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Preparation and Structure of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$, a New Tricobalt Enneacarbonyl Carbon Cluster

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Crystals of triethylamine–(tricobalt enneacarbonyl carbon)oxyborane, $Co_3(CO)_{10}BH_2N(C_2H_5)_3$, have been prepared by the reaction of triethylamine–borane with dicobalt octacarbonyl in benzene. The molecular and crystal structures of this compound have been determined from three-dimensional X-ray counter data. Crystals of the dark red, air-stable compound are monoclinic with cell dimensions of $a = 11.75 \pm 0.01$ Å, $b = 11.29 \pm 0.01$ Å, $c = 8.81 \pm 0.01$ Å, and $\beta = 93.31 \pm 0.05°$. The space group is $P2_1/m$ with two formula species per cell imposing C_s point symmetry on the individual molecules. The structure has been refined by least squares to a conventional R of 0.087. The basic structural feature consists of a triangular cluster of cobalt atoms each coordinated to three carbonyl groups and an apical triply bridging CO group with a C–O bond distance of 1.28 Å. The infrared and mass spectral data are given for this material, and the structure is compared with the related $Co_3(CO)_9CCH_3$ and $Co_3(CO)_9S$ molecular complexes.

Introduction

Many complexes of the type $Co_3(CO)_9CY$ ($Y = H$, CH_3, Cl, etc.) have been described[2] which are universally distinguished by high air stability and striking ease of formation. The salient structural features of this class of compounds have been elucidated by Sutton and Dahl[2] from an X-ray structure investigation on the C-methyl-substituted compound, $Co_3(CO)_9CCH_3$. They consist of a tetrahedral Co_3C cluster arrangement with a triangular array of three basal cobalt atoms and an apical carbon atom. A very similar geometrical arrangement was found in $SCo_3(CO)_9$, structurally characterized by Wei and Dahl.[3] Presumably, heteroatoms other than carbon and sulfur might also interact with a $Co_3(CO)_9$ entity to give an $XCo_3(CO)_9$ cluster. Some preliminary reports in the literature claiming examples for X = Si[4] and X = Sn[5] ostensibly support this assumption.[6] The authenticity of the reported compound $C_4H_9SnCo_3(CO)_9$ has already been disputed.[7]

While exploring the possibilities of incorporating a boron atom (or a BH group) into a metal cluster fragment such as $Co_3(CO)_9$, we synthesized the compound $Co_3(CO)_{10}BH_2N(C_2H_5)_3$. This article describes the properties and structural characterization of this compound.

Synthesis and Properties

Trialkylamine–boranes react with dicobalt octacarbonyl in hydrocarbon solvents to give compounds of composition $CO_3(CO)_{10}BH_2NR_3$ ($R = CH_3, C_2H_5$). These complexes are crystalline solids, black in reflected light and dark red in transmitted light, which are stable to air at room temperature and can be heated to about 80° *in vacuo* without gross decomposition. Having established the composition from analytical and molecular weight data, we expected the presumed molecular asymmetry arising from a cobalt to carbonyl ratio of 3:10 to be reflected in the CO stretching region of the infrared spectra. This, however, was not the case. The observed spectra under high resolution were virtually identical with those of $Co_3(CO)_9CCl$. In particular, no evidence indicating a bridging CO group in $Co_3(CO)_{10}BH_2NR_3$ was found in the 2000–1500-cm^{-1} region of the spectrum. There were two bands of moderate intensity at 2415 and 2365 cm^{-1} suggestive of a BH_2 group. Evidence for the presence of a BH_2 group in the molecule was also obtained from a broadened triplet in the B^{11} nmr spectrum.

Before the X-ray investigation was begun, we made an effort to derive structural information from the mass spectrometric fragmentation pattern of $Co_3(CO)_{10}BH_2N(CH_3)_3$. Not surprisingly, the observed spectra are highly complex, quite unlike those obtained for $Co_3(CO)_9CCl$.[8] While we were reasonably certain about the consecutive loss of nine CO groups from the parent ion, the spectra did not permit definitive conclusions about the mode of attachment of the tenth carbonyl group. However, it was apparent from a consideration of all of the spectroscopic data that the structure of $Co_3(CO)_{10}BH_2NR_3$ had to be highly symmetrical and that a structural fragment $Co_3(CO)_9$, analogous to the one established for $SCo_3(CO)_9$[3] and $Co_3(CO)_9CCH_3$,[2] was likely. A necessary corollary of this assumption was to postulate one unique CO group triply bridging three cobalt atoms. There exists sufficient precedence in metal cluster chemistry to render such a hypothesis justifiable (*vide infra*). Furthermore, it is well known that triply bridging CO groups do not always lead to easily identifiable infrared absorptions.[9]

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[1] To whom inquiries should be addressed.

[2] For a complete listing of references, see P. W. Sutton and L. F. Dahl, *J. Am. Chem. Soc.*, **89**, 261 (1967).

[3] C. H. Wei and L. F. Dahl, *Inorg. Chem.*, **6**, 1229 (1967).

[4] S. F. A. Kettle and I. Khan, *Proc. Chem. Soc.*, 82 (1962); *J. Organometal. Chem.* (Amsterdam), **5**, 588 (1966).

[5] S. D. Ibewke and M. F. Newland, *Chem. Commun.*, 114 (1965).

[6] We have been unable to reproduce the synthesis of $[SiCo_3(CO)_9]_2$ reported by Kettle and Khan.[4] Under the reaction conditions described by these authors, *i.e.*, elution of $Co_2(CO)_8$ *with* CCl_4 from an alumina column, the known cluster $Co_3(CO)_9CCl$ is invariably formed. The incomplete analytical data cited by Kettle and Khan as supporting $[SiCo_3(CO)_9]_2$ are in approximate agreement with the ones required for $Co_3(CO)_9CCl$, and the published ir spectrum is in explicit agreement with that of authentic $Co_3(CO)_9CCl$. We therefore suggest that these authors have misidentified their reaction product.

[7] D. F. Patmore and W. A. G. Graham, *Inorg. Chem.*, **5**, 2222 (1966).

[8] R. B. King, *J. Am. Chem. Soc.*, **88**, 2075 (1966).

[9] An example for this is the $Fe_4(CO)_{13}^{2-}$ anion; see R. F. Doedens and L. F. Dahl, *ibid.*, **88**, 4847 (1966).

Since the chemical and spectroscopic evidence gave only very fragmentary information about the molecular structure, an X-ray investigation was undertaken.

Crystal Data and Structure Determination

Crystals of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$ are monoclinic with cell parameters of $a = 11.75 \pm 0.01$, $b = 11.29 \pm 0.01$, $c = 8.81 \pm 0.01$ Å, and $\beta = 93.31 \pm 0.05°$. These parameters were obtained from a least-squares refinement of powder diffraction data recorded on a Hägg–Guinier camera using a KCl internal standard ($a_{25°} = 6.2931$ Å). The observed (by flotation) and calculated densities for two formula units per cell are 1.63 g/cm^3. The only systematic absence observed on Weissenberg and precession films was $\{0k0\}$, $k = 2n + 1$, indicating that the space group is either $P2_1/m$ or $P2_1$. Our successful refinement in $P2_1/m$ indicates that this is the correct space group. The space group $P2_1/m$ with $Z = 2$ imposes C_s point symmetry on the individual molecules. Atoms were placed in the general positions[10] $\pm(x, y, z; x, {}^1/_2 - y, z)$ and the special positions $\pm(x, {}^1/_4, z)$.

A four-circle diffractometer was used to measure intensities from a crystal of dimensions 0.08 × 0.14 × 0.29 mm mounted with the b axis coincident with the φ axis of the diffractometer. The data were measured using Zr-filtered Mo radiation (λ 0.7107 Å) and the θ–2θ scan technique with a symmetric scan range of 1.5°, a scan rate of 1°/min, and a takeoff angle of 3°. Individual backgrounds of 20 sec were recorded before and after each scan. A total of 1815 reflections was measured including $\{hk0\}$ and $\{\bar{h}k0\}$ data which were averaged. Data were measured out to 45° in 2θ; there was only limited scattering beyond this.

The data were corrected for Lorentz and polarization effects in the usual way and for absorption using Prewitt's program[11] ACACA. For Mo Kα the linear absorption coefficient is 22.5 cm^{-1}. The crystal was described by six plane faces for the absorption correction. The minimum and maximum calculated transmission factors were 0.69 and 0.85, respectively. The structure factor errors were assigned according to a scheme reported earlier.[12]

An unsharpened Patterson function was calculated from which the Co atom positions were located. The Co positions were used to calculate a superposition function from which all of the carbonyl atom positions were located. After refining the scale factor, the R was 0.372 based on a model containing three Co atoms and ten carbonyl groups, one of which had been misplaced. An electron density difference map showed all of the remaining atom positions. In this map the boron and ethyl group atoms appeared at one-half of their expected heights. The positions of the boron and ethyl groups were such that these atoms had to be placed in disordered positions with respect to the mirror plane in the space group $P2_1/m$. The R was 0.126 after two cycles of least squares with all atoms except boron included in the structure factor calculations. The B atom position was confirmed on an electron density difference map, and, after two more cycles of least-squares including all nonhydrogen atoms, the R was 0.118. The refinement was continued by allowing all ordered atoms to assume anisotropic temperature factors keeping isotropic temperature factors for the disordered atoms (B, C_7, C_8, C_9, C_{10}, C_{11}, C_{12}). The refinement was stopped after four more cycles of least squares. The final R factors for the 1489 observed reflections were

$$R = \frac{\sum||F_o| - |F_c||}{\sum|F_o|} = 0.087$$

$$wR = \left[\frac{\sum w||F_o| - |F_c||^2}{\sum w|F_o|^2}\right]^{1/2} = 0.076$$

The corresponding values for all reflections were $R = 0.095$ and $wR = 0.077$. The maximum and minimum peaks in the final electron density difference map were 0.8 and −0.9 e$^-$/Å^3 consistent with a good refinement. The final "error of fit" was

$$\left[\frac{\sum w||F_o| - |F_c||^2}{m - n}\right]^{1/2} = 2.00$$

The disorder was handled in the refinement by placing atoms B and C_7 through C_{12} in general positions with atom multiplicities of 0.5. The function minimized in least-squares was $\Sigma w||F_o| - |F_c||^2$. The atom form factors used were those of the neutral atoms.[13] The anomalous scattering effects of Co were included using the values reported by Templeton.[14] Various local computer programs were used including the Fourier program FOUR and Prewitt's least-squares program[11] SFLS5.

We did not make any serious effort to refine this structure in the noncentric, polar space group $P2_1$. If the mirror plane were not present it would not be necessary for the $-BH_2N(C_2H_5)_3$ portion of this structure to be disordered. A refinement of the scale factor with the molecule in one of the acentric orientations gave a wR of 0.078 and a corresponding increase in the "error of fit." We feel this, coupled with the success of our refinement, indicates that $P2_1/m$ is the correct space group. As a matter of curiosity we did attempt one cycle of refinement of positional parameters in the space group $P2_1$ with the result that all of the meaningful shifts were consistent within experimental error with the presence of the mirror plane. There were some meaningless shifts resulting from the usual singularity problems associated with the refinement of atoms on or in close proximity to special positions.

The final atom parameters are given in Table I where the numbering system corresponds to that shown in Figure 1. A list of observed and calculated structure factors is given in Table II. The interatomic distances

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[10] "International Tables for X-Ray Crystallography," Vol. I, The Kynoch Press, Birmingham, England, 1965, p 93.

[11] C. T. Prewitt, local unpublished computer program, 1968.

[12] L. J. Guggenberger, *Inorg. Chem.*, **7**, 2260 (1968).

[13] H. P. Hansen, F. Herman, J. D. Lea, and S. Skillman, *Acta Cryst.*, **17**, 1040 (1964).

[14] D. H. Templeton, "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Brimingham, England, 1962, p 215.

TABLE I

POSITIONAL AND THERMAL PARAMETERS[a]

Atom	x	y	z	β_{11} or B	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Co_1	0.1330 (1)	0.1394 (1)	0.2521 (1)	0.0077 (1)	0.0082 (1)	0.0133 (2)	−0.0007 (1)	0.0012 (1)	0.0005 (1)
Co_2	0.2034 (1)	0.25	0.0314 (2)	0.0078 (2)	0.0097 (2)	0.0114 (3)	0.0	0.0021 (2)	0.0
C_1	0.1326 (11)	0.1186 (10)	0.4518 (15)	0.0194 (15)	0.0110 (13)	0.0205 (22)	−0.0002 (11)	0.0045 (15)	0.0020 (14)
O_1	0.1338 (12)	0.1016 (9)	0.5786 (10)	0.0448 (22)	0.0213 (14)	0.0139 (14)	−0.0032 (14)	0.0074 (15)	0.0018 (12)
C_2	−0.0134 (10)	0.1096 (9)	0.1789 (12)	0.0109 (11)	0.0097 (11)	0.0203 (20)	−0.0006 (9)	0.0018 (12)	−0.0013 (12)
O_2	−0.1031 (7)	0.0915 (8)	0.1349 (11)	0.0089 (7)	0.0177 (11)	0.0397 (22)	−0.0033 (8)	−0.0027 (10)	−0.0000 (12)
C_3	0.2014 (9)	0.0082 (11)	0.2140 (12)	0.0106 (11)	0.0104 (12)	0.0179 (18)	0.0001 (9)	0.0008 (11)	0.0039 (13)
O_3	0.2503 (7)	−0.0788 (8)	0.1944 (9)	0.0171 (10)	0.0127 (9)	0.0248 (16)	0.0034 (8)	0.0004 (10)	0.0029 (10)
C_4	0.0718 (15)	0.25	−0.0912 (17)	0.0128 (18)	0.0200 (25)	0.0115 (24)	0.0	0.0008 (17)	0.0
O_4	−0.0080 (12)	0.25	−0.1674 (14)	0.0146 (15)	0.0398 (29)	0.0172 (22)	0.0	−0.0031 (14)	0.0
C_5	0.2875 (10)	0.1344 (10)	−0.0436 (12)	0.0130 (12)	0.0112 (12)	0.0179 (18)	−0.0021 (10)	0.0056 (12)	−0.0005 (13)
O_5	0.3427 (8)	0.0638 (8)	−0.0933 (10)	0.0207 (11)	0.0152 (11)	0.0311 (18)	0.0036 (9)	0.0123 (11)	−0.0050 (12)
C_6	0.2560 (11)	0.25	0.2414 (14)	0.0069 (12)	0.0074 (13)	0.0146 (21)	0.0	0.0008 (13)	0.0
O_6	0.3547 (8)	0.25	0.3106 (12)	0.0072 (8)	0.0143 (12)	0.0238 (19)	0.0	−0.0017 (11)	0.0
N	0.5563 (8)	0.25	0.3786 (11)	3.88 (21)					
B	0.4412 (18)	0.1697 (20)	0.3317 (24)	4.25 (45)					
C_7	0.5421 (14)	0.3293 (16)	0.5126 (20)	4.69 (40)					
C_8	0.5120 (14)	0.2815 (17)	0.6571 (19)	5.53 (49)					
C_9	0.5849 (15)	0.3207 (17)	0.2453 (21)	4.88 (40)					
C_{10}	0.6150 (13)	0.2645 (42)	0.1015 (18)	6.45 (55)					
C_{11}	0.6485 (15)	0.1543 (17)	0.4153 (21)	5.05 (41)					
C_{12}	0.7711 (13)	0.2106 (14)	0.4619 (18)	4.71 (43)					

[a] The anisotropic thermal ellipsoid is of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. The standard deviations of the least significant digits are given in parentheses.

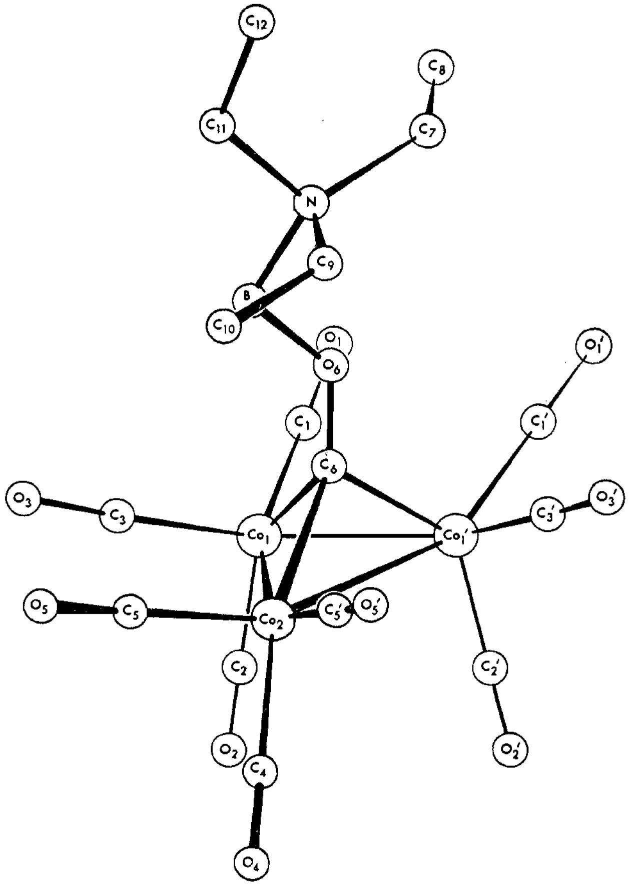

Figure 1.—Molecular configuration of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$.

and angles are given in Tables III and IV, respectively. The errors in the distances were calculated using the variances, but not the covariances, in the positional and cell parameters. The distances were not corrected for thermal motion.

Description of the Structure

The crystal structure of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$ consists of the packing of discrete units such as the one shown in Figure 1. Atoms identified with a prime are related to the unprimed atoms by the molecular mirror plane. Figure 2 shows the anisotropic thermal ellipsoids[15] illustrating a rather general feature that terminal carbonyl groups have relatively large temperature factors. Atoms lying on the molecular mirror plane are N, O_6, C_6, Co_2, C_4, and O_4. The crystal structure is disordered in the sense that half of the units in the crystal have the N tetrahedron inverted with respect to the mirror plane. The operation of inverting the N tetrahedron gives an energetically equivalent unit with the B and three C_2H_5 groups on the opposite side of the mirror plane. All intermolecular distances were calculated, but no abnormal contacts were found.

The basic structural feature of this molecule is the Co_3C tetrahedron where each Co atom assumes a closed-shell electronic configuration by forming two Co–Co bonds, three Co–CO bonds, and one Co–C bond. The structure of the $Co_3C(CO)_9$ core is depicted in Figure 3 where it is seen that this part of the structure has the idealized C_{3v} point symmetry. This portion of the structure is virtually identical with the structures found by Dahl and coworkers for $Co_3(CO)_9CCH_3$[2] and $Co_3(CO)_9S$.[3] Other structures with a carbonyl carbon atom bridging three metal atoms are $(C_5H_5)_3Ni_3(CO)_2$,[16] $Rh_6(CO)_{16}$,[17] and $Fe_4(CO)_{13}^{2-}$.[9]

All of the details concerning the stereochemistry of

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(15) C. K. Johnson, Report No. 3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

(16) A. A. Hook and O. S. Mills in "Advances in the Chemistry of Coordination Compounds," S. Kirschner, Ed., The Macmillan Co., New York, N. Y., 1961, p 640.

(17) E. R. Corey, L. F. Dahl, and W. Beck, *J. Am. Chem. Sco.*, **85**, 1702 (1963).

TABLE II: OBSERVED AND CALCULATED STRUCTURE FACTORS (×10) FOR $Co_3(CO)_{10}BH_2N(C_2H_5)_3$

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  K,L= 0, 0
  H  FOB  FCA
  1  968 1025
  2  648  676
  3  965  948
  4  646  654
  5   26   45*
  6  169  177
  7  221  238
  8  363  387
  9  347  367
 10   10   11*
 11  117  114
 12  192  207

  K,L= 1, 0
  H  FOB  FCA
  1 1196 1258
  2 1265 1199
  3   53   49
  4  188  182
  5  684  714
  6  197  180
  7   56   27
  8   77   17
  9   89   89
 10  117  154
 11   87   92
 12   49   35

  K,L= 2, 0
  H  FOB  FCA
  0 1006  971
  1  221  194
  2  125  115
  3  324  305
  4  130  116
  5  346  343
  6   23   29*
  7   81   95
  8   59   27
  9  113  100
 10   41   26*
 11   55   11
 12   78   90

  K,L= 3, 0
  H  FOB  FCA
  1   13    6*
  2   15   30*
  3  617  623
  4   13   17*
  5  457  495
  6  558  565
  7   11   15*
  8  257  266
  9  214  238
 10   82   92
 11   63   61
 12  137  122

  K,L= 4, 0
  H  FOB  FCA
  0  566  552
  1  584  725
  2  253  226
  3  299  288
  4  825  834
  5  711  731
  6  102   95
  7  636  690
  8  440  465
  9   67   42
 10  169  176
 11  166  173

  K,L= 5, 0
  H  FOB  FCA
  1  138  151
  2  606  599
  3  454  482
  4   37   47*
  5  210  242
  6  599  738
  7  268  287
  8  249  275
  9  250  283
 10  120  145
 11   47   13*

  K,L= 6, 0
  H  FOB  FCA
  0  404  408
  1   37   30*
  2  220  216
  3  132  136
  4  277  288
  5  238  237
  6   30   54*
  7  280  294
  8  105  100
  9   28   23*
 10  115   95

  K,L= 7, 0
  H  FOB  FCA
  1  312  332
  2  182  192
  3  116  106
  4  165  162
  5  109  101
  6  103   87
  7   46   33*
  8   83   30
  9   78   25
 10   21    7*

  K,L= 8, 0
  H  FOB  FCA
  0  609  608
  1  332  345
  2  156  176
  3  359  359
  4  167  179
  5   56   50
  6  112  124
  7   76   90
  8   71   83
  9   26   64*

  K,L= 9, 0
  H  FOB  FCA
  1  462  490
  2  411  428
  3   78    9
  4   86  124
  5  121  140
  6  126  122
  7   98   92
  8   40    9*

  K,L=10, 0
  H  FOB  FCA
  0  311  308
  1   27   58*
  2  114  112
  3  137  150
  4  142  143
  5   71   36
  6   70   32
  7   65   54

  K,L=11, 0
  H  FOd  FCA
  1   46   93*
  2  104   70
  3   55   37*
  4   30   34*
  5   44    9*

  K,L=12, 0
  H, FOB  FCA
  0   82   43
  1   73   66
  2   31   38*

  K,L= 0, 1
  H  FOB  FCA
-12  164  155
-10  477  448
 -9  414  408
 -8   35   12
 -7  433  424
 -6  408  408
 -5  135  135
 -4  192  185
 -3  187  154
 -2  475  434
 -1 1319 1334
  0  557  625
  1  566  607
  2 1590 1552
  3  615  561
  4  787  722
  5  669  702
  6  391  375
  7   72   56
  8  216  214
  9  334  329
 10   76   41
 11   44    7
 12   10   35*

  K,L= 1, 1
  H  FOB  FCA
-12  153  138
-11  225  204
-10   76  112
 -9  344  337
 -8  505  477
 -7  104  105
 -6  244  221
 -5  353  338
 -4  545  480
 -3   56   51
 -2   83   98
 -1  232  190
  0 1260 1210
  1  884  821
  2  252  245
  3 1075  989
  4 1202 1135
  5  118  136
  6  346  289
  7  513  531
  8   88   73
  9   58   24
 10  112  107
 11   25   40*
 12  110  101

  K,L= 2, 1
  H  FOB  FCA
-12   84   57
-11    0   13*
-10  216  201
 -9  102   93
 -8  174  167
 -7  156  152
 -6   62   77
 -5  103  119
 -4  297  294
 -3  115  110
 -2  139  146
 -1  604  593
  0  101   91
  1  374  353
  2  396  380
  3  431  421
  4  106   83
  5  600  579
  6  137  135
  7  111  116
  8   74   58
  9  134  121
 10  127  114
 11   64   26
 12   55   75

  K,L= 3, 1
  H  FOB  FCA
-12   93   14
-11    0    3*
-10  101   86
 -9   85   76
 -8    0   22*
 -7  331  297
 -6  319  296
 -5   58   35
 -4  854  796
 -3  421  374
 -2 1561 1492
 -1 1263 1362
  0  127   87
  1  184  196
  2  277  272
  3  285  282
  4  105   83
  5  462  430
  6   99   83
  7  275  252
  8  346  343
  9  173  177
 10    0   20*
 11  175  179
 12   53   71

  K,L= 4, 1
  H  FOB  FCA
-11   72   10
-10   99   92
 -9  189  197
 -8  302  294
 -7   59   17
 -6  664  631
 -5  582  671
 -4   65   55
 -3  826  817
 -2  782  782
 -1   94   77
  0  475  420
  1  446  466
  2  352  313
  3   47   58
  4  282  231
  5   51   58
  6  427  435
  7  369  358
  8   64   64
  9  243  265
 10  246  247
 11   55   22

  K,L= 5, 1
  H  FOB  FCA
-11  134  117
-10   91   32
 -9   68   29
 -8  159  168
 -7  500  483
 -6  356  339
 -5  286  279
 -4  794  752
 -3  414  430
 -2  345  303
 -1  754  726
  0  698  545
  1  157  155
  2  204  198
  3  284  265
  4  182  171
  5  297  280
  6   25   36*
  7  268  261
  8  396  384
  9  198  172
 10  140  145
 11  235  221

  K,L= 6, 1
  H  FOB  FCA
-10   65   21
 -9   92   21
 -8   92   73
 -7  143  145
 -6  273  248
 -5  413  394
 -4  130   97
 -3  466  451
 -2  563  534
 -1   85   83
  0  415  405
  1  202  203
  2  204  224
  3  309  300
  4   18    5*
  5   50   38
  6  218  219
  7  187  183
  8   73   47
  9  109  120
 10  176  169

  K,L= 7, 1
  H  FOB  FCA
-10    0   29*
 -9  147  135
 -8  127  115
 -7   79   34
 -6   53   71
 -5  116  105
 -4  166  135
 -3  177  173
 -2   46   56
 -1   26   18*
  0  177  187
  1  170  172
  2   46   22
  3  196  185
  4  239  223
  5   65   73
  6  213  192
  7  120  116
  8   44   18
  9  110   94
 10   63   16

  K,L= 8, 1
  H  FOB  FCA
 -9  113  110
 -8   45   43
 -7  143  137
 -6  110  102
 -5   96  103
 -4   90   53
 -3   65    4
 -2  157  152
 -1  373  359
  0  149  143
  1  240  241
  2  410  415
  3  219  214
  4  235  224
  5  377  388
  6  108  113
  7   74   15
  8   75   88
  9   60   57

  K,L= 9, 1
  H  FOB  FCA
 -8  208  199
 -7   87   92
 -6   81   13
 -5  158  128
 -4  122  133
 -3  145  156
 -2  221  200
 -1    0    6*
  0  430  429
  1  349  330
  2    0   50*
  3  321  325
  4  315  290
  5   78   29
  6  114  113
  7  178  162
  8  125  105

  K,L=10, 1
  H  FOB  FCA
 -7  162  134
 -5  102   56
 -4   39   29
 -3   49   37
 -2   87   81
 -1  181  193
  0  127  104
  1  113  113
  2  319  320
  3  164  172
  4   65   48
  5  169  176
  6  143  129

  K,L=11, 1
  H  FOB  FCA
 -5   32   21*
 -4   86   15
 -3   84   39
 -2   53   41
 -1  103  108
  0   39   49
  1   39   70
  2   70   42
  3   12   54*
  4  138  107
  5   10   30*

  K,L=12, 1
  H  FOB  FCA
 -1   87   39
  0   72   55
  1   71   57

  K,L= 0, 2
  H  FOB  FCA
-12    0   19*
-11  214  200
-10  256  253
 -9  129  117
 -8  551  566
 -7  553  556
 -6  112  116
 -5  582  561
 -4   61   37
 -3  323  295
 -2   54   36
 -1  541  527
  0  582  588
  1  540  532
  2  501  469
  3  828  801
  4  955  965
  5  146  141
  6  262  247
  7  637  652
  8  491  507
  9   40   11
 10  169  174
 11  179  179
 12    0   32*

  K,L= 1, 2
  H  FOB  FCA
-12  132  109
-11  116  114
-10  278  287
 -9  396  398
 -8  150  152
 -7  331  341
 -6  579  573
 -5  402  374
 -4   99  110
 -3  151  153
 -2  259  219
 -1  234  230
  0  131  155
  1  171  205
  2  165  186
  3  881  854
  4   98  113
  5  462  471
  6  528  551
  7  143  131
  8  116  113
  9  392  407
 10   47   19
 11   36   59
 12  102   53

  K,L= 2, 2
  H  FOB  FCA
-12   51   45
-11   60   65
-10  212  228
 -9   57    2
 -8  319  322
 -7  238  229
 -6   52   18
 -5  423  412
 -4  352  353
 -3  421  395
 -2  480  459
 -1  218  216
  0  303  286
  1   67   59
  2  528  511
  3  305  316
  4  434  425
  5  322  345
  6   56   56
  7  314  336
  8  148  156
  9  109  112
 10   84   89
 11   63    3
 12   87   87

  K,L= 3, 2
  H  FOB  FCA
-12    0   10*
-11  157  161
-10   75   93
 -9    0    1*
 -8   47   20
 -7  211  226
 -6    0   45*
 -5  312  313
 -4  541  536
 -3  105  105
 -2 1060 1073
 -1  529  522
  0   34   45
  1  369  336
  2   59   42
  3  105   80
  4  265  271
  5  131  134
  6   52   62
  7   70   12
  8   31   46
  9  114  133
 10   92  115
 11   51   52

  K,L= 4, 2
  H  FOB  FCA
-11   89  114
-10   79   56
 -9   85   99
 -8  155  151
 -7  231  224
 -6   94   58
 -5  177  179
 -4  516  540
 -3  666  671
 -2   47   50
 -1  670  705
  0  625  658
  1  374  376
  2  312  323
  3  560  570
  4  167  197
  5  109   57
  6  143  143
  7  133  129
  8  160  175
  9  188  195
 10   28   58*
 11  229  218

  K,L= 5, 2
  H  FOB  FCA
-11  102  108
-10  150  112
 -9  121  146
 -8   79   29
 -7   58   20
 -6  150  170
 -5  369  381
 -4  305  303
 -3  382  412
 -2  769  789
 -1  561  564
  0   19    7*
  1  594  638
  2  514  554
  3   52    6
  4  306  314
  5  265  268
  6  178  203
  7   98   83
  8   10   40*
  9   91  100
 10  203  219
 11  114   84

  K,L= 6, 2
  H  FOB  FCA
-10  120  109
 -9   70   64
 -8   54    4
 -7   45   37
 -6   68   38
 -5  276  284
 -4  487  506
 -3  589  624
 -2   76   87
 -1  300  316
  0  396  431
  1  222  235
  2  193  218
  3  286  283
  4   57   62
  5   39   39
  6   95  105
  7   65   31
  8   58   55
  9  122   98
 10   78   53

  K,L= 7, 2
  H  FOB  FCA
-10    0   43*
 -9  122  129
 -8   97  116
 -7  117  122
 -6   88   98
 -5   36   33
 -4  124  153
 -3   98   77
 -2  167  196
 -1  111   75
  0  135  133
  1  252  260
  2  114  120
  3  140  139
  4  135  145
  5  119  126
  6  128  148
  7   82   66
  8   84   92
  9   90   63

  K,L= 8, 2
  H  FOB  FCA
 -9  114   67
 -8  248  252
 -7  120  122
 -6   35   51
 -5  174  173
 -4  112  143
 -3  106   77
 -2   94  101
 -1   67   82
  0  104  121
  1  186  188
  2   80   87
  3   80   88
  4  310  323
  5  145  127
  6  111  110
  7  200  209
  8  134  118

  K,L= 9, 2
  H  FOB  FCA
 -8  117   92
 -7  191  179
 -6  228  238
 -5   73  118
 -4   91   73
 -3  207  199
 -2  110  114
 -1  114  147
  0    0    8*
  1  142  146
  2  253  280
  3  205  229
  4   56   32
  5  256  258
  6  219  225
  7   79   83

  K,L=10, 2
  H  FOB  FCA
 -6   25   12*
 -5  206  188
 -4  141  159
 -3  108   91
 -2    0   29*
 -1   75   92
  0   89   65
  1  111   98
  2   68   59
  3   32   60
  4  208  213
  5  134  145
  6   45    4

  K,L=11, 2
  H  FOB  FCA
 -4   31   73*
 -3   94   55
 -2    0   53*
 -1    0    9*
  0   79   40
  1    0   62*
  2   18   38*
  3  105  133
  4   23   59*

  K,L= 0, 3
  H  FOB  FCA
-12    0   13*
-11   39   32
-10  113  115
 -9  399  407
 -8  307  308
 -7   91  102
 -6  668  675
 -5  659  672
 -4  277  273
 -3  778  770
 -2  830  835
 -1 1690 1709
  0  218  211
  1  714  702
  2  348  354
  3  255  253
  4   73   97
  5  338  365
  6  425  418
  7  392  425
  8  115   99
  9  303  304
 10  149  160
 11   52   26

  K,L= 1, 3
  H  FOB  FCA
-12   51   15
-11   15   43*
-10  109  112
 -9   67   93
 -8  195  189
 -7  458  485
 -6  342  341
 -5  372  368
 -4  730  717
 -3  407  390
 -2  191  198
 -1 1019  993
  0  525  552
  1  205  207
  2    0   22*
  3    0    8*
  4  315  313
  5  442  464
  6  132  127
  7  327  331
  8  372  379
  9   22   30*
 10  153  170
 11  203  199

  K,L= 2, 3
  H  FOB  FCA
-12   43   38
-11  107   98
-10   98   92
 -9  118   87
 -8  236  235
 -7   81   58
 -6  276  277
 -5  170  160
 -4  111  110
 -3  608  576
 -2  291  295
 -1  161  142
  0  634  611
  1  218  212
  2  406  401
  3   75   54
  4  187  207
  5  121  111
  6  189  169
  7  275  293
  8   71   17
  9  213  213
 10   90   72
 11   70   34

  K,L= 3, 3
  H  FOB  FCA
-11  129  143
-10   15    9*
 -9  215  194
 -8  167  146
 -7    0   25*
 -6   64   60
 -5  292  288
 -4   77   50
 -3  197  184
 -2  241  252
 -1   64   35
  0  648  643
  1  291  306
  2  123  127
  3  435  450
  4  454  463
  5   32   43
  6  443  455
  7  230  219
  8   69   62
  9   30    2
 10    0   19*
 11   58   39

  K,L= 4, 3
  H  FOB  FCA
-11  131  119
-10  220  229
 -9  264  255
 -8   50   35
 -7  226  246
 -6  268  254
 -5  275  281
 -4  121  105
 -3    0    5*
 -2  665  635
 -1  600  611
  0  470  426
  1  482  465
  2  741  717
  3  345  356
  4  124  130
  5  510  524
  6  314  338
  7    0   32*
  8   80   79
  9  100   57
 10   87  104
 11   91   52

  K,L= 5, 3
  H  FOB  FCA
-11  165  188
-10   21    3*
 -9  171  160
 -8  267  260
 -7  290  299
 -6  212  237
 -5   71   13
 -4  189  174
 -3  407  387
 -2  262  262
 -1  113  121
  0  612  617
  1  576  566
  2   67   38
  3  683  688
  4  365  369
  5  115  121
  6  260  244
  7  241  239
  8  104   59
  9    0   25*
 10    0    6*

  K,L= 6, 3
  H  FOB  FCA
-10  161  147
 -9   63   57
 -8  156  125
 -7  156  140
 -6   72   23
 -5   60    4
 -4  125  104
 -3  243  237
 -2  250  236
 -1   97   66
  0   66   63
  1  236  241
  2  432  446
  3  152  143
  4  172  156
  5  340  348
  6  133  116
  7  157  156
  8  106  106
  9   35    9
 10   77   42

  K,L= 7, 3
  H  FOB  FCA
 -9  106  115
 -8   82   39
 -7  119  102
 -6  201  198
 -5  120  140
 -4  102   80
 -3   69   70
 -2  150  141
 -1  255  242
  0   76   71
  1  123  101
  2  102   70
  3  203  202
  4   50    3
  5   87   82
  6  121  122
  7   85   48
  8   97  118
  9    0    5*

  K,L= 8, 3
  H  FOB  FCA
 -8  192  169
 -7   43   11
 -6  207  217
 -5  164  162
 -4  101   75
 -3  246  255
 -2  341  317
 -1  176  183
  0  111  150
  1   99   92
  2   50   23
  3  110  122
  4   27   16*
  5   99   82
  6  198  205
  7  122  130
  8    0   40*

  K,L= 9, 3
  H  FOB  FCA
 -7  252  224
 -6  112   93
 -5  258  233
 -4  232  224
 -3  229  205
 -2   64   23
 -1  265  276
  0  213  217
  1  135  123
  2   85   67
  3   93   93
  4  152  134
  5  123  123
  6   66   57
  7   94  108

  K,L=10, 3
  H  FOB  FCA
 -6  208  180
 -5  181  192
 -4    0   19*
 -3  225  198
 -2  142  146
 -1   52   58
  0  170  167
  1  135  148
  2   50    7
  3   85   75
  4   77   63
  5   15   23*

  K,L=11, 3
  H  FOB  FCA
 -3   67   16
 -2   69   55
 -1  107   92
  0    0    4*
  1   37   47
  2   59   13
  3   42   30

  K,L= 0, 4
  H  FOB  FCA
-11  164  131
-10    0   43*
 -9   15   44*
 -8  120   90
 -7  366  365
 -6  259  251
 -5   74   59
 -4  529  539
 -3  695  668
 -2  293  318
 -1  473  509
  0  737  715
  1  380  367
  2  675  672
  3  790  801
  4   74   68
  5  113  108
  6   55   32
  7  129  134
  8  283  281
  9   98   86
 10  105   91
 11  208  194

  K,L= 1, 4
  H  FOB  FCA
-11  115  102
-10  103   98
 -9  106   64
 -8  107  104
 -7   62   21
 -6  231  233
 -5  567  567
 -4  251  257
 -3  301  334
 -2  830  791
 -1  606  604
  0  228  230
  1  694  705
  2  394  414
  3   57   20
  4  175  185
  5    0   51*
  6  129  147
  7  146  161
  8   70   35
  9  170  176
 10  167  151
 11   96  102

  K,L= 2, 4
  H  FOB  FCA
-11   60   22
-10  103   91
 -9   43   16
 -8  152  159
 -7   77   30
 -6  232  233
 -5   40   28
 -4  296  292
 -3  443  436
 -2  340  331
 -1  603  597
  0  274  299
  1  259  263
  2  282  286
  3   79   42
  4  166  163
  5   14   53*
  6  176  173
  7   85   97
  8  159  142
  9   92   91
 10   80   16

  K,L= 3, 4
  H  FOB  FCA
-11    0   18*
-10  193  165
 -9  236  227
 -8  100   52
 -7  241  250
 -6  167  153
 -5    0   18*
 -4  154  130
 -3  411  435
 -2  314  315
 -1   48   11
  0  291  272
  1   90   87
  2  358  364
  3  211  195
  4  168  196
  5  393  410
  6  431  435
  7   60   72
  8  136  121
  9   69   48
 10    0    5*

  K,L= 4, 4
  H  FOB  FCA
-10  165  149
 -9    0   55*
 -8  310  328
 -7  402  406
 -6   64   13
 -5  360  393
 -4  349  336
 -3    0   11*
 -2  284  252
 -1   60   27
  0  343  294
  1  339  335
  2  188  205
  3  369  399
  4  498  526
  5  286  286
  6  144  142
  7  344  363
  8  141  140
  9   43   30
 10  136  131

  K,L= 5, 4
  H  FOB  FCA
-10  213  205
 -9  244  253
 -8  116  125
 -7  209  214
 -6  349  359
 -5  275  261
 -4  234  294
 -3  239  245
 -2  145  152
 -1  222  237
  0  255  236
  1   54  105
  2  532  553
  3  247  243
  4   52   55
  5  457  486
  6  354  363
  7   84   67
  8  142  135
  9  159  179

  K,L= 6, 4
  H  FOB  FCA
 -9  149  120
 -8  198  181
 -7  126  103
 -6   99  100
 -5  206  197
 -4  165  151
 -3    0   10*
 -2   52   43
 -1   50   36
  0  250  250
  1  172  179
  2  271  285
  3  157  160
  4  361  364
  5  196  193
  6  130  101
  7  353  226
  8   37   40
  9   77   21

  K,L= 7, 4
  H  FOB  FCA
 -9   34   33*
 -8   89   31
 -7  143  150
 -6   90   35
 -5   54   78
 -4  176  148
 -3   78   68
 -2  229  227
 -1  205  227
  0   69  106
  1  201  196
  2  124  111
  3   79   45
  4  113  133
  5   92  125
  6    0    4*
  7  105   95
  8   16   39*

  K,L= 8, 4
  H  FOB  FCA
 -8   64   24
 -7  103   85
 -6  197  182
 -5   50   46
 -4  184  187
 -3  253  254
 -2  157  177
 -1  230  271
  0  312  298
  1  133  121
  2  181  173
  3  158  158
  4   76   95
  5    0   25*
  6   55    8
  7   51   24

  K,L= 9, 4
  H  FOB  FCA
 -6  153  137
 -5  197  217
 -4  116  121
 -3   94  127
 -2  242  236
 -1  290  292
  0  110   36
  1  315  321
  2  183  193
  3   53   21
  4  127  136
  5   77   49
  6  114   67

  K,L=10, 4
  H  FOB  FCA
 -5   17   59*
 -4  175  170
 -3  187  175
 -2    0   28*
 -1  213  202
  0  196  193
  1   63   70
  2   71  104
  3   99  110
  4   90   21

  K,L=11, 4
  H  FOB  FCA
 -1   53   30
  0  110   95

  K,L= 0, 5
  H  FOB  FCA
-11  125  134
-10  294  295
 -9  197  179
 -8   87   58
 -7   95   90
 -6  216  210
 -5  110  109
 -4   30   60
 -3   37    7
 -2  558  582
 -1  760  757
  0  234  256
  1  343  340
  2  909  959
  3  317  313
  4  506  503
  5  491  515
  6  214  230
  7   51   72
  8   59   48
  9   97   76
 10   96   84

  K,L= 1, 5
  H  FOB  FCA
-11  202  232
-10   64   31
 -9  185  194
 -8  208  212
 -7   28   35*
 -6   72   42
 -5   18   63*
 -4   10   17*
 -3  403  364
 -2  395  438
 -1  253  277
  0  643  647
  1  400  409
  2   83   99
  3  529  564
  4  320  316
  5   69   91
  6  128  167
  7   91   74
  8  115  118
  9   72    5
 10   26    1*

  K,L= 2, 5
  H  FOB  FCA
-10   65   80
 -9   57   23
 -8  131  135
 -7   54    7
 -6  132  114
 -5   75   27
 -4  146  150
 -3  139  124
 -2  149  134
 -1  265  246
  0   54   11
  1  213  226
  2  205  199
  3   69   59
  4  218  207
  5   92   97
  6   51    4
  7   51   19
  8   59   21
  9   47   51
 10   48   12

  K,L= 3, 5
  H  FOB  FCA
-10  149  131
 -9    0   24*
 -8  121   94
 -7  255  265
 -6  181  152
 -5  202  219
 -4  390  424
 -3  297  290
 -2   97   85
  1  153  161
  2  143  134
  3  129  136
  4  211  214
  5  235  225
  6   30   73*
  7  159  180
  8   63   99
  9   56    8

  K,L= 4, 5
  H  FOB  FCA
-10  119   95
 -9  216  228
 -8  238  219
 -7  122  113
 -6  364  378
 -5  483  478
 -4   81   35
 -3  602  631
 -2  445  464
 -1   89   58
  0   55   54
  1   79   70
  2  246  264
  3  187  184
  4  136  150
  5  223  219
  6  303  317
  7  133  133
  8   76   37
  9  238  243

  K,L= 5, 5
  H  FOB  FCA
 -9   42    2
 -8  224  228
 -7  254  266
 -6  153  155
 -5  293  299
 -4  621  638
 -3  230  224
 -2  266  272
 -1  243  241
  0  175  169
  1  134  123
  2    0   14*
  3  123  103
  4  338  337
  5  212  196
  6   18   29*
  7  218  215
  8  196  185
  9   55   58

  K,L= 6, 5
  H  FOB  FCA
 -9  108   95
 -8   91   92
 -7  141  129
 -6  171  158
 -5  235  226
 -4    0   27*
 -3  279  309
 -2  163  173
 -1   82   92
  0   81  120
  1    0    9*
  2   75   89
  3   30   14
  4  184  186
  5   67   94
  6  165  162
  7    0   13*
  8   51   37

  K,L= 7, 5
  H  FOB  FCA
 -8   50   10
 -7   86   43
 -6   71    1
 -5   97  109
 -4   60   62
 -3   99   87
 -2  180  195
 -1  104   40
  0  142  131
  1  146  125
  2   75   57
  3  175  168
  4   66   16
  5    0   19*
  6   68   52
  7   49   34

  K,L= 8, 5
  H  FOB  FCA
 -7   93   59
 -6   33   13*
 -5   68   38
 -4  149  124
 -3   39   13
 -2  198  195
 -1  269  265
  0  102  113
  1  236  239
  2  253  247
  3  115  115
  4  157  186
  5  147  147
  6  108   68

  K,L= 9, 5
  H  FOB  FCA
 -5   59   29
 -4  113  121
 -3  173  152
 -2   89   93
 -1  102  136
  0  263  250
  1  231  229
  2   51   67
  3  279  274
  4  150  172

  K,L=10, 5
  H  FOB  FCA
 -3   33    1*
 -2  131  126
 -1  149  127
  0   45    6
  1  172  164
  2  182  164

  K,L= 0, 6
  H  FOB  FCA
-10  163  146
 -9  106  136
 -8  373  392
 -7  223  222
 -6  224  235
 -5  326  345
 -4  411  413
 -3   39   36
 -2  271  275
 -1   65   13
  0  405  407
  1  482  491
  2   73   67
  3  463  471
  4  604  631
  5  154  152
  6  214  203
  7  180  189
  8   64   55
  9  108   68

  K,L= 1, 6
  H  FOB  FCA
-10  148  159
 -9  267  277
 -8   79   50
 -7  232  237
 -6  286  283
 -5   65   42
 -4  179  167
 -3   80   71
 -2  332  355
 -1  255  248
  0  130  102
  1  177  198
  2  415  439
  3  291  303
  4   84   52
  5  296  289
  6  252  262
  7   64   17
  8  196  181
  9   78  110

  K,L= 2, 5
  H  FOB  FCA
-10   81   52
 -9   69   41
 -8  151  141
 -7   86   73
 -6  271  275
 -5  107  114
 -4    0   45*
 -3  152  153
 -2   96   90
 -1   82   57
  0   58   52
  1  139  133
  2   93  116
  3  117  109
  4  195  191
  5   97   60
  6   46   45
  7   95   85
  8   53   16
  9   81   70

  K,L= 3, 6
  H  FOB  FCA
 -9   55   22
 -8  103   93
 -7   99   31
 -6    0   17*
 -5  301  302
 -4  197  186
 -3  313  308
 -2  367  407
 -1  238  253
  0  266  259
  1  349  372
  2   63   45
  3   90   89
  4   43   37
  5  124   93
  6   94   96
  7   53   57
  8   59   45

  K,L= 4, 6
  H  FOB  FCA
 -9    0   22*
 -8  118  113
 -7  177  180
 -6  226  238
 -5   95   90
 -4  456  474
 -3  480  481
 -2   86   76
 -1  459  484
  0  385  383
  1   61   12
  2  217  229
  3  163  141
  4  168  152
  5   75   80
  6   26   20*
  7   84   72
  8  145  138

  K,L= 5, 6
  H  FOB  FCA
 -8  110  131
 -7   72   34
 -6  203  198
 -5  273  282
 -4  174  153
 -3  287  280
 -2  463  474
 -1  154  142
  0  201  201
  1  268  255
  2  182  191
  3   74    7
  4   80   68
  5  124  115
  6  145  120
  7   81   51

  K,L= 6, 6
  H  FOB  FCA
 -8   84   19
 -7   49   45
 -6  112  112
 -5    0   11*
 -4  126  139
 -3  193  182
 -2   72   29
 -1  279  283
  0  170  164
  1   84   39
  2  144  135
  3   35   13
  4   82   33
  5   76   15
  6   88   11
  7   42   43

  K,L= 7, 6
  H  FOB  FCA
 -7  137   98
 -6   69   56
 -5  133  100
 -4   95   83
 -3   30    6*
 -2    0   12*
 -1   65   51
  0  111   92
  1   28    5*
  2   78   91
  3  139  114
  4   47   31
  5  111   78
  6  109   53

  K,L= 8, 6
  H  FOB  FCA
 -5  115  117
 -4   22   27*
 -3   65   65
 -2   64   57
 -1   75   40
  0  153  120
  1  141  123
  2   89   51
  3  174  152
  4  210  185

  K,L= 9, 6
  H  FOB  FCA
 -3    0   22*
 -2  136   95
 -1  107   98
  0   77   63
  1  103   75
  2  192  193

  K,L= 0, 7
  H  FOB  FCA
 -9  178  205
 -8  149  160
 -7  193  179
 -6  373  397
 -5  152  192
 -4  271  264
 -3  187  192
 -2  256  270
 -1  174  155
  0   51   27
  1  131  124
  2  226  232
  3  103  110
  4    0    6*
  5  173  196
  6  220  219
  7  106  109

  K,L= 1, 7
  H  FOB  FCA
 -9    0   27*
 -8  187  190
 -7  281  308
 -6   57   34
 -5  274  276
 -4  302  310
 -3   59   18
 -2  169  176
 -1   87  104
  0  131  109
  1    0   21*
  2   59   72
  3   67   63
  4  108  114
  5  182  177
  6   68   23
  7  197  199

  K,L= 2, 7
  H  FOB  FCA
 -8   81   86
 -7   65   52
 -6  206  211
 -5  128  133
 -4  157  154
 -3   41   14
 -2   93  100
 -1  160  135
  0   51   33
  1   56   53
  2  100   75
  3   38   18
  4  139  132
  5   19   24*
  6   56   88
  7  116  104

  K,L= 3, 7
  H  FOB  FCA
 -8   51   20
 -7  112   81
 -6   70   26
 -5   71   46
 -4    0   26*
 -3  203  224
 -2  146  141
 -1  172  147
  0  233  241
  1  127  121
  2  246  265
  3  198  195
  4  116   99
  5   55   17
  6   20   34*
  7    0   23*

  K,L= 4, 7
  H  FOB  FCA
 -7    0    6*
 -6   31   70*
 -5  119  103
 -4   85   65
 -3  131  124
 -2  247  260
 -1  257  264
  0  100   89
  1  326  322
  2  245  251
  3   77   38
  4  153  152
  5  156  160
  6  117   57

  K,L= 5, 7
  H  FOB  FCA
 -7   36   18
 -6   52   62
 -5   55   46
 -4  165  151
 -3  173  175
 -2  124  115
 -1  179  158
  0  318  324
  1  176  179
  2  192  175
  3  233  227
  4  199  178
  5   11    8*
  6   73   83

  K,L= 6, 7
  H  FOB  FCA
 -5   55   19
 -5   77   54
 -4   61   74
 -3   98   47
 -2  142  143
 -1  250  226
  0   49   30
  1  205  179
  2  162  167
  3   65   37
  4   64   72
  5  151   91

  K,L= 7, 7
  H  FOB  FCA
 -4   22   55*
 -3    0   41*
 -2   86   54
 -1    0    5*
  0   59   73
  1  111   77
  2   33   37
  3   34   31

  K,L= 8, 7
  H  FOB  FCA
 -2   58   44
 -1    0   21*
  0  102   21
  1   58   26

  K,L= 0, 8
  H  FOB  FCA
 -7  165  170
 -6  189  166
 -5  140  113
 -4  306  293
 -3  277  268
 -2   55   75
 -1  170  164
  0  150  157
  1    0    1*
  2   40   60
  3  166  142
  4  133  127
  5  124   85
  6   45   85

  K,L= 1, 8
  H  FOB  FCA
 -7   73   56
 -6  166  134
 -5  196  222
 -4   69   28
 -3  149  133
 -2  268  268
 -1   78   75
  0  125  120
  1  142  132
  2  108  118
  3  112   77
  4   80   15
  5   43   54
  6   83   48

  K,L= 2, 8
  H  FOB  FCA
 -7   72   72
 -6  126  122
 -5   77    6
 -4  151  153
 -3  145  142
 -2  116  124
 -1  171  170
  0  111   84
  1  162  151
  2  122  119
  3   46   13
  4  100   72
  5   57   20

  K,L= 3, 8
  H  FOB  FCA
 -6   96   41
 -5    0   18*
 -4   67    4
 -3  103   82
 -2  100   46
 -1  129  118
  0  110  122
  1   89   32
  2  133  124
  3  183  168
  4   95   53
  5   86   95

  K,L= 4, 8
  H  FOB  FCA
 -5   57   82
 -4  142  121
 -3   81   90
 -2  114   76
 -1    0   21*
  0  140  125
  1  236  231
  2   41   29
  3  171  152
  4  204  196

  K,L= 5, 8
  H  FOB  FCA
 -4    0   17*
 -3   82   68
 -2   90   74
 -1  128  116
  0   96   99
  1   57   89
  2  209  208
  3  147  165

  K,L= 6, 8
  H  FOB  FCA
 -3   57   10
 -2   30   10*
 -1   16   16*
  0  104   91
  1  177  157
  2   71   15

  K,L= 0, 9
  H  FOB  FCA
 -4  151  128
 -3   58   69
 -2  259  257
 -1  217  213
  0   40   14
  1  241  234
  2  266  243
  3   66   74

  K,L= 1, 9
  H  FOB  FCA
 -4   87   96
 -3  238  253
 -2   81   90
 -1  180  168
  0  269  263
  1  105  119
  2  113  114
  3  162  140

  K,L= 2, 9
  H  FOB  FCA
 -4  116   82
 -3   78   27
 -2  118  118
 -1  103   96
  0   72   39
  1  131  133
  2   47   76

  K,L= 3, 9
  H  FOB  FCA
 -3   97    6
 -2   66   30
 -1   24    6*
  0   24    1*
  1   60   44
```

TABLE III

INTRAMOLECULAR BOND DISTANCES (Å)[a]

Co_1–Co_1'	2.498 (2)	C_1–O_1	1.13 (2)
Co_1–Co_2	2.492 (2)	C_2–O_2	1.12 (1)
		C_3–O_3	1.16 (1)
	2.495	C_4–O_4	1.12 (2)
Co_1–C_1	1.78 (1)	C_5–O_5	1.13 (1)
Co_1–C_2	1.83 (1)		
			1.13
Co_1–C_3	1.73 (1)		
Co_2–C_4	1.84 (2)	N–C_7	1.50 (2)
Co_2–C_5	1.79 (1)	N–C_9	1.47 (2)
		N–C_{11}	1.55 (2)
	1.79		
			1.51
Co_1–C_6	1.92 (1)		
Co_2–C_6	1.92 (1)	C_7–C_8	1.44 (3)
		C_9–C_{10}	1.48 (3)
	1.92	C_{11}–C_{12}	1.60 (3)
C_6–O_6	1.28 (2)		
			1.51
O_6–B	1.37 (2)		
B–N	1.66 (2)		

[a] The standard deviations of the least significant figures are given in parentheses. These calculated errors result from the errors in the positional parameters whereas more realistic values for the Co–C distances would be twice those quoted.

TABLE IV

BOND ANGLES (DEG)[a]

Co_1–Co_2–Co_1'	60.2	Co_1–Co_2–C_5'	150.5
Co_2–Co_1–Co_1'	59.9	Co_1'–Co_1–C_3	149.0
		Co_2–Co_1–C_1	149.3
	60.0 (1)		
			149.6 (10)
Co_1–C_6–Co_1'	81.4		
Co_1–C_6–Co_2	81.1	Co_1–Co_2–C_5	97.9
		Co_1'–Co_1–C_1	97.6
	81.3 (2)	Co_2–Co_1–C_3	95.7
C_6–Co_1–Co_1'	49.3		
			97.1 (10)
C_6–Co_1–Co_2	49.5		
C_6–Co_2–Co_1	49.4	Co_1–Co_2–C_4	98.8
		Co_1'–Co_1–C_2	100.6
	49.4 (2)	Co_2–Co_1–C_2	99.5
C_6–Co_1–C_1	100.4		
			99.6 (10)
C_6–Co_1–C_3	100.9		
C_6–Co_2–C_5	101.7	Co_1–C_6–O_6	130.0
		Co_2–C_6–O_6	133.9
	101.0 (5)		
			132.0 (10)
C_6–Co_1–C_2	142.9		
C_6–Co_2–C_4	141.5	B–N–C_7	113.3
		B–N–C_9	108.4
	142.2 (6)	B–N–C_{11}	102.8
C_1–Co_1–C_2	105.6		
			108.2 (30)
C_3–Co_1–C_2	102.3		
C_5–Co_2–C_4	104.4	C_7–N–C_9	110.2
		C_9–N–C_{11}	110.8
	104.1 (15)	C_{11}–N–C_7	111.2
C_1–Co_1–C_3	96.1		
			110.7 (30)
C_5–Co_2–C_5'	93.9		
		N–C_7–C_8	121.0
	95.0 (15)	N–C_9–C_{10}	121.8
Co_1–C_1–O_1	177.7	N–C_{11}–C_{12}	112.5
Co_1–C_3–O_3	176.9		
			118.4 (30)
Co_2–C_5–O_5	177.8		
		C_6–O_6–B	135.4 (30)
	177.5 (15)	O_6–B–N	105.2 (30)
Co_1–C_2–O_2	179.6		
Co_2–C_4–O_4	179.3		
	179.5 (15)		

[a] The estimated standard deviations of the average angles are given in parentheses.

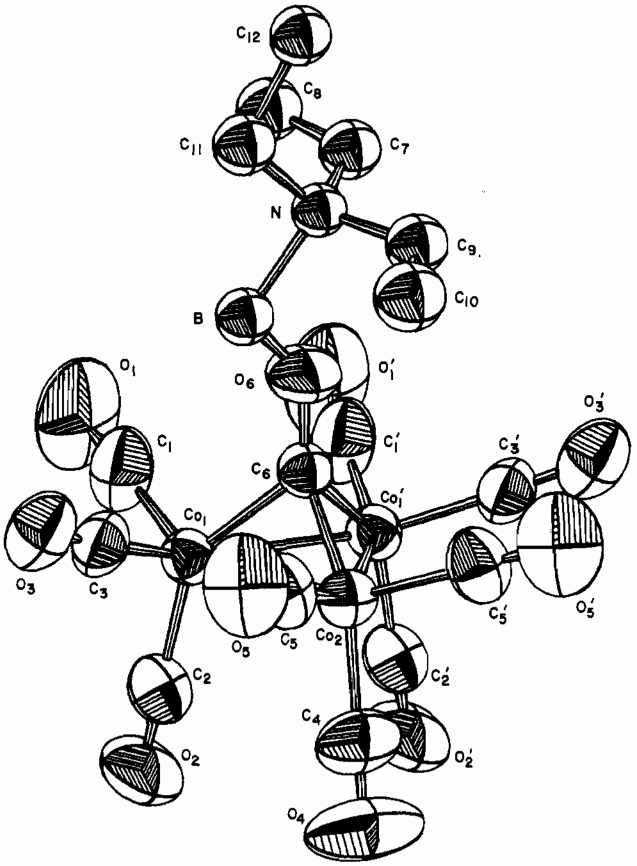

Figure 2.—The molecular configuration of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$ depicting thermal ellipsoids.

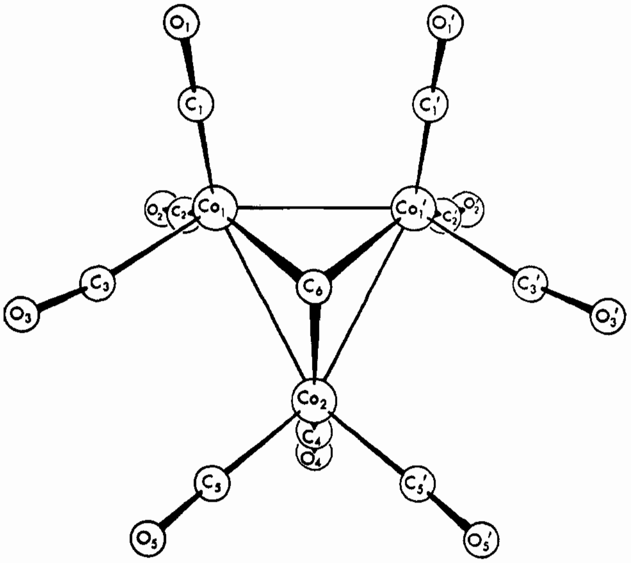

Figure 3.—The metal atom cluster portion of the $Co_3(CO)_{10}BH_2N(C_2H_5)_3$ structure showing C_{3v} point symmetry.

the $Co_3(CO)_9C$ core will not be given here since Dahl and coworkers have commented[2,3] on these at some length. The essential point here is that the carbonyl group configurations are a result of the Co orbitals hybridizing to give maximum Co_3C core overlaps consistent with the very acute angles of 60.0° for Co–Co–Co and 49.4° for C_6–Co–Co. To achieve very acute angles in the Co_3C core the OC–Co–CO angles increase from 90° with the result that the average equatorial–

Co–equatorial angles (C_3–Co–C_1) are 95.0° and the average equatorial–Co–axial angles (C_1–Co_1–C_2) are 104.1°. The corresponding angles in the $Co_3(CO)_9CCH_3$ structure[2] were 97.2 and 102.2°, and in the $Co_3(CO)_9S$ structure[3] they were 101.1 and 100.8°. The average dihedral angles between the planes defined by the equatorial carbonyl groups and the Co atoms to which they are attached and the plane of the three Co atoms is 32° in this structure and 29° in the $Co_3(CO)_9CCH_3$ and $Co_3(CO)_9S$ structures.[2,3]

As for bond distances, the Co–Co distance of 2.495 Å here compares well with the 2.467-Å distance in $Co_3(CO)_9CCH_3$ and with the other observed Co–Co distances as listed by Sutton and Dahl.[2] The Co–Co distance in $Co_3(CO)_9S$[3] was found to be abnormally long at 2.637 Å. The Co–C_6 distance of 1.92 Å is equivalent to the 1.90-Å distance found in the $Co_3(CO)_9CCH_3$ structure. The axial Co–C carbonyl distances are longer than the equatorial Co–C distances, but, on comparison with the $Co_3(CO)_9CCH_3$[2] and $Co_3(CO)_9S$[3] structures, one must conclude that these differences are probably not significant. The average distances for the five unique carbonyl groups in this structure are 1.79 Å for Co–C and 1.13 Å for C–O.

After coordination of the $-OBH_2N(C_2H_5)_3$ group to the $Co_3(CO)_9C$ core, the point symmetry is no longer C_{3v}, or even C_3, because the $-OBH_2N(C_2H_5)_3$ group is not directly above the $Co_3(CO)_9C$ core but is tilted toward Co_2 maintaining the molecular mirror symmetry. This tilt of 14° (angle C_6–O_6–N is 166.0°) minimizes intramolecular repulsions in this case. The distances involving the ethyl groups attached to N are not expected to be good in view of the disorder problem. The B–N distance of 1.66 Å is essentially a normal B–N single bond and can be compared with 1.58 Å for the sum of the covalent radii,[18] 1.60 Å found in H_3NBF_3,[19] and 1.64 Å found in CH_3CNBF_3.[20] The tetrahedral boron atom with the O_6–B–N angle of 105.2° has two hydrogen atoms completing the tetrahedron which are not shown in Figure 1. No attempt was made to locate the hydrogen atoms because with the disorder their peak heights on electron density maps would be less than 0.5 $e^-/Å^3$.

The B–O bond length of 1.37 Å is to be compared with the mean values[21] of 1.37 Å found in trigonal borates and 1.48 Å found in tetrahedral borates. The B–O distance here might be expected to be closer to the value found for tetrahedral borates, but it is short here because of increased π-bond character through the O_6 p_z orbital. This interpretation is consistent with the multiple-bond character of the C_6–O_6 bond. The O_6 atom approaches trigonal hybridization with the C_6–O_6–B angle of 135.4°. This angle is determined primarily by a minimization of intramolecular repulsive forces. The C_6–O_6 length of 1.28 Å approximates a C–O double bond in the valence-bond sense. Interestingly, the C–O bond length is equivalent to that found in metal bisacetylacetonates.[22] In the molecular orbital sense there is extensive electron delocalization extending from B through to the $Co_3(CO)_9C$ core.

The elucidation of the structure of $Co_3(CO)_{10}BH_2N(C_2H_5)_3$ demonstrates the facile formation of the Co_3C tetrahedral cluster skeleton under conditions that might have been considered favorable for the incorporation of a boron atom into the cluster framework. The synthesis of the $Co_3(CO)_{10}BH_2NR_3$ complexes is unique in that the source of the carbon atom in the Co_3C tetrahedron is a carbonyl group of the $Co_2(CO)_8$ itself. In all other known syntheses of compounds of the type $Co_3(CO)_9CY$, the carbon atom is derived from an extraneous source, usually an organic halide. In a formal sense, the $Co_3(CO)_{10}BH_2NR_3$ complex might be visualized as a $Co_3(CO)_9C$-substituted derivative of a hydroxyborane R_3NBH_2O-. There is some justification for such a view in the comparatively short B–O distance (*vide supra*) and the rather long C_6–O_6 distance of 1.28 Å which is significantly longer than the C–O distances of 1.19–1.20 Å found in other triply bridging CO groups.[9,16,17] As noted earlier, we were unable to detect an infrared absorption band between 2000 and 1500 cm^{-1} associated with the triply bridging CO group. It is possible, however, that this band lies below 1500 cm^{-1}, but a definite assignment is precluded by the abundance of other absorptions in this region.

Experimental Section

$(C_2H_5)_3NBH_2Co_3(CO)_{10}$.—Dicobalt octacarbonyl (6.0 g, 0.018 mol) and triethylamine–borane (11.4 g, 0.100 mol) in 200 ml of benzene were stirred for 3 hr at 60–65°. The black-violet mixture was cooled to room temperature and filtered to remove insoluble decomposition products. Benzene was then removed from the filtrate using an aspirator pump. The resultant black sludgy residue was extracted with *ca.* 500 ml of petroleum ether (bp 30–60°) to give a deep violet solution from which the solvent was again evaporated using a rotating evaporator. A violet oil remained. A solid product was separated from the attendant excess of triethylamine–borane by distilling off the latter during 16 hr at room temperature and 10^{-4} mm. The resulting solid was then recrystallized from 200 ml of petroleum ether. Needle-like crystals (2.4 g) precipitated when the solution was chilled to −78°. The compound melted at 103–105° into a black liquid and sublimed partly at 80° (0.0001 mm), substantial decomposition occurring during the sublimation.

Anal. Calcd for $(C_2H_5)_3NBH_2Co_3(CO)_{10}$: C, 33.7; H, 3.1; N, 2.5; O, 27.9; Co, 31.0; B, 1.9; mol wt, 571. Found: C, 33.4; H, 3.0; N, 2.5; O, 27.8; Co, 30.9; B, 2.2.

The cryoscopic molecular weight in benzene was 565. The electronic spectrum in cyclohexane displayed one peak at 5160 Å (ϵ 2000). The infrared spectrum in methylcyclohexane showed B–H absorption at 2415 and 2365 cm^{-1} and CO absorption at 2100 (23), 2048 (100), 2034 (100), and 2008 (45) cm^{-1}. Numerical intensities are given in brackets. The B^{11} nmr spectrum shows a poorly resolved triplet centered at 18 ppm (from external $B(OCH_3)_3$). This may be compared with the triplet exhibited by $(C_2H_5)_3NBH_2Cl$ at 22.8 ppm (J_{BH} = 125 cps).

$(CH_3)_3NBH_2Co_3(CO)_{10}$.—The same procedure as above was followed using 13.0 g (0.038 mol) of dicobalt octacarbonyl and

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(18) L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960.

(19) J. L. Hoard, S. Geller, and W. M. Cashin, *Acta Cryst.*, **4**, 396 (1951).

(20) J. L. Hoard, T. B. Owen, A. Buzzell, and O. N. Salmon, *ibid.*, **3**, 130 (1950).

(21) V. F. Ross and J. O. Edwards in "The Chemistry of Boron and Its Compounds," E. L. Muetterties, Ed., John Wiley & Sons, Inc., New York, N. Y., 1967, p 196.

(22) For several examples see "Tables of Interatomic Distances and Configuration in Molecules and Ions," Special Publication No. 18, The Chemical Society, London, 1965, pp M162s, M163s.

12.0 g (0.165 mol) of trimethylamine–borane in 650 ml of benzene. The crude reaction product was subjected to sublimation at 90° (0.001 mm) where most of the excess $(CH_3)_3NBH_3$ sublimed off. The sublimation residue was twice extracted with 500 ml of petroleum ether. Concentration and cooling of the extract gave a total of 8.2 g of needlelike crystals, mp 124–125°, in two crops. Some additional $(CH_3)_3NBH_3$ as a contaminant in the second crop was removed by sublimation as above.

Anal. Calcd for $(CH_3)_3NBH_2Co_3(CO)_{10}$: C, 29.5; H, 2.1; N, 2.7; O, 30.3; Co, 33.4; B, 2.1. Found: C, 29.6; H, 2.2; N, 2.7; O, 30.6; Co, 33.0; B, 1.7.

The compound absorbed in the visible region of the spectrum at 5150 Å (ϵ 2100). Characteristic infrared absorptions occurred at 2400 and 2370 cm^{-1} (BH stretching) and at 2092, 2040, 2025, and 2000 cm^{-1} (CO stretching).

Mass Spectra.—Mass spectra were obtained with a Consolidated Electrodynamics (CEC) 21-110B high-resolution instrument operated at a 100-μA anode current, an ionizing current of 70 eV, and a 45–80° source temperture. The spectra were recorded oscillographically with and without perfluorokerosene as mass reference. Samples were introduced directly into the ionization source using the solid injection technique. In Table V observed ion peaks with $m/e > 177$ are listed, and probable assignments are given. Where more than one formula is ascribed to a particular mass number, the first is the most likely. The loss of two hydrogen atoms (probably from the BH_2 group) first becomes evident after scission of one CO group from the parent ion. Cleavage of hydrogen atoms becomes much more prominent after the loss of two CO groups, however. Moreover, the loss of two hydrogen atoms leads to peaks of higher intensity than loss of only one H atom. There is a reversal in the relative abundance of the ions corresponding to loss of CO groups *vs.* loss of CO groups plus hydrogen atoms occurring at *m/e* 445 and 443. For all lower fragments hydrogen atom retainment on the BH_2 group becomes much less important.

The elemental composition of the *m/e* 387 ion was established as the fragment $Co_3(CO)_5{}^{11}BN(CH_3)_3{}^+$ with a mass of 386.8569 *vs.* the fragment $Co_3(CO)_6{}^{11}BH_2NCH_3{}^+$ with a mass of 386.8933. The *m/e* 387 ion was measured using the "peak matching" technique and resolution of 14,000. A measured value of 386.8575 was obtained which is within 2 ppm of the calculated value for the $Co_3(CO)_5{}^{11}BN(CH_3)_3{}^+$ fragment. Owing to the thermal instability under the operating conditions, additional fragment ion masses were not measured. In addition to the ions listed in Table V for the ^{11}B and ^{12}C isotope ions, the correct abundances for the ^{10}B and ^{13}C isotope ions were present in the spectra.

Relative abundances were measured only qualitatively. Of all of the ions with a mass >175, the most abundant one occurs at *m/e* 387, corresponding to the loss of five CO groups and two hydrogen atoms from the parent (P). Other peaks at *m/e* 415 (P − 4CO − 2H), 501 (P − CO), 359 (P − 6CO − 2H), 331 (P − 7CO − 2H), 303 (P − 8CO − 2H), and 275 (P − 9CO − 2H) had about one-third to one-half the abundance of the peak at *m/e* 387. The parent ion at *m/e* 529 and the peaks at *m/e* 473 (P − 2CO) and 445 (P − 3CO) showed only 10–20% of the abundance of the *m/e* 387 peak. The abundance of the *m/e* 471 ion (P − 2CO − 2H) was in turn approximately one-third of the peak at *m/e* 473 (P − 2CO).

TABLE V

MASS SPECTRA OF $Co_3(CO)_{10}BH_2N(CH_3)_3$[a]

m/e	Ion Species	m/e	Ion Species
$\underline{529}$	$Co_3(CO)_{10}BH_2N(CH_3)_3{}^+$	304	$Co_3(CO)_2BHN(CH_3)_3{}^+$
$\underline{501}$	$Co_3(CO)_9BH_2N(CH_3)_3{}^+$	303*	$Co_3(CO)_2BN(CH_3)_3{}^+$
499*	$Co_3(CO)_9BN(CH_3)_3{}^+$	297*	Unidentified
$\underline{473}$	$Co_3(CO)_8BH_2N(CH_3)_3{}^+$	292*	$Co_3(CO)_2N(CH_3)_3{}^+$ (r)
472	$Co_3(CO)_8BHN(CH_3)_3{}^+$	289	$Co_3(C)COBH_2N(CH_3)_3{}^+$
471	$Co_3(CO)_8BN(CH_3)_3{}^+$	277	$Co_3COBH_2N(CH_3)_3{}^+$
460*	$Co_3(CO)_8N(CH_3)_3{}^+$ (r)	276	$Co_3COBHN(CH_3)_3{}^+$
456*	Unidentified; B absent	$\underline{275}$	$Co_3COBN(CH_3)_3{}^+$
$\underline{445}$	$Co_3(CO)_7BH_2N(CH_3)_3{}^+$	267*	Unidentified
444	$Co_3(CO)_7BHN(CH_3)_3{}^+$	264*	$Co_3CON(CH_3)_3{}^+$ (r)
$\underline{443}$	$Co_3(CO)_7BN(CH_3)_3{}^+$	261	$Co_3COBN(H)(CH_3)_2{}^+$ (r), $Co_3CBH_2N(CH_3)_3{}^+$ (r)
432	$Co_3(CO)_7N(CH_3)_3{}^+$ (r)	249	$Co_3COBH_2N(H)_2CH_3{}^+$ (r), $Co_3(CO)CON(H)_2{}^+$ (r)
429*	$Co_3(CO)_6(C)BH_2N(CH_3)_3{}^+$	248*	$Co_3COBHN(H)_2CH_3{}^+$ (r), $Co_3(CO)_2NH^+$ (r)
428*	Unidentified, B absent	$\underline{247}$	$Co_3COBN(H)_2CH_3{}^+$ (r), $Co_3(CO)_2N^+$ (r)
417	$Co_3(CO)_6BH_2N(CH_3)_3{}^+$	246	$Co_3COBN(H)CH_3{}^+$ (r), $Co_3(CO)_2BH_2{}^+$
$\underline{416}$	$Co_3(CO)_6BHN(CH_3)_3{}^+$	$\underline{245}$	$Co_3COBNCH_3{}^+$, $Co_3(CO)_2BH^+$
$\underline{415}$	$Co_3(CO)_6BN(CH_3)_3{}^+$	244	$Co_3(CO)_2B$
404*	$Co_3(CO)_6N(CH_3)_3{}^+$ (r)	$\underline{243}$	Could contain 1B
401*	$Co_3(CO)_5(C)BH_2N(CH_3)_3{}^+$	$\underline{233}$	$Co_3COBNH_3{}^+$(r), $Co_3(CO)_2{}^+$, $Co_2(CO)_2N(CH_3)_3$
389	$Co_3(CO)_5BH_2N(CH_3)_3{}^+$	232*	$Co_3COBNH_2{}^+$ (r)
$\underline{388}$	$Co_3(CO)_5BHN(CH_3)_3{}^+$	231*	Co_3COBNH^+ (r)
$\underline{387}$	$Co_3(CO)_5BN(CH_3)_3{}^+$	230*	Co_3COBN^+, $Co_3(C)COBH_2{}^+$
376*	$Co_3(CO)_5N(CH_3)_3{}^+$ (r)	229*	$Co_3(C)COBH^+$
373	$Co_3(CO)_4(C)BH_2N(CH_3)_3{}^+$	218	$Co_3COBH_2{}^+$
361	$Co_3(CO)_4BH_2N(CH_3)_3{}^+$	217	Co_3COBH^+
360	$Co_3(CO)_4BHN(CH_3)_3{}^+$	216	Co_3COB^+
$\underline{359}$	$Co_3(CO)_4BN(CH_3)_3{}^+$	$\underline{205}$	$Co_3CO^{+\cdot}$
348*	$Co_3(CO)_4N(CH_3)_3{}^+$ (r)	202	Co_3CBH^+ (r)
345	$Co_3(CO)_3(C)BH_2N(CH_3)_3{}^+$	$\underline{190}$	Co_3CH^+ (r)
335*	Unidentified	$\underline{189}$	Co_3C^+
333*	$Co_3(CO)_3BH_2N(CH_3)_3{}^+$	$\underline{177}$	$Co_3{}^+$
332	$Co_3(CO)_3BHN(CH_3)_3{}^+$		
$\underline{331}$	$Co_3(CO)_3BN(CH_3)_3{}^+$		
320*	$Co_3(CO)_3N(CH_3)_3{}^+$ (r)		
317	$Co_3(CO)_2(C)BH_2N(CH_3)_3{}^+$		
316	$Co_3(CO)_2(C)BHN(CH_3)_3{}^+$		
305*	$Co_3(CO)_2BH_2N(CH_3)_3{}^+$		

[a] An asterisk indicates a small peak; underlined mass numbers denote the most abundant ions. A rearrangement ion is indicated by (r).

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