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THE CRYSTAL STRUCTURE OF  $\text{Cs}_2(\text{B}_9\text{C}_2\text{H}_{11})\text{Co}(\text{B}_8\text{C}_2\text{H}_{10}) \text{Co}(\text{B}_9\text{C}_2\text{H}_{11})\cdot\text{H}_2\text{O}$ , A SALT OF A THREE-ICOSAHEDRAL- FRAGMENT METALLOCARBORANE

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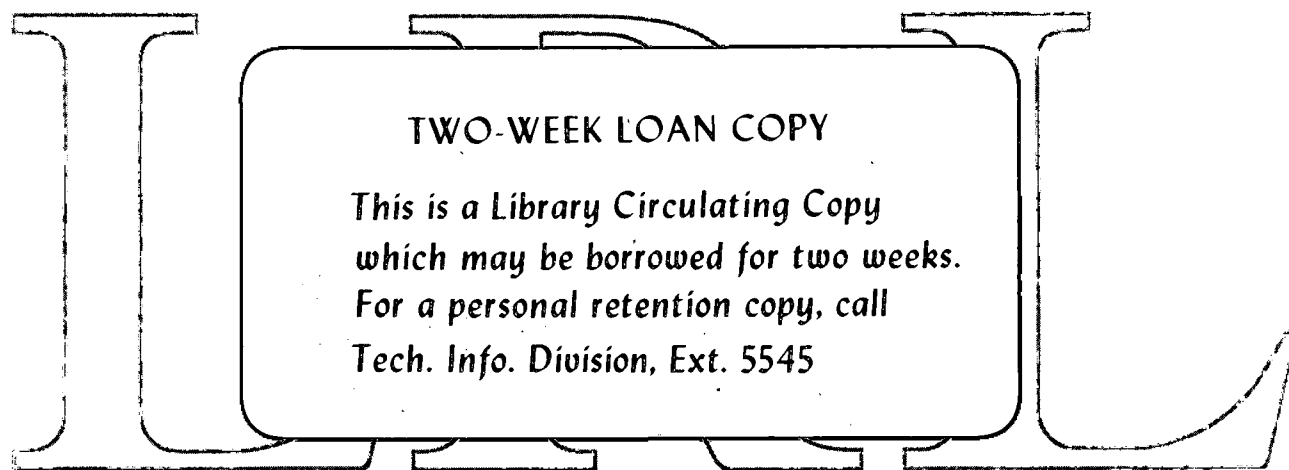
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A SALT OF A THREE-ICOSAHEDRAL-FRAGMENT METALLOCARBORANE

David St. Clair, Allan Zalkin, and David H. Templeton

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CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,  
UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

The Crystal Structure of  $\text{Cs}_2(\text{B}_9\text{C}_2\text{H}_{11})\text{Co}(\text{B}_8\text{C}_2\text{H}_{10})\text{Co}(\text{B}_9\text{C}_2\text{H}_{11})\cdot\text{H}_2\text{O}$ , a Salt of  
a Three-Icosahedral-Fragment Metallocarborane<sup>1</sup>

By David<sup>J</sup> St. Clair<sup>o</sup>, Allan Zalkin,<sup>†</sup> and David H. Templeton<sup>✓</sup>

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(1) Work done under the auspices of the U. S. Atomic Energy Commission.

The crystal structure of the monohydrate of the cesium salt of the bis- $\eta$ -[(3)-1,2-dicarbollylcobalt]- $\eta$ -(3,6)-1,2-dicarbocyanastide ion,  $\text{Cs}_2\text{Co}_2\text{B}_{26}\text{C}_{63}\text{H}_{32}\cdot\text{H}_2\text{O}$ , has been determined from 3649 independent X-ray data obtained from a single-crystal by counter methods. The crystals are monoclinic, space group  $P2_1/c$ . Four molecules are in the unit cell with  $a = 7.089 \text{ \AA}$ ,  $b = 19.240 \text{ \AA}$ ,  $c = 20.682 \text{ \AA}$ ,  $\beta = 98.00^\circ$ . The calculated density is  $\rho = 1.871 \text{ g/cc}$ . The structure was refined by least squares to a conventional R factor of 3.4%. The carborane anion,  $[(\text{B}_9\text{C}_2\text{H}_{11})\text{Co}(\text{B}_8\text{C}_2\text{H}_{10})\text{Co}(\text{B}_9\text{C}_2\text{H}_{11})]^{-2}$ , consists of three linked icosahedra. Two corners of a central ten light atom icosahedron are occupied by cobalts each of which is common to another icosahedron containing eleven light atoms. Five of the positions in the icosahedra are occupied by carbons but, because of disorder, two positions contain half-boron-half-carbon atoms. The cages are staggered across the cobalts and all carbons are as close to each other as is possible in this configuration. All hydrogens except those in the water molecule were located.

Introduction

A novel anion of the carborane type,  $[(B_9C_2H_{11})Co(B_8C_2H_{10})Co(B_9C_2H_{11})]^{-2}$ , the [bis[undecahydro-1,2-dicarba-3-cobalta-closo-dodecaborano]-[3',3';3,6]-comno-decahydro-1,2-dicarba-3,6-dicobalta-closo-dodecaborate](2-) ion, has recently been prepared<sup>2,3</sup>. Francis and Hawthorne proposed that the anion

(2) J. M. Francis and M. F. Hawthorne, J. Am. Chem. Soc., 90, 1663(1968).

(3) This name is an extension of the ACS-approved nomenclature rules given in Inorg. Chem., 7, 1945(1968), which do not explicitly cover the naming of ions in which a single polyhedron is completed by two identical groups attached at two different positions in the cage. The name originally given by Francis and Hawthorne in reference 2 is the bis- $\pi$ -[(3)-1,2-dicarbollylcobalt]- $\pi$ -(3,6)-1,2-dicarbacanastide ion. Both names fail to describe the rotational isomerism of the  $\pi$  bonding.

consisted of two of the previously known eleven atom icosahedral fragments each having a cobalt atom bonded to the open face with the cobalts linked together via a new ten atom icosahedral fragment. In this paper, we report the results of an X-ray analysis of the cesium salt of this anion,  $Cs_2(Co_2-B_{26}C_6H_{32}) \cdot H_2O$ , shown in Figure 1. We confirm the structure assigned for the anion by Francis and Hawthorne. We show that the rotational configuration of the cages in the crystal are those in which the cages are staggered and that the carbons are as close to each other as possible in this staggered arrangement.

Experimental

The dark red crystals were received from Professor Hawthorne of the University of California, Riverside. One was cut to the dimensions 0.11 x 0.12 x 0.23 mm and was glued to a glass fiber with its b axis coincident with the rotation axis of the fiber.

The space group and approximate cell dimensions were determined from oscillation, Weissenberg and precession photographs. Using a manual General Electric XRD-5 diffractometer, space group absences were checked and accurate unit cell dimensions were obtained from measurements of the h00, 0k0, and 00l reflections with the b axis of the crystal parallel to the  $\phi$  axis of the goniostat. Intensity data were measured with this manually operated diffractometer using a quarter circle, Eulerian cradle goniostat, and a scintillation counter equipped with a pulse height discriminator for detection of the diffracted beam. Molybdenum  $K\alpha$  X-rays were used ( $Mo K\alpha_1$ ;  $\lambda = 0.70926 \text{ \AA}$ .) after the  $K\beta$  radiation was filtered out by a 0.003 inch thick Zr filter placed in front of the receiving slit. One complete set of independent reflections (including space group absences) with  $2\theta \leq 45^\circ$  ( $\sin\theta/\lambda = 0.995$ ) was measured using the stationary crystal, stationary counter technique by taking ten second counts at the maximum intensity settings. Excluding space group extinctions, there were 3649 independent reflections of which 344 had intensities less than their estimated standard deviations.

The absorption coefficient is  $\mu = 38 \text{ cm}^{-1}$ . Absorption effects on the data were neglected. It is estimated that the scale factors which would be applied to the intensity data would vary by less than 12 %.

Calculations were done on a CDC 6600 computer. Lorentz and polarization corrections were applied and the data were converted to structure factors. Refinements of parameters were done with our least squares program which

minimizes the quantity  $\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2$  where  $F_o$  and  $F_c$  are the observed and calculated structure factors, respectively. The weighting factor,  $w = [\sigma(F_o)]^{-2}$ , is derived from the standard deviation of the intensity,  $\sigma(I)$ , estimated by the equation,  $\sigma(I) = [I + 2I_b + (0.05I)^2]^{1/2}$ , where  $I_b$  is the number of counts in the background and  $I$  is the number of counts on the peak minus  $I_b$ .

All atoms were considered to be in their neutral valence state. The scattering factors of Cromer and Waber<sup>4</sup> were used for all except the hydrogen

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

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atoms whose scattering factors are those of Stewart, Davidson and Simpson<sup>5</sup>.

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(5) R. F. Stewart, E. R. Davidson and W. T. Simpson, Table II, J. Chem. Phys., 42, 3175(1965).

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Cromer's corrections for anomalous dispersion<sup>6</sup> applied to the scattering

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

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factors of the Cs and Co are  $\Delta f' = -0.5$  and  $\Delta f'' = +2.6$  electrons and  $\Delta f' = +0.4$  and  $\Delta f'' = +1.0$  electrons, respectively. The anisotropic temperature factors were of the form  $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$ .



100

100

Crystal Data

The crystals were found to be monoclinic with unit cell dimensions, measured at room temperature ( $\sim 22^\circ$ ),  $a = 7.089 \pm 0.004 \text{ \AA}$ ,  $b = 19.240 \pm 0.014 \text{ \AA}$ ,  $c = 20.682 \pm 0.004 \text{ \AA}$ ,  $\beta = 98.00 \pm 0.06$  degrees. There are four formula units,  $\text{Cs}_2\text{Co}_2\text{B}_{26}\text{C}_6\text{OH}_{34}$ , per unit cell. The density is calculated to be  $\rho = 1.87 \text{ g/cc}$ . No density measurement was made because the sample was lost.

The observed extinction rules,  $h0l$ ,  $l = 2n$  and  $0k0$ ,  $k = 2n$ , correspond to space group  $P2_1/c$ . All atoms lie on the general symmetry related positions,  $\pm (x, y, z \text{ and } x, \frac{1}{2}-y, \frac{1}{2}+z)$ .

Determination of the Structure

The positions of the cesium and cobalt atoms were deduced from sections through a three-dimensional Patterson function synthesis. Full matrix least squares refinement of a scale factor and the positional and isotropic thermal parameters of the Cs and Co atoms gave  $R = 0.208$ . These atoms were used to calculate approximate phases for the reflections and a three-dimensional difference Fourier was synthesized from which the coordinates of the thirty two boron and carbon atoms were found. Since a water of crystallization was not expected, the oxygen atom which appeared in this Fourier was disregarded. When the positions and thermal parameters of these thirty two light atoms, all with the scattering power of boron, were incorporated into the refinement, the R value dropped to 0.105. Inspection of the bond distances and thermal parameters allowed location of five of the six carbon atoms and gave indications that the sixth carbon was disordered between only two atomic positions in one of the icosahedral cages. So in all further refinements, five of these atoms were given the scattering power of carbon and two were given the scattering power of half carbon and half boron. Following another series of refinements in which the Cs and Co atoms were given anisotropic thermal parameters, another difference Fourier was prepared in which again appeared the oxygen atom as well as thirty two of the thirty four hydrogen atoms. The two hydrogens in the water molecule did not appear. After some blunders in the intensity data were corrected, a final series of full matrix least squares refinement was carried out in which the scattering factors of the Cs and Co atoms were corrected for anomalous dispersion and zero weight was assigned to all reflections which had  $I < \sigma(I)$ . A scale factor, three positional parameters for each of the sixty nine atoms, six anisotropic temperature parameters for each Cs and Co atom, individual isotropic temperature parameters for the thirty three light

atoms excluding hydrogens, and an average temperature parameter for all thirty two hydrogens were refined. The conventional R value for all non-zero-weighted data reached  $R = 0.034$  for 3305 data, the weighted R value was  $R_2 = 0.039$  and the conventional R value calculated with all 3649 data was  $R = 0.041$ . The standard deviation of observation of unit weight was 1.2 and the r.m.s. values of  $(|F_o| - |F_c|)/\sigma(F_o)$  averaged over small intensity intervals approximated this value over the entire intensity range. No parameter in the final refinement differed from that of the previous refinement by an amount greater than one tenth of its estimated standard deviation.

Results and Discussion of the Structure

The final values for positional and thermal parameters are given in Table I for the heavy atoms, Table II for the oxygen, carbon, and boron atoms and in Table III for the hydrogen atoms. The observed and calculated structure factors are given in Table IV.

A drawing of the anion skeleton is given in Figure 1. It consists of two eleven-light atom icosahedral fragments, arbitrarily labeled cages I and III, each having a cobalt ion completing the icosahedron by bonding to the apex position of each cage. The cobalts are linked via the basket-shaped canastide ion completing a third icosahedron, labeled cage II, by bonding to the open faces in this bridging fragment. The cages are staggered with respect to each other across the cobalts. That is, atoms number 7 and 22 are equidistant from atoms 12 and 13. The two carbon atoms in each of cages I and II were located at positions 7 and 8 and 12 and 13, respectively. Disorder was found, however, in cage III, one carbon being localized at position 22 and the other being equally distributed between positions 23 and 26 adjacent to position 22. This disorder indicates the presence of three rotational isomers in the crystal differing only in the directions of rotation of the end cages. Beginning from a hypothetical configuration in which the carbons are all eclipsed, each end cage is rotated  $36^\circ$  either to the right or to the left. The isomers consist of an enantiomorphic pair with point symmetry  $\underline{2}$  and a third isomer with point symmetry  $\underline{n}$ . Since the external shape is not sensitive to the carbon positions, one might expect disorder at both ends of the anion. We have no explanation for the fact that all the disorder seems to be at cage III. Ignoring the distinction between boron and carbon, the anion has approximately  $\underline{2mm}$  ( $C_{2v}$ ) point symmetry in which one of the mirror planes contains atoms in positions 12, 13, 18, and 19 of cage II and the other mirror plane is perpendicular to

TABLE I

Coordinates( $\times 10^5$ ) and Thermal Parameters( $\times 10^2$ )<sup>a</sup> of Heavy Atoms<sup>b</sup>

| Atom  | <u>x</u>  | <u>y</u> | <u>z</u> | <u>B<sub>11</sub></u> | <u>B<sub>22</sub></u> | <u>B<sub>33</sub></u> | <u>B<sub>12</sub></u> | <u>B<sub>13</sub></u> | <u>B<sub>23</sub></u> |
|-------|-----------|----------|----------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|
| Cs(1) | 21468(6)  | 27505(2) | 17044(2) | 434(2)                | 336(2)                | 318(2)                | 017(1)                | 054(1)                | -012(1)               |
| Cs(2) | 05321(6)  | 02870(2) | 34230(2) | 503(2)                | 280(2)                | 449(2)                | 031(2)                | 106(2)                | -044(1)               |
| Co(1) | 23932(10) | 76225(4) | 58130(3) | 250(3)                | 195(3)                | 229(3)                | 020(2)                | -002(2)               | 010(2)                |
| Co(2) | 25769(10) | 57752(4) | 64795(3) | 231(3)                | 214(3)                | 239(3)                | -002(2)               | 002(2)                | 019(2)                |

(a) Anisotropic thermal parameters, B, in units of  $\text{\AA}^2$ , are given by  $\underline{B} = 4\beta_{-ij} / a_i^* a_j^*$  where  $a_i^*$  is the i<sup>th</sup> reciprocal cell length.

(b) Estimated standard deviations are given in parentheses following the parameter.

TABLE II

Coordinates( $\times 10^4$ ) and Thermal Parameters( $\times 10^2$ ) of the Light Atoms<sup>a, b</sup>

| Atom             | X        | Y       | Z       | B( $\text{\AA}^2$ ) |
|------------------|----------|---------|---------|---------------------|
| O(1)             | 7075(8)  | 5640(3) | 8419(3) | 643(12)             |
| Atoms in Cage I  |          |         |         |                     |
| B(1)             | 3781(10) | 8967(4) | 4548(3) | 329(13)             |
| B(2)             | 3175(10) | 8098(4) | 4321(3) | 321(13)             |
| B(3)             | 5330(10) | 8283(4) | 4825(3) | 326(13)             |
| B(4)             | 4890(9)  | 8951(4) | 5360(3) | 294(12)             |
| B(5)             | 2461(9)  | 9202(4) | 5183(3) | 292(12)             |
| B(6)             | 1410(10) | 8660(4) | 4538(3) | 309(13)             |
| C(7)             | 3825(8)  | 7631(3) | 5016(3) | 293(11)             |
| C(8)             | 4806(8)  | 8116(3) | 5604(3) | 295(11)             |
| B(9)             | 3196(9)  | 8664(3) | 5870(3) | 278(12)             |
| B(10)            | 0994(9)  | 8496(3) | 5351(3) | 266(12)             |
| B(11)            | 1482(9)  | 7788(4) | 4818(3) | 294(12)             |
| Atoms in Cage II |          |         |         |                     |
| C(12)            | 1639(7)  | 6581(3) | 5850(3) | 232(9)              |
| C(13)            | 3550(8)  | 6763(3) | 6316(2) | 236(10)             |
| B(14)            | 3294(9)  | 7459(3) | 6797(3) | 264(12)             |
| B(15)            | 0822(9)  | 7708(4) | 6597(3) | 299(12)             |
| B(16)            | -0137(9) | 7143(3) | 5945(3) | 251(11)             |
| B(17)            | 3360(9)  | 6575(3) | 7117(3) | 264(12)             |
| B(18)            | 1585(10) | 7179(4) | 7298(3) | 318(13)             |
| B(19)            | -0546(9) | 6978(3) | 6762(3) | 275(12)             |
| B(20)            | -0047(9) | 6259(3) | 6267(3) | 260(11)             |
| B(21)            | 0951(9)  | 6289(3) | 7108(3) | 284(12)             |

Atoms in Cage III

|        |          |         |         |         |
|--------|----------|---------|---------|---------|
| C(22)  | 4005(8)  | 5193(3) | 5879(3) | 297(11) |
| BC(23) | 5182(9)  | 5261(3) | 6618(3) | 292(11) |
| B(24)  | 3661(9)  | 5032(3) | 7168(3) | 256(12) |
| B(25)  | 1423(9)  | 4807(3) | 6691(3) | 265(12) |
| BC(26) | 1816(9)  | 4931(3) | 5893(3) | 306(12) |
| B(27)  | 5659(11) | 4569(4) | 6115(4) | 363(14) |
| B(28)  | 5409(10) | 4434(4) | 6947(3) | 337(13) |
| B(29)  | 3074(9)  | 4136(3) | 6982(3) | 284(12) |
| B(30)  | 1870(10) | 4088(4) | 6165(3) | 312(13) |
| B(31)  | 3488(10) | 4355(4) | 5649(4) | 339(14) |
| B(32)  | 4321(10) | 3856(4) | 6337(3) | 325(13) |

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(a) Numbering system used is shown in Figure 1.

(b) BC refers to disordered atoms, half boron and half carbon.

TABLE III

Coordinates ( $\times 10^3$ ) of the Hydrogen Atoms <sup>a, b, c</sup>

| Atom  | <u>x</u> | <u>y</u> | <u>z</u> | Atom  | <u>x</u> | <u>y</u> | <u>z</u> |
|-------|----------|----------|----------|-------|----------|----------|----------|
| H(1)  | 410      | 931      | 415      | H(17) | 473      | 650      | 745      |
| H(2)  | 326      | 787      | 385      | H(18) | 142      | 741      | 786      |
| H(3)  | 677      | 687      | 973      | H(19) | -200     | 703      | 687      |
| H(4)  | 615      | 927      | 556      | H(20) | -123     | 592      | 601      |
| H(5)  | 200      | 975      | 526      | H(21) | 038      | 601      | 753      |
| H(6)  | 038      | 885      | 415      | H(22) | 420      | 551      | 552      |
| H(7)  | 430      | 725      | 498      | H(23) | 623      | 559      | 667      |
| H(8)  | 603      | 793      | 587      | H(24) | 379      | 516      | 767      |
| H(9)  | 352      | 892      | 635      | H(25) | 000      | 484      | 684      |
| H(10) | 956      | 864      | 548      | H(26) | 105      | 507      | 556      |
| H(11) | 049      | 738      | 458      | H(27) | 701      | 455      | 589      |
| H(12) | 189      | 642      | 544      | H(28) | 662      | 430      | 733      |
| H(13) | 457      | 671      | 617      | H(29) | 269      | 376      | 738      |
| H(14) | 439      | 777      | 697      | H(30) | 067      | 372      | 601      |
| H(15) | 027      | 821      | 675      | H(31) | 331      | 424      | 518      |
| H(16) | 860      | 722      | 555      | H(32) | 466      | 332      | 628      |

(a) Number refers to the position in the cage occupied by the atom to which the hydrogen atom is attached. See Figure 1.

(b) Refined average temperature factor is  $B = 2.6 \pm 0.2 \text{ \AA}^2$ .

(c) Standard deviations for x, y, and z are  $\pm 0.007$ ,  $\pm 0.003$ , and  $\pm 0.003$ , respectively.



TABLE IV

Observed and Calculated Structure Factors

Reflections given zero weight in the final refinement are marked with an asterisk.

OBSERVED AND CALCULATED STRUCTURE FACTORS

| L FOD FCA             |  | 2 277 275           |  | 8 533 405            |  | 12 128 128  |  | 6 62 52              |  | 6 32 31              |  | 150 146              |  | 14 69 70    |  | -12 0 15*   |  | 9 33 32     |  | 4 350 355            |  | 11 41 33    |  | -17 23 17   |  | 12 130 133           |  |                      |  |
|-----------------------|--|---------------------|--|----------------------|--|-------------|--|----------------------|--|----------------------|--|----------------------|--|-------------|--|-------------|--|-------------|--|----------------------|--|-------------|--|-------------|--|----------------------|--|----------------------|--|
| H <sub>K</sub> -1, -0 |  | 8 288 84            |  | 10 213 205           |  | 7 134 136   |  | 7 55 64              |  | -6 20 23             |  | -6 275 269           |  | -13 0 0     |  | 11 21 222   |  | 9 133 133   |  | 6 105 101            |  | 14 175 174  |  | 15 75 80    |  | 13 50 49             |  |                      |  |
| 21 0 8*               |  | 4 401 403           |  | 10 108 107           |  | 14 268 266  |  | 8 267 263            |  | 8 44 44              |  | -5 0 14*             |  | -5 251 245  |  | -12 160 160 |  | -10 23 23   |  | 10 112 106           |  | 8 394 392   |  | 15 126 127  |  | -15 55 58            |  | H <sub>K</sub> 3, 14 |  |
| H <sub>K</sub> -1, -5 |  | 11 78 77            |  | 12 116 115           |  | 17 167 167  |  | 10 111 111           |  | 10 0 15*             |  | -3 149 143           |  | -3 302 299  |  | -10 219 223 |  | -8 346 356  |  | 12 163 163           |  | 17 163 157  |  | 17 78 80    |  | -13 86 92            |  | -13 104 102          |  |
| 21 62 65              |  | 6 67 63             |  | 12 116 108           |  | 17 167 167  |  | 10 111 111           |  | 10 0 15*             |  | -3 149 143           |  | -3 302 299  |  | -10 219 223 |  | -8 346 356  |  | 12 163 163           |  | 17 163 157  |  | 17 78 80    |  | -13 86 92            |  | -13 104 102          |  |
| H <sub>K</sub> -1, -4 |  | 7 137 137           |  | 13 178 170           |  | 17 0 0      |  | 11 169 171           |  | 11 209 200           |  | -2 24 19             |  | -2 44 46    |  | -9 16 21    |  | -7 0 16*    |  | 10 56 13             |  | 14 358 360  |  | 10 0 11*    |  | -12 70 75            |  | -12 68 74            |  |
| 21 26 110             |  | 4 13 20             |  | 10 104 104           |  | 17 167 167  |  | 10 111 111           |  | 10 0 15*             |  | -3 149 143           |  | -3 302 299  |  | -10 219 223 |  | -8 346 356  |  | 12 163 163           |  | 17 163 157  |  | 17 78 80    |  | -13 86 92            |  | -13 104 102          |  |
| H <sub>K</sub> -1, -3 |  | 8 84 84             |  | 15 91 92             |  | 19 42 48    |  | 13 40 37             |  | 13 88 91             |  | 0 88 90              |  | 0 189 188   |  | -7 0 16*    |  | -5 141 142  |  | 15 75 78             |  | 18 70 75    |  | -20 10 10   |  | -10 93 95            |  | -10 31 28            |  |
| 21 0 1                |  | 10 91 92            |  | H <sub>K</sub> 0, 16 |  | 20 73 72    |  | 14 168 167           |  | 14 21 17             |  | 1 0 16*              |  | 1 329 325   |  | -6 217 218  |  | -4 415 419  |  | H <sub>K</sub> 3, 14 |  | 1 19 23     |  | 25 17 17    |  | -19 53 55            |  | -19 53 55            |  |
| 20 2 13*              |  | 11 270 272          |  | 0 228 226            |  | 17 17 17    |  | 15 265 266           |  | 15 221 218           |  | 2 303 307            |  | 2 255 246   |  | -5 10 7*    |  | -3 227 230  |  | -16 50 55            |  | -21 174 178 |  | -10 109 112 |  | -10 109 112          |  | -10 109 112          |  |
| 21 33 37              |  | 13 0 10*            |  | 2 129 130            |  | -20 30 39   |  | 17 137 131           |  | 17 243 237           |  | 4 49 48              |  | 4 236 239   |  | -3 130 130  |  | -1 112 115  |  | -14 59 61            |  | -19 0 0     |  | -16 210 204 |  | -6 0 9*              |  | -6 0 9*              |  |
| 22 93 100             |  | 14 87 84            |  | 3 103 98             |  | 19 46 49    |  | 18 0 2*              |  | 24 24 25             |  | 5 20 32              |  | 5 173 174   |  | -2 95 92    |  | -1 149 151  |  | -13 66 74            |  | -18 141 136 |  | -15 89 87   |  | -5 12 7              |  | -5 12 7              |  |
| H <sub>K</sub> -1, -1 |  | 15 101 103          |  | 4 116 115            |  | -18 113 114 |  | 7 11 7               |  | H <sub>K</sub> 1, 11 |  | 6 41 47              |  | -1 103 106  |  | -1 103 106  |  | -12 113 113 |  | -12 113 113          |  | -14 215 216 |  | -14 215 216 |  | -14 215 216          |  | -14 215 216          |  |
| 21 80 79              |  | 16 150 156          |  | 5 55 49              |  | -17 0 21*   |  | 20 50 50             |  | -18 91 90            |  | 7 61 63              |  | 7 272 275   |  | 0 288 289   |  | 2 277 275   |  | -11 78 78            |  | -12 159 117 |  | -13 43 38   |  | -3 55 53             |  | -3 54 54             |  |
| 22 17 19*             |  | 17 116 124          |  | 6 266 261            |  | -16 20 34   |  | H <sub>K</sub> 0, 17 |  | -17 0 2*             |  | 7 64 66              |  | 8 335 331   |  | 1 168 157   |  | 3 213 216   |  | -10 120 124          |  | -14 261 257 |  | -12 73 75   |  | -12 73 75            |  | -12 73 75            |  |
| H <sub>K</sub> 0, 0   |  | 18 66 63            |  | 7 112 107            |  | -20 94 91   |  | -16 93 95            |  | 9 15 45              |  | 2 214 218            |  | 2 214 218   |  | -9 39 31    |  | -13 0 7*    |  | -15 151 151          |  | -10 239 281 |  | 0 80 81     |  | 0 80 81              |  | 0 80 81              |  |
| 2 199 181             |  | 19 54 50            |  | 8 40 37              |  | -14 170 164 |  | -19 88 94            |  | -15 36 41            |  | 10 31 32             |  | 10 95 100   |  | 3 161 178   |  | 5 278 282   |  | -7 241 241           |  | -11 166 169 |  | -9 36 45    |  | 1 139 142            |  | 1 58 59              |  |
| 4 237 261             |  | 20 104 99           |  | 9 0 21*              |  | -13 12 6    |  | -18 127 129          |  | -14 130 134          |  | 11 115 107           |  | 11 218 220  |  | 4 255 253   |  | 6 284 282   |  | -7 241 241           |  | -11 166 169 |  | -9 36 45    |  | 1 139 142            |  | 1 58 59              |  |
| 6 536 546             |  | H <sub>K</sub> 0, 8 |  | 10 114 114           |  | -12 168 168 |  | -17 40 44            |  | -13 143 143          |  | 12 130 128           |  | 12 0 0      |  | 5 216 216   |  | 1 177 185   |  | -6 306 311           |  | -10 69 60   |  | -8 163 167  |  | 2 136 131            |  | 2 59 61              |  |
| 10 341 353            |  | 1 501 501           |  | 12 148 142           |  | -10 140 140 |  | -15 32 24            |  | -11 130 140          |  | 14 94 92             |  | 14 62 59    |  | 7 0 15*     |  | 9 213 203   |  | -4 20 23             |  | -8 243 237  |  | -6 65 69    |  | 4 0 3*               |  | 4 0 3*               |  |
| 12 531 539            |  | 2 28 25             |  | 13 80 85             |  | -9 98 97    |  | -14 263 271          |  | -10 100 103          |  | H <sub>K</sub> 1, 16 |  | 15 0 19*    |  | 8 267 305   |  | 10 73 70    |  | -3 154 157           |  | -7 22 21    |  | -5 266 276  |  | 5 135 130            |  | 5 0 9*               |  |
| 14 62 61              |  | 3 37 37             |  | 14 82 75             |  | -8 206 206  |  | -13 186 193          |  | -9 256 262           |  | -14 0 18*            |  | 9 196 194   |  | 11 58 61    |  | -2 216 216  |  | -6 310 327           |  | -4 310 327  |  | -4 310 327  |  | -4 310 327           |  | -4 310 327           |  |
| 18 68 74              |  | 5 586 590           |  | 1 37 40              |  | -6 446 448  |  | -11 52 55            |  | -7 169 176           |  | -12 142 149          |  | 18 22 34    |  | 11 117 116  |  | 13 178 177  |  | 0 271 270            |  | -4 357 362  |  | -2 210 220  |  | 8 159 155            |  | 8 78 75              |  |
| 20 42 53              |  | 6 97 100            |  | 2 195 197            |  | -5 240 237  |  | -10 136 143          |  | -6 12 12             |  | -6 12 12             |  | 16 88 88    |  | 14 88 88    |  | 1 130 130   |  | 3 198 197            |  | -1 334 333  |  | 9 33 39     |  | 9 16 23              |  | 9 16 23              |  |
| 22 38 37              |  | 7 422 421           |  | 3 50 49              |  | -6 106 102  |  | -9 196 196           |  | -5 126 124           |  | -10 139 143          |  | 20 176 173  |  | 13 51 51    |  | 15 91 88    |  | 2 14 7               |  | -2 340 343  |  | 0 191 193   |  | 10 46 47             |  | 10 54 62             |  |
| H <sub>K</sub> 0, 1   |  | 8 115 115           |  | 4 121 120            |  | -3 323 323  |  | -8 233 238           |  | -4 350 357           |  | -7 44 47             |  | 2 142 142   |  | 16 23 26    |  | 3 0 13*     |  | -1 82 88             |  | 1 292 300   |  | 11 176 171  |  | 11 70 69             |  | 11 70 69             |  |
| 3 84 76               |  | 8 192 193           |  | 6 20 44              |  | -5 465 467  |  | -7 170 163           |  | -7 170 163           |  | -8 44 44             |  | -8 44 44    |  | -8 44 44    |  | -8 44 44    |  | -8 44 44             |  | -8 44 44    |  | -8 44 44    |  | -8 44 44             |  | -8 44 44             |  |
| 4 365 356             |  | 11 336 340          |  | 7 113 105            |  | -10 139 134 |  | -5 44 44             |  | -10 139 134          |  | -5 44 44             |  | -10 139 134 |  | -5 44 44    |  | -10 139 134 |  | -5 44 44             |  | -10 139 134 |  | -5 44 44    |  | -10 139 134          |  | -5 44 44             |  |
| 5 201 196             |  | 12 126 125          |  | 8 113 132            |  | 1 508 503   |  | -4 241 240           |  | C 305 305            |  | -6 163 166           |  | -18 152 149 |  | 18 60 60    |  | 19 182 192  |  | 7 90 85              |  | 3 311 326   |  | 5 291 294   |  | 15 102 97            |  | 12 67 63             |  |
| 6 68 85               |  | 10 120 148          |  | 4 42 10              |  | 3 204 200   |  | -2 341 341           |  | 2 202 200            |  | -3 21 21             |  | -16 52 55   |  | -21 0 14*   |  | -17 112 117 |  | 9 178 177            |  | 5 90 87     |  | 7 152 152   |  | H <sub>K</sub> 3, 10 |  | -10 131 137          |  |
| 8 295 291             |  | 15 25 21            |  | 11 168 166           |  | 4 321 313   |  | -1 207 204           |  | 3 231 234            |  | -2 41 37             |  | -15 43 45   |  | -21 0 14*   |  | -17 112 117 |  | 9 178 177            |  | 5 90 87     |  | 7 152 152   |  | H <sub>K</sub> 3, 10 |  | -10 131 137          |  |
| 9 325 320             |  | 17 196 200          |  | H <sub>K</sub> 0, 18 |  | 6 423 414   |  | 1 303 297            |  | 5 241 236            |  | 0 196 190            |  | 13 66 61    |  | -19 154 154 |  | -14 26 32   |  | 12 65 64             |  | 8 93 94     |  | 10 0 3*     |  | -16 36 31            |  | -7 44 49             |  |
| 11 46 42              |  | 16 0 4*             |  | 0 82 80              |  | 7 266 270   |  | 2 128 130            |  | 6 197 194            |  | 1 0 8*               |  | -12 47 48   |  | -18 22 18   |  | -13 25 27   |  | 13 33 34             |  | 8 93 96     |  | 11 246 247  |  | -15 61 64            |  | -6 46 46             |  |
| 12 168 168            |  | 16 40 40            |  | 1 51 51              |  | 15 165 163  |  | 6 21 16              |  | 10 58 51             |  | 5 49 53              |  | -9 465 471  |  | -14 0 15*   |  | -9 65 70    |  | -13 28 17            |  | 13 148 147  |  | 15 110 110  |  | -11 240 232          |  | -2 58 61             |  |
| 13 105 103            |  | 20 73 76            |  | 2 0 1*               |  | 19 174 174  |  | 4 153 150            |  | 8 150 157            |  | 3 0 3*               |  | -10 86 89   |  | -16 65 67   |  | -11 95 98   |  | H <sub>K</sub> 2, 15 |  | 11 53 56    |  | 13 162 164  |  | -13 252 252          |  | -4 66 67             |  |
| 14 34 31              |  | H <sub>K</sub> 3, 9 |  | 3 60 56              |  | 10 299 253  |  | 5 93 92              |  | 9 57 55              |  | 4 15 11              |  | -9 49 54    |  | -15 87 91   |  | -10 13 18*  |  | -14 67 73            |  | 12 53 52    |  | 14 66 64    |  | -12 23 25            |  | -3 104 105           |  |
| 15 164 168            |  | 1 752 748           |  | 15 95 96             |  | 11 165 163  |  | 6 21 16              |  | 10 58 51             |  | 5 49 53              |  | -9 465 471  |  | -14 0 15*   |  | -9 65 70    |  | -13 28 17            |  | 13 148 147  |  | 15 110 110  |  | -11 240 232          |  | -2 58 61             |  |
| 16 107 107            |  | 16 40 40            |  | 5 139 126            |  | 4 109 107   |  | 15 192 195           |  | 8 150 157            |  | 3 0 3*               |  | -10 86 89   |  | -16 65 67   |  | -11 95 98   |  | H <sub>K</sub> 2, 15 |  | 11 53 56    |  | 13 162 164  |  | -13 252 252          |  | -4 66 67             |  |
| 17 41 30              |  | 3 217 241           |  | 6 50 44              |  | -7 287 287  |  | 8 238 239            |  | 12 212 208           |  | 7 15 23              |  | -6 276 276  |  | -12 285 291 |  | -7 454 468  |  | -11 35 32            |  | 15 0 30*    |  | 17 51 54    |  | -29 21 0             |  | 0 2 0                |  |
| 18 93 95              |  | 4 0 3*              |  | 7 76 73              |  | 14 78 81    |  | 9 219 215            |  | 13 87 82             |  | 8 62 66              |  | -5 271 272  |  | -11 214 222 |  | -6 125 131  |  | -10 148 152          |  | 16 22 33    |  | 18 68 82    |  | -6 67 66             |  | 1 195 197            |  |
| 20 122 121            |  | 6 20 18             |  | 9 10 10              |  | 16 74 75    |  | 11 168 168           |  | 15 104 113           |  | 10 51 48             |  | -3 19 19    |  | -9 75 77    |  | -4 17 27    |  | -8 0 0               |  | 5 16 10     |  | -20 6 2     |  | -6 55 54             |  | 3 83 77              |  |
| 21 64 63              |  | 5 22 24             |  | 10 107 102           |  | 17 110 109  |  | 12 118 117           |  | 16 64 62             |  | 11 23 22             |  | -2 463 475  |  | -6 157 160  |  | -3 393 400  |  | -7 71 72             |  | 16 76 81    |  | -19 89 83   |  | -5 346 348           |  | 4 0 6*               |  |
| 22 34 34              |  | 2 187 187           |  | 4 109 107            |  | 14 106 104  |  | 14 24 24             |  | 10 0 13*             |  | 13 94 92             |  | 0 560 562   |  | -6 299 304  |  | -5 108 107  |  | -21 34 23            |  | -17 183 182 |  | -3 113 115  |  | 10 6 52 49           |  | 10 6 52 49           |  |
| 0 117 142             |  | 10 167 156          |  | 2 114 109            |  | 23 67 67    |  | 15 48 55             |  | H <sub>K</sub> 1, 12 |  | H <sub>K</sub> 1, 17 |  | 1 236 232   |  | -5 301 311  |  | 0 50 47     |  | -4 137 139           |  | -2 92 84    |  | -16 26 26   |  | -2 102 100           |  | 7 115 117            |  |
| 0 216 199             |  | 11 146 147          |  | 3 78 52              |  | 21 34 34    |  | 16 37 35             |  | -18 81 86            |  | -17 95 100           |  |             |  |             |  |             |  |                      |  |             |  |             |  |                      |  |                      |  |

|      |     |     |     |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |     |     |      |      |      |      |     |     |    |    |    |
|------|-----|-----|-----|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|-----|-----|------|------|------|------|-----|-----|----|----|----|
| H.K. | 4   | 1   | -19 | 110 | 105 | -4   | 153 | 161 | 13   | 74  | 67  | 3    | 145 | 143 | 8    | 17  | 17  | 1    | 79  | 78  | -13  | 172 | 172 | 12   | 126 | 124 | -11  | 122 | 122 | 3    | 114 | 114 | -2   | 150 | 154 | 6    | 81  | 89  | -1   | 63   | 55   | -4   | 62  | 64  |    |    |    |
| 8    | 144 | 140 | -16 | 56  | 59  | -3   | 21  | 31  | 14   | 0   | 3*  | 4    | 25  | 23  | 9    | 71  | 72  | 2    | 27  | 28  | -12  | 183 | 183 | 13   | 86  | 85  | H.K. | 5   | 10  | 4    | 16  | 0   | -1   | 48  | 47  | 7    | 70  | 75  | 0    | 42   | 44   | -3   | 0   | 8*  |    |    |    |
| 9    | 75  | 76  | -13 | 22  | 34  | -2   | 0   | 18* | 15   | 94  | 97  | 5    | 140 | 137 | H.K. | 4   | 15  | 3    | 0   | 10* | -11  | 122 | 122 | H.K. | 5   | 7   | -14  | 29  | 30  | 9    | 0   | 9*  | 0    | 9*  | 8   | 16   | 23  | 1   | 139  | 132  | -2   | 58   | 56  |     |    |    |    |
| 10   | 28  | 32  | -16 | 0   | 14* | -1   | 33  | 31  | H.K. | 4   | 9   | 7    | 79  | 82  | -11  | 143 | 145 | 4    | 121 | 123 | -10  | 48  | 44  | -16  | 182 | 144 | -13  | 86  | 86  | 6    | 50  | 49  | 1    | 0   | 1*  | 9    | 96  | 88  | 3    | 9    | 4    | -1   | 0   | 11* |    |    |    |
| 11   | 20  | 18  | -15 | 220 | 220 | 0    | 266 | 267 | -17  | 29  | 33  | 7    | 90  | 87  | -10  | 50  | 46  | 5    | 140 | 144 | -9   | 72  | 69  | -15  | 77  | 74  | -12  | 23  | 19  | 7    | 17  | 7   | 2    | 211 | 207 | 10   | 83  | 91  | 6    | 4    | 36   | 0    | 76  | 73  |    |    |    |
| 12   | 21  | 18  | -14 | 201 | 200 | 1    | 89  | 89  | -16  | 88  | 86  | 8    | 0   | 12* | -5   | 76  | 77  | 6    | 150 | 159 | -9   | 254 | 254 | -14  | 99  | 105 | -11  | 114 | 110 | H.K. | 5   | 14  | 3    | 21  | 30  | H.K. | 6   | 6   | 4    | 28   | 32   | 1    | 23  | 10  |    |    |    |
| 13   | 0   | 3*  | -1  | 259 | 260 | 2    | 70  | 70  | -15  | 45  | 39  | 0    | 131 | 136 | -8   | 89  | 93  | 7    | 48  | 54  | -7   | 143 | 133 | -13  | 124 | 123 | -10  | 0   | 3*  | -9   | 66  | 60  | 4    | 117 | 115 | -14  | 17  | 13  | 5    | 91   | 85   | 2    | 0   | 7*  |    |    |    |
| 14   | 49  | 46  | -12 | 183 | 182 | 3    | 51  | 56  | -14  | 83  | 78  | 10   | 106 | 106 | -7   | 84  | 93  | 8    | 130 | 133 | -6   | 95  | 94  | -12  | 127 | 135 | -9   | 109 | 109 | -8   | 130 | 130 | 5    | 63  | 67  | -13  | 44  | 44  | 6    | 100  | 98   | 3    | 86  | 81  |    |    |    |
| 15   | 0   | 19* | -11 | 90  | 92  | 4    | 139 | 141 | -13  | 22  | 22  | 11   | 0   | 14* | -6   | 73  | 77  | 0    | 89  | 86  | -4   | 117 | 121 | -11  | 37  | 38  | -8   | 77  | 80  | -7   | 74  | 68  | 6    | 92  | 90  | -12  | 43  | 46  | 7    | 66   | 65   | 4    | 100 | 136 |    |    |    |
| 16   | 118 | 116 | -10 | 182 | 178 | 5    | 130 | 130 | -12  | 101 | 97  | 12   | 33  | 29  | -5   | 84  | 85  | 10   | 75  | 76  | -3   | 172 | 178 | -10  | 109 | 109 | -7   | 0   | 4*  | -6   | 23  | 20  | 7    | 16  | 13  | -11  | 46  | 45  | H.K. | 6    | 10   | 5    | 34  | 29  |    |    |    |
| 17   | 0   | 10* | -9  | 257 | 260 | 6    | 45  | 39  | -11  | 141 | 143 | 13   | 159 | 159 | -4   | 22  | 30  | 11   | 115 | 116 | -2   | 68  | 65  | -9   | 166 | 168 | -6   | 15  | 13  | -5   | 111 | 110 | 9    | 63  | 62  | -10  | 60  | -10 | 29   | 22   | H.K. | 7    | 3   |     |    |    |    |
| H.K. | 4   | 2   | -8  | 328 | 323 | 7    | 178 | 172 | -10  | 177 | 180 | H.K. | 4   | 12  | -3   | 116 | 117 | 12   | 16  | 3   | -1   | 219 | 216 | -8   | 32  | 39  | -5   | 218 | 219 | -4   | 144 | 146 | 10   | 50  | 52  | -9   | 125 | 119 | -9   | 196  | 199  | -11  | 48  | 47  |    |    |    |
| -19  | 52  | 51  | -7  | 250 | 254 | 8    | 54  | 84  | -9   | 63  | 64  | -14  | 109 | 107 | -2   | 16  | 19  | 13   | 16  | 10  | 0    | 171 | 174 | -7   | 77  | 78  | -4   | 58  | 60  | -3   | 77  | 77  | 11   | 42  | 39  | -8   | 70  | 71  | -8   | 0    | 1*   | -10  | 44  | 40  |    |    |    |
| -18  | 95  | 88  | -6  | 17  | 28  | 0    | 0   | 0   | -8   | 31  | 34  | -13  | 83  | 87  | -1   | 32  | 36  | 14   | 47  | 46  | 1    | 40  | 42  | -6   | 60  | 61  | -3   | 88  | 90  | -2   | 85  | 77  | 3    | 7   | 7   | H.K. | 6   | 3   | -7   | 123  | 119  | -9   | 33  | 24  |    |    |    |
| -17  | 22  | 25  | -4  | 76  | 77  | 10   | 39  | 38  | -7   | 121 | 122 | -12  | 42  | 35  | C    | 22  | 22  | H.K. | 5   | 2   | 2    | 0   | 10* | -5   | 63  | 65  | -2   | 15  | 16  | -1   | 40  | 39  | -15  | 34  | 28  | -6   | 0   | 16* | -6   | 56   | 55   | -8   | 40  | 35  |    |    |    |
| -16  | 68  | 67  | -3  | 315 | 327 | 11   | 74  | 72  | -6   | 79  | 81  | -11  | 31  | 32  | 1    | 233 | 231 | -18  | 38  | 37  | 3    | 197 | 198 | -4   | 176 | 172 | -1   | 95  | 93  | 0    | 37  | 38  | -14  | 77  | 77  | -5   | 53  | 58  | -14  | 44   | 44   |      |     |     |    |    |    |
| -15  | 21  | 16  | -2  | 271 | 278 | 12   | 121 | 119 | -5   | 95  | 96  | -10  | 77  | 82  | 2    | 0   | 43* | -17  | 0   | 10* | 4    | 143 | 136 | -3   | 74  | 73  | 0    | 26  | 12  | 1    | 57  | 59  | -13  | 81  | 75  | -4   | 37  | 35  | -4   | 28   | 34   | -6   | 54  | 91  |    |    |    |
| -14  | 32  | 24  | -1  | 107 | 108 | 13   | 105 | 107 | -4   | 75  | 72  | -9   | 78  | 78  | 3    | 66  | 67  | -16  | 53  | 46  | 5    | 95  | 98  | -2   | 24  | 34  | 1    | 125 | 126 | 2    | 128 | 128 | -12  | 0   | 11* | -3   | 170 | 174 | -236 | 235  | -5   | 46   | 43  |     |    |    |    |
| -13  | 28  | 25  | 0   | 168 | 165 | 14   | 40  | 43  | -3   | 121 | 124 | -8   | 143 | 143 | 4    | 46  | 48  | -15  | 16  | 3   | 6    | 0   | 3   | 6    | 0   | 3   | 6    | 0   | 3   | 112  | 117 | -11 | 116  | 110 | -2  | 66   | 62  | -28 | 19   | -4   | 61   | 64   |     |     |    |    |    |
| -12  | 45  | 40  | 1   | 182 | 183 | 15   | 29  | 28  | -2   | 27  | 18  | -7   | 229 | 231 | 5    | 155 | 147 | -14  | 105 | 103 | 7    | 0   | 15* | 0    | 182 | 183 | 3    | 57  | 55  | 4    | 114 | 111 | -10  | 31  | 29  | -1   | 37  | 32  | -1   | 16   | 5    | -3   | 16  | 15  |    |    |    |
| -11  | 23  | 15  | 0   | 254 | 259 | 16   | 115 | 112 | -1   | 0   | 0   | 1    | 33  | 30  | 4    | 33  | 36  | 3    | 26  | 27  | 8    | 56  | 61  | -12  | 45  | 47  | 4    | 41  | 42  | 5    | 29  | 27  | 9    | 31  | 45  | 0    | 146 | 143 | 0    | 46   | 48   | -2   | 104 | 101 |    |    |    |
| -10  | 172 | 165 | 3   | 150 | 156 | H.K. | 4   | 7   | 0    | 133 | 133 | -5   | 99  | 95  | 7    | 92  | 93  | -12  | 119 | 118 | 9    | 92  | 81  | 2    | 49  | 51  | 5    | 22  | 14  | H.K. | 5   | 15  | -8   | 64  | 62  | 1    | 86  | 79  | 1    | 167  | 164  | -1   | 0   | 10* |    |    |    |
| -9   | 10  | 8   | 4   | 252 | 255 | -18  | 50  | 49  | 0    | 143 | 145 | -4   | 170 | 170 | 8    | 34  | 23  | -11  | 0   | 15* | 10   | 95  | 95  | 3    | 120 | 122 | 6    | 16  | 21  | -6   | 0   | 10* | -7   | 107 | 106 | 2    | 83  | 87  | 2    | 49   | 45   | 0    | 54  | 51  |    |    |    |
| -8   | 116 | 116 | 5   | 48  | 42  | -17  | 95  | 93  | 2    | 14  | 3   | -14  | 216 | 216 | H.K. | 4   | 16  | -10  | 234 | 231 | 11   | 0   | 1*  | 4    | 70  | 61  | 7    | 135 | 138 | -5   | 56  | 58  | 36   | 42  | 3   | 121  | 111 | 3   | 92   | 92   | 1    | 16   | 12  |     |    |    |    |
| -7   | 69  | 68  | 6   | 29  | 25  | 4    | 182 | 185 | 3    | 147 | 152 | -2   | 129 | 125 | -9   | 53  | 53  | -9   | 86  | 88  | 12   | 16  | 26  | 5    | 0   | 14* | 8    | 51  | 47  | -4   | 47  | 46  | -5   | 91  | 86  | 4    | 117 | 116 | 4    | 0    | 8*   | 2    | 0   | 6*  |    |    |    |
| -6   | 238 | 244 | 7   | 78  | 73  | -15  | 15  | 24  | 3    | 168 | 174 | -1   | 167 | 198 | -8   | 143 | 140 | 0    | 0   | 4*  | 13   | 47  | 47  | 6    | 15  | 12  | 9    | 40  | 44  | -3   | 17  | 12  | -4   | 120 | 122 | 5    | 78  | 76  | 5    | 29   | 18   | 3    | 17  | 5*  |    |    |    |
| -5   | 179 | 180 | 8   | 33  | 34  | -14  | 34  | 26  | 5    | 44  | 44  | 0    | 50  | 52  | -7   | 94  | 96  | 7    | 72  | 67  | 14   | 29  | 26  | 7    | 15  | 7   | 15   | 34  | 22  | -2   | 60  | 58  | -3   | 0   | 14  | 6    | 16  | 1*  | 6    | 24   | 26   |      |     |     |    |    |    |
| -4   | 50  | 51  | 0   | 107 | 108 | -13  | 110 | 110 | 5    | 44  | 49  | 1    | 0   | 18* | -4   | 112 | 108 | -6   | 168 | 175 | H.K. | 5   | 9   | 0    | 46  | 5   | H.K. | 5   | 11  | -1   | 47  | 46  | -10  | 0   | 4*  | 0    | 4*  | 5   | 51   | H.K. | 5    | 11   | 5   | 24  | 30 |    |    |
| -3   | 60  | 64  | 10  | 130 | 140 | -12  | 140 | 141 | 7    | 77  | 72  | 2    | 44  | 45  | -5   | 28  | 35  | -2   | 44  | 45  | -5   | 28  | 35  | 9    | 0   | 2*  | -13  | 47  | 48  | 0    | 29  | 34  | -1   | 138 | 134 | 8    | 33  | 34  | -9   | 54   | 51   | H.K. | 7   | 4   |    |    |    |
| -2   | 12  | 17* | 11  | 21  | 18  | -11  | 155 | 152 | 8    | 113 | 113 | 3    | 161 | 176 | -4   | 111 | 104 | -4   | 128 | 129 | -16  | 63  | 49  | 10   | 28  | 32  | -12  | 103 | 97  | 1    | 34  | 29  | 0    | 0   | 13* | 9    | 117 | 117 | -8   | 0    | 3*   | -10  | 110 | 108 |    |    |    |
| -1   | 67  | 70  | 12  | 72  | 80  | -17  | 174 | 180 | 9    | 123 | 125 | 4    | 103 | 101 | -3   | 153 | 155 | -3   | 142 | 142 | -15  | 36  | 34  | 11   | 36  | 32  | 2    | 38  | 42  | 1    | 132 | 131 | H.K. | 6   | 7   | 7    | 17  | 24  | -9   | 17   | 24   |      |     |     |    |    |    |
| 0    | 114 | 114 | 13  | 0   | 2*  | 9    | 0   | 11* | 10   | 11  | 3   | 5    | 176 | 179 | -2   | 192 | 186 | -4   | 127 | 151 | -14  | 120 | 120 | 12   | 62  | 61  | -10  | 96  | 94  | H.K. | 6   | 0   | 2    | 105 | 108 | -13  | 93  | 96  | -6   | 33   | 34   | -8   | 60  | 68  |    |    |    |
| 1    | 86  | 82  | 14  | 91  | 91  | -8   | 58  | 56  | 11   | 16  | 10  | 6    | 0   | 10* | -1   | 28  | 20  | -1   | 49  | 49  | -13  | 0   | 9*  | H.K. | 5   | 8   | -9   | 101 | 97  | -16  | 38  | 30  | 3    | 15  | 8   | -12  | 17  | 21  | -5   | 1    | 5    | 10*  | -7  | 76  | 79 |    |    |
| 2    | 39  | 42  | 15  | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38  | 42  | 15   | 38   | 42   | 15   | 38  | 42  | 15 | 38 | 42 |

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the first one and bisects the bonds between atoms 12 to 13 and 18 to 19. The 2-fold rotation axis is along the intersection of these two mirror planes. If the carbon atom in position 8 of cage I were disordered equally between positions 8 and 11, the anion would have  $2mm$  point symmetry with respect to all atoms including borons and carbons. The axis of cage I, Co(1) to B(1), makes an angle of  $117^\circ$  with the axis of cage III, Co(2) to B(32). The cages are arranged such that all the carbon atoms lie on the interior side of this angle and are as close to each other as is possible in this staggered configuration.

The bond distances between all the atoms in the icosahedral framework except hydrogens are listed in Table V and the distances between the hydrogen atoms and the atoms to which they are bonded are given in Table VI. The standard deviations quoted on the bond distances in these tables are calculated from the least squares estimates of the accuracy of the final positional parameters. Probably a better estimate of their accuracy is given by the larger root-mean-square deviations of equivalent bond distances from their average values, given in Table VII. A complete list of bond angles would be a rather long list indeed. Therefore, since both the cages I and III are analogous to the previously investigated eleven-atom icosahedral fragments<sup>7</sup>, only the angles involving the

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

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ten-atom fragment, cage II, are reported in Table VIII.

The molecular packing in the crystal is shown in Figure 2 by a framework drawing of the projection of the contents of the unit cell seen from a view along the a axis. This drawing together with the distances given in Table IX reveals a series of interactions between the cesium ions, the oxygens of the water molecules and the hydrogen atoms attached to the anion. The cesium ions

TABLE V

## Bond Distances

| <u>Atom 1</u> | <u>Atom 2</u> | <u>Distance (<math>\text{\AA}</math>)</u> | <u>Atom 1</u> | <u>Atom 2</u> | <u>Distance (<math>\text{\AA}</math>)</u> |
|---------------|---------------|---|---------------|---------------|---|
| Co(1)         | C(8)          | 2.051 $\pm$ .006                          | C(7)          | B(2)          | 1.702 $\pm$ .009                          |
| Co(1)         | C(7)          | 2.051 $\pm$ .006                          | B(11)         | B(10)         | 1.815 $\pm$ .009                          |
| Co(1)         | B(11)         | 2.094 $\pm$ .007                          | B(11)         | B(2)          | 1.787 $\pm$ .009                          |
| Co(1)         | B(10)         | 2.113 $\pm$ .007                          | B(11)         | B(6)          | 1.772 $\pm$ .009                          |
| Co(1)         | B(9)          | 2.081 $\pm$ .007                          | B(10)         | B(9)          | 1.797 $\pm$ .009                          |
| Co(1)         | C(12)         | 2.070 $\pm$ .005                          | B(10)         | B(6)          | 1.775 $\pm$ .009                          |
| Co(1)         | C(13)         | 2.061 $\pm$ .005                          | B(10)         | B(5)          | 1.774 $\pm$ .009                          |
| Co(1)         | B(14)         | 2.070 $\pm$ .006                          | B(9)          | B(5)          | 1.776 $\pm$ .009                          |
| Co(1)         | B(15)         | 2.100 $\pm$ .007                          | B(9)          | B(4)          | 1.792 $\pm$ .009                          |
| Co(1)         | B(16)         | 2.072 $\pm$ .006                          | B(3)          | B(2)          | 1.762 $\pm$ .010                          |
| Co(2)         | C(12)         | 2.065 $\pm$ .005                          | B(3)          | B(4)          | 1.754 $\pm$ .009                          |
| Co(2)         | C(13)         | 2.065 $\pm$ .005                          | B(2)          | B(6)          | 1.759 $\pm$ .009                          |
| Co(2)         | B(17)         | 2.053 $\pm$ .007                          | B(6)          | B(5)          | 1.772 $\pm$ .009                          |
| Co(2)         | B(20)         | 2.072 $\pm$ .006                          | B(5)          | B(4)          | 1.776 $\pm$ .009                          |
| Co(2)         | B(21)         | 2.101 $\pm$ .007                          | B(1)          | B(3)          | 1.759 $\pm$ .010                          |
| Co(2)         | C(22)         | 2.043 $\pm$ .006                          | B(1)          | B(2)          | 1.775 $\pm$ .010                          |
| Co(2)         | BC(23)        | 2.080 $\pm$ .006                          | B(1)          | B(6)          | 1.779 $\pm$ .009                          |
| Co(2)         | B(24)         | 2.087 $\pm$ .007                          | B(1)          | B(5)          | 1.774 $\pm$ .009                          |
| Co(2)         | B(25)         | 2.105 $\pm$ .007                          | B(1)          | B(4)          | 1.754 $\pm$ .010                          |
| Co(2)         | BC(26)        | 2.055 $\pm$ .006                          | C(12)         | C(13)         | 1.561 $\pm$ .007                          |
| C(8)          | C(7)          | 1.613 $\pm$ .008                          | C(12)         | B(16)         | 1.719 $\pm$ .008                          |
| C(8)          | B(9)          | 1.700 $\pm$ .008                          | C(12)         | B(20)         | 1.713 $\pm$ .008                          |
| C(8)          | B(3)          | 1.734 $\pm$ .009                          | C(13)         | B(14)         | 1.695 $\pm$ .008                          |
| C(8)          | B(4)          | 1.688 $\pm$ .009                          | C(13)         | B(17)         | 1.720 $\pm$ .008                          |
| C(7)          | B(11)         | 1.682 $\pm$ .008                          | B(14)         | B(15)         | 1.808 $\pm$ .009                          |

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|        |        |              |        |        |              |
|--------|--------|--------------|--------|--------|--------------|
| C(7)   | B(3)   | 1.727 ± .009 | B(14)  | B(17)  | 1.823 ± .009 |
| B(14)  | B(18)  | 1.783 ± .009 | BC(23) | B(28)  | 1.728 ± .009 |
| B(15)  | B(16)  | 1.791 ± .009 | B(24)  | B(25)  | 1.801 ± .009 |
| B(15)  | B(18)  | 1.791 ± .009 | B(24)  | B(28)  | 1.797 ± .009 |
| B(15)  | B(19)  | 1.768 ± .009 | B(24)  | B(29)  | 1.804 ± .009 |
| B(16)  | B(19)  | 1.783 ± .009 | B(25)  | BC(26) | 1.729 ± .009 |
| B(16)  | B(20)  | 1.824 ± .009 | B(25)  | B(29)  | 1.790 ± .009 |
| B(17)  | B(18)  | 1.790 ± .009 | B(25)  | B(30)  | 1.814 ± .009 |
| B(18)  | B(19)  | 1.786 ± .009 | BC(26) | B(30)  | 1.715 ± .009 |
| B(19)  | B(20)  | 1.786 ± .009 | BC(26) | B(31)  | 1.747 ± .009 |
| B(21)  | B(17)  | 1.792 ± .009 | B(27)  | B(28)  | 1.772 ± .010 |
| B(21)  | B(18)  | 1.800 ± .009 | B(27)  | B(31)  | 1.748 ± .010 |
| B(21)  | B(19)  | 1.783 ± .009 | B(28)  | B(29)  | 1.764 ± .009 |
| B(21)  | B(20)  | 1.786 ± .009 | B(29)  | B(30)  | 1.787 ± .009 |
| C(22)  | BC(23) | 1.643 ± .008 | B(30)  | B(31)  | 1.749 ± .010 |
| C(22)  | BC(26) | 1.636 ± .008 | B(32)  | B(27)  | 1.764 ± .010 |
| C(22)  | B(27)  | 1.702 ± .009 | B(32)  | B(28)  | 1.775 ± .010 |
| C(22)  | B(31)  | 1.706 ± .009 | B(32)  | B(29)  | 1.784 ± .009 |
| BC(23) | B(24)  | 1.729 ± .009 | B(32)  | B(30)  | 1.782 ± .009 |
| BC(23) | B(27)  | 1.752 ± .009 | B(32)  | B(31)  | 1.750 ± .010 |



TABLE VI  
Hydrogen to Carbon or Boron Distances ( $\text{\AA}$ )<sup>a</sup>

| Atoms in Cage I |                 | Atoms in Cage II |                 | Atoms in Cage III |                 |
|-----------------|-----------------|------------------|-----------------|-------------------|-----------------|
| <u>Atom</u>     | <u>Distance</u> | <u>Atom</u>      | <u>Distance</u> | <u>Atom</u>       | <u>Distance</u> |
| C(8)            | 1.02            | C(12)            | 0.94            | C(22)             | 0.99            |
| C(7)            | 0.81            | C(13)            | 0.82            | BC(23)            | 0.97            |
| B(11)           | 1.12            | B(14)            | 1.01            | B(24)             | 1.05            |
| B(10)           | 1.12            | B(15)            | 1.11            | B(25)             | 1.10            |
| B(9)            | 1.10            | B(16)            | 1.14            | BC(26)            | 0.86            |
| B(3)            | 1.11            | B(17)            | 1.12            | B(27)             | 1.12            |
| B(2)            | 1.08            | B(18)            | 1.26            | B(28)             | 1.11            |
| B(6)            | 1.07            | B(19)            | 1.09            | B(29)             | 1.15            |
| B(5)            | 1.13            | B(20)            | 1.14            | B(30)             | 1.12            |
| B(4)            | 1.11            | B(21)            | 1.14            | B(31)             | 0.98            |
| B(1)            | 1.09            |                  |                 | B(32)             | 1.07            |

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(a) Estimated standard deviations on all distances are  $\pm 0.05 \text{ \AA}$ .



TABLE VII

Averages of Equivalent Bond Distances( $\text{\AA}$ )<sup>a, b</sup>

| Type   | Number | Average | $\sigma$ |
|--------|--------|---------|----------|
| Co - C | 7      | 2.06    | 0.01     |
| Co - B | 11     | 2.09    | 0.02     |
| C - B  | 12     | 1.71    | 0.02     |
| B - B  | 48     | 1.78    | 0.02     |
| C - H  | 5      | 0.92    | 0.10     |
| B - H  | 25     | 1.11    | 0.05     |

- (a) An average is given only if there are 5 or more values to include in the calculation of that average.
- (b) The root-mean-square deviation from the average is given by  $\sigma = [\sum \Delta^2 / (n - 1)]^{\frac{1}{2}}$ , where  $\Delta_i$  is the difference between the  $i^{\text{th}}$  bond distance and the average of  $n$  distances of the  $i^{\text{th}}$  type.



-21-  
TABLE VIII

UCRL-18870

## Angles Involving the Cobalts and the Canastide Ion

| <u>Atom 1</u> | <u>Atom 2</u> | <u>Atom 3</u> | <u>Angle(deg)</u> | <u>Atom 1</u> | <u>Atom 2</u> | <u>Atom 3</u> | <u>Angle(deg)</u> |
|---------------|---------------|---------------|-------------------|---------------|---------------|---------------|-------------------|
| C(12)         | Co(1)         | C(13)         | 44.4 ± .2         | Co(1)         | B(16)         | B(15)         | 65.4 ± .3         |
| C(12)         | Co(2)         | C(13)         | 44.4 ± .2         | Co(2)         | B(17)         | B(21)         | 65.9 ± .3         |
| C(12)         | Co(1)         | B(16)         | 49.0 ± .2         | Co(2)         | B(21)         | B(17)         | 63.1 ± .3         |
| C(13)         | Co(1)         | B(14)         | 48.5 ± .2         | Co(2)         | B(21)         | B(20)         | 63.8 ± .3         |
| C(12)         | Co(2)         | B(20)         | 48.9 ± .2         | Co(2)         | B(20)         | B(21)         | 65.5 ± .3         |
| C(13)         | Co(2)         | B(17)         | 49.4 ± .2         | C(12)         | C(13)         | B(14)         | 112.9 ± .4        |
| B(14)         | Co(1)         | B(15)         | 51.4 ± .2         | C(12)         | C(13)         | B(17)         | 112.0 ± .4        |
| B(15)         | Co(1)         | B(16)         | 50.8 ± .2         | C(13)         | C(12)         | B(16)         | 112.0 ± .4        |
| B(17)         | Co(2)         | B(21)         | 51.1 ± .2         | C(13)         | C(12)         | B(20)         | 112.1 ± .4        |
| B(20)         | Co(2)         | B(21)         | 50.7 ± .2         | B(14)         | C(13)         | B(17)         | 64.5 ± .4         |
| Co(1)         | C(12)         | C(13)         | 67.5 ± .2         | B(16)         | C(12)         | B(20)         | 64.2 ± .4         |
| Co(1)         | C(13)         | C(12)         | 68.1 ± .2         | C(13)         | B(14)         | B(17)         | 58.4 ± .3         |
| Co(2)         | C(12)         | C(13)         | 67.8 ± .2         | C(13)         | B(17)         | B(14)         | 57.1 ± .3         |
| Co(2)         | C(13)         | C(12)         | 67.8 ± .2         | C(12)         | B(16)         | B(20)         | 57.7 ± .3         |
| Co(1)         | C(12)         | B(16)         | 65.5 ± .3         | C(12)         | B(20)         | B(16)         | 58.1 ± .3         |
| Co(1)         | C(13)         | B(14)         | 66.1 ± .3         | C(13)         | B(14)         | B(15)         | 104.7 ± .4        |
| Co(2)         | C(12)         | B(20)         | 65.8 ± .3         | C(12)         | B(16)         | B(15)         | 104.7 ± .4        |
| Co(2)         | C(13)         | B(17)         | 64.9 ± .3         | C(13)         | B(17)         | B(21)         | 104.9 ± .4        |
| Co(1)         | B(14)         | C(13)         | 65.5 ± .3         | C(12)         | B(20)         | B(21)         | 105.3 ± .4        |
| Co(1)         | B(16)         | C(12)         | 65.4 ± .3         | B(14)         | B(15)         | B(16)         | 105.5 ± .4        |
| Co(2)         | B(17)         | C(13)         | 65.7 ± .3         | B(17)         | B(21)         | B(20)         | 105.5 ± .4        |
| Co(2)         | B(20)         | C(12)         | 65.3 ± .2         | C(13)         | B(14)         | B(18)         | 103.9 ± .4        |
| Co(1)         | B(14)         | B(15)         | 65.2 ± .3         | C(13)         | B(17)         | B(18)         | 102.5 ± .4        |
| Co(1)         | B(15)         | B(14)         | 63.5 ± .3         | C(12)         | B(16)         | B(19)         | 102.7 ± .4        |
| Co(1)         | B(15)         | B(16)         | 63.8 ± .3         | C(12)         | B(20)         | B(19)         | 102.8 ± .4        |



|       |       |       |            |       |       |       |            |
|-------|-------|-------|------------|-------|-------|-------|------------|
| B(15) | B(14) | B(18) | 59.8 ± .4  | B(16) | B(19) | B(21) | 110.3 ± .4 |
| B(21) | B(17) | B(18) | 60.3 ± .4  | B(18) | B(19) | B(20) | 107.9 ± .5 |
| B(15) | B(16) | B(19) | 59.3 ± .4  | Co(1) | C(13) | B(17) | 125.1 ± .3 |
| B(21) | B(20) | B(19) | 59.9 ± .4  | Co(1) | B(14) | B(17) | 119.1 ± .3 |
| B(14) | B(15) | B(18) | 59.4 ± .4  | Co(1) | B(14) | B(18) | 118.5 ± .4 |
| B(17) | B(21) | B(18) | 59.8 ± .4  | Co(1) | B(15) | B(18) | 116.6 ± .3 |
| B(16) | B(15) | B(19) | 60.1 ± .4  | Co(1) | B(15) | B(19) | 116.9 ± .3 |
| B(20) | B(21) | B(19) | 60.0 ± .4  | Co(1) | B(16) | B(19) | 117.6 ± .3 |
| B(14) | B(17) | B(18) | 59.1 ± .4  | Co(1) | B(16) | B(20) | 118.4 ± .3 |
| B(17) | B(14) | B(18) | 59.5 ± .4  | Co(1) | C(12) | B(20) | 124.3 ± .3 |
| B(16) | B(20) | B(19) | 59.2 ± .4  | Co(2) | C(12) | B(16) | 124.7 ± .3 |
| B(20) | B(16) | B(19) | 59.3 ± .4  | Co(2) | B(20) | B(16) | 118.8 ± .3 |
| B(17) | B(18) | B(15) | 110.0 ± .5 | Co(2) | B(20) | B(19) | 118.0 ± .3 |
| B(17) | B(18) | B(19) | 106.7 ± .5 | Co(2) | B(21) | B(19) | 116.7 ± .3 |
| B(14) | B(18) | B(19) | 106.9 ± .5 | Co(2) | B(21) | B(18) | 116.2 ± .3 |
| B(14) | B(18) | B(21) | 109.3 ± .5 | Co(2) | B(17) | B(18) | 119.1 ± .3 |
| B(15) | B(18) | B(21) | 109.0 ± .5 | Co(2) | B(17) | B(14) | 118.4 ± .3 |
| B(15) | B(19) | B(21) | 110.9 ± .5 | Co(2) | C(13) | B(14) | 124.4 ± .3 |
| B(15) | B(19) | B(20) | 110.3 ± .5 | Co(1) | C(12) | Co(2) | 134.1 ± .2 |
| B(16) | B(19) | B(18) | 108.3 ± .5 | Co(1) | C(13) | Co(2) | 134.7 ± .2 |

and the anion cages are held in their relative orientations by the balance of the attraction of their unlike charges with the repulsive forces of the electrons in the cesium ions and the hydrogen atoms attached to the cages. All of the shortest distances to the oxygen, given in Table IX, correspond to reasonable van der Waals contacts expected for a normal water of hydration.



TABLE IX

Shortest Distances( $\text{\AA}$ ) to Cesiums and Oxygen<sup>a</sup>

| <u>Atom</u> | <u>To O(1)</u> | <u>Atom</u> | <u>To Cs(1)</u> | <u>Atom</u> | <u>To Cs(2)</u> |
|-------------|----------------|-------------|-----------------|-------------|-----------------|
| H(1)        | 2.76           | H(18)       | 2.82            | H(15)       | 2.95            |
| H(24)       | 2.78           | H(32)       | 2.95            | H(4)        | 3.05            |
| H(6)        | 2.78           | H(17)       | 2.99            | H(10)       | 3.08            |
| H(17)       | 2.93           | H(19)       | 3.00            | H(24)       | 3.08            |
| Cs(2)       | 3.029          | H(11)       | 3.12            | H(21)       | 3.10            |
| Cs(1)       | 3.163          | H(6)        | 3.15            | H(6)        | 3.16            |
| H(5)        | 3.21           | H(29)       | 3.23            | H(25)       | 3.25            |
| H(21)       | 3.26           | H(3)        | 3.24            |             |                 |
|             |                | H(30)       | 3.29            |             |                 |

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(a) Estimated standard deviations are all  $\pm 0.05 \text{ \AA}$ . except O(1) to Cs(1) and to Cs(2) which are  $\pm 0.006 \text{ \AA}$ .



Acknowledgment

We wish to thank J. N. Francis and M. F. Hawthorne of the University of California, Riverside, for providing us with the crystals used in this work.

0.20

0.20

Figure Captions

Figure 1: Drawing of the  $(\text{Co}_2\text{B}_{26}\text{C}_6\text{H}_{32})^{-2}$  anion (hydrogen atoms not shown).

Figure 2: Framework drawing of the projection of the unit cell contents down the a axis.

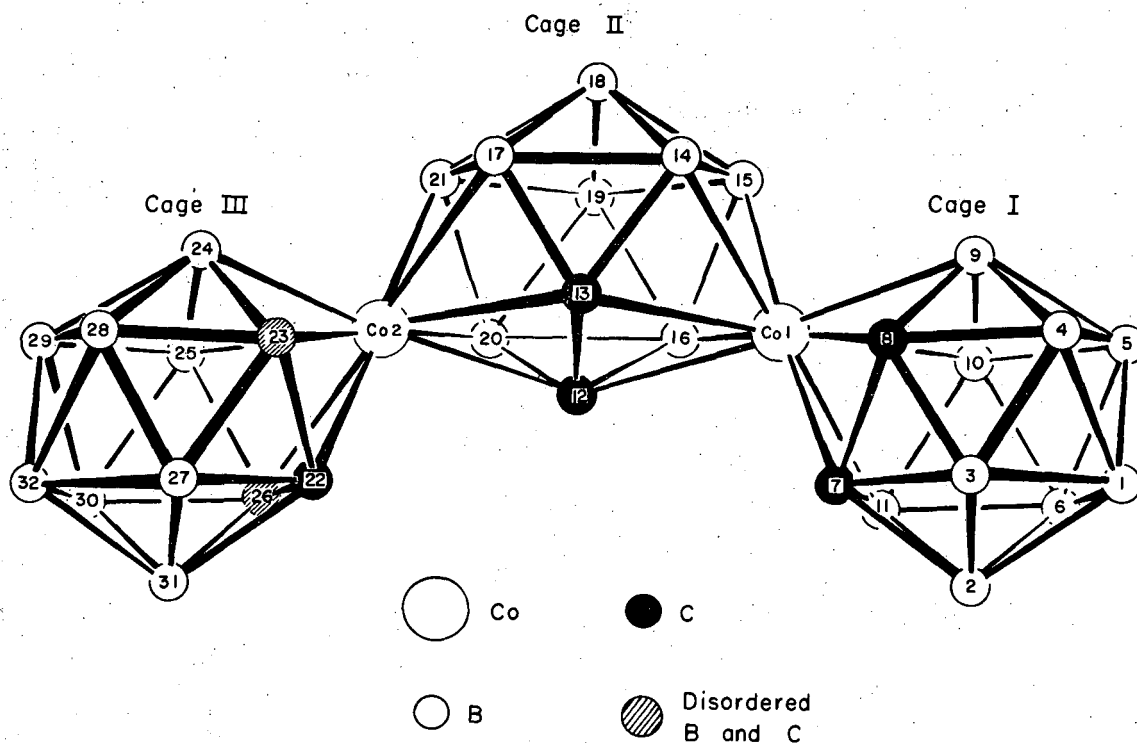
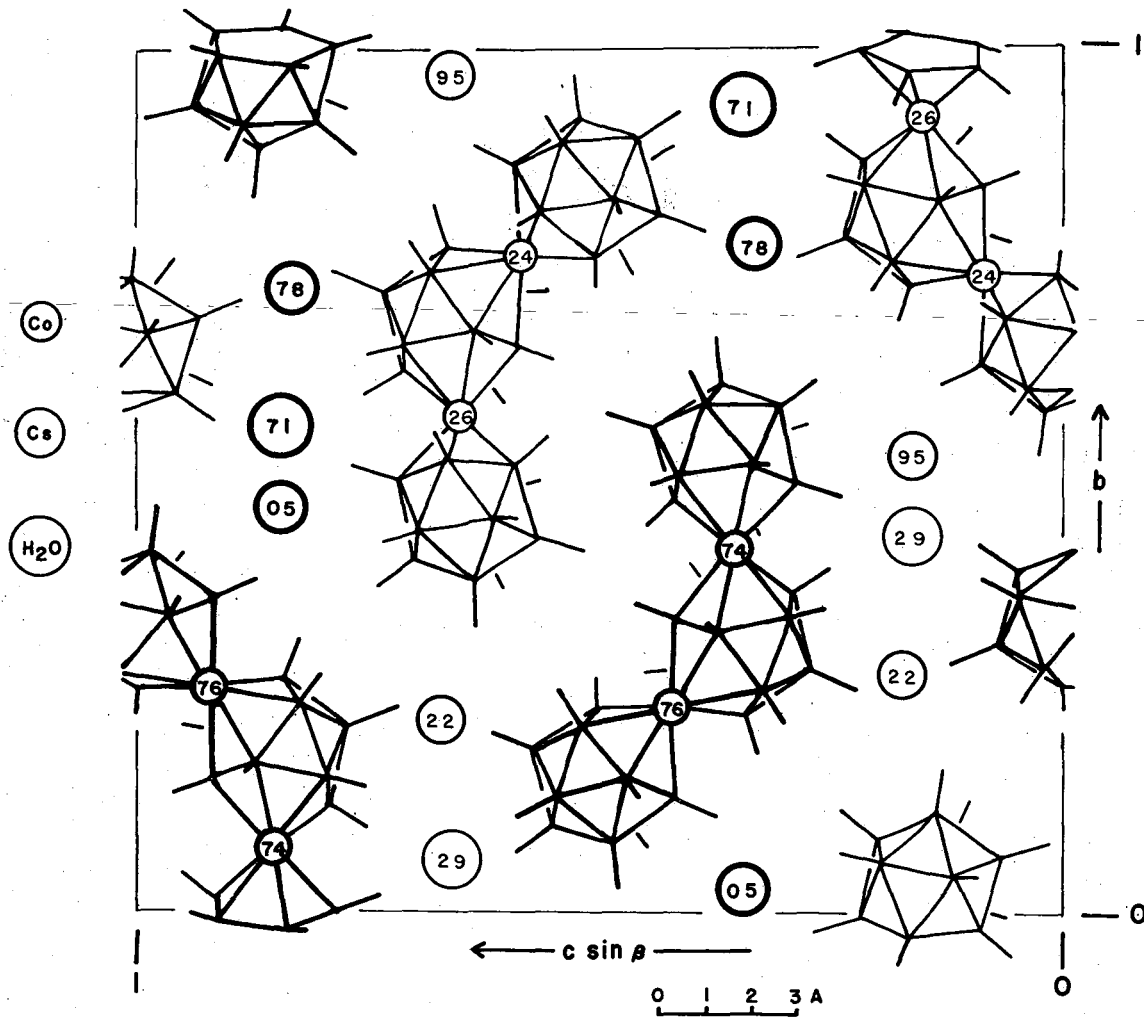


Figure 1

XBL6810-6853





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