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The Crystal Structure of $Cs(B_9C_2H_{11})_2Co$[1]

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The crystal structure of $(B_9C_2H_{11})_2CoCs$ has been determined from an X-ray diffraction study on a single-crystal specimen. The monoclinic cell, space group C2/c, with $a = 11.698$, $b = 7.381$, $c = 21.207$ A, and $\beta = 101.73°$, contains four formula units. The structure consists of a cesium cation and a $(B_9C_2H_{11})_2Co^-$ anion. The latter ion consists of two icosahedra sharing one common apex, which is the cobalt atom on a center of symmetry. The carbon atoms are disordered among the pentagon of atoms adjacent to the cobalt. All 11 hydrogens, one for each boron and carbon atom, were located as well.

Introduction

The salt $Cs(B_9C_2H_{11})_2Co$ was synthesized in the laboratory of Professor M. F. Hawthorne[2] at the University of California at Riverside. A crystal structure study was undertaken to confirm the proposed structure of two icosahedra joined at the cobalt apex; this we proved to be true. The synthesizers of this material had hoped they had produced a compound in which the carbon atoms were not adjacent to each other; unfortunately, we found the carbon atoms to be disordered and therefore could neither affirm nor deny this hypothesis.

Experimental Section

A light brown parallelepiped-shaped crystal of dimensions $\sim 0.15 \times 0.15 \times 0.30$ mm was selected and glued to the end of a Pyrex fiber in air.

A General Electric XRD-5 X-ray diffraction apparatus equipped with a molybdenum X-ray tube (Mo $K\alpha_1$, λ 0.70926 A), a scintillation counter, a pulse height discriminator, and a quarter-circle Eulerian-cradle type of goniostat was used. The X-ray tube was operated at 50 kv and 20 ma; a Zr filter was used on the receiving slit. The crystal was oriented such that the b axis was parallel to the ϕ axis of the instrument. The cell dimensions were obtained from carefully measured 2θ (θ is the Bragg angle) values of the $0k0$, $h00$, and $00l$ reflections whose α_1 and α_2 components were resolved. The β angle was measured directly from the angle between the $h00$ and $00l$ set of reflections.

A total of 2051 independent intensities were measured, of which 144 were recorded as zero. A stationary-crystal, stationary-counter technique using a 10-sec count for every reflection was used. The maximum 2θ angle was 55° [$(\sin\theta)/\lambda = 0.651$]. The maximum observed raw intensity was 46,900 cps for the 200 reflection. Background was plotted as a function of 2θ and these values were used for most of the intensities; in the cases where background was seriously affected by streaking, individual backgrounds were measured. The absorption parameter is 30 cm^{-1}, which we estimate causes the absorption factor to vary by a factor 1.5 between the most extreme cases. Variations of the intensity of 020 when the ϕ setting was changed 90° ($\chi = 90°$) were about 20%. No corrections were made for either absorption or extinction.

Our unpublished least-squares program minimizes the function $\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o^2$, where F_o and F_c are the observed and calculated structure factors and w is the weighting factor. All of the nonzero reflections were given equal weights, and zero observed intensities were given zero weight.

Scattering factors for Cs^+, Co, O, C, B, and H were taken from the "International Tables."[3] The Cs^+ and Co scattering factors were given $\Delta f'$ corrections, the real part of the anomalous dispersion,[4,5] by the amounts -0.5 and $+0.4$ electron, respectively. The imaginary components of the anomalous dispersion, $\Delta f''$, $+2.49$ and $+1.05$ electrons for Cs^+ and Co, were introduced into the least-squares calculations as well. The anisotropic temperature factors used have the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$. B_{ij} values, in units of A^2, where $B_{ij} = 4\beta_{ij}/a_i^*a_j^*$, are reported in this paper.

Results

Unit Cell and Space Group.—Four formula units of $(B_9C_2H_{11})_2CoCs$ are in the monoclinic C-centered unit cell whose dimensions are: $a = 11.698 \pm 0.005$ A, $b = 7.381 \pm 0.004$ A, $c = 21.207 \pm 0.006$ A, and $\beta = 101.73 \pm 0.05°$; the errors are estimates. The calculated X-ray density is 1.692 g/cc. The density measured by flotation is 1.69 g/cc. The observed extinctions correspond to C2/c or Cc, and good agreement of observed and calculated structure factors was achieved in the centric space group, C2/c.

Determination of the Structure.—The Patterson function readily revealed the locations of the cesium and cobalt atoms. The cesium occupies the special fourfold position on the twofold axis, and the cobalt is on a center of symmetry at the origin. A least-squares refinement on these two atoms alone using anisotropic temperature factors gave an R factor of 0.25, where $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. A difference function phased by these two heavy atoms revealed the positions of the 11 boron and carbon atoms as the 11 largest peaks. Three cycles of full-matrix least-squares refinement of the parameters of the havy atoms with anisotropic temperature factors and the other 11 atoms as boron with isotropic temperature factors resulted in $R = 0.069$.

In attempting to locate the two carbon atoms, we used the two criteria of temperature factors and interatomic distances. Figure 1 shows the framework of the $(B_9C_2H_{11})_2Co^-$ group. When all of these atoms with the exception of Co were refined as borons, the isotropic temperature factors of the pentagon of atoms adjacent to the cobalt, *i.e.*, atoms 2–6, ranged from 2.46 to 2.71 A^2; those of the remaining atoms ranged from 2.96 to 3.24 A^2. A carbon atom refined with boron

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[1] Work done under the auspices of the U. S. Atomic Energy Commission.

[2] M. F. Hawthorne and T. D. Andrews, *Chem. Commun.*, **19**, 443 (1965).

[3] J. A. Ibers in "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 202.

[4] D. T. Cromer, *Acta Cryst.*, **18**, 17 (1965).

[5] D. H. Templeton in "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 216.

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS (×2.0) FOR $(B_9C_2H_{11})_2CoCs^{a,b}$

```
OBSERVED AND CALCULATED STRUCTURE FACTORS (X 2.0) OF CS+ (CO (B(9)C(2)H(11))2 )-.
H,K= 0, 0
L FOB FCA
2 14 -11
4 234 250
6 225-235
8 368 393
10 193-198
12 330 344
14 65 -60
16 240 241
18 33 -28
20 147 147
22 15 -9
24 128 119
26 17 20

H,K= 0, 2
L FOB FCA
-0 279 277
1 331 350
2 0 -1*
3 315-335
4 244 255
5 289 307
6 35 33
7 183-198
8 185 198
9 268 280
10 14 26
11 209-209
12 290 300
13 118 120
14 120 123
15 180-182
16 97 93
17 126 127
18 10 -5
19 63 -58
20 94 93
21 42 45
22 19 -19
23 58 -56
24 41 38
25 53 51
26 47 46

H,K= 0, 4
L FOB FCA
-0 10 -10
1 125 134
2 294 317
3 174-180
4 50 -46
5 158 169
6 275 293
7 195-206
8 42 -46
9 108 114
10 160 165
11 91 -92
12 31 -32
13 107 109
14 183 187
15 65 -61
16 0 -9*
17 74 76
18 130 126
19 46 -44
20 0 10*
21 62 55
22 62 59
23 41 -38
24 0 8*

H,K= 0, 6
L FOB FCA
-0 54 -52
1 57 -62
2 195 207
3 63 69
4 68 -73
5 32 -30
6 224 233
7 67 70
8 47 -50
9 58 -62
10 168 170
11 34 34
12 29 -25
13 25 -25
14 108 110
15 34 35
16 27 -24
17 34 -29
18 111 106
19 0 11*
20 0 -1*
21 21 -24

H,K= 0, 8
L FOB FCA
-0 44 45
1 84 -89
2 48 47
3 83 85
4 42 44
5 86 -91
6 44 45
7 81 83
8 61 61
9 67 -68
10 38 36
11 66 66
12 20 23
13 46 -47
14 36 33

H,K= 1, 1
L FOB FCA
-26 92 87
-25 20 6
-24 16 12
-23 19 -18
-22 96 95
-21 54 51
-20 0 -6*
-19 74 -70
-18 155 150
-17 106 106
-16 41 -40
-15 23 -21
-14 280 291
-13 104 103
-12 0 -5*
-11 168-173
-10 268 285
-9 85 93
-8 132-131
-7 201-205
-6 406 427
-5 176 174
-4 248-239
-3 394-384
-2 656 667
-1 157 139
-0 224 193
1 7 -13
2 454 452
3 312 309
4 219-215
5 179-176
6 419 433
7 249 250
8 217-215
9 50 -60
10 279 299
11 137 146
12 32 30
13 183-185
14 286 292
15 9 -6
16 52 -59
17 64 -61
18 154 146
19 82 86
20 14 -10
21 56 -50
22 113 107
23 44 42
24 0 5*
25 20 -16
26 86 76

H,K= 1, 3
L FOB FCA
-25 52 50
-24 16 13
-23 34 -26
-22 0 4*
-21 63 56
-20 97 91
-19 104 -99
-18 37 34
-17 89 87
-16 96 94
-15 143-147
-14 127 128
-13 101 102
-12 159 161
-11 178-182
-10 56 52
-9 258 269
-8 164 168
-7 188-192
-6 70 71
-5 274 276
-4 165 170
-3 230-230
-2 44 43
-1 324 309
-0 248 233
1 433-415
2 137 124
3 209 219
4 144 150
5 260-269
6 109 108
7 265 273
8 223 227
9 197-202
10 75 72
11 150 154
12 155 160
15 152 155
16 62 62
17 78 -74
18 57 52
19 99 96
20 69 67
21 54 -51
22 11 -17
23 59 54
24 43 44
25 61 -57

H,K= 1, 5
L FOB FCA
-23 12 -9
-22 12 -8
-21 26 27
-20 95 93
-19 16 -2
-18 0 -4*
-17 0 -1*
-16 141 141
-15 35 -31
-14 28 -27
-13 29 28
-12 168 174
-11 13 -15
-10 117-124
-9 42 43
-8 221 233
-7 19 -18
-6 8 -6
-5 44 42
-4 300 304
-3 44 -39
-2 105-102
-1 30 -27
-0 329 318
1 100 -97
2 107-105
3 89 88
4 287 293
5 25 23
6 61 -67
7 30 24
8 206 214
9 0 -3*
10 90 -90
11 39 40
12 189 193
13 42 -44
14 42 -41
15 20 11
16 144 146
17 15 -14
18 15 -15
19 19 12
20 101 99
21 0 -8*
22 12 -6

H,K= 1, 7
L FOB FCA
-18 21 22
-17 20 -19
-16 57 58
-15 28 30
-14 16 -11
-13 112-113
-12 93 92
-11 61 66
-10 18 13
-9 50 -51
-8 106 110
-7 86 92
-6 14 4
-5 85 -89
-4 121 124
-3 92 88
-2 0 2*
-1 116-109
-0 128 123
1 119 117
2 31 -28
3 88 -88
4 109 107
5 92 95
6 10 13
7 87 -90
8 122 123
9 62 64
10 0 3*
11 66 -71
12 90 88
13 63 63
14 11 8
15 60 -56
16 58 58
17 40 45

H,K= 1, 9
L FOB FCA
-9 51 -52
-8 0 6*
-7 48 45
-6 59 59
-5 46 -44
-4 0 -4*
-3 59 55
-2 70 68
-1 71 -62
-0 0 1*
1 52 45
2 84 79
3 54 -54
4 17 -14
5 47 47
6 65 65
7 36 -36
8 30 26

H,K= 1, 3
L FOB FCA
13 181-185
14 67 66

H,K= 2, 0
L FOB FCA
-26 12 10
-24 113 106
-22 21 17
-20 178 174
-18 43 -34
-16 137 140
-14 61 -61
-12 330 359
-10 121-123
-8 409 420
-6 165-164
-4 460 455
-2 116 97
-0 840 852
2 340-321
4 447 459
6 188-184
8 365 377
10 115-118
12 319 327
14 91 -90
16 237 236
18 33 -32
20 166 157
22 15 -11
24 106 99
26 0 -1*

H,K= 2, 2
L FOB FCA
-26 36 39
-25 31 -27
-24 23 32
-23 81 76
-22 0 9*
-21 56 -53
-20 97 96
-19 57 57
-18 25 22
-17 71 -75
-16 129 129
-15 116 117
-14 82 84
-13 180-186
-12 196 206
-11 183 185
-10 61 61
-9 220-222
-8 250 254
-7 209 212
-6 48 42
-5 212-208
-4 286 274
-3 378 361
-2 19 -17
-1 322-306
-0 259 243
1 264 254
2 22 -8
3 346-337
4 281 278
5 372 370
6 131 119
7 143-158
8 221 224
9 190 202
10 39 37
11 246-250
12 244 254
13 110 108
14 46 48
15 146-143
16 73 69
17 136 135
18 10 16
19 83 -78
20 99 95
21 92 86
22 0 5*
23 30 -28
24 55 52
25 30 22

H,K= 2, 4
L FOB FCA
-25 17 -18
-24 0 11*
-23 33 30
-22 77 72
-21 38 -36
-20 19 18
-19 50 48
-18 119 119
-17 62 -62
-16 43 40
-15 45 45
-14 178 185
-13 123-127
-12 67 -71
-11 110 113
-10 135 137
-9 44 -43
-8 16 -12
-7 183 183
-6 301 305
-5 150-150
-4 66 -64
-3 151 143
-2 274 259
-1 273-261
-0 30 21
1 90 93
2 320 318
3 135-126
4 78 -75
5 148 148
6 273 278
7 125-127
8 12 -7
9 196 204
10 173 177
11 108-111
12 64 -64
13 58 53
14 135 137
15 73 -73
16 0 -5*
17 73 73
18 130 127
19 38 -36
20 0 -9*
21 64 57
22 67 66
23 52 -45

H,K= 2, 6
L FOB FCA
-21 34 35
-20 0 7*
-19 0 -8*
-18 103 101
-17 19 21
-16 45 -45
-15 53 -54
-14 98 104
-13 0 9*
-12 0 9*
-11 28 -25
-10 156 156
-9 49 47
-8 41 -41
-7 42 -41
-6 229 229
-5 93 92
-4 24 -23
-3 76 -74
-2 241 234
-1 9 -9
-0 85 -83
1 68 -68
2 152 148
3 114 112
4 77 -76
5 19 -21
6 208 210
7 47 43
8 47 -45
9 44 -45
10 167 172
11 37 36
12 23 -21
13 54 -55
14 136 144
15 25 29
16 19 -15
17 11 -16
18 85 81
19 21 21
20 0 -4*

H,K= 2, 8
L FOB FCA
-15 53 -55
-14 34 35
-13 24 21
-12 31 30
-11 54 -52
-10 25 25
-9 73 73
-8 35 27
-7 71 -70
-6 44 42
-5 88 89
-4 49 48
-3 80 -79
-2 37 34
-1 84 79
-0 70 64
1 89 -84
2 77 72
3 89 85
4 38 36
5 79 -76
6 46 37
7 86 84
8 51 55
9 68 -72
10 42 33
11 50 48
12 34 35
13 63 -59

H,K= 3, 1
L FOB FCA
-27 12 -12
-26 63 59
-25 11 8
-24 25 23
-23 15 -21
-22 110 110
-21 41 37
-20 30 27
-19 23 -22
-18 117 129
-17 76 74
-16 53 -53
-15 104-101
-14 216 218
-13 86 86
-12 15 -9
-11 59 -55
-10 320 322
-9 70 72
-8 49 36
-7 239-224
-6 454 445
-5 233 212
-4 134-124
-3 178-169
-2 391 384
-1 198 185
-0 159-152
1 214-207
2 446 432
3 167 162
4 74 -75
5 131-126
6 373 370
7 153 155
8 88 -84
9 120-118
10 332 340
11 131 132
12 35 -37
13 148-149
14 192 192
15 32 36
16 56 -52
17 44 -43
18 169 163
19 78 75
20 0 10*
21 34 -27
22 108 103
23 23 25
24 20 -17
25 44 -42

H,K= 3, 3
L FOB FCA
-26 32 28
-25 49 49
-24 38 33
-23 19 -19
-22 44 44
-21 38 37
-20 92 86
-19 74 -71
-18 62 65
-17 90 92
-16 111 110
-15 137-140
-14 41 41
-13 65 66
-12 100 100
-11 134-136
-10 91 89
-9 210 212
-8 142 140
-7 198-198
-6 71 76
-5 284 267
-4 217 207
-3 183-182
-2 73 71
-1 217 215
-0 221 208
1 320-305
2 74 67
3 222 218
4 176 174
5 214-220
6 191 190
7 261 262
8 198 199
9 180-183
10 0 -4*
14 45 41
15 151 154
16 89 90
17 87 -84
18 53 52
19 94 90
20 53 54
21 37 -32
22 31 26
23 59 53

H,K= 3, 5
L FOB FCA
-23 0 1*
-22 12 -17
-21 23 18
-20 77 81
-19 11 -4
-18 15 13
-17 0 11*
-16 124 128
-15 14 -11
-14 20 -16
-13 24 23
-12 143 144
-11 16 -6
-10 79 -78
-9 40 37
-8 257 254
-7 21 -15
-6 17 9
-5 53 50
-4 250 240
-3 86 -85
-2 95 -90
-1 84 -79
-0 267 263
1 38 -34
2 91 -91
3 116 115
4 245 241
5 17 -14
6 77 -75
7 54 52
8 222 229
9 28 29
10 55 -52
11 10 8
12 167 170
13 49 -47
14 29 -30
15 21 15
16 132 135
17 27 -23
18 30 -30
19 0 8*
20 95 96
21 0 -6*

H,K= 3, 7
L FOB FCA
-18 21 20
-17 20 -24
-16 45 46
-15 0 -12*
-14 22 18
-13 97-100
-12 100 102
-11 73 74
-10 11 -2
-9 39 -40
-8 80 81
-7 93 91
-6 20 20
-5 70 -66
-4 129 127
-3 81 75
-2 0 4*
-1 77 -76
-0 128 118
1 121 116
2 14 -9
3 90 -92
4 98 93
5 69 68
6 0 -4*
7 84 -88
8 105 105
9 62 64
10 0 8*
11 79 -83
12 101 97
13 41 43
14 0 14*
15 24 -21
16 45 43

H,K= 3, 9
L FOB FCA
-8 17 -20
-7 30 23
-6 52 54
-5 36 -39
-4 0 5*
-3 43 43
-2 68 69
-1 78 -73
-0 29 27
1 43 43
2 74 78
3 32 -27
4 17 -20
5 60 56
6 63 62

H,K= 3, 3
L FOB FCA
11 115 113
12 86 83
13 168-166

H,K= 4, 0
L FOB FCA
-26 12 -13
-24 100 109
-22 59 55
-20 149 145
-18 47 -51
-16 123 125
-14 14 9
-12 289 293
-10 61 -63
-8 407 404
-6 67 -67
-4 385 374
-2 245-236
-0 380 373
2 19 -26
4 479 475
6 118-116
8 420 427
10 59 -59
12 201 200
14 94 -95
16 216 216
18 34 -29
20 148 142
22 20 -17
24 87 81

H,K= 4, 2
L FOB FCA
-26 40 35
-25 0 -12*
-24 54 57
-23 45 43
-22 34 34
-21 62 -60
-20 89 89
-19 52 50
-18 60 62
-17 72 -77
-16 88 85
-15 72 73
-14 12 -14
-13 98 -96
-12 168 168
-11 144 144
-10 139 136
-9 257-248
-8 252 244
-7 218 212
-6 113 102
-5 55 -64
-4 283 265
-3 262 255
-2 30 -25
-1 286-276
-0 208 201
1 210 206
2 32 28
3 241-235
4 251 244
5 231 232
6 99 94
7 230-231
8 197 198
9 179 179
10 0 -2*
11 137-137
12 146 148
13 126 126
14 26 26
15 115-114
16 104 103
17 99 100
18 33 35
19 64 -62
20 101 96
21 91 89
22 26 28
23 58 -58

H,K= 4, 4
L FOB FCA
-24 17 19
-23 23 14
-22 67 66
-21 25 -26
-20 32 33
-19 51 50
-18 123 123
-17 25 -25
-16 29 25
-15 51 50
-14 114 112
-13 86 -91
-12 25 -23
-11 102 101
-10 151 146
-9 75 -74
-8 14 -9
-7 65 63
-6 257 253
-5 134-129
-4 13 -16
-3 130 126
-2 222 216
-1 166-160
-0 19 -8
1 165 166
2 274 273
3 124-125
4 0 -11*
5 101 102
6 256 257
7 56 -50
8 21 -18
9 150 149
10 136 132
11 140-143
12 54 -57
13 63 59
14 131 131
15 61 -59
16 0 -2*
17 54 52
18 91 106
19 42 -39
20 0 6*
21 45 41

H,K= 4, 6
L FOB FCA
-21 0 4*
-20 0 4*
-19 0 -7*
-18 71 73
-17 25 24
-16 22 -19
-15 46 -43
-14 126 124
-13 18 13
-12 23 23
-11 17 -15
-10 134 135
-9 45 41
-8 14 -10
-7 29 -23
-6 183 182
-5 58 57
-4 23 -20
-3 81 -80
-2 182 179
-1 26 20
-0 81 -79
1 23 -22
2 143 139
3 75 73
4 53 -53
5 52 -54
6 176 177
7 37 37
8 23 -18
9 41 -39
10 148 145
11 18 24
12 19 -13
13 25 -21
14 146 147
15 44 50
16 16 -19
17 27 -26
18 50 59

H,K= 4, 8
L FOB FCA
-14 36 40
-13 36 32
-12 31 32
-11 33 -34
-10 0 8*
-9 50 50
-8 23 18
-7 50 -52
-6 46 45
-5 79 75
-4 33 31
-3 74 -74
-2 44 42
-1 70 67
-0 112 104
1 58 -53
2 78 74
3 74 79
4 16 11
5 65 -72
6 23 19
7 55 53
8 37 39
9 69 -66
10 38 37
11 47 49

H,K= 5, 1
L FOB FCA
-27 0 -11*
-26 47 48
-25 0 1*
-24 46 43
-23 35 -30
-22 89 87
-21 15 15
-20 35 32
-19 26 -29
-18 129 128
-17 22 16
-16 18 -20
-15 54 -53
-14 153 153
-13 140 139
-12 39 33
-11 44 -40
-10 307 304
-9 24 21
-8 46 34
-7 115-106
-6 305 294
-5 155 143
-4 93 -90
-3 117-119
-2 247 249
-1 110 105
-0 31 -35
1 252-235
2 421 410
3 83 85
4 70 -72
5 119-117
6 300 306
7 117 116
8 29 27
9 50 -47
10 232 231
11 132 132
12 95 -93
13 96 -95
14 182 178
15 62 59
16 17 -15
17 50 -49
18 130 126
19 45 44
20 16 8
21 43 -39
22 92 89
23 0 3*

H,K= 5, 3
L FOB FCA
-25 38 36
-24 56 57
-23 20 -20
-22 53 49
-21 24 18
-20 66 67
-19 56 -54
-18 64 63
-17 78 77
-16 54 49
-15 65 -64
-14 20 18
-13 79 79
-12 139 134
-11 143-147
-10 71 67
-9 88 89
-8 104 99
-7 138-136
-6 157 157
-5 232 227
-4 208 212
-3 138-147
-2 37 36
-1 215 217
-0 135 133
1 144-142
2 86 84
3 141 138
4 183 180
5 227-226
6 110 112
7 174 177
8 86 88
9 131-133
11 106 107
12 91 91
13 119-118
14 64 66
15 109 111
16 96 94
17 75 -76
18 45 43
19 76 75
20 63 63
21 43 -41
22 30 34

H,K= 5, 5
L FOB FCA
-23 24 -25
-22 21 -12
-21 0 0*
-20 77 79
-19 19 15
-18 24 25
-17 32 28
-16 100 100
-15 15 -4
-14 14 8
-13 22 22
-12 142 144
-11 0 5*
-10 19 -17
-9 13 -10
-8 185 187
-7 63 -65
-6 30 -25
-5 29 29
-4 175 181
-3 35 -35
-2 74 -72
-1 0 -5*
-0 208 206
1 18 11
2 15 -21
3 56 52
4 211 209
5 18 -22
6 45 -43
7 45 45
8 199 203
9 24 -24
10 44 -44
11 27 20
12 134 133
13 21 17
14 19 -19
15 0 7*
16 104 103
17 43 -47
18 24 -20
19 0 2*

H,K= 5, 7
L FOB FCA
-18 12 15
-17 21 -28
-16 46 52
-15 0 3*
-14 34 31
-13 45 -42
-12 76 77
-11 55 55
-10 15 12
-9 37 -38
-8 77 79
-7 70 66
-6 15 12
-5 61 -61
-4 77 82
-3 71 69
-2 28 21
-1 39 -34
-0 132 125
1 82 79
2 0 1*
3 92 -92
4 67 66
5 44 44
6 0 1*
7 70 -71
8 87 88
9 54 54
10 0 2*
11 60 -59
12 78 79
13 61 61
14 17 20

H,K= 5, 9
L FOB FCA
-4 0 10*
-3 32 24
-2 68 67
-1 53 -51
-0 36 33
1 55 54

H,K= 5, 3
L FOB FCA
10 13 -16

H,K= 6, 0
L FOB FCA
-26 12 14
-24 87 85
-22 30 31
-20 122 117
-18 25 28
-16 142 144
-14 42 38
-12 236 235
-10 32 -33
-8 216 208
-6 67 -69
-4 283 286
-2 143-135
-0 367 363
2 94 78
4 329 328
6 42 -46
8 324 324
10 89 -92
12 169 165
14 26 -24
16 119 117
18 58 -55
20 133 131
22 12 4

H,K= 6, 2
L FOB FCA
-26 44 42
-25 12 -4
-24 63 62
-23 11 5
-22 15 13
-21 71 -71
-20 62 62
-19 30 27
-18 71 67
-17 33 -34
-16 49 46
-15 99 98
-14 0 4*
-13 51 -48
-12 178 174
-11 63 56
-10 115 109
-9 139-134
-8 177 176
-7 178 172
-6 118 114
-5 128-135
-4 175 181
-3 149 149
-2 39 35
-1 145-143
-0 206 205
1 139 143
2 28 22
3 197-202
4 118 115
5 109 109
6 50 47
7 132-130
8 164 166
OBSERVED AND CALCULATED STRUCTURE FACTORS (X 2.0) OF CS+ (CO (B(9)C(2)H(11))2 )-.
9 201 203
10 0 6*
11 84 -86
12 121 120
13 93 93
14 65 64
15 87 -86
16 111 109
17 64 63
18 11 18
19 65 -60
20 74 73
21 53 49

H,K= 6, 4
L FOB FCA
-24 21 22
-23 0 8*
-22 53 56
-21 0 -10*
-20 35 35
-19 27 30
-18 93 89
-17 21 -17
-16 20 18
-15 49 52
-14 117 121
-13 43 -44
-12 34 35
-11 39 39
-10 98 92
-9 122-125
-8 9 -3
-7 27 26
-6 221 218
-5 41 -40
-4 12 6
-3 133 133
-2 129 126
-1 43 -44
-0 28 24
1 128 127
2 262 258
3 138-142
4 18 11
5 68 72
6 159 161
7 59 -55
8 19 -15
9 53 55
10 127 125
11 82 -82
12 25 -25
13 83 81
14 98 102
15 57 -55
16 0 3*
17 38 37
18 92 94
19 36 -35

H,K= 6, 6
L FOB FCA
-20 12 7
-19 0 6*
-18 58 61
-17 20 18
-16 11 15
-15 25 -18
-14 89 98
-13 30 26
-12 24 19
-11 21 -11
-10 118 119
-9 25 17
-8 0 -4*
-7 36 -34
-6 113 112
-5 30 30
-4 14 -13
-3 31 -30
-2 134 135
-1 40 39
-0 31 -33
1 30 -26
2 137 138
3 30 33
4 20 -20
5 47 -46
6 145 144
7 21 16
8 21 -15
9 31 -29
10 84 88
11 55 58
12 0 -4*
13 23 12
14 121 123
15 17 19

H,K= 6, 8
L FOB FCA
-12 44 41
-11 32 -36
-10 27 27
-9 33 36
-8 31 26
-7 35 -34
-6 23 28
-5 40 41
-4 23 25
-3 50 -52
-2 59 59
-1 65 63
-0 82 82
1 38 -37
2 39 37
3 48 49
4 20 17
5 65 -65
6 24 24
7 38 41
8 27 28

H,K= 7, 1
L FOB FCA
-26 53 49
-25 0 2*
-24 26 25
-23 37 -42
-22 50 51
-21 11 -7
-20 53 51
-19 10 -17
-18 125 120
-17 29 25
-16 0 6*
-15 9 9
-14 142 137
-13 111 111
-12 83 78
-11 31 -29
-10 168 165
-9 42 39
-8 43 -43
-7 99 -98
-6 204 207
-5 42 41
-4 30 29
-3 49 -41
-2 243 247
-1 50 46
-0 50 42
1 174-172
2 254 253
3 73 71
4 67 -67
5 36 -35
6 226 224
7 129 128
8 30 23
9 30 -32
10 152 150
11 71 66
12 0 -10*
13 41 -40
14 161 158
15 48 49
16 47 -44
17 74 -74
18 81 79
19 12 13
20 21 18
21 24 -27

H,K= 7, 3
L FOB FCA
-24 51 53
-23 12 -14
-22 20 13
-21 0 4*
-20 31 31
-19 37 -40
-18 58 61
-17 60 60
-16 67 64
-15 10 -17
-14 62 63
-13 41 36
-12 104 103
-11 126-130
-10 31 32
-9 55 57
-8 129 129
-7 84 -80
-6 152 158
-5 115 124
-4 111 109
-3 69 -75
-2 34 33
-1 192 192
-0 138 133
1 105-104
2 69 66
3 53 56
4 89 86
5 155-160
6 42 44
7 118 120
8 91 89
9 92 -93
10 49 50
11 86 86
14 68 67
15 72 70
16 72 74
17 48 -48
18 17 17
19 53 53

H,K= 7, 5
L FOB FCA
-21 0 5*
-20 66 68
-19 12 5
-18 28 29
-17 11 14
-16 80 82
-15 0 2*
-14 18 24
-13 15 19
-12 108 111
-11 0 -7*
-10 0 -1*
-9 22 -20
-8 106 106
-7 27 -30
-6 14 -12
-5 37 38
-4 144 144
-3 9 -2
-2 31 -29
-1 23 20
-0 180 179
1 10 -7
2 17 18
3 17 10
4 141 146
5 20 -20
6 0 -11*
7 0 -3*
8 144 148
9 0 -16*
10 43 -45
11 66 67
12 92 92
13 16 15
14 0 -8*
15 20 -18
16 77 84

H,K= 7, 7
L FOB FCA
-16 34 36
-15 24 26
-14 24 21
-13 23 -20
-12 62 68
-11 11 13
-10 36 33
-9 32 -32
-8 58 59
-7 40 40
-6 16 9
-5 40 -42
-4 68 72
-3 58 57
-2 36 29
-1 31 -34
-0 80 82
1 47 43
2 19 13
3 51 -48
4 69 70
5 41 35
6 11 9
7 49 -48
8 54 56
9 47 50
10 0 3*
11 34 -31

H,K= 7, 3
L FOB FCA
12 94 94
13 82 -83

H,K= 8, 0
L FOB FCA
-24 40 41
-22 31 33
-20 113 108
-18 55 52
-16 118 118
-14 69 64
-12 154 150
-10 70 -71
-8 90 87
-6 36 33
-4 255 251
-2 23 -18
-0 286 284
2 60 54
4 176 173
6 48 -55
8 181 182
10 10 -15
12 191 186
14 44 -44
16 35 32
18 0 -8*

H,K= 8, 2
L FOB FCA
-25 12 -7
-24 39 38
-23 16 -13
-22 11 -6
-21 33 -34
-20 48 48
-19 34 31
-18 68 65
-17 14 -12
-16 60 59
-15 67 67
-14 52 50
-13 37 -37
-12 129 129
-11 48 48
-10 62 57
-9 64 -61
-8 127 131
-7 30 27
-6 94 93
-5 122-121
-4 141 144
-3 142 143
-2 103 100
-1 74 -79
-0 147 148
1 65 65
2 24 -25
3 112-110
4 52 51
5 106 109
6 59 56
7 57 -57
8 145 149
9 102 106
10 44 41
11 76 -77
12 106 106
13 68 65
14 68 65
15 74 -72
16 66 68
17 37 34
18 0 -6*
19 32 -32

H,K= 8, 4
L FOB FCA
-22 34 33
-21 17 -15
-20 34 35
-19 11 -7
-18 89 85
-17 15 -15
-16 41 41
-15 31 37
-14 90 94
-13 32 -29
-12 33 31
-11 0 10*
-10 73 68
-9 66 -69
-8 16 17
-7 52 57
-6 134 143
-5 0 -7*
-4 13 12
-3 78 82
-2 111 112
-1 32 -34
-0 40 41
1 46 47
2 149 150
3 81 -82
4 17 10
5 54 55
6 129 129
7 69 -73
8 21 19
9 35 36
10 86 94
11 22 -21
12 11 -8
13 45 47
14 81 83
15 37 -34
16 12 5
17 46 46

H,K= 8, 6
L FOB FCA
-18 48 48
-17 0 12*
-16 20 21
-15 16 -2
-14 43 49
-13 0 7*
-12 0 11*
-11 11 -13
-10 94 91
-9 32 30
-8 15 15
-7 21 -18
-6 94 98
-5 0 6*
-4 24 16
-3 24 -19
-2 87 91
-1 28 23
-0 0 7*
1 11 -17
2 117 117
3 34 28
4 0 -10*
5 31 -30
6 83 91
7 20 15
8 11 -16
9 0 2*
10 55 63
11 34 39
12 0 6*

H,K= 8, 8
L FOB FCA
-7 27 -28
-6 24 22
-5 24 25
-4 30 29
-3 21 -21
-2 34 31
-1 36 33
-0 30 30
1 32 -33

H,K= 9, 1
L FOB FCA
-24 24 21
-23 20 -24
-22 47 48
-21 11 4
-20 50 42
-19 11 -8
-18 80 79
-17 21 21
-16 42 41
-15 17 16
-14 115 116
-13 55 51
-12 45 44
-11 18 -16
-10 73 70
-9 16 14
-8 0 -5*
-7 89 -90
-6 164 180
-5 45 47
-4 82 74
-3 12 -13
-2 182 185
-1 15 -8
-0 54 50
1 81 -78
2 134 137
3 94 90
4 44 -45
5 0 -4*
6 133 134
7 55 52
8 29 25
9 53 -52
10 138 138
11 43 41
12 30 25
13 19 -16
14 83 82
15 0 8*
16 28 -25
17 49 -51
18 72 70

H,K= 9, 3
L FOB FCA
-23 17 5
-22 0 -4*
-21 0 8*
-20 46 45
-19 30 -33
-18 77 77
-17 19 20
-16 68 70
-15 15 -15
-14 55 57
-13 25 20
-12 69 65
-11 59 -59
-10 48 46
-9 37 39
-8 113 110
-7 47 -51
-6 70 79
-5 68 71
-4 76 72
-3 33 -36
-2 66 68
-1 81 87
-0 82 81
1 65 -66
2 32 33
3 71 75
4 80 75
5 73 -78
7 54 53
8 97 95
9 54 -58
10 56 57
12 68 66
13 69 -72
14 42 46
15 44 47
16 29 30

H,K= 9, 5
L FOB FCA
-19 0 -9*
-18 26 35
-17 12 8
-16 53 54
-15 11 -11
-14 0 7*
-13 0 5*
-12 76 76
-11 30 25
-10 21 17
-9 39 37
-8 69 70
-7 0 -11*
-6 27 31
-5 23 -14
-4 123 125
-3 15 -13
-2 0 3*
-1 15 10
-0 107 105
1 30 -26
2 0 6*
3 15 6
4 109 108
5 0 -1*
6 19 16
7 11 8
8 83 86
9 16 6
10 28 -28
11 24 27
12 69 73
13 0 -8*

H,K= 9, 7
L FOB FCA
-12 45 48
-11 12 2
-10 34 33
-9 17 -8
-8 51 54
-7 23 21
-6 29 30
-5 29 -25
-4 49 53
-3 31 28
-2 17 8
-1 31 -33
-0 60 59
1 49 48
2 36 34
3 0 4*
4 49 47
5 24 25

H,K= 9, 3
L FOB FCA
6 57 58
11 52 55

H,K=10, 0
L FOB FCA
-24 40 37
-22 37 34
-20 54 59
-18 47 44
-16 90 88
-14 38 37
-12 97 94
-10 10 7
-8 96 93
-6 59 54
-4 148 143
-2 37 36
-0 195 195
2 9 11
4 84 81
6 10 -14
8 135 134
10 37 36
12 109 107
14 37 -39
16 59 60

H,K=10, 2
L FOB FCA
-23 17 15
-22 21 18
-21 12 -3
-20 40 38
-19 11 -2
-18 47 49
-17 24 -20
-16 66 66
-15 31 32
-14 57 55
-13 23 -23
-12 71 71
-11 33 32
-10 54 49
-9 44 -46
-8 86 97
-7 13 6
-6 67 63
-5 30 -33
-4 107 116
-3 71 70
-2 79 79
-1 64 -64
-0 74 76
1 66 66
2 14 12
3 31 -31
4 74 72
5 52 52
6 56 51
7 46 -46
8 100 100
9 55 50
10 36 36
11 60 -61
12 64 61
13 23 19
14 37 39
15 40 -37

H,K=10, 4
L FOB FCA
-20 48 48
-19 12 -12
-18 67 69
-17 16 -11
-16 30 29
-15 0 12*
-14 59 60
-13 15 -5
-12 42 42
-11 34 34
-10 62 56
-9 0 -4*
-8 10 12
-7 36 37
-6 88 94
-5 34 -33
-4 46 47
-3 10 16
-2 71 71
-1 44 -49
-0 0 -4*
1 25 25
2 106 108
3 0 -3*
4 63 63
5 44 44
6 91 94
7 52 -51
8 11 13
9 32 33
10 61 66
11 20 -18
12 0 10*
13 17 19

H,K=10, 6
L FOB FCA
-14 12 15
-13 0 -5*
-12 17 16
-11 20 23
-10 73 77
-9 37 38
-8 43 44
-7 30 -29
-6 80 88
-5 0 -12*
-4 20 18
-3 20 -9
-2 53 57
-1 16 11
-0 30 26
1 0 -4*
2 66 65
3 29 32
4 12 -17
5 12 -4
6 56 60
7 17 14

H,K=11, 1
L FOB FCA
-22 34 37
-21 0 -10*
-20 20 15
-19 16 -18
-18 48 52
-17 19 18
-16 42 42
-15 0 -3*
-14 67 64
-13 0 -3*
-12 57 55
-11 0 -4*
-10 94 95
-9 28 27
-8 26 33
-7 26 -22
-6 92 95
-5 34 28
-4 46 46
-3 31 -30
-2 110 113
-1 20 12
-0 36 35
1 20 -11
2 88 86
3 29 26
4 18 17
5 36 -31
6 95 94
7 32 29
8 22 23
9 27 -25
10 91 85
11 23 17
12 17 12
13 12 -14
14 59 62

H,K=11, 3
L FOB FCA
-20 42 49
-19 27 -27
-18 45 51
-17 12 3
-16 41 48
-15 16 -9
-14 41 42
-13 24 20
-12 47 48
-11 21 -20
-10 50 51
-9 15 21
-8 58 62
-7 27 -23
-6 44 46
-5 27 32
-4 64 68
-3 39 -38
-2 36 38
-1 31 30
-0 59 58
1 0 -10*
2 82 84
3 65 64
4 73 72
5 48 -44
6 33 37
7 32 30
8 31 69
9 26 -27
10 33 35
11 0 9*
12 24 22

H,K=11, 5
L FOB FCA
-16 27 34
-15 12 -18
-14 17 14
-13 20 20
-12 61 62
-11 50 49
-10 26 27
-9 25 27
-8 52 62
-7 28 -30
-6 48 54
-5 22 -22
-4 75 79
-3 0 -9*
-2 0 -6*
-1 16 -13
-0 53 51
1 20 -19
2 20 20
3 33 31
4 75 83
5 20 18
6 17 17
7 12 5
8 47 53

H,K=12, 0
L FOB FCA
-20 32 33
-18 39 36
-16 32 36
-14 38 35
-12 137 135
-10 79 77
-8 38 41
-6 10 4
-4 85 81
-2 37 38
-0 78 79
2 26 23
4 105 105
6 19 25
8 70 72
10 20 27
12 61 60

H,K=12, 2
L FOB FCA
-19 12 -11
-18 24 27
-17 16 -6
-16 49 49
-15 0 5*
-14 32 34
-13 19 -18
-12 47 49
-11 27 25
-10 56 59
-9 11 -12
-8 62 66
-7 18 17
-6 50 49
-5 11 -9
-4 68 71
-3 30 29
-2 37 36
-1 21 -20
-0 74 73
1 30 25
2 67 70
3 45 -44
4 54 55
5 32 28
6 16 18
7 16 -7
8 52 54
9 12 13
10 12 15
11 46 -45

H,K=12, 4
L FOB FCA
-16 34 32
-15 0 6*
-14 43 43
-13 0 5*
-12 31 37
-11 16 19
-10 44 48
-9 0 -4*
-8 23 25
-7 0 6*
-6 56 64
-5 32 -29
-4 28 32
-3 0 -4*
-2 25 31
-1 25 -28
-0 25 22
1 34 37
2 91 97
3 20 21
4 44 46
5 17 15
6 51 53
7 27 -22

H,K=13, 1
L FOB FCA
-18 24 34
-17 17 11
-16 26 25
-15 23 -19
-14 55 54
-13 0 -4*
-12 77 79
-11 22 24
-10 71 75
-9 29 30
-8 16 16
-7 0 -4*
-6 45 49
-5 0 8*
-4 31 31
-3 11 -8
-2 47 51
-1 11 15
-0 39 35
1 37 -33
2 90 92
3 23 -21
4 41 41
5 12 -5
6 42 42
7 27 28
8 21 17

H,K=13, 3
L FOB FCA
-15 0 -6*
-14 21 20
-13 0 9*
-12 39 41
-11 12 -18
-10 52 56
-9 12 -5
-8 38 38
-7 16 -13
-6 36 37
-5 28 27
-4 48 55
-3 0 -9*
-2 36 39
-1 11 13
-0 70 67
1 0 -7*
2 58 60
3 31 29
4 29 28
5 12 -13
6 27 29

H,K=14, 0
L FOB FCA
-14 60 58
-12 91 97
-10 35 37
-8 16 13
-6 11 8
-4 53 48
-2 20 21
-0 53 55
2 53 55
4 67 70

H,K=14, 2
L FOB FCA
-14 21 22
-13 0 2*
-12 36 43
-11 0 8*
-10 44 48
-9 17 -16
-8 42 45
-7 20 20
-6 45 51
-5 12 15
-4 48 49
-3 12 12
-2 29 30
-1 26 -26
-0 66 68
1 17 -12
2 51 52
3 17 -18

H,K=15, 1
L FOB FCA
-8 21 24
-7 0 7*
-6 36 38
-5 0 1*
-4 27 27
-3 12 -10
```

[a] Asterisked data are the zero observed and zero weighted reflections. [b] As anomalous dispersion has been properly introduced into the calculations, the reported values FCA are the absolute values of the structure factors with the sign of the real part.

TABLE II

ATOMIC[a] AND THERMAL PARAMETERS[b] IN $(B_9C_2H_{11})_2CoCs$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cs	0^c	0.4060	$1/4^c$	7.46	3.05	4.30	0^c	−1.11	0^c
Co(1)	0^c	0^c	0^c	1.75	2.30	2.13	−0.17	0.35	−0.13
BC(2)[d]	0.1224	−0.1774	0.0476	2.5	3.3	2.9	0.3	0.3	0.0
BC(3)[d]	0.1415	0.0369	0.0743	2.5	3.3	3.0	−0.4	−0.1	0.0
B(4)	0.0121	0.1172	0.0904	3.0	2.7	2.2	−0.2	0.4	−0.2
BC(5)[d]	0.4146	0.4396	0.0744	2.9	2.8	2.6	−0.2	1.0	0.0
BC(6)[d]	0.4823	0.2579	0.0470	2.8	2.7	2.7	−0.3	0.5	0.2
B(7)	0.1959	−0.1381	0.1256	2.4	4.0	3.3	−0.1	−0.1	0.5
B(8)	0.1267	0.4838	0.1538	3.5	3.5	2.5	−0.8	−0.1	−0.3
B(9)	0.4810	0.4845	0.1537	3.4	3.2	2.4	−0.4	0.8	−0.2
B(10)	0.4613	0.2594	0.1260	2.8	3.3	2.5	−0.6	0.4	0.2
B(11)	0.0949	−0.3173	0.1087	3.1	3.0	3.4	0.0	0.2	0.3
B(12)	0.0962	−0.1757	0.1761	3.3	3.3	2.8	−0.2	0.0	0.2
H(2)	0.173	−0.233	0.015	2.2^e					
H(3)	0.197	0.111	0.062	2.5					
H(4)	0.490	−0.262	0.085	1.9					
H(5)	0.319	0.456	0.061	3.0					
H(6)	0.436	0.152	0.019	2.0					
H(7)	0.281	−0.165	0.139	2.1					
H(8)	0.177	0.139	0.186	0.6					
H(9)	0.432	−0.458	0.191	0.4					
H(10)	0.397	0.170	0.142	2.6					
H(11)	0.128	−0.455	0.113	1.1					
H(12)	0.128	−0.227	0.221	0.9					

[a] Estimated standard deviations on the atomic positions for Co, B or BC, and H are ±0.0005, ±0.005, and ±0.05 A, respectively. [b] The thermal parameters have the units A^2. The estimated standard deviations on the thermal parameters of Co or Cs, B or BC, and H are ±0.04, ±0.2, and ±1.4 A^2, respectively. [c] Parameter fixed by the space group symmetry. [d] Half boron and half carbon. [e] Isotropic B in the case for hydrogen.

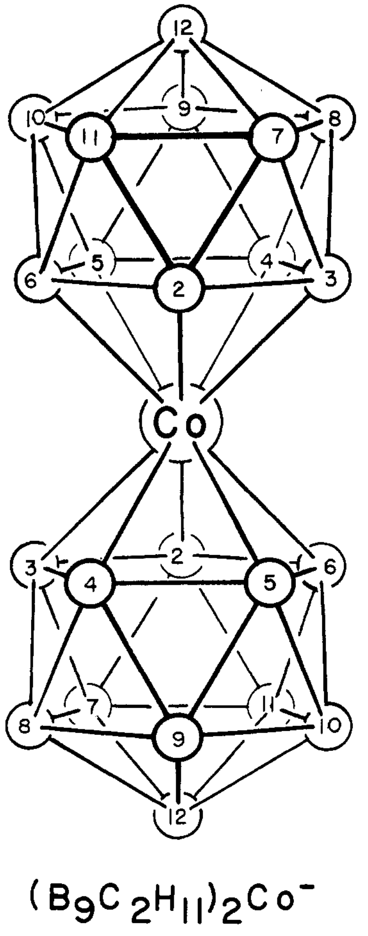

Figure 1.—Framework of the $(B_9C_2H_{11})_2Co^-$ ion. Hydrogen atoms, which are on each of the atoms with the exception of cobalt, are not shown.

TABLE III

HYDROGEN DISTANCES (A) IN THE $(B_9C_2H_{11})_2Co^-$ ION[a]

H(2)	1.08	H(8)	1.05
H(3)	0.93	H(9)	1.15
H(4)	0.93	H(10)	1.11
H(5)	1.10	H(11)	1.09
H(6)	1.06	H(12)	1.03
H(7)	1.00		

[a] Estimated standard deviations of these distances are ±0.06 A. These distances are shorter than expected for the internuclear B–H distance; the X-ray method gives values for such bonds which are systematically shortened by about 0.1 A. See: W. Smith and W. N. Lipscomb, *J. Chem. Phys.*, **43**, 1060 (1965); W. N. Lipscomb, *ibid.*, **22**, 985 (1954). The standard deviations given by least squares, and stated in Table III, do not include this systematic effect.

scattering factors is expected to receive an abnormally low thermal parameter. No two atoms stand out as carbon atoms on this basis, but the above results are consistent with the carbon atoms being distributed in the pentagon adjacent to the cobalt. A better guide to locating the carbons are the interatomic distances. Figure 2 shows the icosahedra spread out with all of the interatomic distances in the icosahedra. In our previous study on $B_9C_2H_{11}Re(CO)_3Cs$,[6] which has a boron–carbon network quite similar to this structure, the bond distances were found to be: C–C, 1.61 A; C–B, 1.72 A; B–B 1.78 A. In Figure 2, the top half of the icosahedra, atoms 7–12, appear to have standard B–B distances. In the lower pentagon, atoms 2–6, the distances are all more or less B–C-type distances.

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(6) A. Zalkin, T. E. Hopkins, and D. H. Templeton, *Inorg. Chem.*, **5**, 1189 (1966).

TABLE IV

ANGLES (IN DEGREES) IN $Co(B_9C_2H_{11})_2^{-}$ [a]

BC(2)–Co–BC(3)	48.3	Co–BC(5)–B(4)	65.5	B(4)–B(9)–BC(5)	59.2
BC(3)–Co–B(4)	49.2	B(4)–BC(5)–B(9)	61.1	BC(5)–B(9)–B(10)	59.6
B(4)–Co–BC(5)	49.0	B(9)–BC(5)–B(10)	60.9	B(10)–B(9)–B(12)	60.2
BC(5)–Co–BC(6)	48.8	B(10)–BC(5)–BC(6)	60.6	B(12)–B(9)–B(8)	60.0
BC(6)–Co–B(2)	48.8	BC(6)–BC(5)–Co	65.5	B(8)–B(9)–B(4)	60.2
Co–BC(2)–BC(6)	66.5	Co–BC(6)–BC(2)	64.8	BC(5)–B(10)–BC(6)	59.2
BC(6)–BC(2)–B(11)	61.1	BC(2)–BC(6)–B(11)	60.4	BC(6)–B(10)–B(11)	59.7
B(11)–BC(2)–B(7)	61.1	B(11)–BC(6)–B(10)	60.9	B(11)–B(10)–B(12)	59.8
B(7)–BC(2)–BC(3)	60.9	B(10)–BC(6)–BC(5)	60.2	B(12)–B(10)–B(9)	60.5
BC(3)–BC(2)–Co	66.1	BC(5)–BC(6)–Co	65.7	B(9)–B(10)–BC(5)	59.4
Co–BC(3)–BC(2)	65.6	BC(2)–B(7)–BC(3)	58.3	BC(2)–B(11)–B(7)	59.1
BC(2)–BC(3)–B(7)	60.9	BC(3)–B(7)–B(8)	59.6	B(7)–B(11)–B(12)	59.8
B(7)–BC(3)–B(8)	61.3	B(8)–B(7)–B(12)	60.6	B(12)–B(11)–B(10)	60.4
B(8)–BC(3)–B(4)	61.6	B(12)–B(7)–B(11)	60.3	B(10)–B(11)–BC(6)	59.4
B(4)–BC(3)–Co	66.1	B(11)–B(7)–BC(2)	59.7	BC(6)–B(11)–BC(2)	58.5
Co–B(4)–BC(3)	64.7	BC(3)–B(8)–B(4)	59.0	B(7)–B(12)–B(8)	59.8
BC(3)–B(4)–B(8)	59.4	B(4)–B(8)–B(9)	59.6	B(8)–B(12)–B(9)	59.6
B(8)–B(4)–B(9)	60.2	B(9)–B(8)–B(12)	60.4	B(9)–B(12)–B(10)	59.3
B(9)–B(4)–B(5)	59.7	B(12)–B(8)–B(7)	59.6	B(10)–B(12)–B(11)	59.9
B(5)–B(4)–Co	65.5	B(7)–B(8)–BC(3)	59.2	B(11)–B(12)–B(7)	59.9

[a] Estimated standard deviations for all angles are ±0.3°.

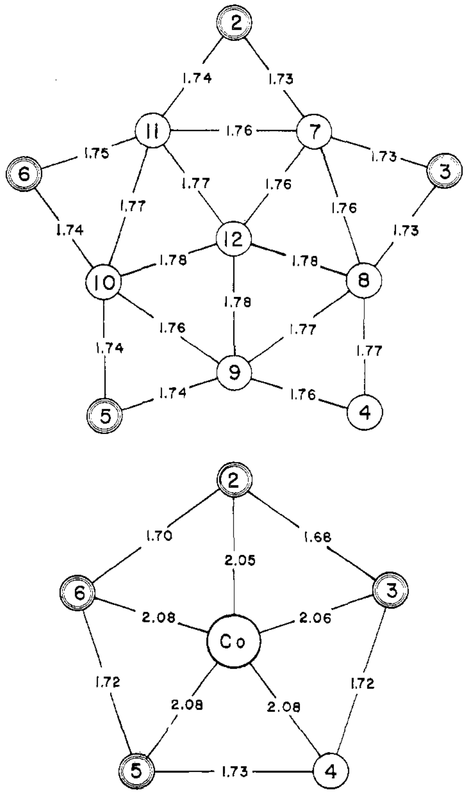

Figure 2.—Interatomic distances in the $(B_9C_2H_{11})_2Co$ icosahedra. Multiple circles indicate the disordered positions containing half boron–half carbon atoms; single circles represent borons. The estimated standard deviations on these bond lengths are ±0.01 A.

These results as well as the temperature factors indicate the carbons to be present in the lower pentagon in a disordered manner. Since atom 4 had distances to atoms 8 and 9 which are B–B type, we finally chose a model in which atoms 2, 3, 5, and 6 are half borons and half carbons and atoms 4, 7, 8, 9, 10, 11, and 12 are borons. Admittedly this is not unique but is one of the many ways of "playing the game." Various models using different ratios of B and C in the disordered positions were tried; temperature factors were affected, but no significant changes in the distances in the icosahedral structure resulted.

A difference function was calculated, and it indicated the 11 hydrogen atoms among the top 17 peaks. Six of these peaks were apparent anisotropies near Cs, Co, and B(8).

The final least squares, performed with all 24 atoms of the structure included, resulted in $R = 0.042$. The hydrogen atoms were refined with isotropic temperature factors, and all of the other atoms were refined with anisotropic temperature factors. The real and imaginary components of the anomalous dispersion correction were applied to the Cs and Co atoms. Atoms 2, 3, 5, and 6 in the icosahedron were treated as half boron and half carbon atoms. The B_{12} and B_{23} temperature factors of Cs were restrained to zero as required by the symmetry of the special position. The last shift in the parameter in each case was less than 0.001 of the estimated standard deviation of that parameter. The list of the observed and calculated structure factors is shown in Table I. The final set of parameters is shown in Table II. The sets of positions are: Cs in 4(e): $\pm(0, y, {}^1/_4;\ {}^1/_2, {}^1/_2 + y, {}^1/_4)$; Co in 4(a): $(0, 0, 0;\ 0, 0, {}^1/_2;\ {}^1/_2, {}^1/_2, 0;\ {}^1/_2, {}^1/_2, {}^1/_2)$; B and C in 8(f): $\pm(x, y, z;\ x, \bar{y}, {}^1/_2 + z;\ {}^1/_2 + x, {}^1/_2 + y, z;\ {}^1/_2 + x, {}^1/_2 - y, {}^1/_2 + z)$.

The anisotropic temperature factor refinements were carried out primarily for the purpose of looking for hydrogen atoms. It has been our experience that it is often necessary to remove the anisotropies of the bonding atom in order to see the hydrogens in the difference Fouriers. With the exception of Cs, all of the temperature factors appear to be fairly isotropic. We offer no explanation for the sizable anisotropy of Cs.

Description of Structure.—The structure consists of a Cs^+ cation and a $(B_9C_2H_{11})_2Co^-$ anion. The cesium ion is surrounded by ten hydrogen atoms at distances from 3.00 to 3.34 A; the next nearest neighbors to the cesium atom are two boron atoms, B(10), at 3.67 A.

The framework of the $(B_9C_2H_{11})_2Co^-$ group is shown in Figure 1. The structure consists of two icosahedra in a staggered configuration joined at a common apex, which is the cobalt atom. The two carbon atoms are shown in disordered positions in the pentagon of atoms adjacent to the cobalt, occupying mainly positions 2, 3, 5, and 6. The interatomic distances in the framework are shown in Figure 2.

Each atom with the exception of cobalt in the framework shown in Figure 1 has bonded to it a hydrogen atom. The hydrogen bond lengths are shown in Table III; the differences among them are not experimentally significant.

The above icosahedral structure is very similar to that found in $(B_9C_2H_{11})Fe(C_5H_5)$[7] and in $(B_9C_2H_{11})Re(CO)_3$,[6] where there is no disorder of the carbon atoms. Because of the disorder in $(B_9C_2H_{11})_2CoCs$, we cannot say if the carbon atoms are adjacent or not in each molecule, nor indeed if the crystal contains a single isomer or more than one isomer.

Table IV shows a list of angles in the $(B_9C_2H_{11})_2Co^-$ group.

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Notes

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The Preparation and Some Reactions of Phosphoramidic Difluoride[1]

BY SANTAD KONGPRICHA AND WILLIAM C. PREUSSE

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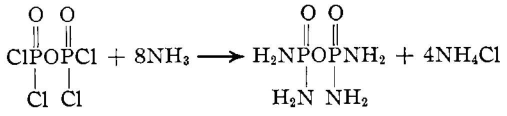
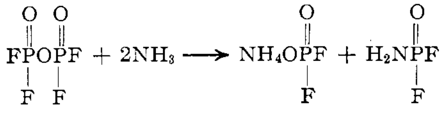
The reaction of ammonia with pyrophosphoryl tetrachloride[2] was previously reported to give pyrophosphoryl tetramide, $P_2O_3(NH_2)_4$. In this reaction the preferential attack by ammonia on the P–Cl bond rather than the P–O–P bond was noted



$$\mathrm{Cl\overset{O}{\underset{Cl}{P}}O\overset{O}{\underset{Cl}{P}}Cl + 8NH_3 \longrightarrow H_2N\overset{O}{\underset{H_2N}{P}}O\overset{O}{\underset{NH_2}{P}}NH_2 + 4NH_4Cl}$$

The recently published facile preparation of pyrophosphoryl tetrafluoride[3] prompted us to investigate its reaction with ammonia to determine whether the substitution of the phosphorus-bonded fluorine atoms by amido groups or the heterolytic cleavage of the P–O–P bond by ammonia occurred predominantly.

When pyrophosphoryl tetrafluoride was treated with ammonia, ammonium difluorophosphate[4,5] and phosphoramidic difluoride were obtained

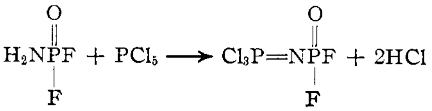


$$\mathrm{F\overset{O}{\underset{F}{P}}O\overset{O}{\underset{F}{P}}F + 2NH_3 \longrightarrow NH_4O\overset{O}{\underset{F}{P}}F + H_2N\overset{O}{\underset{F}{P}}F}$$

The novel phosphoramidic difluoride was characterized by infrared and ^{31}P and ^{19}F nuclear magnetic resonance spectrometry. The reaction of ammonia with pyrophosphoryl tetrafluoride is indeed different from that with pyrophosphoryl tetrachloride. The highly electronegative fluorine atoms in $P_2O_3F_4$ probably withdraw electrons toward themselves, thus weakening the POP bond. Since phosphoryl trifluoride[6] in ethyl ether reacted rapidly with ammonia, even at −110°, to form phosphoryl triamide, phosphoramidic difluoride would, thus, be expected to react further with excess of ammonia.

The reactions of phosphoramidic difluoride with phosphorus pentachloride and with triphenylphosphorus dichloride were investigated.

Phosphoramidic difluoride reacted with an equivalent amount of phosphorus pentachloride to yield trichlorophosphazophosphoryl difluoride



$$\mathrm{H_2N\overset{O}{\underset{F}{P}}F + PCl_5 \longrightarrow Cl_3P{=}N\overset{O}{\underset{F}{P}}F + 2HCl}$$

Its infrared spectrum is quite similar to that of $Cl_3P{=}NPOCl_2$[7] (mp 35.5°; bp 102° (1 mm)), except for the additional P—F stretching frequency in $Cl_3P{=}NPOF_2$.

Trichlorophosphazophosphoryl difluoride decomposed in an atmosphere of nitrogen at 200° to liberate gaseous products which were mainly OPF_2Cl and $OPCl_3$. The residue was an opaque elastomeric material with an empirical formula of $(NPFCl)_n$. It was not determined if the elastomer was a mixture of

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[1] Presented in part at the 148th National Meeting of the American Chemical Society, Chicago, Ill., Sept 1964; Abstracts, p 3K.

[2] (a) M. Becke-Goehring and K. Niedenzu, *Chem. Ber.*, **89**, 1771 (1956); (b) M. Becke-Goehring and K. Niedenzu, *ibid.*, **90**, 151 (1957); (c) R. Klement and L. Benek, *Z. Anorg. Allgem. Chem.*, **287**, 12 (1956).

[3] E. A. Robinson, *Can. J. Chem.*, **40**, 1725 (1962).

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