stoichiometry, but a modifying principle can be introduced which will make some distributions more likely than others by assuming that the oxygen ions will prefer identical or nearly identical cation environments. The ion distribution around oxygens characterized by 0112 and 0121 is that of an inverse spinel. The cation arrangement of $MgGa_2S_4$[18] is an example of the case $n = 4$, which is a partially inverted structure of solution V, and the cation arrangement is listed in Table VI. The number of integral formula weights per unit cell

(18) C. Romers, B. A. Blaisse, and D. J. W. Ijdo, *Acta Cryst.*, **23**, 634 (1967).

will be given by the expression $I[\text{lcm}(4,n)/4]$, where I is an integer and the numerator is the least common multiplier of 4 and n.

It is quite possible that the local energy around oxygen due to the ionic charges becomes higher as n increases. If n is small, *e.g.*, 1, then each oxygen ion has the same cation environment. For $n = 2$ the cation environment becomes different for the inverted spinel structure and these differences among oxygen ions would become more pronounced as n increases. The fact that spinel, olivine, and Ag_2HgI_4 structures occur so frequently indicates that structures in which the local surroundings of anions differ greatly are unstable.

The Crystal Structure of Cesium Tetrakis(hexafluoroacetylacetonato)yttrate(III). A Novel Stereoisomer Having Dodecahedral Eight-Coordination[1]

By M. J. BENNETT, F. A. COTTON, P. LEGZDINS,[2] and S. J. LIPPARD

The crystal and molecular structures of cesium tetrakis(hexafluoroacetylacetonato)yttrate(III), $Cs[Y(CF_3COCHCOCF_3)_4]$, have been determined from 1650 independent, nonzero reflections collected with a counter diffractometer. The compound crystallizes in the orthorhombic space group Pbcn with four molecules in a unit cell of dimensions: $a = 8.679 \pm 0.005$ Å, $b = 21.518 \pm 0.008$ Å, and $c = 17.553 \pm 0.008$ Å ($\rho_{obsd} = 2.10$ g cm^{-3}; $\rho_{calcd} = 2.12$ g cm^{-3}). The structure, excluding hydrogen atoms, was solved from Patterson and electron density maps and refined by least-squares methods to a conventional R factor of 6.5%. The molecules are monomeric with the yttrium being dodecahedrally coordinated to the eight essentially equivalent oxygen atoms (average Y–O, 2.323 (4) Å). Both coplanar ligands show dihedral folding of 7.6° along the O–O line in the chelate rings. In contrast with other dodecahedral ML_4 (L = bidentate ligand) structures, each ligand in this structure spans adjacent vertices *between* the two bisphenoids constituting the dodecahedron, resulting in over-all D_2 symmetry. The monomeric anions are linked together in infinite columns parallel to the crystallographic a axis by a close Cs–F association (3.2–3.7 Å), but other intermolecular distances are not unusual. The structure is in accord with the mass spectral evidence for the existence of a strong ion pair.

Introduction

During the course of our investigation of the solid-state and solution properties of several anionic eight-coordinate β-diketonate complexes of yttrium(III),[3,4] it was discovered that certain large but flexible organic cations, such as $(C_6H_5)_4As^+$, gave rise to a polymorphism that was not observed for the simple inorganic cations (Na^+, K^+, Cs^+).[4,5] It was considered possible that this polymorphism might correspond to geometric isomerism for the eight-coordinate anions. This type of isomerism, although considered to be a possibility,[6] has never been observed in tetrachelate metal atom complexes.[7] As part of an investigation to test this

structural possibility, it was decided to carry out a single-crystal X-ray analysis of $Cs[Y(HFA)_4]$, where HFA represents the hexafluoroacetylacetonate ion, $CF_3COCHCOCF_3^-$.

Experimental Procedures

Crystalline $Cs[Y(HFA)_4]$ was prepared by the reaction of $Cs(HFA)$ with YCl_3 in aqueous ethanol as described previously[8] and was recrystallized from the same solvent. *Anal.* Calcd for $CsYC_{20}H_4O_8F_{24}$: C, 22.85; H, 0.38; F, 43.43. Found: C, 22.71; H, 0.46; F, 43.27. The analyses were performed by Galbraith Microanalytical Laboratories, Knoxville, Tenn.

The well-formed, transparent, needle-like crystals showed mmm morphology under optical examination, and preliminary Weissenberg (0kl and 1kl levels) and precession (h0l, h1l, hk0, and hk1 levels) photographs, taken with Cu Kα radiation, confirmed this as the Laue symmetry. Moreover, the observed systematic absences—0kl for $k \neq 2n$, h0l for $l \neq 2n$, and hk0 for $h + k \neq 2n$—suggested the unique choice of the orthorhombic space group Pbcn (no. 60). The following unit cell dimensions were obtained, at 22°, by a method described earlier,[9] with 2θ values measured on a General Electric XRD-5 manually operated single-crystal diffractometer using Cu Kα radiation (λ(Kα₁)

(1) Work supported by the National Science Foundation under Grant No. 7034X.

(2) Arthur D. Little Predoctoral Fellow, 1966–1967.

(3) F. A. Cotton, P. Legzdins, and S. J. Lippard, *J. Chem. Phys.*, **45**, 3461 (1966).

(4) S. J. Lippard, Proceedings of the 9th International Conference on Coordination Chemistry, St. Moritz, 1966, p 47.

(5) H. Bauer, J. Blanc, and D. L. Ross, *J. Am. Chem. Soc.*, **86**, 5125 (1964).

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

(7) For general discussions of the stereochemistry of eight-coordinate complexes see: (a) S. J. Lippard, *Progr. Inorg. Chem.*, **8**, 109 (1967); (b) E. L. Muetterties and C. M. Wright, *Quart. Rev.* (London), **21**, 109 (1967).

(8) S. J. Lippard, *J. Am. Chem. Soc.*, **88**, 4300 (1966).

(9) M. J. Bennett, F. A. Cotton, and J. Takats, *ibid.*, **90**, 903 (1968).

1.5405 Å, λ(Kα₂) 1.5443 Å): $a = 8.679 \pm 0.005$ Å, $b = 21.518 \pm 0.008$ Å, $c = 17.553 \pm 0.008$ Å. The pycnometrically observed density of 2.10 g cm⁻³ agrees with the calculated density of 2.12 g cm⁻³ for a formula weight of 1049.8, $Z = 4$, and a unit cell volume of 3278 Å³.

Preliminary intensity data were collected by the equiinclination Weissenberg technique for $0kl$ through $6kl$ using Cu Kα radiation, and the results of this study have been briefly reported.[10] Since further refinement of the data did not proceed satisfactorily, it was decided to collect a new data set. Laue photographs of a series of optically satisfactory crystals indicated that disorder was variable from crystal to crystal. Consequently, the best crystal, having well-developed {100}, {010}, and {001} faces and approximate dimensions $0.1 \times 0.1 \times 0.3$ mm, was selected for the subsequent work.

Intensity data were collected on the diffractometer with the crystal oriented so that a^* was coincident with the ϕ axis of the instrument.[11] The crystal was placed 5.73 in. from the source while the distance from the crystal to the detector with a circular aperture of 2° was 7.05 in. The integrated intensities of 3342 independent reflections within the sphere $\theta \leq 70°$ were measured using nickel-filtered Cu Kα radiation. The intensities were measured using a scintillation counter (the linear counting rate of which was never exceeded) with a pulse height discriminator set to accept 95% of the Cu Kα radiation with the window centered on the Cu Kα peak. The data were collected using a θ-2θ scan technique at a 2θ scan rate of 4°/min. The peak counts, P, were obtained from a 2θ scan of 2.66° from $2\theta_{calcd} - 1.33°$ to $2\theta_{calcd} + 1.33°$. Stationary background counts, B_1 and B_2, of 20 sec were taken at each of the limits of the scan. From these readings the intensity, I, assuming a linear change in background, or its equivalent, between the two limits of the scan, is given by: $I = P - (B_1 + B_2)$. Periodic checks of three standard reflections showed a variation in intensity ($\pm 2\%$) having no 2θ dependence, which is consistent with fluctuations in the electronic circuits rather than with crystal decomposition.

The experimental values of I were processed using the program pmmo written by M. J. Bennett. A number of reflections (1692) were rejected as unreliable using the two criteria: (1) $I < 0$; (2) $I < 2(P + [B_1 + B_2])^{1/2}$. The remaining 1650 independent intensities were next assigned standard deviations, $\sigma(F)$, according to Ibers[12] with $p^2 = 0.002$, and corrections on I and $\sigma(F)$ were made for Lorentz and polarization effects.

Appreciable variations in the intensities of the $h00$ reflections were observed as a function of crystal orientation, ϕ scans of several of these reflections indicating a variation of 20%. Consequently, absorption corrections ($\mu = 130.0$ cm⁻¹), based on the equations of the crystal faces, were applied to I and $\sigma(F)$ and a set of $|F_o|^2$ and $|F_o|$ values (on a relative scale) was thus obtained.

Solution and Refinement of the Structure

From the three-dimensional map of the Patterson function, both the cesium and yttrium atoms were located on twofold axes in the crystal. The rest of the structure was solved by successive conventional least-squares and Fourier calculations. The scattering factors used during the analysis were those of Cromer and Waber,[13] and the anomalous dispersion corrections[14] for Cs⁺ ($\Delta f' = -1.80$ e⁻, $\Delta f'' = 8.00$ e⁻) and Y ($\Delta f'$

= -0.75 e⁻, $\Delta f'' = 2.20$ e⁻), applied to F_c, were taken from a standard source.[15] The function minimized in the least-squares refinements was $\Sigma w(|F_o| - |F_c|)^2$, where $|F_o|$ is the observed structure amplitude, $|F_c|$ is the calculated structure amplitude, and $w = [\sigma(F)]^{-2}$.

Five cycles of refinement of coordinates and isotropic temperature parameters for all atoms converged to a value of 0.192 for the residual R_1 (defined as $\Sigma ||F_o| - |F_c||/\Sigma |F_o|$). At this stage of the refinement, the fluorine temperature parameters ranged from 12 to 17 Å² and a subsequent difference Fourier synthesis, phased on all the atoms, indicated unassigned electron density (~ 2 e⁻/Å³) in the regions of the terminal CF₃ groups. Consequently, even with the best-ordered crystal that we could find, evidence from the thermal parameters and electron density maps suggested that the CF₃ moieties were behaving as hindered rotors. Since the fluorine parameters are not of great importance in this study and should have negligible correlation with the parameters of primary interest, this motion was approximated by anisotropic temperature factors of the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. Because of limitations in the core storage of the IBM 7094 computer, two cycles of refinement were then executed using anisotropic temperature factors for the CF₃ groups only, after which R_1 dropped to 0.108 and the weighted R factor, $R_2 = \{\Sigma w[|F_o| - |F_c|]^2/\Sigma w|F_o|^2\}^{1/2}$, was 0.114.

With the advent of the IBM 360 computer, we were able to perform three final cycles of anistropic refinement for all atoms which led to convergence and $R_1 = 0.065$ and $R_2 = 0.076$. The chosen weighting scheme satisfied Cruickshank's criterion[16] as $\Sigma w[|F_o| - |F_c|]^2$ was substantially the same in different ranges of both $|F_o|$ and $(\sin \theta)/\lambda$. A comparison of the final values of F_o and F_c suggested that a correction for extinction was not necessary.

During the final cycle of refinement, no positional or thermal parameter shifted more than one-fourth or one-half, respectively, of its estimated standard deviation. The final value of the standard deviation of an observation of unit weight was 1.030. While this was satisfactorily close to the expected value of unity, it was nevertheless considered to be too low since the data undoubtedly had systematic errors due to various nonisotropic effects and it thus suggested that the use of a smaller p factor might have been preferable. A final difference Fourier map showed peaks of 0.94 and 0.87 e⁻/Å³ in the vicinity of the cesium and yttrium atoms, respectively. All other peaks were less than 0.70 e⁻/Å³; no attempt was made to deduce the hydrogen atom positions. The observed and final calculated structure amplitudes are given in Table I.

A comparison of the final parameters of this study with those obtained from the film data showed the former to have smaller estimated standard deviations and lower temperature factors for all atoms, thereby indicating that the original crystal had a higher degree

(10) S. J. Lippard, F. A. Cotton, and P. Legzdins, *J. Am. Chem. Soc.*, **88**, 5930 (1966).

(11) For this experiment, the conditions for multiple diffraction were maximized: W. A. Zachariasen, *Acta Cryst.*, **18**, 705 (1965). Since agreement between observed and calculated structure factors was later found to be very good, the effects are probably small in this case.

(12) P. W. R. Corfield, R. J. Doedens, and J. A. Ibers, *Inorg. Chem.*, **6**, 197 (1967).

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

(14) C. T. Prewitt, Ph.D. Thesis, Massachusetts Institute of Technology, 1962, p 163.

(15) C. H. Dauben and D. H. Templeton, *Acta Cryst.*, **8**, 841 (1955).

(16) D. W. J. Cruickshank in "Computing Methods in Crystallography," J. S. Rollett, Ed., Pergamon Press Inc., New York, N. Y., 1965, p 113.

TABLE I

OBSERVED AND CALCULATED STRUCTURE AMPLITUDES (×10) (IN ELECTRONS) FOR $Cs[Y(HFA)_4]$

K	L	FOBS	FCAL
*****H =	0******		
0	2	3334	3056
0	4	1243	1013
0	6	1284	1254
0	8	1254	1340
0	10	1405	1407
0	12	1179	1162
0	14	737	765
0	16	634	632
0	18	299	307
0	20	213	253
2	0	2993	2741
2	1	1968	1917
2	2	2309	2301
2	3	1439	1404
2	4	3605	3604
2	5	365	372
2	6	1597	1638
2	7	364	402
2	8	1947	1903
2	9	665	907
2	10	755	811
2	11	762	769
2	12	964	934
2	13	442	452
2	14	551	537
2	15	427	415
2	16	347	345
2	17	301	322
2	18	243	231
2	20	263	236
4	0	1982	1863
4	1	291	295
4	2	311	299
4	3	3941	3763
4	4	1514	1469
4	5	2302	2265
4	6	1518	1490
4	7	1531	1571
4	8	884	845
4	9	775	810
4	10	633	623
4	11	931	917
4	12	403	434
4	13	864	844
4	14	172	82
4	15	313	350
4	16	222	155
4	17	356	305
4	19	213	209
4	20	101	96
6	0	1272	1216
6	1	1729	1667
6	2	542	521
6	3	1519	1533
6	4	197	121
6	5	1775	1775
6	6	150	145
6	7	1162	1135
6	9	1600	1575
6	11	879	956
6	12	164	133
6	13	337	333
6	14	146	194
6	15	178	216
6	17	271	256
6	19	190	140
8	0	508	496
8	1	987	985
8	3	1020	1004
8	4	201	214
8	5	151	193
8	6	387	409
8	7	158	142
8	8	799	765
8	9	1245	1223
8	10	461	515
8	11	576	559
8	13	277	302
8	14	146	164
8	16	183	121
10	0	388	358
10	1	197	177
10	2	715	687
10	3	252	149
10	5	227	202
10	7	163	145
10	8	411	374
10	9	400	359
10	11	171	115
10	12	174	173
10	13	148	166
10	14	132	138
10	15	147	125
12	0	402	380
12	1	178	200
12	2	428	397
12	3	132	160
12	4	304	268
12	5	252	231
12	6	175	213
12	7	147	142
12	8	305	308
12	9	141	178
12	14	216	166
14	0	452	395
14	1	239	239
14	2	174	152
14	3	329	366
14	4	587	615
14	5	150	98
14	6	673	654
14	7	215	225
14	8	280	279
14	9	161	214
14	10	282	229
14	11	264	286
14	12	385	355
14	13	193	152
14	16	174	108
14	18	116	91
16	0	156	115
16	1	611	540
16	2	282	283
16	3	760	746
16	4	260	259
16	5	332	281
16	6	227	250
16	7	231	256
16	8	531	516
16	9	575	639
16	10	244	274
16	11	572	531
16	12	195	173
16	13	152	159
16	14	117	116
16	16	132	94
16	17	81	87
18	0	131	178
18	1	435	372
18	3	535	533
18	5	673	645
18	7	759	754
18	8	176	173
18	9	448	463
18	11	326	348
18	13	260	246
18	15	147	156
20	0	342	308
20	1	322	323
20	2	342	297
20	3	513	519
20	4	434	487
20	5	513	507
20	6	354	390
20	7	254	316
20	8	289	259
20	9	265	256
20	10	213	220
20	11	225	231
20	13	183	169
20	14	105	93
22	0	962	864
22	1	217	205
22	2	579	583
22	4	185	215
22	5	121	85
22	6	279	298
22	8	361	378
22	10	214	224
22	12	161	170
24	0	511	535
24	1	213	229
24	2	421	432
24	3	107	100
24	4	218	241
24	5	103	91
24	6	147	133
24	8	156	160
24	9	86	91
26	0	45	93
26	1	240	220
26	2	85	94
26	3	238	273
26	4	119	108
*****H =	1******		
0	2	465	472
0	4	1988	2032
0	6	222	205
0	8	400	357
0	12	132	179
0	16	175	146
1	0	459	359
1	1	259	249
1	2	383	347
1	3	327	349
1	4	151	186
1	5	1592	1735
1	7	518	511
1	8	280	284
1	9	157	145
1	10	201	249
1	11	269	264
1	13	140	84
2	1	270	277
2	2	154	81
2	3	871	944
2	4	967	923
2	5	225	216
2	6	583	571
2	7	224	232
2	10	279	270
2	12	144	147
2	13	175	151
3	0	665	630
3	1	629	626
3	2	865	860
3	3	1621	1626
3	4	1363	1432
3	5	1184	1145
3	6	99	95
3	7	791	762
3	9	635	613
3	10	422	462
3	11	437	461
3	12	391	485
3	13	345	295
3	14	201	202
3	15	132	172
3	16	208	157
3	17	170	161
3	18	122	150
4	1	217	362
4	2	3306	3252
4	3	999	1017
4	4	112	76
4	5	182	195
4	6	374	399
4	7	631	657
4	8	118	134
4	10	302	287
4	11	283	265
4	12	239	195
4	13	166	177
5	0	707	664
5	1	1869	1626
5	2	537	467
5	3	1457	1396
5	4	128	107
5	5	2210	2234
5	6	483	450
5	7	1020	1085
5	8	214	225
5	9	658	675
5	10	282	234
5	11	1064	1009
5	13	623	660
5	15	400	355
5	16	159	80
5	17	220	219
5	20	97	57
6	1	637	581
6	2	605	594
6	3	107	82
6	4	302	294
6	5	519	495
6	6	98	101
6	7	169	160
6	9	246	240
6	10	369	337
6	11	117	65
7	0	1537	1489
7	1	1652	1621
7	2	1148	1065
7	3	1699	1624
7	4	1236	1167
7	5	1499	1360
7	6	505	490
7	7	1735	1610
7	8	154	170
7	9	1434	1471
7	10	230	269
7	11	744	739
7	13	529	505
7	14	293	261
7	15	414	404
7	16	181	134
7	17	283	276
7	19	157	169
8	1	358	244
8	2	353	359
8	3	814	729
8	4	200	216
8	5	943	970
8	6	435	432
8	7	201	196
8	8	207	205
8	9	166	90
8	11	320	327
8	12	127	126
9	0	1964	1705
9	1	2122	1945
9	2	1644	1681
9	3	625	618
9	4	1425	1502
9	5	1038	1092
9	6	1337	1393
9	7	997	967
9	8	1248	1253
9	9	543	538
9	10	667	619
9	11	716	708
9	12	416	355
9	13	482	439
9	14	364	378
9	15	226	241
9	16	252	302
9	17	168	196
9	18	219	209
9	19	108	124
10	1	331	335
10	2	386	375
10	4	741	732
10	5	229	256
10	8	128	126
10	10	174	164
10	12	227	179
10	18	129	86
11	0	1431	1402
11	1	328	297
11	2	1768	1784
11	3	186	164
11	4	2046	2055
11	5	271	255
11	6	1440	1389
11	9	906	869
11	10	750	734
11	12	636	620
11	13	175	97
11	14	540	524
11	16	361	378
11	18	221	224
12	1	270	274
12	3	196	215
12	5	272	305
12	7	346	412
12	11	151	148
12	13	199	161
13	0	1415	1415
13	1	746	724
13	2	1297	1225
13	3	593	535
13	4	1328	1286
13	5	604	580
13	6	1111	1093
13	7	419	449
13	8	680	727
13	9	141	135
13	10	468	499
13	11	252	246
13	12	434	381
13	13	297	297
13	14	389	382
13	15	199	203
13	16	313	277
13	17	103	87
13	18	154	159
14	3	168	144
14	4	169	122
14	6	205	254
14	7	141	151
14	10	148	101
15	0	418	362
15	1	918	930
15	2	597	601
15	3	724	694
15	4	486	483
15	5	503	528
15	6	425	481
15	7	665	628
15	8	448	491
15	9	654	686
15	10	133	105
15	11	400	412
15	12	132	64
15	13	304	290
15	14	159	110
15	15	261	229
15	16	112	96
15	17	146	142
16	3	155	195
16	4	143	182
16	5	275	298
16	7	148	144
17	1	519	494
17	3	447	442
17	4	147	71
17	5	552	576
17	7	512	545
17	8	340	312
17	9	356	332
17	11	310	308
17	13	258	233
17	15	143	134
18	1	132	87
18	2	330	289
18	4	218	188
18	6	132	137
18	8	174	182
19	0	175	126
19	2	162	136
19	3	311	321
19	4	581	541
19	5	177	171
19	6	363	358
19	8	129	107
19	9	125	98
19	10	192	208
19	11	154	89
19	12	143	184
20	2	131	150
21	0	186	143
21	1	129	133
21	2	182	175
21	3	168	191
21	4	127	111
21	12	98	75
21	13	76	54
23	1	384	367
23	6	120	81
23	7	124	109
23	9	127	116
25	0	217	225
25	2	218	229
25	3	93	59
25	4	181	174
25	6	116	86
*****H =	2******		
0	0	1520	1448
0	2	3358	3492
0	4	1486	1533
0	6	2982	3180
0	8	1888	2005
0	10	1302	1292
0	12	904	922
0	14	828	802
0	16	468	444
0	18	318	303
0	20	117	168
1	1	123	131
1	2	1921	1989
1	3	294	286
1	4	318	352
1	5	499	518
1	6	362	334
1	7	679	733
1	8	118	88
1	10	218	243
2	0	1719	1653
2	1	2487	2574
2	2	3041	3089
2	3	858	841
2	4	1541	1582
2	5	1105	1151
2	6	2405	2459
2	7	817	837
2	8	1347	1353
2	9	702	698
2	10	1122	1213
2	11	539	586
2	12	759	766
2	13	436	414
2	14	601	608
2	15	265	295
2	16	322	331
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6	3	807	836
6	5	474	490
6	6	160	108
6	7	496	506
6	9	558	559
6	11	512	463
6	13	311	299
6	15	171	169
7	1	148	153
7	3	242	230
7	5	177	184
7	7	164	171
7	12	124	66
7	14	111	71
8	0	295	254
8	1	397	362
8	2	260	228
8	3	365	380
8	4	182	223
8	5	503	497
8	6	165	178
8	7	279	248
8	8	184	165
8	9	184	141
8	10	168	152
8	11	293	267
8	12	209	180
8	13	177	104
9	2	296	240
9	3	153	170
9	4	171	161
10	0	133	143
10	1	154	135
10	2	131	132
10	3	172	166
10	4	311	326
10	6	340	370
11	2	278	275
11	4	245	234
11	6	332	336
11	10	126	93
12	0	167	122
12	1	133	85
12	3	134	89
12	4	188	168
12	5	203	194
12	6	285	281
14	1	132	95
14	2	285	239
14	3	215	183
14	4	363	365
14	5	270	251
14	6	255	275
14	8	255	238
14	10	146	150
14	11	134	80
14	12	111	72
15	1	249	205
15	3	234	265
16	0	221	224
16	1	413	366
16	2	162	161
16	3	345	319
16	4	167	161
16	5	262	284
16	6	181	198
16	7	276	281
16	8	170	153
16	9	224	207
16	10	97	103
18	1	514	527
18	3	345	364
18	5	293	263
18	7	245	248
19	2	136	91
20	0	316	324
20	1	222	207
20	2	266	275
20	3	249	265
*****H =	7******		
0	8	159	105
0	10	234	244
0	12	123	84
1	1	157	183
1	3	361	357
1	6	237	238
1	7	135	83
1	8	305	266
2	4	219	196
2	7	135	124
2	11	164	114
3	0	142	179
3	1	158	135
3	2	230	214
3	3	370	359
3	4	252	265
3	5	495	526
3	6	215	233
3	7	460	450
3	8	156	169
3	9	304	290
3	10	279	304
3	11	163	196
3	12	220	196
4	3	149	160
4	4	167	183
4	6	134	88
4	7	189	206
5	0	140	138
5	1	252	228
5	2	280	253
5	3	860	874
5	5	824	836
5	7	552	543
5	8	152	126
5	9	453	471
5	11	365	342
5	12	191	129
5	13	269	245
6	5	131	90
6	6	132	133
7	0	417	396
7	1	898	878
7	2	364	343
7	3	644	652
7	4	251	229
7	5	581	617
7	6	322	336
7	7	567	607
7	8	200	222
7	9	509	479
7	10	124	56
7	11	454	395
7	12	112	82
7	13	337	297
8	1	194	178
8	2	288	333
8	5	184	153
8	6	130	163
9	0	950	872
9	1	440	469
9	2	710	694
9	3	557	571
9	4	556	538
9	5	397	455
9	6	528	573
9	7	346	377
9	8	389	380
9	9	412	365
9	10	363	322
9	11	235	253
9	12	219	226
9	13	177	184
11	0	853	794
11	2	720	720
11	4	724	716
11	6	755	718
11	8	459	472
11	10	313	313
11	12	262	263
12	3	151	94
13	0	659	528
13	1	249	226
13	2	617	629
13	3	332	331
13	4	589	537
13	5	237	227
13	6	456	434
13	7	213	173
13	8	277	308
13	9	183	161
13	10	226	222
14	1	145	57
14	2	162	124
15	0	329	311
15	1	437	400
15	2	170	216
15	3	403	389
15	4	178	207
15	5	342	328
15	6	203	162
15	7	312	298
15	8	130	106
17	1	279	270
17	3	296	301
17	5	269	249
*****H =	8******		
0	0	1219	1163
0	2	916	980
0	4	702	694
0	6	580	641
0	8	753	786
0	10	539	503
0	12	361	305
1	1	279	312
1	3	166	217
2	0	931	878
2	1	516	538
2	2	738	728
2	3	361	357
2	4	739	788
2	5	250	277
2	6	580	607
2	7	346	378
2	8	462	427
2	9	352	368
2	10	391	371
2	11	224	210
2	12	353	304
3	6	169	121
4	0	562	525
4	1	491	472
4	2	349	386
4	3	732	691
4	4	330	344
4	5	635	652
4	6	305	272
4	7	452	447
4	8	206	185
4	9	415	387
4	10	115	135
4	11	371	340
5	2	140	95
5	4	134	112
5	5	129	99
6	1	503	532
6	3	457	435
6	5	472	441
6	7	402	415
6	8	120	61
6	9	332	338
6	11	313	259
7	3	153	205
7	7	142	80
7	8	137	124
8	0	135	164
8	1	262	250
8	2	221	208
8	3	244	259
8	4	197	147
8	5	146	140
8	6	123	104
8	7	197	172
8	8	116	145
8	9	196	175
8	10	136	146
8	11	168	151
9	3	152	113
10	0	201	155
11	4	159	135
12	0	140	155
13	3	148	113
13	4	112	70
14	0	129	124
14	2	141	180
14	4	218	215
14	5	144	65
*****H =	9******		
1	0	229	130
3	1	340	317
3	2	159	140
3	3	175	140
3	4	214	216
3	5	195	214
3	6	157	140
3	7	149	137
3	8	129	97
3	9	120	120
4	5	143	125
4	6	120	74
5	1	546	510
5	3	377	411
5	5	313	339
5	6	117	122
5	7	235	239
7	0	278	223
7	1	527	532
7	2	234	197
7	3	377	385
7	4	156	160
7	5	399	421
7	6	159	103
7	7	334	350
9	0	437	421
9	1	257	254
9	2	440	405
9	3	309	324
9	4	383	365
9	5	246	276
9	6	264	310
11	0	442	477
11	2	474	477
*****H =	10******		
0	0	506	507
0	2	461	531
0	4	507	526
2	0	466	396
2	1	334	311
2	2	425	403
2	3	252	255
4	1	352	374
4	2	153	142

of disorder. This was subsequently confirmed by examination of a Laue photograph of the original crystal.

The following programs for the IBM 7094 and 360 computers were used in the structure analysis and interpretation: (1) D. P. Shoemaker, MIT X-ray goniometer package, MIXG-2 (1962); (2) R. C. Elder, convenient listing of diffractometer settings, PDATA2; (3) M. J. Bennett, general data reduction program, PMMO; (4) a modified version of W. G. Sly, D. P. Shoemaker, and J. H. van der Hende, two- and three-dimensional crystallographic Fourier summation, MIFR-2 (1962);

(5) a modified version of A. Zalkin, Fourier analysis program, FORDAP; (6) W. C. Hamilton, absorption correction program, GONO9; (7) C. T. Prewitt, full-matrix crystallographic least-squares programs, SFLSQ3 (1962) and SFLS5 (1966); (8) J. S. Wood, molecular geometry with estimated standard deviations, MGEOM (1964); (9) D. P. Shoemaker, crystallographic bond distance, bond angle, and dihedral angle program, DISTAN (1963); (10) D. P. Shoemaker and R. C. Srivastava, program for analysis of anisotropic temperature factors, VIBELL (1963); (11) R. C. Elder, data presentation program, PUBTAB.

Results

The molecular structure and atom numbering system are shown in Figure 1. Figures 2 and 3 show selected
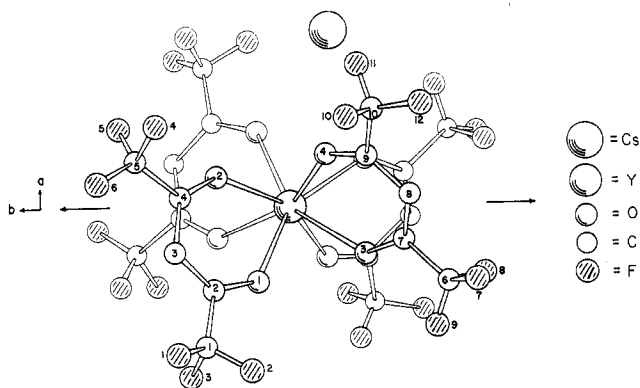
Figure 1.—The molecular structure projected on the *ab* plane. The arrows indicate the crystallographic twofold axis which passes through the yttrium atom.
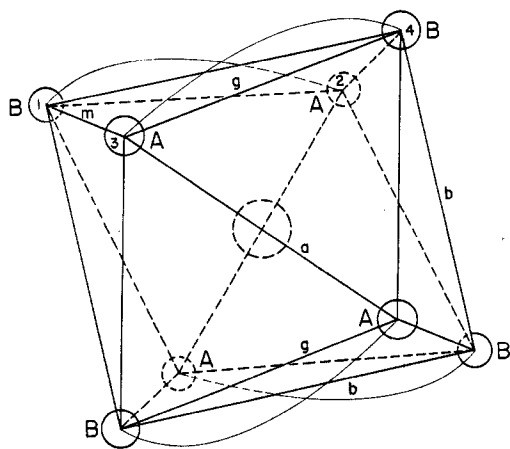
Figure 2.—View of coordination dodecahedron down the twofold axis through yttrium (from −*b* direction).

portions of the molecule as (010) and (100) projections, respectively. Final atomic positional coordinates are given in Table II with the listed standard deviations being derived from the inverse matrix of the final least-squares refinement cycle. Anisotropic thermal parameters and the root-mean-square amplitudes of vibration with their direction cosines are listed in Table III. Intramolecular distances and bond angles are given in Tables IV and V, while Table VI shows some selected intermolecular contacts. The characteristic param-
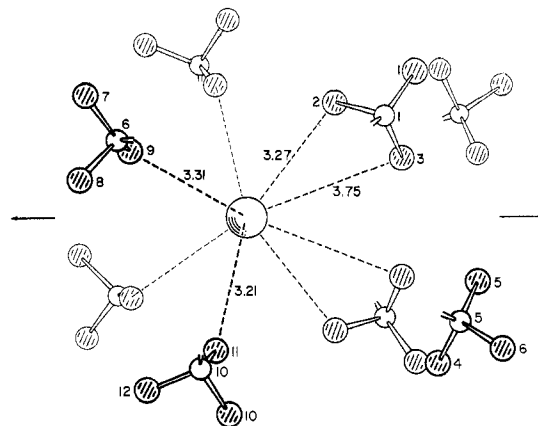
Figure 3.—(100) projection of the fluorine environment about the cesium atom. The arrows indicate the crystallographic twofold axis which passes through Cs.

TABLE II
FINAL ATOMIC POSITIONAL COORDINATES[a]

Atom	x	y	z
Cs	0.5000 (0)	0.27070 (5)	0.2500 (0)
Y	0.0000 (0)	0.31652 (5)	0.2500 (0)
O_1	−0.2121 (8)	0.3542 (3)	0.3160 (4)
O_2	0.0875 (8)	0.4005 (3)	0.3202 (4)
O_3	−0.1349 (8)	0.2311 (3)	0.2998 (4)
O_4	0.1476 (9)	0.2754 (3)	0.3472 (4)
C_1	−0.3967 (16)	0.4138 (6)	0.3798 (10)
C_2	−0.2385 (12)	0.4035 (5)	0.3547 (6)
C_3	−0.1257 (15)	0.4483 (5)	0.3765 (6)
C_4	0.0264 (11)	0.4422 (5)	0.3577 (7)
C_5	0.1319 (18)	0.4931 (6)	0.3848 (10)
C_6	−0.2168 (20)	0.1348 (6)	0.3494 (13)
C_7	−0.0966 (14)	0.1870 (5)	0.3452 (7)
C_8	0.0329 (11)	0.1806 (6)	0.3879 (7)
C_9	0.1425 (14)	0.2280 (5)	0.3887 (6)
C_{10}	0.2735 (20)	0.2205 (9)	0.4442 (12)
F_1	−0.4221 (11)	0.4467 (6)	0.4376 (7)
F_2	−0.4692 (11)	0.3636 (5)	0.3963 (9)
F_3	−0.4832 (13)	0.4331 (9)	0.3269 (8)
F_4	0.2273 (11)	0.4707 (5)	0.4386 (7)
F_5	0.2237 (12)	0.5119 (4)	0.3335 (6)
F_6	0.0662 (11)	0.5381 (4)	0.4197 (8)
F_7	−0.2115 (17)	0.1021 (6)	0.4050 (8)
F_8	−0.1897 (21)	0.0953 (7)	0.2991 (10)
F_9	−0.3430 (12)	0.1494 (6)	0.3357 (15)
F_{10}	0.2505 (15)	0.2515 (10)	0.5033 (8)
F_{11}	0.4019 (12)	0.2383 (6)	0.4216 (6)
F_{12}	0.2930 (12)	0.1659 (5)	0.4717 (8)

[a] Numbers in parentheses are the estimated standard deviations occurring in the last digits listed.

eters of the coordination dodecahedron about yttrium are summarized in Table VII, and Table VIII gives the best weighted least-squares planes through portions of the polyhedron.

Discussion

The crystal structure consists of the close packing of discrete monomeric $Y(HFA)_4^-$ anions and Cs^+ cations in infinite chains parallel to the crystallographic *a* axis. Each Cs^+ ion is surrounded by eight fluorine atoms from two neighboring $Y(HFA)_4^-$ moieties, as shown in Figure 3. Primary interactions between parallel chains appear to be due to fluorine–fluorine contacts, the shortest of which are listed in Table VI.

TABLE III

A. Anisotropic Temperature Factors[a] ($\times 10^4$)

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	Equiv[b] isotropic B, Å^2
Cs	134 (2)	41 (1)	64 (1)	0	7 (1)	0	6.49
Y	104 (2)	18 (1)	44 (1)	0	1 (1)	0	3.95
O$_1$	141 (11)	24 (2)	50 (3)	2 (4)	11 (5)	1 (2)	4.94
O$_2$	157 (12)	24 (2)	58 (4)	2 (4)	3 (5)	−10 (2)	5.44
O$_3$	165 (12)	20 (2)	49 (3)	−7 (4)	−3 (5)	6 (2)	4.93
O$_4$	182 (14)	29 (2)	50 (3)	−12 (4)	−15 (6)	3 (2)	5.68
C$_1$	172 (25)	30 (4)	81 (8)	18 (8)	15 (8)	−5 (4)	6.90
C$_2$	127 (17)	28 (3)	40 (4)	4 (6)	8 (7)	3 (3)	4.69
C$_3$	165 (20)	28 (3)	43 (5)	−8 (6)	1 (8)	−7 (3)	5.15
C$_4$	96 (24)	24 (2)	57 (5)	−3 (5)	−3 (7)	−5 (3)	4.79
C$_5$	215 (27)	27 (3)	83 (9)	−14 (8)	5 (13)	−13 (5)	7.21
C$_6$	244 (32)	22 (3)	124 (12)	−26 (8)	−50 (16)	19 (5)	8.88
C$_7$	177 (21)	20 (2)	52 (5)	−10 (6)	8 (9)	−2 (3)	5.14
C$_8$	115 (21)	33 (3)	52 (5)	−13 (6)	6 (6)	1 (3)	5.37
C$_9$	173 (21)	32 (3)	44 (5)	8 (7)	−4 (8)	3 (3)	5.51
C$_{10}$	220 (29)	49 (5)	73 (9)	−4 (10)	−48 (14)	12 (6)	8.25
F$_1$	221 (17)	111 (6)	148 (8)	−18 (8)	65 (10)	−83 (6)	15.1
F$_2$	240 (21)	64 (4)	217 (11)	−38 (7)	127 (12)	−26 (6)	15.2
F$_3$	270 (22)	187 (11)	113 (7)	139 (13)	24 (10)	42 (7)	18.8
F$_4$	310 (21)	67 (4)	108 (6)	−33 (7)	−66 (9)	−18 (4)	11.6
F$_5$	423 (24)	53 (3)	111 (6)	−100 (8)	51 (10)	−14 (3)	12.0
F$_6$	257 (16)	39 (2)	201 (11)	−13 (5)	2 (10)	−58 (4)	13.2
F$_7$	668 (42)	77 (4)	149 (9)	−164 (12)	−116 (15)	58 (6)	17.5
F$_8$	833 (57)	91 (6)	179 (11)	−209 (16)	92 (20)	−65 (7)	21.3
F$_9$	193 (18)	52 (4)	413 (23)	−45 (7)	−55 (17)	72 (8)	22.0
F$_{10}$	415 (32)	183 (11)	93 (6)	65 (13)	−101 (12)	−67 (7)	19.3
F$_{11}$	201 (17)	123 (7)	108 (7)	−28 (9)	−41 (9)	63 (6)	14.0
F$_{12}$	417 (25)	71 (4)	163 (9)	−7 (9)	−136 (13)	53 (5)	15.2

B. Root-Mean-Square Amplitudes of Vibrations (Å) and Their Direction Cosines[c]

Atom	Minor axis			Medium axis			Major axis		
Cs		0.224			0.309			0.317	
	(0.994,	0.000,	−0.107)	(0.000,	1.000,	0.000)	(0.107,	0.000.	0.994)
Y		0.199			0.206			0.261	
	(0.999,	0.000,	−0.013)	(0.000,	1.000,	0.000)	(0.013,	0.000,	0.999)
O$_1$		0.226			0.237			0.284	
	(0.921,	−0.290,	−0.260)	(0.244,	0.950,	−0.195)	(0.304,	0.116,	0.946)
O$_2$		0.218			0.246			0.314	
	(−0.204,	0.888,	0.412)	(0.979,	0.198,	0.058)	(0.030,	−0.415,	0.909)
O$_3$		0.204			0.252			0.286	
	(0.245,	0.926,	−0.288)	(0.944,	−0.160,	0.290)	(−0.223,	0.343,	0.913)
O$_4$		0.237			0.260			0.304	
	(0.750,	0.621,	0.228)	(0.328,	−0.648,	0.688)	(−0.575,	0.442,	0.689)
C$_1$		0.219			0.291			0.360	
	(0.739,	−0.649,	−0.182)	(0.656,	0.755,	−0.026)	(0.154,	−0.100,	0.983)
C$_2$		0.215			0.243			0.270	
	(0.942,	−0.073,	−0.327)	(0.194,	−0.677,	0.710)	(0.274,	0.732,	0.624)
C$_3$		0.226			0.252			0.285	
	(0.422,	0.710,	0.563)	(0.851,	−0.096,	−0.517)	(−0.312,	0.697,	−0.645)
C$_4$		0.189			0.234			0.303	
	(0.980,	0.186,	0.072)	(−0.197,	0.950,	0.241)	(−0.023,	−0.251,	0.968)
C$_5$		0.224			0.292			0.372	
	(0.344,	0.901,	0.263)	(0.929,	−0.286,	−0.233)	(0.135,	−0.325,	0.936)
C$_6$		0.192			0.288			0.467	
	(0.309,	0.940,	−0.145)	(0.893,	−0.233,	0.385)	(−0.328,	0.249,	0.911)
C$_7$		0.208			0.259			0.292	
	(0.340,	0.940,	0.022)	(0.846,	−0.296,	−0.443)	(0.411,	−0.169,	0.896)
C$_8$		0.198			0.287			0.288	
	(0.944,	0.311,	−0.115)	(0.247,	−0.429,	0.869)	(−0.221,	0.849,	0.481)
C$_9$		0.243			0.266			0.283	
	(0.721,	−0.493,	0.487)	(−0.574,	−0.031,	0.818)	(0.388,	0.869,	0.306)
C$_{10}$		0.240			0.323			0.389	
	(0.789,	−0.183,	0.587)	(0.438,	0.837,	−0.327)	(−0.431,	0.516,	0.740)
F$_1$		0.237			0.328			0.641	
	(0.753,	−0.366,	−0.547)	(0.643,	0.587,	0.492)	(−0.141,	0.722,	−0.677)
F$_2$		0.231			0.373			0.623	
	(0.936,	0.205,	−0.287)	(−0.118,	0.949,	0.293)	(0.332,	−0.240,	0.912)

TABLE III (*Continued*)

Atom	Minor axis			Medium axis			Major axis		
F_3	0.239			0.395			0.709		
	(0.936,	−0.341,	0.087)	(−0.163,	−0.201,	0.966)	(0.312,	0.918,	0.244)
F_4	0.254			0.416			0.452		
	(0.739,	0.441,	0.510)	(−0.561,	0.821,	0.103)	(−0.373,	−0.362,	0.854)
F_5	0.216			0.385			0.513		
	(0.642,	0.766,	−0.037)	(−0.332,	0.321,	0.887)	(0.691,	−0.557,	0.461)
F_6	0.211			0.316			0.599		
	(0.193,	0.909,	0.370)	(0.981,	−0.171,	−0.093)	(0.022,	−0.381,	0.924)
F_7	0.221			0.394			0.680		
	(0.511,	0.822,	−0.251)	(0.569,	−0.105,	0.816)	(0.644,	−0.560,	−0.521)
F_8	0.213			0.475			0.734		
	(0.517,	0.811,	0.276)	(−0.536,	0.055,	0.843)	(0.668,	−0.583,	0.462)
F_9	0.214			0.333			0.826		
	(0.797,	0.599,	−0.082)	(−0.598,	0.763,	−0.245)	(−0.084,	0.244,	0.966)
F_{10}	0.250			0.411			0.708		
	(0.540,	0.197,	0.818)	(0.805,	−0.404,	−0.434)	(0.245,	0.893,	−0.377)
F_{11}	0.255			0.316			0.607		
	(0.900,	−0.122,	0.418)	(−0.415,	−0.533,	0.737)	(−0.132,	0.837,	0.531)
F_{12}	0.245			0.396			0.602		
	(0.599,	−0.531,	0.599)	(0.679,	0.734,	−0.028)	(−0.425,	0.423,	0.800)

[a] Numbers in parentheses are estimated standard deviations occurring in the last digits listed, neglecting correlations.   [b] The equivalent isotropic B's were calculated by the program VIBELL.   [c] Direction cosines in parentheses are referred to the Cartesian coordinate system *abc*.

TABLE IV
INTRAMOLECULAR DISTANCES[a] IN Cs[Y(HFA)$_4$]

Atoms	Dist, Å	Atoms	Dist, Å
Cs–Y	4.450 (1)	C_1–F_1	1.26 (2)
		C_1–F_2	1.28 (2)
Y–O_1	2.321 (7)	C_1–F_3	1.27 (2)
Y–O_2	2.315 (7)	C_5–F_4	1.34 (2)
Y–O_3	2.347 (7)	C_5–F_5	1.27 (2)
Y–O_4	2.310 (7)	C_5–F_6	1.28 (2)
		C_6–F_7	1.21 (2)
O_1–C_2	1.28 (1.3)	C_6–F_8	1.25 (3)
O_2–C_4	1.23 (1.3)	C_6–F_9	1.16 (2)
O_3–F_7	1.28 (1.3)	C_{10}–F_{10}	1.25 (3)
O_4–C_9	1.26 (1.3)	C_{10}–F_{11}	1.24 (2)
		C_{10}–F_{12}	1.28 (2)
C_2–C_3	1.43 (2)		
C_3–C_4	1.37 (2)	Cs–F_2	3.27 (1.4)
C_7–C_8	1.36 (2)	Cs–F_3	3.75 (2)
C_8–C_9	1.39 (2)	Cs–F_9	3.31 (2)
		Cs–F_{11}	3.21 (1)
C_1–C_2	1.46 (2)		
C_4–C_5	1.51 (2)	O_1–O_2	2.79 (1)
C_6–C_7	1.53 (2)	O_3–O_4	2.76 (1)
C_9–C_{10}	1.51 (2)		

Atoms	Av dist, Å[b]	Weighted av dist, Å[c]
Y–O	2.323 (8)	2.323 (4)
O–C	1.263 (12)	1.263 (7)
C–C(ring)	1.386 (15)	1.386 (8)
C–CF$_3$	1.501 (16)	1.498 (9)
C–F	1.26 (1)	. . .
O–O	2.772 (13)	2.780 (7)

[a] Numbers in parentheses are estimated standard deviations occurring in the last digits listed. No significance can be attached to C–F distances or to their standard deviations because both librational corrections (∼0.1 Å) and parameter correlations were neglected. [b] Standard deviations of averages calculated from: $\sigma^2 = [1/n(n-1)]\Sigma_i(x_i - \bar{x})^2$. [c] Weighted average distances were computed from: $\mu_{av} = \Sigma(\mu_i/\sigma_i{}^2)/\Sigma(1/\sigma_i{}^2)$ and $\sigma_{av} = \sqrt{1/\Sigma_i(1/\sigma_i{}^2)}$ (where μ_i are the individual observations, and σ_i are the standard deviations for them) when the equation in footnote b suggested it was valid to do so.

These distances are as expected in view of the van der Waals radius for fluorine[17] of 1.35 Å. There are no significant cesium–fluorine contacts between neigh-

TABLE V
INTRAMOLECULAR ANGLES[a] IN Cs[Y(HFA)$_4$]

Atoms	Angle, deg	Atoms	Angle, deg
O_1–Y–O_2	73.8 (3)	O_3–Y–O_4	72.7 (3)
Y–O_1–O_2	53.0 (2)	Y–O_3–O_4	53.1 (2)
Y–O_2–O_1	53.2 (2)	Y–O_4–O_3	54.3 (2)
Y–O_1–C_2	134.2 (6)	Y–O_3–C_7	133.0 (7)
Y–O_2–C_4	135.3 (8)	Y–O_4–C_9	136.1 (7)
O_1–C_2–C_1	117 (1)	O_3–C_7–C_6	113 (1)
O_1–C_2–C_3	125 (1)	O_3–C_7–C_8	129 (1)
C_1–C_2–C_3	118 (1)	C_6–C_7–C_8	118 (1)
C_2–C_3–C_4	122 (1)	C_7–C_8–C_9	120 (1)
C_3–C_4–O_2	128 (1)	C_8–C_9–O_4	128 (1)
C_3–C_4–C_5	116 (1)	C_8–C_9–C_{10}	116 (1)
O_2–C_4–C_5	116 (1)	O_4–C_9–C_{10}	116 (1)
C_2–C_1–F_1	120 (1.4)	C_7–C_6–F_7	116 (1.7)
C_2–C_1–F_2	114 (1.2)	C_7–C_6–F_8	110 (1.6)
C_2–C_1–F_3	113 (1.4)	C_7–C_6–F_9	116 (2.1)
F_1–C_1–F_2	102 (1.5)	F_7–C_6–F_8	100 (1.6)
F_1–C_1–F_3	108 (1.4)	F_7–C_6–F_9	111 (1.4)
F_2–C_1–F_3	99 (1.4)	F_8–C_6–F_9	102 (2.2)
C_4–C_5–F_4	110 (1.2)	C_9–C_{10}–F_{10}	111 (1.6)
C_4–C_5–F_5	113 (1.4)	C_9–C_{10}–F_{11}	116 (1.5)
C_4–C_5–F_6	116 (1.2)	C_9–C_{10}–F_{12}	116 (1.4)
F_4–C_5–F_5	103 (1.2)	F_{10}–C_{10}–F_{11}	104 (1.8)
F_4–C_5–F_6	102 (1.5)	F_{10}–C_{10}–F_{12}	101 (1.4)
F_5–C_5–F_6	112 (1.3)	F_{11}–C_{10}–F_{19}	106 (1.6)

[a] Numbers in parentheses are estimated standard deviations occurring in the last digits listed.

boring columns. This relatively weak intermolecular interaction due to ion–dipole and van der Waals forces is consistent with the observation from mass spectrometry that this compound readily sublimes as an ion pair.[4]

The molecular structure of Cs[Y(HFA)$_4$] is shown in Figure 1. Eight essentially equidistant (2.310–2.347 Å) oxygen atoms surround the central yttrium atom in a slightly distorted dodecahedral configuration.[18] Figure 2 shows the two interpenetrating

(17) L. Pauling, "The Nature of the Chemical Bond," Cornell University Press, Ithaca, N. Y., 1960, p 260.

(18) See ref 6 and 7 for explanations and definitions of the nomenclature used in this discussion and summarized in Figure 2.

TABLE VI
SELECTED INTERMOLECULAR CONTACTS

Atom I	Atom J	Vector from molecule containing I to molecule containing J	Dist, Å
F_1	F_1	$(-1 - x, 1 - y, 1 - z)$	3.45
F_1	F_4	$(-x, 1 - y, 1 - z)$	3.23
F_1	F_7	$(-1/2 - x, 1/2 + y, z)$	3.59
F_1	F_{12}	$(-1/2 + x, 1/2 - y, 1 - z)$	3.45
F_2	F_{10}	$(-1/2 + x, 1/2 - y, 1 - z)$	3.59
F_2	F_{12}	$(-1/2 + x, 1/2 - y, 1 - z)$	3.31
F_3	F_3	$(-1 - x, y, 1/2 - z)$	2.72
F_4	F_6	$(-x, 1 - y, 1 - z)$	3.57
F_4	F_7	$(1/2 + x, 1/2 - y, 1 - z)$	3.21
F_5	F_8	$(1/2 + x, 1/2 + y, 1/2 - z)$	3.03
F_6	F_6	$(-x, 1 - y, 1 - z)$	3.46
F_6	F_7	$(-1/2 - x, 1/2 + y, z)$	3.38
F_6	F_9	$(-1/2 - x, 1/2 + y, z)$	3.41
F_6	F_{12}	$(1/2 - x, 1/2 + y, z)$	3.14
F_7	F_{10}	$(-1/2 + x, 1/2 - y, 1 - z)$	3.55
F_{10}	F_{11}	$(-1/2 + x, 1/2 - y, 1 - z)$	3.31

TABLE VII
CHARACTERISTIC PARAMETERS OF THE
COORDINATION DODECAHEDRON[a]

Atoms	Distances — Type	Value, Å[b]	Angles
O_3–O_3'	a	2.92	Angle between
O_2–O_2'	a	2.90	trapezoids = 88.3°
O_1–O_4	b	3.60	θ_A = 38.6°
O_1–O_4'	b	3.38	θ_B = 68.5°
O_1–O_3	m	2.75	
O_2–O_4	m	2.78	
O_1–O_2	g	2.79	
O_3–O_4	g	2.76	
O_1–O_2'	g	2.81	
O_3–O_4'	g	2.75	

[a] See especially Figure 2 for the labeling scheme. Primes denote symmetry-related atoms. [b] All standard deviations are 0.01 Å.

TABLE VIII
BEST WEIGHTED LEAST-SQUARES PLANES.[a]
$LX + MY + NZ = D$[b]

Atoms	L	M	N	D	Mean Dev, Å	Mean σ, Å
Planes through Trapezoids						
O_4'–O_2'–O_2–O_4	0.8169	0.0	0.5769	11.1389	0.07	0.01
O_1–O_3–O_3'–O_1'	−0.5523	0.0	0.8336	8.5780	0.07	0.01
Planes through Chelate Rings						
C_1–C_2–O_1–C_3–C_4–O_2–C_5	−0.1717	0.5113	0.8421	12.6043	0.01	0.01
C_6–C_7–O_3–C_8–C_9–O_4–C_{10}	0.4641	−0.4965	0.7336	9.9628	0.04	0.01
O_1–Y–O_2	−0.2117	0.6099	0.7637	11.5430	0.0	0.0
O_3–Y–O_4	0.4567	−0.6023	0.6548	8.2237	0.0	0.0

[a] Direction cosines of the planes refer to the Cartesian axis system abc. [b] $X = ax$, $Y = by$, $Z = cz$.

trapezoids BAAB as viewed down the crystallographic twofold axis (idealized unique $\bar{4}$ axis of the dodecahedron). In other dodecahedral ML_4 structures,[7] where L is a bidentate ligand, the chelate rings are closed across the m edges of a single trapezoid and $g > a = m$. However, in $Cs[Y(HFA)_4]$, the β-diketonate groups span adjacent A–B vertices of the two bisphenoids along g edges of the dodecahedron, thereby resulting in overall idealized D_2 symmetry for the anion and $g = m < a$. This novel structural feature has also recently been found[19] in the structure of $(NH_4)Pr[C_8H_4F_3O_2S]_4 \cdot H_2O$.

The characteristic parameters of the coordination dodecahedron are summarized in Table VII. θ_A and θ_B (angles made by bonds Y–A and Y–B with the unique C_2 axis of the dodecahedron, which coincides with the crystallographic twofold axis in this case) are 38.6 and 68.5°, respectively, again reflecting the dependence of the coordination geometry upon intermolecular packing and ligand steric requirements. The angle between the best least-squares planes through each of the trapezoids BAAB is 88.3°. Ideally (for D_2 symmetry) this would be 90°.

The essentially coplanar β-diketonate ligands exhibit the folding along the oxygen–oxygen line so often found in these chelate rings,[20] with the dihedral angles between the ligand plane and the plane defined by the O–Y–O group being 7.6° in both cases. This folding apparently aids packing within and between molecules. Tables IV and V contain all pertinent intramolecular distances and angles.

As shown in Table IV, the standard deviations for the average bond lengths, estimated from a spread of values, are comparable to those obtained from the inverse matrix during least-squares refinement, with the notable exception of the C–F bonds. This appears to confirm our postulate that the approximation of the CF_3 motion by anisotropic temperature factors would lead to a negligible effect on the parameters of primary interest. Finally, it should be noted that the bond distances and angles within the chelate rings show no significant deviations from the average values for other acetylacetonate structures which have been accurately determined by three-dimensional methods.[20−22]

(19) R. A. Lalancette, M. Cefola, W. C. Hamilton, and S. J. LaPlaca *Inorg. Chem.*, **6**, 2127 (1967).
(20) F. A. Cotton and J. S. Wood, *ibid.*, **3**, 245 (1964).
(21) E. C. Lingafelter and R. L. Braun, *J. Am. Chem. Soc.*, **88**, 2951 (1966).
(22) F. A. Cotton and R. Eiss, *ibid.*, **90**, 38 (1968).