-8	126	-125
11	-8	110	-105
12	-8	30	-39
13	-8	65	-55
1	-9	163	137
3	-9	132	101
5	-9	76	61
6	-9	30	-32
7	-9	32	36
8	-9	54	-48

peak nor the negative regions, so these were attributed to series termination effects.

The final calculated and observed structure factors are listed in Table I. The atomic coordinates and isotropic temperature factors are given in Table II.

TABLE II
ATOMIC PARAMETERS OF $Y(C_5H_7O_2)_3 \cdot 3H_2O$ WITH ESTIMATED STANDARD DEVIATIONS

	x	*y*	*z*	*B*, A²
Y	0.2690 (2)	0.02950 (6)	0.1180 (2)	3.04 (5)
O(1)	0.8639 (12)	0.0078 (5)	0.1161 (12)	3.5 (2)
O(2)	0.6278 (14)	0.4888 (6)	0.0538 (16)	5.3 (3)
O(3)	0.4719 (12)	0.0512 (5)	0.2286 (14)	3.6 (2)
O(4)	0.2882 (12)	0.1284 (5)	0.0269 (14)	4.0 (2)
O(5)	0.0747 (11)	0.0786 (5)	0.1266 (13)	3.7 (2)
O(6)	0.2797 (13)	0.0710 (6)	0.3763 (14)	4.3 (3)
O(7)	0.3392 (13)	0.4588 (5)	0.2680 (14)	4.3 (2)
O(8)	0.1294 (12)	0.4349 (5)	0.3866 (14)	4.3 (2)
O(9)	0.8702 (14)	0.4694 (5)	0.3881 (15)	4.5 (3)
C(1)	0.2914 (21)	0.2338 (9)	0.9988 (25)	5.6 (4)
C(2)	0.1302 (19)	0.1836 (8)	0.1235 (21)	4.1 (4)
C(3)	0.0636 (18)	0.1358 (8)	0.1608 (21)	4.1 (4)
C(4)	0.2339 (23)	0.1775 (9)	0.0523 (24)	4.0 (4)
C(5)	−0.0636 (23)	0.1477 (10)	0.2382 (28)	6.2 (5)
C(6)	0.5135 (21)	0.0903 (8)	0.3305 (23)	4.6 (4)
C(7)	0.4592 (21)	0.1275 (9)	0.4271 (25)	5.1 (4)
C(8)	0.3459 (20)	0.1150 (8)	0.4478 (23)	4.9 (4)
C(9)	0.6505 (23)	0.0990 (10)	0.3458 (27)	6.2 (5)
C(10)	0.7870 (21)	0.3479 (10)	0.0702 (26)	5.2 (5)
C(11)	0.1611 (19)	0.3801 (8)	0.3796 (21)	3.7 (3)
C(12)	0.0775 (21)	0.3319 (9)	0.4209 (23)	5.0 (4)
C(13)	0.2664 (18)	0.3607 (8)	0.3318 (20)	3.8 (3)
C(14)	0.3602 (20)	0.3990 (8)	0.2807 (22)	4.0 (4)
C(15)	0.4639 (23)	0.3739 (10)	0.2213 (26)	5.6 (5)

Discussion of Structure

Figure 1 shows one complete molecule projected onto (001). The bond lengths and bond angles are listed in Table III,[8] and the average dimensions of an yttrium acetylacetonate ring are shown in Figure 2. Each yttrium atom is bonded to eight oxygen atoms, contributed by three bidentate acetylacetonate groups and two water molecules [O(1) and O(9)].

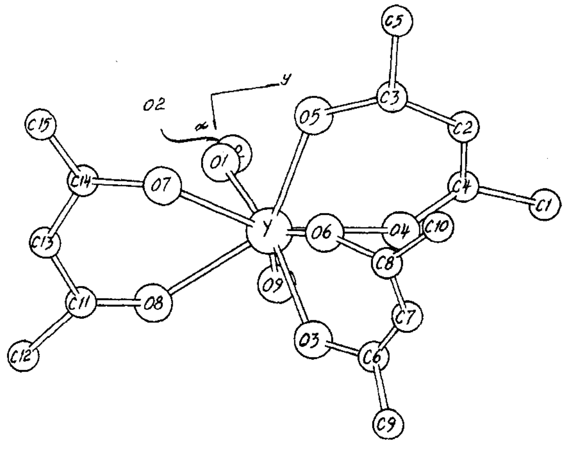

Figure 1.—Projection of one molecule onto (001).

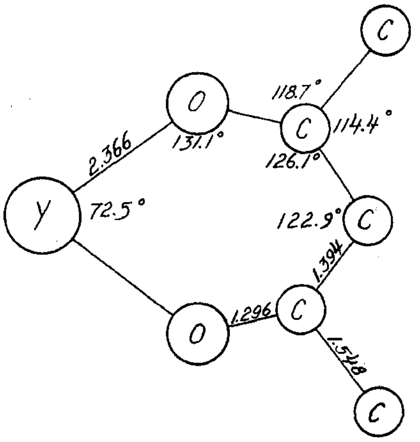

Figure 2.—Average dimensions of a ring in $Y(C_5H_7O_2)_3 \cdot 3H_2O$.

The average yttrium to carbonyl oxygen distance is some 0.04 A shorter than the value of 2.409 A for the average distance of yttrium to coordinated water. However, the ranges of values do not allow any significance to be attached to this result. The average O–O separation in a ring is 2.80 A, and the average ring dimensions are comparable to those found in other acetylacetonates.[9]

The mean deviations of the carbon and oxygen atoms from the least-squares planes of the acetylacetonate groups are 0.084, 0.071, and 0.034 A for the rings containing O(3)–O(6), O(4)–O(5), and O(7)–O(8), respectively. The deviations of the yttrium ion from the planes are 0.73, 0.68, and 0.45 A, respectively. Each ring is folded about a line passing through its two oxygen atoms; the mean angle of folding is 18.9°. The O(3)–O(6) ring is folded away from the O(7)–O(8) ring and toward the O(4)–O(5) ring. The O(4)–O(5) ring is folded away from the coordinated water molecules, and the O(7)–O(8) ring is folded toward the water molecules. The average sum of the interior angles of a ring is 709.7°, as compared with 720° required for planarity.

A hydrogen bond (2.80 A) links the coordinated water

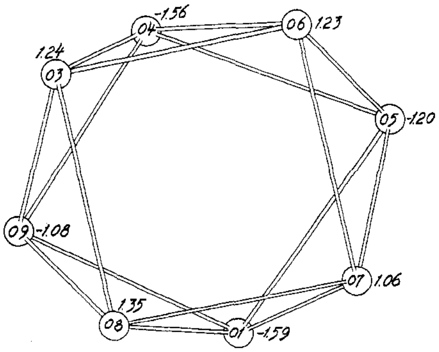

Figure 3.—Coordination polyhedron formed by the eight oxygens bonded to yttrium. The distance of each atom from the central plane is given.

---

[8] Distances, angles, and standard deviations were calculated using W. R. Busing, K. O. Martin, and H. A. Levy's "ORFFE, a Fortran Crystallographic Function and Error Program," ORNL-TM-306, 1964.

[9] R. B. Roof, Jr., *Acta Cryst.*, **9**, 781 (1956); R. P. Dodge, D. H. Templeton, and A. Zalkin, *J. Chem. Phys.*, **35**, 55 (1961); H. Montgomery and E. C. Lingafelter, *Acta Cryst.*, **16**, 748 (1963); J. V. Silverton and J. L. Hoard, *Inorg. Chem.*, **2**, 243 (1963); B. Morosin and J. R. Brothovde, *Acta Cryst.*, **17**, 705 (1964).

TABLE III

BOND LENGTHS AND ANGLES

Lengths, A

Y–O(1)	2.397 ± 0.011	O(8)–C(11)	1.27 ± 0.02
Y–O(3)	2.451 ± 0.012	C(2)–C(3)	1.36 ± 0.03
Y–O(4)	2.348 ± 0.012	C(2)–C(4)	1.41 ± 0.03
Y–O(5)	2.456 ± 0.013	C(7)–C(8)	1.35 ± 0.03
Y–O(6)	2.349 ± 0.012	C(7)–C(6)	1.38 ± 0.03
Y–O(7)	2.302 ± 0.014	C(13)–C(14)	1.48 ± 0.03
Y–O(8)	2.394 ± 0.013	C(13)–C(11)	1.38 ± 0.03
Y–O(9)	2.421 ± 0.015	C(1)–C(4)	1.51 ± 0.03
O(1)–O(2)	2.802 ± 0.018	C(3)–C(5)	1.70 ± 0.03
O(4)–C(4)	1.29 ± 0.02	C(6)–C(9)	1.54 ± 0.03
O(5)–C(3)	1.31 ± 0.02	C(8)–C(10)	1.56 ± 0.03
O(3)–C(6)	1.25 ± 0.02	C(11)–C(12)	1.51 ± 0.03
O(6)–C(8)	1.31 ± 0.02	C(14)–C(15)	1.46 ± 0.03
O(7)–C(14)	1.35 ± 0.02		

Angles, deg

O(3)–Y–O(6)	70.8 ± 0.4	C(6)–C(7)–C(8)	118.6 ± 2.0
O(4)–Y–O(5)	74.3 ± 0.4	C(3)–C(2)–C(4)	123.4 ± 1.9
O(7)–Y–O(8)	72.5 ± 0.5	C(11)–C(13)–C(14)	126.6 ± 1.6
Y–O(3)–C(6)	128.7 ± 1.3	O(3)–C(6)–C(9)	112.8 ± 1.9
Y–O(6)–C(8)	130.5 ± 1.3	O(6)–C(8)–C(10)	115.0 ± 2.0
Y–O(4)–C(4)	131.4 ± 1.3	O(4)–C(4)–C(1)	110.2 ± 1.0
Y–O(5)–C(3)	123.8 ± 1.1	O(5)–C(3)–C(5)	110.7 ± 1.6
Y–O(7)–C(14)	137.4 ± 1.3	O(7)–C(14)–C(15)	119.0 ± 1.8
Y–O(8)–C(11)	134.4 ± 1.4	O(8)–C(11)–C(12)	118.4 ± 1.8
O(3)–C(6)–C(7)	132.8 ± 2.2	C(9)–C(6)–C(7)	114.4 ± 1.9
O(6)–C(8)–C(7)	124.7 ± 2.1	C(10)–C(8)–C(7)	120.3 ± 1.9
O(4)–C(4)–C(2)	127.4 ± 1.9	C(1)–C(4)–C(2)	118.4 ± 1.8
O(4)–C(3)–C(2)	128.6 ± 1.9	C(5)–C(3)–C(2)	120.1 ± 1.8
O(7)–C(14)–C(13)	117.9 ± 1.8	C(12)–C(11)–C(13)	116.5 ± 1.6
O(8)–C(11)–C(13)	125.0 ± 1.9	C(15)–C(14)–C(13)	122.5 ± 1.7

TABLE IV

DIMENSIONS OF COORDINATION POLYHEDRON

Distances, A[a]

O(3)–O(6)	2.72	O(9)–O(4)	2.71	O(8)–O(9)	3.02
O(6)–O(7)	2.98	O(3)–O(4)	2.97	O(9)–O(3)	2.93
O(7)–O(8)	2.78	O(4)–O(6)	3.23	O(3)–O(7)	4.06
O(8)–O(3)	2.92	O(6)–O(5)	2.83	O(6)–O(8)	3.99
O(4)–O(5)	2.90	O(5)–O(7)	2.91	O(4)–O(1)	3.57
O(5)–O(1)	2.83	O(7)–O(1)	2.99	O(5)–O(9)	4.32
O(1)–O(9)	2.76	O(1)–O(8)	3.23		

Angles, deg[b]

O(8)–O(3)–O(6)	90.1	O(4)–O(6)–O(5)	56.8	O(3)–O(9)–O(8)	60.6
O(3)–O(6)–O(7)	90.7	O(6)–O(5)–O(4)	68.6	O(9)–O(3)–O(4)	54.6
O(6)–O(7)–O(8)	87.6	O(5)–O(4)–O(6)	54.7	O(3)–O(4)–O(9)	61.9
O(7)–O(8)–O(3)	91.0	O(6)–O(5)–O(7)	62.6	O(4)–O(9)–O(3)	63.5
O(4)–O(5)–O(1)	74.8	O(5)–O(7)–O(6)	57.4	O(5)–O(7)–O(1)	60.6
O(5)–O(1)–O(9)	97.6	O(7)–O(6)–O(5)	60.1	O(7)–O(1)–O(5)	58.4
O(1)–O(9)–O(4)	81.6	O(7)–O(1)–O(8)	52.9	O(1)–O(5)–O(7)	60.9
O(9)–O(4)–O(5)	100.6	O(1)–O(8)–O(7)	59.0	O(8)–O(1)–O(9)	56.1
O(3)–O(4)–O(6)	51.9	O(8)–O(7)–O(1)	68.1	O(1)–O(9)–O(8)	70.4
O(4)–O(3)–O(6)	69.0	O(9)–O(8)–O(3)	61.0	O(9)–O(8)–O(1)	53.5
O(4)–O(6)–O(3)	59.2	O(8)–O(3)–O(9)	58.4		

[a] Standard deviations, 0.02 A. [b] Standard deviations, 0.4–0.5°.

molecule O(1) to the uncoordinated water molecule O(2). This O(2) is 2.90 A from another O(2), related by a center of symmetry, which is linked to O(1) of another molecule. The molecules are thus connected in pairs by the sequence Y–O(1)–O(2)–O(2)′–O(1)′–Y′, where the primed and unprimed atoms are related by a center of symmetry. Interpretation of the O(2)–O(2)′ linkage as a hydrogen bond implies a disordered structure in which the center of symmetry is preserved by randomly assigning the shared hydrogen atom to one oxygen atom or the other. No significant accumulation of electron density in this vicinity was observed on the difference map, although there was an indication of the hydrogen atom contributed by O(1) to the O(1)–O(2) bond.

Other than the hydrogen bonds, the shortest intermolecular distances are methyl group contacts of 3.96 A.

The polyhedron formed about the yttrium ion by the eight coordinated oxygen atoms is a distorted square antiprism. In Figure 3 the coordinates have been transformed to a Cartesian system, and the view is along the vector from the yttrium ion to the center of the quadrilateral O(3)–O(6)–O(7)–O(8). The distances and angles of this polyhedron are listed in Table IV. In addition to distortions in lengths and angles, the O(1)–O(5)–O(4)–O(9) quadrilateral is folded about the O(1)–O(4) diagonal.

In spite of the deviations from $\bar{8}2m$ symmetry, the average parameters for this polyhedron agree well with those calculated by Hoard and Silverton for minimization of the ligand repulsive energy.[10] In the notation of Hoard and Silverton, we have $l = 1.270$, $s = 1.195$, $l/s = 1.064$, and $\theta = 57.1°$, as compared with their values 1.258, 1.190, 1.057, and 57.3°.

The distortion of the polyhedron can be attributed to the presence of two chemically different ligands bonded to the same central metal atom. The geometric constraints of the bidentate acetylacetonate groups *vs.* the monodentate water molecules undoubtedly contribute to the deformation, as does the difference in the mutual repulsion of the two ligands.

(10) J. L. Hoard and J. V. Silverton, *Inorg. Chem.*, **2**, 235 (1963).

CONTRIBUTION FROM THE CHEMISTRY DIVISION, RESEARCH DEPARTMENT,
U. S. NAVAL ORDNANCE TEST STATION, CHINA LAKE, CALIFORNIA

Crystal Structure of Potassium Permanganate

BY GUS J. PALENIK[1]

Received July 14, 1966

Potassium permanganate forms deep purple, orthorhombic crystals with $a = 9.105$, $b = 5.720$, and $c = 7.425$ A. The space group is Pnma and there are four molecules per unit cell. The intensity data were measured using an automatic diffractometer and molybdenum radiation. The structure was refined by least-squares methods with anisotropic thermal parameters for each atom. The final residual (R) was 4.6% for the 561 observed reflections used in the analysis. After a correction for libration, the three independent Mn–O distances are 1.622, 1.625, and 1.634 A. The average Mn–O distance in the ion is 1.629 ± 0.008 A. The average O–Mn–O angle is 109.4 ± 0.7°. Therefore, the permanganate ion can be described as a manganese atom surrounded by four oxygen atoms at the corners of a regular tetrahedron.

Introduction

Manganese is unique in that it forms three oxy anions: permanganate, manganate, and hypomanganate. They are all tetrahedral MnO_4 ions with a charge of -1, -2, and -3, respectively. The permanganate ion has been the subject of several papers[2] discussing the electronic structure of the ion. It is generally agreed that the extra electrons in the manganate and hypomanganate ions occupy an antibonding orbital. The Mn–O bond should therefore lengthen in going from permanganate to manganate to hypomanganate. A study of these ions should provide a comparison of the effect of the antibonding electrons on the size and shape of the MnO_4 tetrahedron.

The crystal structure of potassium permanganate was originally studied by Mooney,[3] who reported the ion to be "a nearly regular tetrahedron" with Mn–O distances of 1.68, 1.52, 1.58, and 1.58 A, the average distance being 1.59 A. More recently Ramaseshan, Venkatesan, and Mani,[4] in the course of studying anomalous dispersion with zonal data, reported Mn–O distances of 1.56, 1.56, 1.54, and 1.54 A with an average of 1.55 A. Since neither previous study was sufficiently precise to detect small changes in bond distances, a redetermination of the crystal structure of potassium permanganate was undertaken.

Collection and Reduction of the X-Ray Data

Crystals were grown by slowly cooling a warm aqueous solution of potassium permanganate in a dewar. A crystal was cleaved to give a parallelepiped of approximately 0.15 × 0.15 × 0.15 mm. The crystal was mounted on a glass fiber with the $\bar{b}$ axis parallel to the fiber axis and dipped in liquid nitrogen to minimize extinction effects.

The unit cell and intensity measurements were carried out with the General Electric single-crystal orienter, using molybdenum radiation (α_1 0.70926, α_2 0.71354 A). The unit cell dimensions were determined using a very narrow beam. The α_1–α_2 doublet could be resolved for values greater than about 27°, and the average values from these measurements are: $a = 9.105 \pm 0.005$, $b = 5.720 \pm 0.003$, $c = 7.425 \pm 0.004$ A. The reported density[5] is 2.703 g cm^{-3}, and the value calculated for four molecules per unit cell is 2.714 g cm^{-3}.

A wide beam was used for the intensity measurements which were made with a scintillation counter. A linear amplifier–pulse height selector combination together with a zirconium filter provided almost mono-

---

[1] Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada.

[2] (a) H. Basch, A. Viste, and H. B. Gray, *J. Chem. Phys.*, **44**, 10 (1966); (b) L. Oleari, G. de Michelis, and L. di Sipio, *Mol. Phys.*, **10**, 111 (1966), and references therein.

[3] R. C. L. Mooney, *Phys. Rev.*, **37**, 1306 (1931).

[4] S. Ramaseshan, K. Venkatesan, and N. V. Mani, *Proc. Indian Acad. Sci.*, **A46**, 95 (1957).

[5] "Handbook of Chemistry and Physics," 44th ed, Chemical Rubber Publishing Co., Cleveland, Ohio, 1963, p 632.