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

The Crystal and Molecular Structure of Tris(acetylacetonato)aquoytterbium(III) Hemibenzene[1]

BY EDWARD D. WATKINS, II, JAMES A. CUNNINGHAM, THEODORE PHILLIPS, II,
DONALD E. SANDS,[2] AND WILLIAM F. WAGNER

Received June 13, 1968

The crystal structure of tris(acetylacetonato)aquoytterbium(III) hemibenzene, $Yb(C_5H_7O_2)_3(H_2O)\cdot 0.5C_6H_6$, has been determined from three-dimensional X-ray diffraction data. Two formula units are contained in a triclinic unit cell of space group $P\bar{1}$ and dimensions $a = 8.728 \pm 0.009$ Å, $b = 8.435 \pm 0.009$ Å, $c = 15.623 \pm 0.016$ Å, $\alpha = 95.95 \pm 0.02°$, $\beta = 107.72 \pm 0.09°$, and $\gamma = 96.18 \pm 0.09°$. A total of 2625 intensities above background were collected by counter methods. The structure was refined by least-squares methods to a conventional R factor of 0.080; during the final cycle of refinement, corrections for anomalous dispersion were included. The ytterbium atom is surrounded by seven oxygen atoms at the vertices of a capped trigonal prism. These tris(acetylacetonato)aquoytterbium(III) molecules are linked in pairs by hydrogen bonds. The benzene molecule occupies a center of symmetry; all of the intermolecular contacts involving benzene carbon atoms and acetylacetonate carbon or oxygen atoms exceed 3.5 Å.

Introduction

The preceding paper described the structure of $Yb(C_5H_7O_2)_3(H_2O)$.[3] In the course of preparing and studying the hydrates, it was observed that the crystals recovered from a solution of $Yb(C_5H_7O_2)_3\cdot 3H_2O$ in benzene or acetone contained one molecule of the solvent for two molecules of $Yb(C_5H_7O_2)_3(H_2O)$.[4] The infrared spectra of these solvates differed from the spectrum of the monohydrate only in the appearance of additional bands characteristic of the solvent molecule. Interest in the possible effect of crystal packing on the molecular geometry prompted the determination of the structure of this benzene solvate.

Experimental Section

Crystals of $Yb(C_5H_7O_2)_3(H_2O)\cdot 0.5C_6H_6$ were prepared by Dr. Richardson.[4] A crystal of dimensions 0.28 × 0.23 × 0.20 mm was sealed in a glass capillary with the 0.28-mm dimension parallel to the capillary, and approximate unit cell dimensions and the diffraction symmetry were obtained from oscillation, Weissenberg, and precession photographs. Accurate unit cell dimensions and the intensity data were measured on a General Electric X-ray spectrometer equipped with a single-crystal orienter and a scintillation counter. The crystals are triclinic with dimensions (based on Mo Kα = 0.7107 Å) $a = 8.728 \pm 0.009$ Å, $b = 8.435 \pm 0.009$ Å, $c = 15.623 \pm 0.016$ Å, $\alpha = 95.95 \pm 0.02°$, $\beta = 107.72 \pm 0.09°$, and $\gamma = 96.18 \pm 0.09°$. The long dimension of the crystal was parallel to a. With two molecules of $Yb(C_5H_7O_2)_3(H_2O)\cdot 0.5C_6H_6$ per unit cell the calculated density is 1.62 g cm^{-3}.

The intensities were measured manually by the stationary-crystal, stationary-counter technique using Mo Kα radiation and 40-sec counts. The crystal was oriented about a^*, and the settings were calculated for the Kα_1 line (λ 0.70930 Å). Conversion of peak heights to integrated intensities were based upon several intensities measured by a manual ω scan. Background corrections were obtained by computer interpolation of a plot of intensity *vs.* angle, constructed from measurements made with the crystal out of reflecting position. No corrections were made for absorption (μ = 45.9 cm^{-1}) or extinction. The estimated maximum variation in the ratio of any two intensities due to absorption is 10.5%.

It was initially planned to measure all reflections for which 2θ was less than 50°. However, problems in electronic stability which arose during the measurements indicated the advisability of completing the data collection as soon as possible, so the maximum 2θ was decreased to 35°. A total of 2963 reflections were measured, and these included about 60% of the accessible reflections between 35 and 50°. Of the measured reflections, 2625 were at least 7% above background and were classified as observed. The variation in the reflected beam intensity was 32%.

Structure Determination and Refinement

Space group $P\bar{1}$ was assumed and was ultimately confirmed by the structure. Trial ytterbium positions were deduced from the three-dimensional Patterson function. Further interpretation of the Patterson function was not feasible. The ytterbium atoms occupy the $P\bar{1}$ general positions 2(i): $\pm(x, y, z)$ with $x = 0.012$, $y = 0.190$, $z = 0.165$. The ytterbium contributions to the structure factors were calculated, and a difference Fourier synthesis was computed using the 2539 reflections for which $|F_c| \geq 0.40|F_o|$. This difference Fourier map revealed the positions of the seven oxygen atoms. Two additional difference Fourier maps were required before all of the carbon atoms were located.

Least-squares refinements were carried out using a local IBM 360 adaptation of the program of Busing, Martin, and Levy.[5] The refinements included one scale factor, the positional parameters for one ytterbium atom, seven oxygen atoms, and eighteen carbon atoms, anisotropic temperature factors for the ytterbium atom, and isotropic temperature factors for the oxygen and carbon atoms. The atomic form factors used were those of Cromer, Larson, and Waber[6] for neutral ytterbium and from the compilation of Ibers[7] for oxy-

---

[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] Author to whom correspondence should be addressed.

[3] J. A. Cunningham, D. E. Sands, W. F. Wagner, and M. F. Richardson, *Inorg. Chem.*, **8**, 22 (1969).

[4] M. F. Richardson, Ph.D. Dissertation, University of Kentucky, 1967.

[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

MAGNITUDES OF OBSERVED AND CALCULATED STRUCTURE FACTORS FOR OBSERVED DATA, EACH MULTIPLIED BY 10 [$10F(000) = 4680$]

K	L	FO	|FC|
	H=	0	
1	0	232	220
2	0	179	184
3	0	348	355
5	0	380	380
6	0	171	180
7	0	136	150
8	0	117	128
0	1	509	558
1	1	617	648
2	1	727	741
3	1	219	228
4	1	432	424
5	1	234	227
6	1	92	114
7	1	202	219
8	1	32	58
9	1	61	74
-1	1	814	868
-2	1	142	138
-3	1	386	421
-4	1	392	396
-5	1	76	89
-6	1	279	290
-7	1	41	54
-8	1	99	123
-9	1	90	89
0	2	372	409
1	2	811	873
2	2	392	395
3	2	244	249
4	2	480	513
5	2	77	69
6	2	236	244
7	2	96	98
9	2	93	92
-1	2	354	380
-2	2	611	605
-3	2	227	224
-4	2	489	488
-5	2	258	273
-6	2	104	110
-7	2	195	185
-9	2	72	90
0	3	443	496
1	3	102	82
2	3	417	416
3	3	582	614
4	3	250	266
5	3	293	297
6	3	101	107
7	3	29	57
8	3	121	133
-1	3	51	51
-2	3	657	699
-3	3	286	319
-4	3	172	177
-5	3	365	358
-6	3	149	156
-7	3	138	142
-8	3	131	150
0	4	397	388
1	4	199	202
2	4	391	413
3	4	230	222
4	4	299	306
5	4	188	179
6	4	19	34
7	4	136	152
8	4	55	63
9	4	40	52
-1	4	530	576
-2	4	122	120
-3	4	504	557
-4	4	183	180
-6	4	308	308
-7	4	57	75
-8	4	120	139
-9	4	121	133
0	5	163	150
1	5	194	249
2	5	178	150
3	5	330	337
4	5	302	317
5	5	36	46
6	5	159	171
7	5	89	94
8	5	28	46
9	5	43	67
-1	5	393	432
-2	5	440	488
-3	5	70	84
-4	5	435	479
-5	5	141	129
-7	5	196	199
-9	5	127	150
0	6	399	430
1	6	188	205
2	6	206	224
3	6	275	273
4	6	32	31
5	6	212	228
6	6	139	149
8	6	65	85
-2	6	463	483
-3	6	306	329
-4	6	166	187
-6	6	44	33
-8	6	126	140
0	7	338	335
1	7	238	279
2	7	384	415
3	7	62	47
4	7	214	233
5	7	166	161
7	7	102	117
8	7	27	35
-1	7	161	176
-2	7	119	124
-4	7	235	256
-5	7	230	253
-8	7	84	108
-9	7	95	94
0	8	70	33
1	8	376	373
2	8	172	160
3	8	151	160
4	8	214	247
5	8	36	74
6	8	116	119
7	8	57	67
8	8	33	48
-2	8	241	267
-4	8	310	314
-5	8	88	94
-6	8	196	220
-7	8	133	151
0	9	281	308
1	9	184	201
2	9	117	116
3	9	209	211
4	9	79	68
5	9	165	186
6	9	67	87
-1	9	66	62
-2	9	316	313
-3	9	231	239
-5	9	211	226
-7	9	123	133
0	10	277	289
1	10	85	74
2	10	173	164
3	10	79	49
4	10	126	126
5	10	145	159
7	10	61	71
-1	10	288	297
-2	10	78	85
-3	10	236	215
-4	10	149	178
-5	10	98	103
-6	10	174	188
-8	10	54	82
0	11	89	90
1	11	244	239
2	11	100	126
3	11	108	105
4	11	162	150
6	11	68	71
-1	11	257	260
-2	11	186	200
-3	11	54	75
-4	11	175	180
-5	11	131	153
-6	11	132	121
-7	11	111	123
0	12	284	264
1	12	166	176
2	12	62	73
3	12	162	149
4	12	27	37
5	12	84	83
6	12	48	53
-1	12	47	46
-2	12	205	219
-3	12	117	124
-4	12	52	58
-5	12	215	218
-7	12	97	111
-8	12	53	82
0	13	184	183
2	13	179	180
3	13	61	76
4	13	82	71
5	13	81	70
-1	13	261	247
-5	13	107	113
-6	13	86	112
0	14	30	33
1	14	169	152
2	14	103	110
3	14	58	70
4	14	110	106
-1	14	189	195
-5	14	25	35
-7	14	68	91
0	15	153	150
1	15	108	101
2	15	39	36
3	15	127	122
4	15	33	47
-5	15	118	127
0	16	115	96
2	16	112	110
-1	16	102	89
1	17	86	81
-1	17	123	106
-2	17	57	62
5	-4	170	168
6	-5	172	191
7	-6	133	145
5	-6	395	422
3	-7	396	416
6	-7	208	225
1	-8	308	327
3	-8	69	88
9	-8	41	72
4	-9	58	63
8	-9	89	85
6	-14	66	78
	H=	1	
0	0	962	1022
1	0	286	260
2	0	176	174
3	0	338	317
4	0	127	157
5	0	401	427
6	0	48	30
7	0	103	120
8	0	114	136
-1	0	450	409
-2	0	334	297
-3	0	417	399
-5	0	318	323
-6	0	210	218
-7	0	105	115
-8	0	123	139
0	1	175	177
1	1	368	360
2	1	680	694
3	1	116	139
4	1	412	388
5	1	163	161
6	1	165	188
7	1	171	182
8	1	27	52
9	1	58	72
-1	1	585	547
-2	1	519	469
-3	1	450	456
-4	1	359	343
-6	1	238	235
-7	1	57	71
-9	1	99	119
0	2	183	189
1	2	733	754
2	2	129	133
3	2	350	323
4	2	327	324
5	2	123	119
6	2	228	233
7	2	89	93
8	2	57	69
9	2	62	79
-1	2	58	53
-2	2	662	664
-3	2	107	92
-4	2	463	463
-5	2	243	255
-6	2	29	52
-7	2	137	170
-10	2	49	68
0	3	513	502
1	3	67	48
2	3	576	591
3	3	604	626
4	3	81	128
5	3	224	186
6	3	90	75
7	3	50	68
8	3	95	106
-1	3	277	267
-2	3	312	331
-3	3	471	483
-4	3	54	37
-5	3	356	361
-6	3	176	205
-7	3	47	69
-8	3	124	176
0	4	268	285
1	4	424	444
2	4	558	530
4	4	275	268
5	4	195	210
6	4	59	37
7	4	117	114
9	4	51	42
-1	4	478	507
-2	4	202	196
-3	4	302	295
-4	4	461	470
-5	4	137	160
-6	4	287	295
-7	4	75	97
-8	4	104	142
-9	4	92	131
0	5	195	182
1	5	592	594
3	5	369	360
4	5	266	276
5	5	19	17
6	5	157	135
7	5	51	66
-1	5	444	461
-2	5	527	533
-3	5	205	201
-4	5	427	436
-5	5	213	226
-6	5	164	178
-7	5	165	174
-9	5	104	146
0	6	549	557
1	6	262	254
2	6	364	340
3	6	234	211
4	6	19	38
5	6	249	263
6	6	110	128
8	6	56	66
-2	6	436	447
-3	6	353	382
-4	6	34	35
-5	6	341	345
-6	6	73	74
-7	6	164	179
-8	6	147	175
0	7	427	414
1	7	307	292
2	7	335	329
3	7	81	73
4	7	223	211
5	7	172	173
6	7	29	42
7	7	73	75
-1	7	324	332
-2	7	25	59
-3	7	320	356
-4	7	274	274
-5	7	142	156
-6	7	252	283
-8	7	101	112
-9	7	77	101
0	8	151	125
1	8	345	331
2	8	160	143
3	8	148	132
4	8	193	186
6	8	122	135
7	8	42	52
-1	8	324	317
-2	8	278	274
-3	8	83	86
-4	8	285	303
-5	8	100	93
-6	8	178	189
-7	8	207	223
-8	8	54	57
-9	8	67	81
0	9	338	322
1	9	171	156
2	9	113	103
3	9	170	143
4	9	30	54
5	9	123	122
6	9	62	67
-1	9	40	38
-2	9	367	362
-3	9	176	165
-4	9	82	93
-5	9	234	266
-6	9	103	96
-7	9	84	109
-8	9	136	148
0	10	225	205
1	10	72	81
2	10	174	149
4	10	84	95
5	10	103	117
-1	10	307	303
-2	10	79	97
-3	10	226	212
-4	10	179	191
-5	10	130	124
-6	10	211	218
-8	10	99	112
0	11	68	62
1	11	219	184
2	11	75	78
3	11	101	111
4	11	114	100
6	11	47	63
-1	11	219	195
-2	11	205	197
-3	11	62	83
-4	11	296	302
-5	11	130	128
-6	11	106	103
-7	11	110	126
0	12	257	236
1	12	105	88
2	12	43	62
3	12	146	138
5	12	75	75
-4	12	139	135
-5	12	203	189
-7	12	72	96
-8	12	74	94
0	13	160	146
1	13	53	78
2	13	144	135
3	13	43	58
4	13	74	70
5	13	51	50
-1	13	198	194
-5	13	86	110
-6	13	97	109
1	14	149	138
2	14	68	73
3	14	57	68
4	14	74	84
-1	14	160	154
-6	14	69	79
1	15	78	72
2	15	24	51
3	15	104	98
-5	15	121	118
0	16	92	92
1	16	30	47
-1	16	95	94
-1	17	100	86
-2	17	68	71
0	-1	511	481
1	-1	658	654
2	-1	346	309
3	-1	381	369
4	-1	335	323
5	-1	149	150
6	-1	244	241
8	-1	107	131
9	-1	76	81
-1	-1	366	321
-2	-1	536	514
-3	-1	77	74
-4	-1	451	444
-5	-1	280	278
-6	-1	51	29
-7	-1	185	199
-8	-1	45	55
-9	-1	60	67
0	-2	208	207
1	-2	484	469
2	-2	110	73
3	-2	74	68
4	-2	425	398
5	-2	194	177
6	-2	178	198
7	-2	164	168
9	-2	54	60
-1	-2	716	753
-2	-2	166	166
-3	-2	452	425
-4	-2	438	442
-5	-2	28	13
-6	-2	263	264
-7	-2	121	133
-8	-2	55	52
-9	-2	63	75
0	-3	628	624
1	-3	295	305
2	-3	196	209
3	-3	226	228
4	-3	116	125
5	-3	355	353
6	-3	78	71
7	-3	162	166
8	-3	104	103
-1	-3	92	115
-2	-3	232	221
-3	-3	575	486
-4	-3	71	92
-5	-3	266	299
-6	-3	160	180
-7	-3	54	62
-8	-3	96	126
0	-4	229	229
1	-4	619	603
2	-4	21	29
3	-4	229	224
4	-4	75	69
5	-4	128	121
6	-4	296	283
7	-4	58	51
8	-4	106	113
9	-4	87	88
-1	-4	222	230
-2	-4	319	345
-3	-4	239	263
-4	-4	292	295
-5	-4	192	197
-6	-4	68	66
-7	-4	136	164
-8	-4	77	111
-9	-4	26	40
0	-5	221	228
1	-5	459	461
2	-5	310	344
3	-5	164	187
4	-5	296	261
5	-5	242	251
6	-5	137	121
7	-5	188	187
9	-5	77	104
-1	-5	237	251
-2	-5	108	128
-3	-5	188	197
-4	-5	282	290
-5	-5	54	74
-6	-5	178	187
-7	-5	91	101
-9	-5	49	90
0	-6	325	395
1	-6	136	144
2	-6	458	484
3	-6	238	268
4	-6	137	122
5	-6	364	342
6	-6	147	143
7	-6	130	118
8	-6	97	98
-1	-6	119	126
-2	-6	208	221
-3	-6	324	373
-4	-6	35	25
-5	-6	187	183
-6	-6	114	111
-8	-6	82	97
0	-7	189	214
1	-7	327	383
2	-7	132	102
3	-7	517	508
4	-7	219	197
5	-7	119	116
6	-7	242	217
7	-7	43	48
8	-7	112	106
9	-7	65	74
-1	-7	231	258
-2	-7	328	357
-3	-7	180	136
-4	-7	237	232
-5	-7	111	105
-7	-7	98	129
-8	-7	22	39
0	-8	123	131
1	-8	404	482
2	-8	286	339
3	-8	60	50
4	-8	321	303
5	-8	128	113
6	-8	142	126
7	-8	144	144
9	-8	80	77
-1	-8	344	382
-2	-8	233	228
-3	-8	206	203
-4	-8	186	202
-6	-8	101	98
-7	-8	75	79
-8	-8	29	60
0	-9	260	278
1	-9	66	62
2	-9	335	364
3	-9	311	337
4	-9	71	89
5	-9	215	226
7	-9	118	96
8	-9	90	95
-1	-9	255	285
-2	-9	132	129
-3	-9	274	284
-5	-9	105	133
-6	-9	111	113
-8	-9	68	85
0	-10	266	292
3	-10	313	346
4	-10	146	182
5	-10	90	104
6	-10	146	152
8	-10	92	70
-1	-10	67	60
-2	-10	241	226
-3	-10	113	118
-4	-10	134	145
-5	-10	146	166
-7	-10	79	75
0	-11	52	57
1	-11	263	268
2	-11	227	233
3	-11	87	101
4	-11	177	187
5	-11	68	122
6	-11	120	147
7	-11	131	127
-1	-11	278	274
-2	-11	68	84
-3	-11	42	52
-4	-11	182	200
-6	-11	59	78
-7	-11	49	64
0	-12	301	278
2	-12	205	223
3	-12	133	157
4	-12	39	61
5	-12	183	203
7	-12	114	143
8	-12	83	88
-1	-12	185	193
-2	-12	64	75
-3	-12	149	143
-4	-12	31	60
-5	-12	57	79
-6	-12	79	80
0	-13	195	181
1	-13	239	253
5	-13	123	142
6	-13	138	159
-2	-13	170	166
-3	-13	78	73
-4	-13	65	81
-5	-13	98	96
0	-14	39	44
1	-14	204	200
4	-14	112	135
7	-14	53	86
-1	-14	163	146
-2	-14	99	109
-3	-14	40	65
-4	-14	116	119
0	-15	145	141
5	-15	143	154
-1	-15	119	122
-3	-15	127	132
-4	-15	39	44
0	-16	115	95
1	-16	92	92
-2	-16	105	114
-3	-16	76	82
1	-17	121	116
2	-17	67	61
-1	-17	83	75
-2	-17	77	71
0	-18	53	47
2	-18	135	111
	H=	2	
0	0	454	448
1	0	308	298
2	0	432	413
3	0	429	416
4	0	125	120
5	0	278	283
6	0	61	56
7	0	96	111
8	0	113	126
-1	0	180	171
-2	0	322	311
-3	0	546	504
-4	0	82	74
-5	0	310	293
-6	0	143	157
-7	0	49	53
0	1	243	246
1	1	144	152
2	1	576	542
3	1	15	6
4	1	230	198
5	1	151	144
6	1	137	150
7	1	157	167
9	1	61	78
-1	1	722	645
-2	1	174	167
-3	1	509	450
-4	1	416	412
-6	1	163	130
-7	1	127	139
0	-1	53	49
1	-1	1055	1021
2	-1	214	177
3	-1	409	386
4	-1	296	281
5	-1	139	135
6	-1	198	180
7	-1	29	25
8	-1	100	118
9	-1	59	78
-1	-1	169	154
-2	-1	433	424
-3	-1	75	59
-4	-1	481	459
-5	-1	345	349
-6	-1	48	28
-7	-1	152	151
0	2	132	141
1	2	545	534
2	2	92	87
3	2	289	280
4	2	185	178
5	2	43	39
6	2	222	238
7	2	41	53
8	2	55	70
9	2	44	66
-1	2	521	482
-2	2	528	506
-3	2	269	249
-4	2	332	303
-5	2	240	260
-6	2	57	67
-7	2	151	173
0	-2	229	217
1	-2	487	462
2	-2	397	387
3	-2	193	193
4	-2	421	381
5	-2	163	155
6	-2	149	171
7	-2	161	167
9	-2	51	71
-1	-2	550	534
-2	-2	154	138
-3	-2	623	549
-4	-2	259	241
-5	-2	25	8
-6	-2	270	281
0	3	461	476
1	3	127	119
2	3	477	432
3	3	297	290
4	3	26	36
5	3	187	177
6	3	76	64
7	3	63	90
8	3	80	102
-1	3	193	194
-2	3	416	377
-3	3	530	482
-4	3	74	58
-5	3	384	371
-6	3	190	204
-8	3	148	157
0	-3	693	641
1	-3	77	48
2	-3	312	316
3	-3	335	326
4	-3	146	129
5	-3	345	335
6	-3	91	54
7	-3	132	133
8	-3	113	115
-1	-3	459	487
-2	-3	269	274
-4	-3	38	41
-5	-3	296	287
-6	-3	207	189
0	4	286	280
1	4	478	467
2	4	346	351
3	4	19	7
4	4	245	232
5	4	165	181
6	4	45	60
7	4	104	107
-1	4	580	529
-2	4	259	238
-3	4	340	340
-4	4	341	321
-5	4	136	121
-6	4	261	272
-7	4	87	111
-8	4	99	112
-9	4	84	102
0	-4	443	431
1	-4	506	500
3	-4	360	340
4	-4	249	228
5	-4	190	183
6	-4	266	269
7	-4	29	39
8	-4	92	83
9	-4	63	68
-1	-4	188	187
-2	-4	247	282
-3	-4	242	185
-4	-4	356	331
-5	-4	249	209
-6	-4	84	97
-9	-4	25	52
0	5	274	268
1	5	539	529
2	5	28	11
3	5	261	259
4	5	215	210
5	5	28	24
6	5	156	155
7	5	23	38
8	5	56	65
-1	5	422	396
-2	5	436	405
-3	5	102	98
-4	5	422	401
-5	5	200	196
-6	5	105	112
-7	5	166	188
-9	5	68	96
0	-5	176	187
1	-5	565	563
2	-5	367	351
4	-5	457	427
5	-5	203	202
6	-5	144	114
7	-5	166	167
9	-5	53	68
-1	-5	380	361
-2	-5	204	219
-3	-5	193	211
-4	-5	314	251
-6	-5	259	254
-8	-5	34	58
-9	-5	55	67
0	6	428	414
1	6	222	225
2	6	281	281
3	6	247	253
4	6	28	37
5	6	224	245
6	6	98	126
8	6	60	53
-1	6	142	136
-2	6	344	352
-3	6	392	402
-4	6	33	23
-5	6	226	238
-6	6	45	60
-7	6	127	138
-8	6	152	168
0	-6	393	408
1	-6	120	143
2	-6	484	476
4	-6	150	137
5	-6	402	375
6	-6	149	151
7	-6	136	134
8	-6	82	98
-1	-6	198	177
-2	-6	196	203
-3	-6	382	407
-4	-6	72	37
-5	-6	259	239
-6	-6	146	124
-8	-6	98	108
-9	-6	44	45
0	7	164	165
1	7	181	166
2	7	318	294
3	7	57	57
4	7	192	199
5	7	183	203
6	7	38	62
7	7	63	77
-1	7	375	380
-3	7	336	327
-4	7	237	221
-5	7	127	123
-6	7	198	221
-7	7	66	80
-8	7	83	107
-9	7	78	101
0	-7	233	240
1	-7	682	567
2	-7	77	86
3	-7	463	437
4	-7	224	210
5	-7	134	121
6	-7	313	297
8	-7	112	112
9	-7	39	55
-1	-7	229	220
-2	-7	390	430
-3	-7	222	226
-4	-7	196	188
-5	-7	194	178
-6	-7	62	92
-7	-7	104	125
-8	-7	44	55
0	8	188	173
1	8	317	316
2	8	100	107
3	8	153	139
4	8	184	175
6	8	138	141
7	8	54	50
-1	8	233	230
-2	8	296	281
-4	8	213	235
-5	8	78	97
-6	8	147	148
-7	8	202	212
-8	8	63	64
-9	8	75	82
0	-8	228	209
1	-8	478	465
2	-8	371	367
3	-8	149	151
4	-8	337	306
5	-8	151	139
6	-8	180	174
7	-8	153	148
9	-8	86	92
-1	-8	429	441
-2	-8	255	244
-3	-8	209	212
-4	-8	260	277
-5	-8	19	8
-6	-8	132	147
-7	-8	65	82
0	9	321	315
1	9	156	153
2	9	131	125
3	9	182	174
5	9	95	91
6	9	73	73
-1	9	55	69
-2	9	186	152
-3	9	202	195
-4	9	106	114
-5	9	276	274
-6	9	109	111
-7	9	84	104
-8	9	124	145
0	-9	378	360
1	-9	63	52
2	-9	502	468
3	-9	277	279
4	-9	138	148
5	-9	245	211
6	-9	35	56
7	-9	160	148
8	-9	71	79
9	-9	32	46
-1	-9	311	290
-2	-9	138	119
-3	-9	380	446
-4	-9	19	14
-5	-9	133	123
-6	-9	119	127
-8	-9	77	91
0	10	188	169
1	10	63	91
2	10	183	171
3	10	38	57
4	10	128	119
5	10	88	79
-1	10	254	242
-3	10	213	220
-4	10	211	202
-5	10	141	120
-6	10	191	195
-8	10	97	110
0	-10	288	276
1	-10	283	263
2	-10	96	97
3	-10	367	352
4	-10	143	147
5	-10	154	141
6	-10	156	146
8	-10	139	123
-1	-10	20	45
-2	-10	338	381
-3	-10	164	172
-4	-10	212	227
-5	-10	151	174
-7	-10	57	75
0	11	68	66
1	11	204	180
2	11	82	70
3	11	107	104
4	11	124	104
-1	11	172	157
-2	11	163	155
-4	11	331	312
-5	11	116	116
-6	11	51	76
-7	11	99	123
0	-11	47	2
1	-11	304	286
2	-11	233	228
3	-11	123	117
4	-11	253	237
6	-11	153	154
7	-11	132	123
-1	-11	258	284
-2	-11	217	225
-3	-11	116	113
-4	-11	193	217
-6	-11	68	76
-7	-11	67	87
0	12	200	173
1	12	104	95
2	12	73	83
3	12	127	124
5	12	58	55
-4	12	98	100
-5	12	150	167
-7	12	70	86
0	-12	243	245
1	-12	20	20
2	-12	252	261
3	-12	159	147
4	-12	143	133
5	-12	129	132
7	-12	173	153
8	-12	79	98
-1	-12	217	246
-2	-12	63	66
-3	-12	197	198
-4	-12	38	67
-5	-12	54	69
-6	-12	21	109
0	13	143	132
1	13	40	52
2	13	138	119
4	13	56	54
-5	13	73	66
-6	13	123	129
0	-13	238	265
1	-13	176	174
2	-13	49	71
3	-13	234	208
4	-13	57	61
5	-13	140	134
6	-13	183	169
-2	-13	183	172
-3	-13	65	81
-4	-13	45	65
-5	-13	96	104
1	14	127	112
2	14	44	40
3	14	31	40
-1	14	152	143
-5	14	25	55
-6	14	72	75
1	-14	196	208
4	-14	192	178
6	-14	166	153
7	-14	83	73
-1	-14	189	182
-2	-14	94	90
-3	-14	57	64
-4	-14	114	123
0	15	99	96
1	15	68	63
-1	15	20	21
-5	15	110	107
0	-15	147	139
1	-15	30	43
5	-15	170	154
-1	-15	146	138
-3	-15	116	116
-4	-15	53	46
0	16	93	75
-1	16	83	81
0	-16	147	134
1	-16	115	115
4	-16	87	83
-2	-16	89	98
-3	-16	74	71
1	-17	140	132
2	-17	34	58
-1	-17	66	72
-2	-17	81	83
0	-18	58	51
1	-18	40	44
2	-18	133	127
-1	-18	66	69
	H=	3	
0	0	287	290
1	0	41	33
2	0	427	425
3	0	365	345
4	0	82	69
5	0	250	239
6	0	74	84
7	0	111	127
8	0	70	82
-1	0	327	305
-2	0	387	374
-3	0	475	432
-4	0	24	30
-5	0	368	355
-6	0	140	151
0	1	78	90
1	1	217	221
2	1	393	383
3	1	134	136
4	1	236	219
5	1	142	120
6	1	118	127
7	1	132	144
-1	1	413	430
-2	1	173	175
-3	1	444	396
-4	1	413	390
-5	1	53	64
-6	1	282	213
0	-1	204	220
1	-1	425	420
2	-1	103	78
3	-1	462	451
4	-1	248	234
5	-1	123	126
6	-1	222	223
8	-1	77	66
9	-1	32	65
-1	-1	243	198
-2	-1	425	416
-3	-1	41	20
-4	-1	560	525
-5	-1	345	324
-6	-1	66	70
0	2	160	172
1	2	366	347
2	2	127	114
3	2	282	284
4	2	175	174
5	2	107	101
6	2	188	179
8	2	47	61
-1	2	400	387
-2	2	601	563
-3	2	353	304
-4	2	311	285
-5	2	275	273
-6	2	87	88
0	-2	155	142
1	-2	388	408
2	-2	548	542
3	-2	280	267
4	-2	330	332
5	-2	144	125
6	-2	136	140
7	-2	145	151
9	-2	81	66
-1	-2	425	435
-2	-2	317	257
-3	-2	596	514
-4	-2	379	365
-5	-2	69	48
-6	-2	243	250
0	3	458	463
1	3	99	63
2	3	358	346
3	3	232	199
4	3	103	106
5	3	193	192
6	3	50	72
7	3	66	92
8	3	54	63
-1	3	183	188
-2	3	253	239
-3	3	570	549
-4	3	23	7
-5	3	365	323
-6	3	196	201
-7	3	100	119
-8	3	138	141
0	-3	618	583
1	-3	72	66
2	-3	549	543
3	-3	198	173
4	-3	118	109
5	-3	266	251
6	-3	52	39
7	-3	143	142
8	-3	107	118
9	-3	29	15
-1	-3	414	472
-2	-3	401	388
-3	-3	602	481
-4	-3	42	36
-5	-3	331	310
-6	-3	229	229
0	4	262	261
1	4	302	300
2	4	411	382
3	4	79	84
4	4	234	214
5	4	111	107
6	4	64	76
7	4	74	81
-1	4	560	527
-2	4	316	294
-3	4	341	309
-4	4	330	318
-5	4	105	104
-6	4	294	281
-7	4	118	133
-8	4	78	101
-9	4	64	65
0	-4	647	632
1	-4	595	593
2	-4	115	120
3	-4	328	300
4	-4	228	232
5	-4	159	155
6	-4	211	201
8	-4	112	94
9	-4	61	47
-1	-4	79	68
-2	-4	[illegible]	[illegible]
-3	-4	75	60
-4	-4	373	342
-5	-4	306	289
0	5	296	313
1	5	474	495
2	5	114	113
3	5	265	245
4	5	157	138
5	5	89	78
6	5	182	148
-1	5	237	222
-2	5	384	380
-3	5	60	61
-4	5	335	301
-5	5	225	221
-6	5	92	95
-7	5	195	202
-9	5	57	64
0	-5	37	22
1	-5	463	445
2	-5	400	398
3	-5	92	98
4	-5	375	337
5	-5	103	98
6	-5	128	123
7	-5	134	130
9	-5	49	68
-1	-5	561	530
-2	-5	423	405
-3	-5	313	297
-4	-5	426	316
-5	-5	42	69
-6	-5	227	227
0	6	406	409
1	6	205	202
2	6	257	238
3	6	212	207
5	6	194	185
6	6	68	71
7	6	66	60
-1	6	187	175
-2	6	297	298
-3	6	400	377
-5	6	232	229
-6	6	96	123
-7	6	72	97
-8	6	132	140
1	-6	68	46
2	-6	438	434
3	-6	207	204
4	-6	190	187
5	-6	305	278
6	-6	93	103
7	-6	111	114
8	-6	78	86
-1	-6	479	428
-2	-6	141	132
-3	-6	389	394
-4	-6	155	148
-5	-6	201	171
0	7	157	149
1	7	242	236
2	7	270	266
3	7	29	35
4	7	180	173
5	7	106	115
6	7	39	42
7	7	73	84
-1	7	339	330
-2	7	64	65
-3	7	298	308
-4	7	233	229
-5	7	31	32
-6	7	189	196
-7	7	96	103
-8	7	96	105
-9	7	87	98
0	-7	446	416
1	-7	422	431
2	-7	50	47
3	-7	379	351
4	-7	128	140
5	-7	186	165
6	-7	228	218
8	-7	107	110
9	-7	42	45
-1	-7	247	199
-2	-7	483	528
-3	-7	265	291
-4	-7	80	68
-5	-7	226	216
0	8	187	169
1	8	296	272
2	8	63	54
3	8	193	181
4	8	137	134
6	8	87	89
-1	8	254	243
-2	8	288	270
-3	8	47	44
-4	8	221	246
-5	8	160	154
-6	8	49	69
-7	8	204	219
0	-8	95	77
1	-8	489	490
2	-8	335	331
3	-8	188	172
4	-8	351	317
5	-8	64	70
6	-8	153	154
7	-8	118	124
8	-8	30	49
-1	-8	419	379
-2	-8	303	292
-3	-8	140	114
-4	-8	252	267
-5	-8	90	98
0	9	329	312
1	9	124	114
2	9	155	155
3	9	182	171
5	9	75	83
6	9	49	52
-1	9	104	103
-2	9	125	124
-3	9	244	235
-4	9	77	84
-5	9	251	253
-6	9	117	112
-7	9	109	108
-8	9	109	137
0	-9	389	356
1	-9	63	44
2	-9	546	527
3	-9	131	114
4	-9	171	160
5	-9	214	208
7	-9	158	141
8	-9	81	98
-1	-9	296	262
-2	-9	113	88
-3	-9	318	344
-4	-9	142	158
0	10	154	140
1	10	97	106
2	10	200	174
4	10	119	121
5	10	84	68
-1	10	265	241
-4	10	164	156
-5	10	98	100
-6	10	164	179
-8	10	91	103
0	-10	323	294
1	-10	274	232
2	-10	233	207
3	-10	316	307
4	-10	78	61
5	-10	188	171
6	-10	160	158
8	-10	119	106
-1	-10	21	15
-2	-10	382	331
-3	-10	182	149
-4	-10	99	128
0	11	98	72
1	11	229	213
2	11	38	33
3	11	100	100
4	11	96	100
-1	11	183	182
-4	11	249	241
-5	11	84	109
-6	11	42	72
-7	11	128	129
0	-11	30	17
1	-11	283	237
2	-11	152	139
3	-11	169	179
4	-11	242	228
6	-11	147	147
7	-11	98	103
8	-11	28	33
-1	-11	248	221
-2	-11	318	307
-3	-11	60	67
0	12	170	149
1	12	80	71
2	12	101	118
3	12	121	109
-4	12	78	83
-5	12	143	165
-6	12	48	58
-7	12	84	90
0	-12	167	142
1	-12	30	64
2	-12	262	256
3	-12	108	98
4	-12	197	189
5	-12	124	103
7	-12	131	133
8	-12	76	72
-1	-12	201	162
-2	-12	40	57
-6	-12	89	109
0	13	120	105
1	13	31	56
2	13	133	120
-1	13	124	121
-5	13	79	79
-6	13	110	121
0	-13	180	171
1	-13	127	94
2	-13	131	111
3	-13	262	226
4	-13	28	37
5	-13	122	126
6	-13	120	107
-5	-13	87	96
1	14	105	91
-1	14	145	134
-5	14	34	49
3	-14	135	118
4	-14	183	164
6	-14	121	117
7	-14	88	76
-4	-14	107	121
-5	-14	37	50
0	15	101	93
2	-15	154	153
3	-15	57	56
4	-15	137	127
5	-15	131	119
-3	-15	109	106
-4	-15	23	43
2	-16	62	56
3	-16	134	127
5	-16	89	92
-2	-16	84	94
-3	-16	51	42
3	-17	77	76
4	-17	135	96
-1	-17	85	97
-2	-17	84	97
0	-18	63	71
-1	-18	90	86
	H=	4	
0	0	436	435
2	0	358	332
3	0	329	286
4	0	95	67
5	0	161	168
6	0	78	102
7	0	74	82
-1	0	177	169
-3	0	457	425
-4	0	74	41
-5	0	251	247
-6	0	279	295
0	1	226	219
1	1	314	310
2	1	315	304
3	1	137	131
4	1	206	186
5	1	107	103
6	1	56	58
7	1	98	118
-1	1	376	366
-2	1	251	223
-3	1	252	227
-4	1	372	376
-5	1	37	24
-6	1	275	278
0	-1	251	262
1	-1	321	312
2	-1	58	52
3	-1	465	454
4	-1	254	251
5	-1	94	77
6	-1	54	167
8	-1	64	70
-1	-1	205	178
-2	-1	506	492
-3	-1	150	138
-4	-1	332	314
-5	-1	279	277
0	2	184	188
1	2	79	281
2	2	54	46
3	2	261	228
4	2	93	79
5	2	77	86
6	2	137	147
7	2	32	29
8	2	34	45
-1	2	353	307
-2	2	508	499
-3	2	187	150
-4	2	330	301
-5	2	280	287
-6	2	52	65
0	-2	67	76
1	-2	320	303
2	-2	619	604
3	-2	187	179
4	-2	311	289
5	-2	117	119
6	-2	118	120
7	-2	115	125
-1	-2	424	435
-2	-2	378	303
-3	-2	308	273
-4	-2	450	454
-5	-2	42	17
-6	-2	181	190
0	3	390	374
1	3	88	61
2	3	278	265
3	3	134	110
4	3	94	93
5	3	169	168
6	3	37	58
7	3	43	63
-1	3	208	204
-2	3	350	343
-3	3	387	383
-4	3	29	5
-5	3	284	269
-6	3	211	222
-7	3	93	107
-8	3	129	157
0	-3	460	445
1	-3	60	54
2	-3	563	568
3	-3	331	330
4	-3	183	156
5	-3	222	202
6	-3	36	38
7	-3	115	121
8	-3	83	90
-1	-3	432	481
-2	-3	216	201
-3	-3	407	359
-4	-3	185	158
-5	-3	269	266
-6	-3	204	189
0	4	204	188
1	4	232	221
2	4	245	226
3	4	53	72
4	4	173	176
5	4	59	69
6	4	51	72
7	4	60	71
-1	4	385	368
-2	4	124	130
-3	4	371	329
-4	4	250	247
-5	4	21	57
-6	4	267	270
-7	4	131	141
-8	4	56	67
-9	4	86	108
0	-4	718	635
1	-4	534	507
2	-4	34	22
3	-4	404	394
4	-4	156	142
5	-4	136	129
6	-4	185	169
8	-4	90	89
-1	-4	112	80
-2	-4	402	458
-3	-4	212	173
-4	-4	287	265
-5	-4	326	285
0	5	271	279
1	5	291	289
2	5	96	112
3	5	173	157
4	5	106	98
5	5	72	81
6	5	95	108
-1	5	145	136
-2	5	356	343
-3	5	40	9
-4	5	255	254
-5	5	167	172
-7	5	194	205
-8	5	31	66
-9	5	61	73
0	-5	59	54
1	-5	389	408
2	-5	281	273
3	-5	180	172
4	-5	339	323
5	-5	106	100
6	-5	137	140
7	-5	103	118
-1	-5	404	380
-2	-5	437	448
-3	-5	208	232
-4	-5	430	325
-5	-5	103	92
0	6	315	308
1	6	39	49
2	6	132	126
3	6	162	149
5	6	118	112
6	6	34	47
-1	6	143	177
-2	6	248	223
-3	6	276	271
-5	6	191	183
-6	6	165	161
-7	6	31	70
-8	6	121	141
0	-6	343	334
1	-6	24	16
2	-6	349	346
3	-6	157	149
4	-6	156	148
5	-6	288	270
6	-6	38	10
7	-6	117	113
8	-6	78	82
-1	-6	542	478
-2	-6	72	86
-3	-6	341	369
-4	-6	173	168
-5	-6	167	133
0	7	77	81
1	7	186	191
2	7	193	206
4	7	131	115
5	7	52	48
-1	7	260	241
-2	7	105	114
-3	7	235	237
-4	7	225	233
-6	7	159	168
-7	7	82	96
-8	7	87	86
0	-7	314	314
1	-7	200	220
2	-7	58	51
3	-7	335	318
4	-7	104	87
5	-7	188	179
6	-7	174	168
8	-7	114	116
-1	-7	19	13
-2	-7	402	460
-3	-7	182	181
-4	-7	93	98
-5	-7	204	178
0	8	130	108
1	8	207	201
3	8	174	174
4	8	96	103
-1	8	65	62
-2	8	301	284
-3	8	45	55
-4	8	247	233
-5	8	167	166
-7	8	158	155
1	-8	328	339
2	-8	283	285
3	-8	118	106
4	-8	245	230
6	-8	160	161
7	-8	131	131
-1	-8	317	321
-2	-8	207	201
-3	-8	122	120
-4	-8	232	244
0	9	218	216
1	9	67	64
2	9	154	154
3	9	123	123
6	9	82	75
-1	9	157	154
-3	9	277	255
-4	9	31	27
-5	9	177	187
-6	9	76	100
-7	9	69	83
0	-9	265	281
1	-9	65	64
2	-9	343	305
3	-9	90	87
4	-9	166	193
5	-9	215	206
7	-9	143	143
8	-9	55	54
-1	-9	253	213
-2	-9	153	133
-3	-9	232	256
-4	-9	104	106
0	10	99	106
1	10	122	123
2	10	147	144
4	10	85	93
-1	10	215	209
-4	10	145	152
-6	10	129	150
0	-10	303	274
1	-10	246	229
2	-10	138	139
3	-10	236	230
5	-10	206	187
6	-10	163	148
8	-10	94	99
-1	-10	37	28
-2	-10	331	330
-3	-10	77	70
0	11	64	75
1	11	169	179
3	11	85	95
-1	11	95	103
-4	11	162	166
-5	11	97	117
-6	11	71	68
1	-11	225	215
2	-11	63	61
3	-11	197	190
4	-11	214	186
6	-11	142	136
7	-11	92	88
-1	-11	332	306
-2	-11	246	222
-3	-11	87	88
0	12	146	133
1	12	34	43
2	12	100	104
-5	12	150	159
-6	12	45	64
0	-12	20	154
1	-12	38	85
2	-12	240	261
3	-12	84	76
4	-12	143	121
5	-12	157	138
7	-12	106	102
-1	-12	228	191
0	13	62	70
1	13	64	63
-1	13	137	121
-5	13	35	55
2	-13	155	129
3	-13	266	249
4	-13	67	57
5	-13	101	102
6	-13	62	74
-1	14	89	90
2	-14	137	121
3	-14	118	111
4	-14	167	158
6	-14	84	76
2	-15	202	175
3	-15	87	82
4	-15	107	115
5	-15	100	104
2	-16	58	45
3	-16	152	165
5	-16	101	101
3	-17	100	97
4	-17	139	116
	H=	5	
0	0	391	364
1	0	122	89
2	0	360	347
3	0	226	226
4	0	110	112
5	0	182	197
6	0	42	77
7	0	77	78
-1	0	318	305
-2	0	198	178
-3	0	359	363
-4	0	88	64
-5	0	229	236
0	1	74	62
1	1	383	357
2	1	249	246
3	1	94	80
4	1	234	226
5	1	125	119
6	1	79	87
7	1	87	87
-1	1	468	443
-2	1	328	274
-3	1	212	213
-4	1	316	317
-5	1	39	16
0	-1	392	379
1	-1	265	264
2	-1	158	138
3	-1	347	351
4	-1	176	178
5	-1	94	77
6	-1	155	162
7	-1	31	21
-1	-1	215	198
-2	-1	432	426
-3	-1	238	212
-4	-1	205	207
-5	-1	222	248
0	2	328	307
1	2	322	313
2	2	39	17
3	2	248	240
4	2	141	122
5	2	107	117
6	2	121	137
-1	2	128	120
-2	2	505	488
-3	2	86	77
-4	2	263	240
-5	2	247	255
-6	2	78	87
0	-2	77	61
1	-2	407	403
2	-2	299	280
3	-2	129	109
4	-2	257	261
5	-2	56	73
6	-2	125	134
7	-2	124	141
-1	-2	409	445
-3	-2	223	198
-4	-2	329	315
-5	-2	20	27
0	3	345	329
1	3	73	72
2	3	206	202
3	3	232	216
4	3	83	116
5	3	170	165
6	3	40	46
7	3	33	50
-1	3	286	257
-2	3	315	291
-3	3	372	380
-4	3	31	16
-5	3	233	227
-6	3	198	211
-7	3	79	81
-8	3	129	153
0	-3	404	397
1	-3	34	6
2	-3	413	415
3	-3	265	275
4	-3	207	194
5	-3	169	164
7	-3	114	124
8	-3	55	69
-1	-3	366	406
-2	-3	160	155
-3	-3	464	406
-4	-3	136	127
-5	-3	165	179
0	4	80	84
1	4	185	169
2	4	257	246
4	4	202	201
5	4	51	68
6	4	37	59
-1	4	346	327
-2	4	110	99
-3	4	331	291
-4	4	248	260
-5	4	32	44
-6	4	236	238
-7	4	112	136
-8	4	47	65
0	-4	405	411
1	-4	362	375
2	-4	86	89
3	-4	408	405
4	-4	46	20
5	-4	127	135
6	-4	127	133
8	-4	97	98
-1	-4	90	84
-2	-4	395	410
-3	-4	225	187
-4	-4	275	234
-5	-4	325	294
0	5	176	164
1	5	265	258
2	5	101	92
3	5	160	159
4	5	111	91
6	5	81	82
-1	5	131	124
-2	5	340	310
-3	5	20	17
-4	5	277	266
-5	5	193	201
-7	5	172	179
-8	5	65	79
0	-5	72	52
1	-5	340	352
2	-5	218	225
3	-5	251	253
4	-5	262	243
5	-5	43	53
6	-5	112	117
7	-5	82	82
-1	-5	495	467
-2	-5	214	242
-3	-5	207	203
-4	-5	531	378
-5	-5	148	139
0	6	215	226
1	6	122	116
2	6	158	153
3	6	140	128
5	6	77	76
-1	6	121	126
-2	6	192	179
-3	6	260	257
-5	6	194	199
-6	6	157	158
-7	6	65	69
-8	6	120	138
0	-6	283	294
1	-6	54	52
2	-6	296	286
3	-6	131	131
4	-6	174	181
5	-6	186	163
7	-6	113	120
8	-6	56	66
-1	-6	433	389
-2	-6	168	156
-3	-6	338	372
-4	-6	159	164
-5	-6	170	162
0	7	151	136
1	7	117	124
2	7	169	162
4	7	104	89
-3	7	231	221
-4	7	252	237
-6	7	193	190
-7	7	62	69
-8	7	47	69
0	-7	345	324
1	-7	228	250
2	-7	77	75
3	-7	310	301

gen and carbon. The contributions to the least-squares sums were weighted by $1/\sigma^2$, where σ, the standard deviation of each structure factor, was computed by a variation of the method of Smith and Alexander,[8] which includes the contribution of each correction factor, such as background measurements, calculation of integrated intensities from peak heights, and correc-

---

[8] G. S. Smith and L. A. Alexander, *Acta Cryst.*, **16**, 462 (1963).

TABLE I (*Continued*)

K	L	FO	|FC|
	H=	5	
4	-7	61	55
5	-7	154	156
6	-7	117	98
7	-7	46	33
8	-7	91	100
-1	-7	34	25
-2	-7	304	343
-3	-7	151	184
-4	-7	131	145
0	8	47	68
1	8	186	175
3	8	113	123
4	8	76	74
-4	8	222	212
-5	8	168	165
-7	8	83	124
0	-8	79	81
1	-8	327	337
2	-8	249	228
3	-8	87	80
4	-8	216	200
6	-8	158	159
7	-8	96	94
8	-8	42	24
-1	-8	314	296
-2	-8	180	192
-3	-8	104	120
-4	-8	215	240
0	9	189	170
1	9	61	44
2	9	149	119
3	9	91	84
-1	9	64	91
-3	9	187	106
-5	9	154	165
-6	9	84	92
-7	9	46	67
0	-9	249	228
1	-9	70	61
2	-9	280	273
3	-9	110	114
4	-9	206	201
5	-9	214	211
7	-9	113	100
-1	-9	296	271
-2	-9	99	89
-3	-9	224	255
0	10	89	78
1	10	62	89
2	10	99	111
-1	10	191	198
-4	10	123	124
-6	10	115	132
0	-10	311	275
1	-10	252	224
2	-10	85	77
3	-10	258	269
5	-10	193	187
6	-10	152	145
-2	-10	300	282
-3	-10	137	161
0	11	52	61
1	11	127	118
-1	11	102	98
-4	11	92	104
-5	11	77	89
0	-11	45	62
1	-11	275	254
2	-11	99	94
3	-11	222	204
4	-11	223	216
5	-11	24	25
6	-11	126	124
7	-11	63	69
-1	-11	335	313
-2	-11	195	182
0	12	103	106
-1	12	39	49
0	-12	171	159
2	-12	249	252
3	-12	87	78
4	-12	136	131
5	-12	158	148
7	-12	89	91
2	-13	167	165
3	-13	260	229
4	-13	59	59
5	-13	114	122
6	-13	71	75
2	-14	100	86
3	-14	130	118
4	-14	150	142
6	-14	114	99
2	-15	194	170
3	-15	22	43
4	-15	116	113
5	-15	100	93
2	-16	89	83
3	-16	123	138
5	-16	100	95
3	-17	95	98
4	-17	116	106
	H=	6	
0	0	281	282
1	0	55	46
2	0	270	276
3	0	153	156
4	0	127	132
5	0	136	138
-1	0	387	370
-2	0	83	68
-3	0	392	335
-4	0	109	110
0	1	67	68
1	1	293	305
2	1	214	214
3	1	84	83
4	1	216	202
6	1	88	74
-1	1	354	360
-2	1	267	229
-3	1	214	204
-4	1	291	305
0	-1	318	313
1	-1	242	247
2	-1	118	111
3	-1	202	205
4	-1	97	97
5	-1	118	126
6	-1	90	91
-1	-1	94	76
-2	-1	398	394
-3	-1	224	208
-4	-1	60	65
0	2	305	318
1	2	256	262
2	2	28	24
3	2	244	244
4	2	60	60
5	2	116	111
6	2	72	89
-1	2	49	63
-2	2	351	372
-3	2	115	122
-4	2	183	181
-5	2	227	246
0	-2	46	26
1	-2	299	314
2	-2	133	138
3	-2	60	72
4	-2	191	197
5	-2	20	32
6	-2	110	121
7	-2	72	76
-1	-2	424	445
-2	-2	368	317
-3	-2	49	58
-4	-2	213	228
0	3	231	236
2	3	238	244
3	3	149	142
4	3	100	117
5	3	110	128
-1	3	236	220
-2	3	195	180
-3	3	313	332
-4	3	54	53
-5	3	199	212
-6	3	166	178
-8	3	145	164
0	-3	326	317
1	-3	85	78
2	-3	240	251
3	-3	167	174
4	-3	138	136
5	-3	145	142
7	-3	86	99
-1	-3	276	298
-2	-3	107	100
-3	-3	455	393
-4	-3	194	169
1	4	227	224
2	4	188	174
3	4	68	55
4	4	147	159
5	4	26	53
-1	4	258	273
-2	4	139	130
-3	4	207	207
-4	4	221	246
-6	4	175	207
-7	4	124	140
-8	4	46	51
0	-4	309	321
1	-4	184	208
2	-4	123	144
3	-4	297	301
4	-4	49	65
5	-4	122	134
6	-4	99	104
-1	-4	102	92
-2	-4	278	308
-3	-4	328	264
-4	-4	208	190
0	5	162	172
1	5	232	226
2	5	46	44
3	5	152	151
4	5	75	75
5	5	49	40
-2	5	240	230
-3	5	40	51
-4	5	204	207
-5	5	229	233
-6	5	26	32
-7	5	152	174
0	-5	66	28
1	-5	313	321
2	-5	129	139
3	-5	183	185
4	-5	220	224
6	-5	84	84
7	-5	54	66
-1	-5	319	316
-2	-5	193	224
-3	-5	158	184
-4	-5	486	367
0	6	236	211
1	6	31	46
2	6	171	167
3	6	77	93
4	6	37	35
-2	6	126	129
-3	6	232	241
-4	6	44	45
-5	6	203	208
-6	6	151	148
-7	6	43	62
0	-6	232	231
1	-6	104	99
2	-6	275	284
3	-6	76	71
4	-6	161	164
5	-6	119	115
7	-6	100	108
-1	-6	347	337
-2	-6	63	72
-3	-6	271	314
-4	-6	137	134
0	7	44	43
1	7	126	119
2	7	118	120
[illegible]	7	95	90
-3	7	182	180
-4	7	215	219
-6	7	175	174
-7	7	55	79
0	-7	339	330
1	-7	156	163
2	-7	106	107
3	-7	253	249
5	-7	96	103
6	-7	103	96
7	-7	38	39
-2	-7	235	280
-3	-7	133	177
0	8	124	121
1	8	137	125
3	8	83	99
-4	8	159	162
-5	8	142	165
0	-8	143	148
1	-8	247	258
2	-8	126	140
3	-8	120	118
4	-8	197	193
6	-8	118	122
7	-8	73	78
-1	-8	267	257
-2	-8	174	188
-3	-8	71	98
0	9	166	154
2	9	90	95
-1	9	80	90
-3	9	142	152
-5	9	142	152
0	-9	188	195
1	-9	143	130
2	-9	234	223
3	-9	91	102
4	-9	175	169
5	-9	151	156
7	-9	82	77
-1	-9	271	237
-2	-9	39	54
0	10	28	57
1	10	65	63
-1	10	176	173
-4	10	101	118
0	-10	282	275
1	-10	164	169
2	-10	104	101
3	-10	206	201
5	-10	165	170
6	-10	121	116
2	-11	111	120
3	-11	145	147
4	-11	176	186
6	-11	98	105
-1	11	76	82
2	-12	197	231
3	-12	44	45
4	-12	137	137
5	-12	100	108
6	-12	39	17
2	-13	129	143
3	-13	195	183
5	-13	97	97
6	-13	81	77
2	-14	37	58
3	-14	122	110
4	-14	108	120
5	-14	27	22
2	-15	166	165
4	-15	103	102
5	-15	61	76
2	-16	99	99
3	-16	101	110
3	-17	98	93
	H=	7	
0	0	214	238
2	0	205	201
3	0	74	82
4	0	93	104
5	0	82	97
-1	0	310	302
-2	0	52	59
0	1	85	77
1	1	243	264
2	1	172	191
3	1	66	67
4	1	126	128
-1	1	232	243
-2	1	164	168
-3	1	140	138
0	-1	241	258
1	-1	163	161
2	-1	55	50
3	-1	162	171
5	-1	101	107
6	-1	56	70
-1	-1	37	19
-2	-1	298	323
0	2	236	247
1	2	196	205
2	2	52	38
3	2	138	157
4	2	41	67
5	2	62	75
-2	2	248	268
-3	2	134	128
-4	2	116	136
-5	2	190	204
-6	2	29	3
0	-2	51	37
1	-2	197	210
2	-2	93	110
3	-2	73	82
4	-2	146	152
6	-2	66	78
-1	-2	320	337
-2	-2	281	261
0	3	196	206
1	3	30	42
2	3	189	196
3	3	84	97
4	3	24	44
-1	3	154	156
-2	3	86	89
-3	3	232	261
-4	3	92	104
-5	3	125	149
-6	3	104	140
0	-3	215	229
1	-3	87	88
2	-3	200	222
3	-3	96	93
4	-3	78	81
5	-3	119	134
-1	-3	170	216
-2	-3	182	152
-3	-3	286	263
1	4	166	167
2	4	93	107
3	4	31	60
4	4	108	116
-2	4	122	126
-3	4	75	103
-4	4	208	231
-5	4	48	45
-6	4	131	153
-7	4	91	113
0	-4	225	265
1	-4	142	154
2	-4	101	103
3	-4	184	194
4	-4	69	96
5	-4	121	137
6	-4	76	89
-1	-4	118	94
-2	-4	268	315
-3	-4	135	140
0	5	172	167
1	5	104	131
2	5	29	30
3	5	119	132
-2	5	183	194
-3	5	86	89
-4	5	107	133
-5	5	195	212
0	-5	39	13
1	-5	262	272
2	-5	111	118
3	-5	108	124
4	-5	194	210
5	-5	25	4
6	-5	85	94
-1	-5	277	270
-2	-5	123	171
0	6	150	165
2	6	103	113
3	6	70	70
-2	6	96	95
-3	6	184	189
-4	6	70	66
-5	6	166	173
-6	6	125	135
0	-6	301	322
1	-6	80	94
2	-6	238	248
3	-6	76	71
4	-6	126	151
5	-6	112	117
-1	-6	231	236
-2	-6	30	26
1	7	91	104
2	7	87	90
-3	7	113	129
-4	7	166	176
0	-7	304	298
1	-7	171	172
2	-7	135	151
3	-7	222	223
4	-7	23	48
5	-7	93	101
6	-7	85	84
-1	-7	90	9
0	8	85	104
1	8	89	87
-4	8	116	128
1	-8	254	266
2	-8	119	132
3	-8	140	149
4	-8	189	187
6	-8	88	92
-1	9	58	60
-3	9	128	142
2	-9	257	254
3	-9	91	84
4	-9	135	138
5	-9	121	130
2	-10	94	96
3	-10	196	204
4	-10	21	30
5	-10	136	141
6	-10	81	93
2	-11	113	122
3	-11	108	111
4	-11	157	178
2	-12	169	199
3	-12	47	52
4	-12	141	145
5	-12	95	103
2	-13	115	131
3	-13	137	141
5	-13	102	116
3	-14	109	110
4	-14	97	93
2	-15	118	120
2	-16	93	100
	H=	8	
0	0	202	230
2	0	149	161
3	0	54	53
4	0	59	90
1	1	197	225
2	1	110	132
3	1	43	47
4	1	102	101
-1	1	187	205
-2	1	98	124
-3	1	92	105
1	-1	158	161
2	-1	30	50
3	-1	144	153
0	2	165	192
1	2	136	151
2	2	30	36
3	2	105	125
-1	2	68	48
-2	2	177	203
-3	2	81	111
-4	2	114	124
-5	2	179	183
1	-2	169	186
2	-2	122	127
3	-2	92	104
4	-2	95	119
0	3	147	171
2	3	118	151
3	3	68	73
-1	3	97	135
-2	3	86	86
-3	3	154	189
-4	3	101	101
-5	3	100	125
-6	3	113	140
1	-3	58	81
2	-3	204	228
3	-3	75	85
4	-3	56	66
5	-3	103	103
0	4	33	13
1	4	108	126
2	4	76	81
-2	4	94	109
-3	4	54	73
-4	4	160	188
-5	4	32	46
1	-4	121	139
2	-4	90	103
3	-4	169	176
4	-4	61	54
5	-4	106	120
0	5	108	139
1	5	90	92
-2	5	146	174
-3	5	62	85
-4	5	89	114
-5	5	123	152
1	-5	239	261
2	-5	68	90
3	-5	103	110
4	-5	165	179
0	6	137	144
-2	6	50	62
-3	6	137	154
1	-6	93	119
2	-6	209	217
3	-6	40	51
4	-6	122	137
5	-6	124	137
1	-7	96	117
2	-7	172	184
3	-7	178	201
5	-7	108	114
1	-8	250	274
2	-8	24	36
3	-8	148	158
4	-8	139	147
2	-9	192	202
3	-9	24	18
4	-9	109	123
5	-9	80	98
2	-10	134	143
3	-10	125	142
5	-10	100	106
2	-11	48	65
3	-11	102	128
4	-11	91	102
2	-12	136	165
4	-12	117	121
2	-13	88	106
3	-13	97	117
3	-14	88	94
2	-15	66	64
	H=	9	
0	0	147	169
1	0	62	64
2	0	116	138
1	1	157	167
2	1	57	76
-1	1	172	183
-2	1	104	125
-3	1	64	80
1	-1	104	118
2	-1	86	84
3	-1	109	122
0	2	131	151
1	2	86	114
-1	2	40	21
-2	2	168	185
-3	2	98	119
-4	2	75	87
1	-2	173	204
2	-2	64	66
3	-2	95	92
0	3	123	134
-1	3	116	135
-2	3	43	39
-3	3	163	180
1	-3	79	91
2	-3	174	191
3	-3	28	44
1	-4	81	108
2	-4	106	113
3	-4	128	149
1	-5	168	200
2	-5	46	69
3	-5	90	110
4	-5	92	102
1	-6	63	66
2	-6	138	169
4	-6	92	111
1	-7	82	99
2	-7	95	130
3	-7	118	148
1	-8	145	186
3	-8	103	123
2	-9	115	137
3	-9	27	10
2	-10	104	113
3	-10	79	101
3	-11	67	92
2	-12	106	129
	H=	10	
1	-3	61	95
1	-4	60	79
1	-5	124	166
1	-6	59	79
1	-7	54	71
1	-8	104	149

tions for instrumental variations, to the error. Unobserved reflections were omitted from the refinements. Poor agreement for the $40\bar{8}$ reflection indicated an error of measurement, and this reflection was discarded. During the last cycle of refinement the ytterbium scattering factors were corrected for anomalous dispersion using values of $\Delta f'$ and $\Delta f''$ from the compilation of Templeton.[9] The final value of the residual, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, was 0.080 for 2624 observed data. Before the anomalous dispersion correction, R was 0.083. The mean positional parameter shift during the last cycle was 0.00041. The standard deviation of an observation of unit weight was 1.49. No attempt was made to locate hydrogen atoms.

The magnitudes of the observed and calculated structure factors are listed in Table I, and the atomic parameters are listed in Table II.

Discussion of the Structure

The projection of the structure along [100] is shown in Figure 1. The bond lengths and angles are listed in Table III; the standard deviations were computed by the method of Sands.[10] Figure 2 shows the average dimensions of an ytterbium acetylacetonate ring.

Each ytterbium atom is bonded to one water molecule, O(1), and to six carbonyl oxygen atoms, donated by three bidentate acetylacetonate groups. The ring dimensions compare favorably with the average found by Lingafelter and Braun[11] in a study of acetylacetonate chelate structural data; the average bite of the rings is 2.750 Å as compared with their average value of 2.781 Å.

The mean deviations of the carbon and oxygen atoms from the least-squares planes of the acetylacetonate groups are 0.134, 0.004, and 0.017 Å for the rings containing O(2)–O(3), O(4)–O(5), and O(6)–O(7), respectively. The respective deviations of the ytterbium atom from each of these planes are 0.13 ± 0.14, 0.554 ± 0.002, and 0.02 ± 0.01 Å. The metal–chelate ring containing O(6)–O(7) is planar; the sum of the interior angles of this ring is 720.0°, which is the value required for planarity. The sum of the interior angles for the ring containing O(2)–O(3) is 718.0°; this ring is folded by 6.5° about the O(2)–O(3) line away from the water molecule. The sum of the interior angles for the O(4)–O(5) ring is 713.5°, and this ring is folded by 18.5° about the O(4)–O(5) line away from O(6)–O(7) toward O(2)–O(3).

TABLE II

ATOMIC PARAMETERS OF $Yb(C_5H_7O_2)_3(H_2O)\cdot0.5C_6H_6$ WITH ESTIMATED STANDARD DEVIATIONS

	X	Y	Z	B.A
YB	0.01210(10)	0.19206(11)	0.16134(6)	a
O(1)	-0.0783(14)	-0.0645(13)	0.01780(7)	3.7(3)
O(2)	-0.1401(14)	0.1107(14)	0.24257(8)	4.0(3)
O(3)	0.1569(15)	0.0281(14)	0.25144(8)	4.7(3)
O(4)	0.2164(16)	0.3661(15)	0.25890(8)	4.7(3)
O(5)	0.2144(16)	0.1760(13)	0.10083(7)	3.7(3)
O(6)	-0.1083(14)	0.2332(14)	0.01718(8)	4.1(3)
O(7)	-0.1134(15)	0.4090(15)	0.17292(8)	4.8(3)
CA(1)	0.2624(28)	0.0341(27)	0.3536(15)	7.1(6)
CA(2)	0.1257(24)	0.0421(23)	0.3118(13)	4.6(5)
CA(3)	0.0130(23)	-0.0231(22)	0.3576(12)	4.1(4)
CA(4)	0.1455(25)	-0.0319(24)	0.3215(14)	5.2(5)
CA(5)	0.2937(29)	-0.1168(27)	0.3726(15)	7.5(6)
CA(6)	0.4686(26)	0.4780(26)	0.3715(14)	6.3(6)
CA(7)	0.3677(27)	0.3799(25)	0.2829(14)	5.6(5)
CA(8)	0.4519(25)	0.3118(25)	0.2325(14)	5.6(5)
CA(9)	0.3730(23)	0.2112(21)	0.1418(12)	3.9(4)
CA(10)	0.4700(26)	0.1321(25)	0.0821(14)	6.2(6)
CA(11)	-0.2485(24)	0.3125(23)	-0.1261(13)	5.3(5)
CA(12)	-0.1980(23)	0.3413(23)	-0.0210(13)	4.5(4)
CA(13)	-0.2427(23)	0.4634(23)	0.0250(13)	4.6(4)
CA(14)	-0.2013(23)	0.4910(22)	0.1176(13)	4.3(4)
CA(15)	-0.2649(24)	0.6289(23)	0.1655(13)	5.1(5)
CB(1)	0.0097(38)	0.4792(38)	0.4124(19)	9.6(8)
CB(2)	0.0986(35)	0.3906(34)	0.4678(23)	9.4(8)
CB(3)	0.0886(33)	0.4008(32)	0.5553(20)	9.6(8)

a) β^{11} = 0.00840(15), β^{22} = 0.01615(18), β^{33} = 0.00439(5)
β^{12} = 0.00462(12), β^{13} = 0.00241(7), β^{23} = 0.00227(7)

The tris(acetylacetonato)aquoytterbium(III) molecules are linked in pairs by hydrogen bonds of length

---

(9) See ref 7, p 216.

(10) D. E. Sands, *Acta Cryst.*, **21**, 868 (1966).

(11) E. C. Lingafelter and R. L. Braun, *J. Am. Chem. Soc.*, **88**, 2951 (1966).

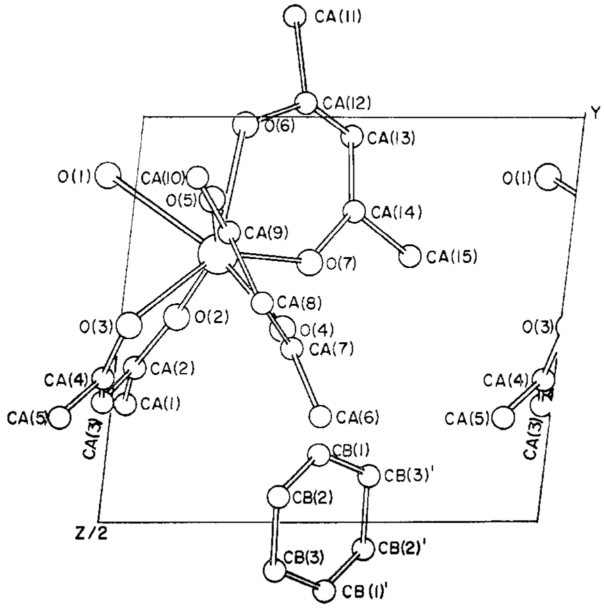

Figure 1.—Projection of a molecule along [100]. The primed and unprimed atoms are related by a center of symmetry.

TABLE III

BOND LENGTHS AND ANGLES, CHEMICALLY EQUIVALENT DISTANCES BEING GROUPED TOGETHER

Lengths, A[a]

Yb-O(1)	2.347(11)	CA(4)-CA(3)	1.44(2)
		CA(7)-CA(8)	1.37(3)
Yb-O(2)	2.204(12)	CA(9)-CA(8)	1.49(2)
Yb-O(3)	2.278(13)	CA(12)-CA(13)	1.35(2)
Yb-O(4)	2.231(12)	CA(14)-CA(13)	1.37(2)
Yb-O(5)	2.252(11)		
Yb-O(6)	2.258(12)	CA(2)-CA(1)	1.53(3)
Yb-O(7)	2.250(13)	CA(4)-CA(5)	1.58(3)
		CA(7)-CA(6)	1.50(3)
O(2)-CA(2)	1.24(2)	CA(9)-CA(10)	1.58(3)
O(3)-CA(4)	1.28(2)	CA(12)-CA(11)	1.55(2)
O(4)-CA(7)	1.25(2)	CA(14)-CA(15)	1.56(3)
O(5)-CA(9)	1.32(2)		
O(6)-CA(12)	1.34(2)	CB(1)-CB(2)	1.33(3)
O(7)-CA(14)	1.28(2)	CB(2)-CB(3)	1.39(3)
		CB(3)-CB(1)'	1.54(3)
CA(2)-CA(3)	1.41(2)		

Angles, degrees

O(2)-Yb-O(3)	74.8(4)	O(4)-CA(7)-CA(6)	120.4(21)
O(4)-Yb-O(5)	77.2(4)	O(5)-CA(9)-CA(10)	111.0(15)
O(6)-Yb-O(7)	74.5(4)	O(6)-CA(12)-CA(11)	112.6(17)
		O(7)-CA(14)-CA(15)	113.5(17)
Yb-O(2)-CA(2)	138.2(13)		
Yb-O(3)-CA(4)	133.9(14)	CA(1)-CA(2)-CA(3)	115.1(18)
Yb-O(4)-CA(7)	135.2(14)	CA(5)-CA(4)-CA(3)	119.0(19)
Yb-O(5)-CA(9)	128.5(11)	CA(6)-CA(7)-CA(8)	115.6(21)
Yb-O(6)-CA(12)	134.7(12)	CA(10)-CA(9)-CA(8)	124.0(17)
Yb-O(7)-CA(14)	136.2(13)	CA(11)-CA(12)-CA(13)	122.4(19)
		CA(15)-CA(14)-CA(13)	120.8(18)
O(2)-CA(2)-CA(3)	125.4(19)		
O(3)-CA(4)-CA(3)	125.1(20)	CA(2)-CA(3)-CA(4)	120.6(18)
O(4)-CA(7)-CA(8)	124.0(21)	CA(7)-CA(8)-CA(9)	123.5(20)
O(5)-CA(9)-CA(8)	125.0(17)	CA(12)-CA(13)-CA(14)	123.8(20)
O(6)-CA(12)-CA(13)	125.0(18)		
O(7)-CA(14)-CA(13)	125.7(19)	CB(3)'-CB(1)-CB(2)	122.1(27)
		CB(1)-CB(2)-CB(3)	117.0(30)
O(2)-CA(2)-CA(1)	119.4(19)	CB(2)-CB(3)-CB(1)'	120.7(26)
O(3)-CA(4)-CA(5)	115.9(19)		

[a] Primed and unprimed atoms are related by a center of symmetry.

O(1)–O(5)′ = O(1)′–O(5) = 2.66 Å, where the primes indicate atoms related to the unprimed atoms by the center of symmetry at 0, 0, 0. The distance between water molecules is O(1)–O(1)′ = 3.27 Å. The closest contact between pairs of molecules is 3.60 Å between O(2) and the CA(10) methyl group in the molecule generated by a lattice translation along *a*.

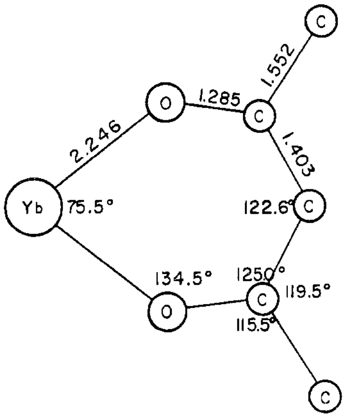

Figure 2.—Average dimensions of a ring in $Yb(C_5H_7O_2)_3(H_2O)\cdot 0.5C_6H_6$.

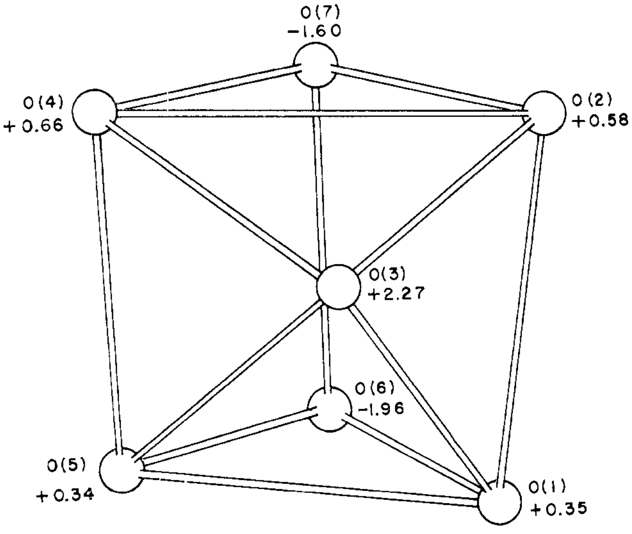

Figure 3.—Coordination polyhedron formed by the seven oxygen atoms bonded to the ytterbium atom. The distance of each atom from the central plane is given. The view is along a vector 30° from Yb–O(6) in the Yb–O(6)–O(7) plane.

TABLE IV

DIMENSIONS OF COORDINATION POLYHEDRON

LENGTHS, A[a]

O(1)-O(2)	3.09	O(4)-O(7)	2.86
O(1)-O(3)	2.86	O(7)-O(2)	2.86
O(1)-O(5)	2.98	O(7)-O(6)	2.73
O(1)-O(6)	2.77	O(2)-O(3)	2.72
O(4)-O(2)	3.51	O(3)-O(5)	2.92
O(4)-O(3)	2.83	O(5)-O(6)	2.84
O(4)-O(5)	2.80	O(2)-O(6)	3.85
O(4)-O(6)	3.93		

ANGLES, DEGREES[b]

O(4)-O(2)-O(3)	52.0	O(1)-O(2)-O(3)	58.6
O(2)-O(3)-O(4)	78.5	O(2)-O(3)-O(1)	67.2
O(2)-O(4)-O(3)	49.4	O(2)-O(1)-O(3)	54.2
O(4)-O(3)-O(5)	58.2	O(1)-O(3)-O(5)	62.0
O(3)-O(5)-O(4)	59.2	O(3)-O(5)-O(1)	58.0
O(3)-O(4)-O(5)	62.6	O(3)-O(1)-O(5)	59.9
O(4)-O(5)-O(6)	88.5	O(1)-O(5)-O(6)	56.7
O(5)-O(6)-O(4)	45.3	O(5)-O(6)-O(1)	64.3
O(5)-O(4)-O(6)	46.2	O(5)-O(1)-O(6)	59.0
O(4)-O(7)-O(6)	89.4	O(1)-O(2)-O(6)	45.3
O(7)-O(6)-O(4)	43.9	O(2)-O(6)-O(1)	52.7
O(7)-O(4)-O(6)	46.7	O(2)-O(1)-O(6)	82.0
O(4)-O(7)-O(2)	75.8	O(2)-O(6)-O(7)	47.8
O(7)-O(2)-O(4)	52.0	O(6)-O(7)-O(2)	87.1
O(7)-O(4)-O(2)	52.1	O(6)-O(2)-O(7)	45.1

a) STANDARD DEVIATION = 0.02 A.
b) STANDARD DEVIATIONS ARE 0.3° to 0.5°.

The coordination polyhedron formed by the seven oxygen atoms around an ytterbium atom is a capped trigonal prism (Figure 3), with O(3) capping the face O(1)–O(2)–O(4)–O(5). The dimensions of the coordination polyhedron are given in Table IV. This polyhedron is identical, within experimental error, with the polyhedron found about Yb(1) in the unsolvated $Yb(C_5H_7O_2)_3(H_2O)$ structure,[3] and confidence in the reality of such features of the polyhedron as the deviations of the ytterbium atom from the mean planes of the ligands may be obtained by comparing the two structures.

The benzene molecule is centered on the center of symmetry at 0, $^1/_2$, $^1/_2$. The mean deviation of the carbon atoms from the mean plane of the benzene ring is 0.019 Å, and the sum of the interior angles of the ring is 719.6°. Discrepancies in the carbon–carbon bond lengths (Table III) of several standard deviations must be interpreted as indicative of systematic errors in the data, such as uncorrected adsorption, rather than as implying any actual distortion of the molecule. The shortest distance between a benzene carbon atom and another carbon or oxygen atom is 3.53 Å for the CB(1)–O(4) contact.

The volume occupied by two tris(acetylacetonato)-aquoytterbium(III) molecules in the unsolvated structure[3] is 931.8 Å^3. The molecular volume of benzene, based on the density of the liquid at 25°, is 147.6 Å^3. The sum of these volumes is 1079.4 Å^3, as compared with a unit cell volume of 1077.9 Å^3 in the crystal structure of the benzene solvate. These figures suggest that the efficiency of packing of the molecules is about the same for the solvated and unsolvated structures.

CONTRIBUTION FROM THE LOS ALAMOS SCIENTIFIC LABORATORY,
UNIVERSITY OF CALIFORNIA, LOS ALAMOS, NEW MEXICO 87544

The Crystal Structure of Ammonium Hexafluorocerate(IV), $(NH_4)_2CeF_6$[1]

BY ROBERT R. RYAN, ALLEN C. LARSON, AND F. H. KRUSE

Received June 5, 1968

The crystal structure of ammonium hexafluorocerate(IV), $(NH_4)_2CeF_6$, has been determined from counter measurements of 561 reflections and refined by full-matrix least squares to an R factor of 0.032. The space group is Pbcn (no. 60); D_x = 3.05 and $D_m = 3.05 \pm 0.02$ g/cm^3. The unit cell has dimensions a = 7.026 (3), b = 12.098 (6), and c = 7.439 (8) Å; Z = 4. Of the 561 reflections measured, 389 were judged to be observed. The structure consists of infinite chains of eightfold coordinated cerium atoms parallel to the c axis, joined by NH_4^+ ions. The fluorine configuration about the Ce can be roughly described as a square antiprism. The average Ce–F distance is 2.26 Å and the average F–F distance for nearest neighbors within an antiprism is 2.77 Å. Hydrogen bonding causes this particular structure to be stabilized over the trigonal-dodecahedral arrangement found in the similar Rb_2UF_6 and K_2ZrF_6 compounds.

Introduction

The preparation of ammonium hexafluorocerate(IV) was first reported by Besse and Capestan,[2] who indexed the powder pattern on the basis of a cubic cell with a' = 10.52 Å and claimed a measured density of 3.27 g/cm^3. For Z = 8, D_x = 3.31 g/cm^3 for this cell. Penneman[3] of this laboratory noticed that these values for the volume and Z give a very low value for the fluorine volume, as compared to similar actinide fluoride compounds. Further, there is no evidence for cubic phases of any other 2:1 NH_4F–actinide(IV) fluoride complexes. He was therefore prompted to prepare this material. Optical examination showed single crystals of $(NH_4)_2CeF_6$ to be biaxial and thus this compound must have orthorhombic or lower symmetry. Subsequent examination of precession films showed the symmetry to be orthorhombic with cell dimensions which are nearly those which would permit indexing of powder data on the basis of the cubic cell of Besse and Capestan,[2] *i.e.*, $a'/a = {}^3/_2$, $a'/b = \sqrt{3}/2$, and $a'/c = \sqrt{2}$.

The single-crystal X-ray investigation of this compound was undertaken for three reasons: (1) No compound of known structure could be found with which it is isostructural. (2) Comparison of the powder patterns indicated it is isostructural with the α phase of $(NH_4)_2UF_6$ for which no single crystals were then available. (Subsequent comparisons of single-crystal data, when they became available, confirmed that the two compounds were indeed isostructural. This is of interest because of the existence of four closely related polymorphs for $(NH_4)_2UF_6$ over a narrow temperature range[4] (~70°), and the elucidation of these structures should provide information concerning the energy relationships between some of the possible coordination polyhedra for uranium.) (3) It is also of interest that after extensive investigation the only phase to be found for the Ce compound is the α phase. It is hoped that the knowledge of this structure plus the structure of the γ-$(NH_4)_2UF_6$ (room-temperature stable phase) compound currently being worked on will provide some information concerning these questions.

Experimental Section

Crystals were prepared by cooling a saturated solution of $(NH_4)_2CeF_6$ in a 15% NH_4F–H_2O solution. A needlelike crystal was selected and cut to a length suitable for examination by X-

---

(1) This work was sponsored by the U. S. Atomic Energy Commission.

(2) J. P. Besse and M. Capestan, *Bull. Soc. Chim. France*, 577 (1966).

(3) R. A. Penneman, unpublished results, to be submitted for publication.

(4) R. A. Penneman, F. H. Kruse, R. S. George, and J. S. Coleman, *Inorg. Chem.*, **3**, 309 (1964).