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Flattening of Rhodium Vertices in Mixed Rhodium–Nickel Carbonyl Clusters: Relationships to Borane and Zintl Ion Structures

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The flattened deltahedra and related polyhedra found in hypoelectronic bare group 13 metal cluster anions are also found in some anionic mixed rhodium–nickel carbonyl clusters. In all cases the rhodium vertices rather than the nickel vertices are involved in the flattening process so that the rhodium vertices contribute four internal orbitals and the nickel vertices three internal orbitals to the skeletal bonding of the cluster. Thus, the 11-vertex cluster $Rh_5Ni_6(CO)_{21}^{3-}$ has a D_{3h} triflattened pentacapped trigonal prismatic structure similar to that found in the In_{11}^{7-} anion of the intermetallic K_8In_{11}. Similarly the polyhedra in the 11-vertex cluster $RhNi_{10}(CO)_{19}^{3-}$ and the 9-vertex cluster $Rh_3Ni_6(CO)_{17}^{3-}$ are both derived from a 10-vertex *isocloso* polyhedron by capping (for $RhNi_{10}(CO)_{19}^{3-}$) or vertex removal (for $Rh_3Ni_6(CO)_{17}^{3-}$) followed by flattening all of the rhodium vertices. A D_{3h} icosahedron with flattened rhodium vertices is found in the 12-vertex cluster $Rh_3Ni_9(CO)_{22}^{3-}$.

1. Introduction

Boron is well-known to form the very stable deltahedral boranes $B_nH_n^{2-}$ ($6 \leq n \leq 12$)[1] as well as isoelectronic carborane derivatives in which one to three of the boron vertices have been replaced by carbon atoms.[2] The heavier group 13 elements form much weaker E−H bonds (E = Al, Ga, In, Tl), and thus, the corresponding deltahedral $E_nH_n^{2-}$ remain unknown. However, highly charged bare deltahedral E_n^{z-} anions are found in alkali-metal intermetallics (Zintl ions) of the heavier group 13 elements [E = Ga (occasionally), In, Tl][3−6] but not in boron or aluminum clusters. The E_n^{z-} ions do not correspond to complete deprotonation products of the hypothetical $E_nH_n^{2-}$ but instead are hypoelectronic with $z < n + 2$. This hypoelectronicity (electron poverty) in E_n^{z-} leads to deltahedra not only different from those found in the deltahedral boranes but also different from those found in similarly hypoelectronic metal carbonyl clusters such as bicapped tetrahedral $Os_6(CO)_{18}$[7] and capped octahedral $Os_7(CO)_{21}$.[8] More specifically, for E_n^{z-} derivatives having 9−11 vertices (i.e., $9 \leq n \leq 11$), each apparent skeletal electron pair less than the $2n + 2$ skeletal electrons required by the Wade−Mingos rules[9,10,11] corresponds to a degree 4 vertex being pushed toward the center of the deltahedron. Originally this flattening process was assumed to be a consequence of the involvement of one of the lone pairs of d electrons in the skeletal bonding/flattened vertex leading to the $2n + 2$ effective skeletal electrons required by the Wade−Mingos rules.[12,13] However, in light of the accumulating evidence for the lack of involvement of d orbitals in the chemical bonding of posttransition elements,[14−19] a more likely source of the extra electron pair contributed by each flattened vertex appears to be "inverted hybridization" similar to that of the bridgehead carbons in the surprisingly stable tricyclo[1.1.1]pentane.[20−24]

A question of interest is whether the same flattened deltahedra can be found in metal clusters other than the

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alkali-metal indium and thallium intermetallics. This paper shows the close relationship between the 11-vertex deltahedra in In_{11}^{7-} and the isoelectronic rhodium−nickel mixed metal carbonyl cluster[25] $Rh_5Ni_6(CO)_{21}^{3-}$. In addition, this paper discusses relationships between other mixed rhodium−nickel carbonyl clusters and group 13 analogues including polyhedral borane and metallaborane frameworks. Rhodium vertices in a mixed metal carbonyl cluster composed primarily of nickel atoms are generally seen to be particularly susceptible to flattening processes to compensate for apparent hypoelectronicity. Furthermore, 3-fold symmetry appears to be preferred for flattened deltahedra even when the corresponding $B_nH_n^{2-}$ deltahedron (e.g., for $n = 10$ or 11) does not have 3-fold symmetry.

2. Genesis of Flattened Deltahedra

All of the rhodium−nickel carbonyl clusters discussed in this paper have structures related to deltahedral three-dimensional aromatic systems.[26,27] The normal vertices in such structures, including the deltahedral boranes $B_nH_n^{2-}$, each contribute three internal orbitals to the skeletal bonding. The resulting chemical bonding topology for a deltahedron having n vertices consists of an n-center core bond and n two-center surface bonds leading to the requirement of $2n + 2$ skeletal electrons in accord with the Wade−Mingos rules.[9−11]

A variety of bare posttransition metal cluster anions (the "Zintl ions"), such as Ge_9^{2-} [28] and $TlSn_9^{3-}$,[29] are isolobal, isoelectronic, and essentially isostructural with the deltahedral boranes having the same number of vertices. Such bare metal cluster ions may be regarded as analogous three-dimensional aromatic systems[27] also with $2n + 2$ skeletal electrons using three internal orbitals from each vertex atom. However, bare metal-cluster anions containing exclusively indium and thallium exhibit apparent skeletal electron counts less than $2n + 2$ and thus are formally hypoelectronic. The geometries of such clusters differ from those of the corresponding deltahedral boranes by having one or more vertices pushed toward the center of the deltahedron by a process conveniently called flattening. Vertices of degree 4 surrounded exclusively by vertices of degrees 5 or higher appear particularly susceptible to such flattening processes. The deltahedra of $B_9H_9^{2-}$ and $B_{10}H_{10}^{2-}$, namely the tricapped trigonal prism and bicapped square antiprism, respectively, have three and two such vertices (Figure 1). Flattening one and two vertices of the $B_9H_9^{2-}$ and $B_{10}H_{10}^{2-}$ deltahedra, respectively, leads to the deltahedra found in the clusters Tl_9^{9-} [30,31] and $E_{10}Zn^{8-}$ ($E = In$[32] and Tl[33]). In some but not all cases, the vertex motion associated with the flattening process leads to some realignment of the polyhedral edges by diamond−square−diamond processes. However, all faces remain triangles so that a deltahedron remains some kind of deltahedron after the flattening process.

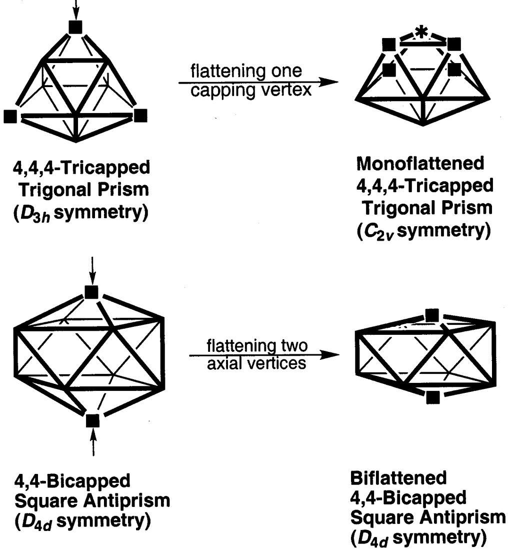

Figure 1. (a) Flattening one degree 4 (capping) vertex in the 4,4,4-tricapped trigonal prism of $B_9H_9^{2-}$ to give the deltahedron found in Tl_9^{9-}. (b) Flattening both axial (degree 4) vertices in the 4,4-bicapped square antiprism of $B_{10}H_{10}^{2-}$ to give the deltahedron found in $In_{10}Zn^{8-}$. In Figures 1 and 2 vertices of degree 3, 4, and 6 are indicated by the symbols ▲, ■, and ∗, respectively. Vertices of degree 5 are not marked.

The other deltahedron subject to flattening is an 11-vertex deltahedron found in triflattened form in the clusters E_{11}^{7-} ($E = Ga$,[34] In,[35] Tl[36,37]) as well as the isoelectronic $In_{10}Hg^{8-}$.[38] However, the starting 11-vertex deltahedron for this flattening process is not the "edge-coalesced icosahedron" found in $B_{11}H_{11}^{2-}$ (Figure 2a) but instead a pentacapped trigonal prism (Figure 2b). The two axial vertices in an unflattened pentacapped trigonal prism have degrees of only three and thus represent sites of localized bonding so that this deltahedron would not be favorable for a fully delocalized aromatic system such as $B_{11}H_{11}^{2-}$. However, the edge realignment accompanying the triflattening of the pentacapped trigonal prism increases the degrees of the two axial vertices from 3 to 6 so that the final triflattened pentacapped trigonal prism has five degree 6 vertices and six degree 4 vertices (Figure 2b). The triflattened pentacapped trigonal prism, such as that found in In_{11}^{7-}, can also be viewed as a trigonal bipyramid of degree 6 vertices surrounded by a trigonal prism of degree 4 vertices.

There is an important correlation between the number of flattened vertices and the electron count in a flattened

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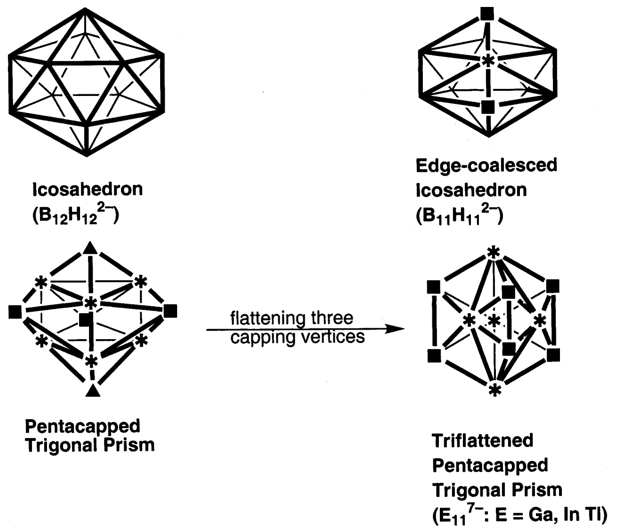

Figure 2. (a) Icosahedron found in $B_{12}H_{12}^{2-}$ and the edge-coalesced icosahedron found in $B_{11}H_{11}^{2-}$. (b) Flattening the three vertices capping the rectangular faces in a pentacapped trigonal prism to give the triflattened pentacapped trigonal prism found in E_{11}^{7-} (E = Ga, In, Tl) and $Rh_5Ni_6(CO)_{21}^{3-}$.

deltahedron. Thus, if all of the vertices in a flattened deltahedral cluster are assumed to contribute the usual three internal orbitals to the skeletal bonding, the apparent number of skeletal electrons is less than the $2n + 2$ of the Wade−Mingos rules;[9−11] i.e., the cluster is apparently hypoelectronic. However, the number of skeletal electrons in the known uncentered flattened deltahedral bare posttransition element clusters increases to $2n + 2$ if each flattened vertex is assumed to contribute an extra pair of skeletal electrons. The vertices in the bare deltahedral clusters of the heavier group 13 elements found in their intermetallics clearly have an external lone pair from their sp^3 bonding manifolds for incorporation into the skeletal bonding by a suitable deltahedral distortion. Thus, flattening of one or more vertices in a deltahedral cluster can be seen as a mechanism for drawing the otherwise external lone pairs from these vertices into the skeletal bonding. Such a flattening process is not possible for the deltahedral boranes $B_nH_n^{2-}$ ($6 \leq n \leq 12$) in which the external electrons are used for the B−H bonds.

3. Flattened Polyhedral Rhodium−Nickel Carbonyl Clusters

3.1. Connection between $Rh_5Ni_6(CO)_{21}^{3-}$ and E_{11}^{7-} (E = Ga, In, Tl). The structures of E_{11}^{7-} [34−36] and $Rh_5Ni_6(CO)_{21}^{3-}$ [25] can both be described as a trigonal bipyramid surrounded by a trigonal prism (Figures 2b and 3). Furthermore, these two species are isoelectronic with 24 skeletal electrons in accord with the $2n + 2$ ($n = 11$) skeletal electrons required by the Wade−Mingos rules for closed deltahedra without degree 3 vertices.[9−11] Thus, first consider electron counting in the E_{11}^{7-} clusters, in which the eight normal vertices can be considered each to be a donor of 1 skeletal electron and the three flattened vertices each to be a donor of 3 skeletal electrons. This leads to $(8)(1) + (3)(3) + 7 = 24 = 2n + 2$ skeletal electrons for $n = 11$ required by the Wade−Mingos rules[9−11] after allowing for the −7 charge on the E_{11}^{7-} anion.

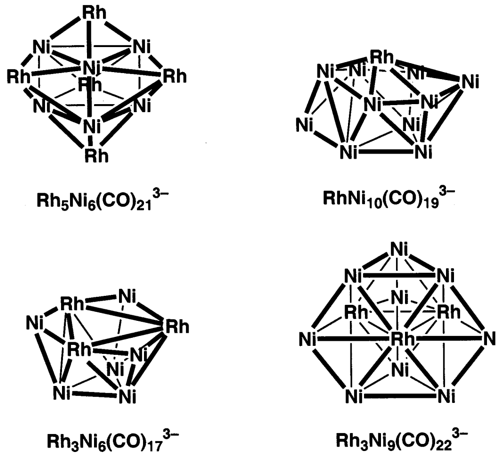

Figure 3. Metal frameworks of the four mixed rhodium−nickel carbonyl cluster anions discussed in this paper.

A completely analogous method can be used for electron counting in the metal carbonyl cluster anion $Rh_5Ni_6(CO)_{21}^{3-}$. For electron counting purposes this structure can be dissected into 5 $Rh(CO)_2$ + 6 $Ni(CO)_2$ − CO + (3−). The three equatorial $Rh(CO)_2$ vertices provide four internal orbitals for the skeletal bonding because of their flattening so that each such vertex becomes a donor of three skeletal electrons. However, the two axial $Rh(CO)_2$ vertices provide only the usual three internal orbitals, and thus, each are donors of only a single skeletal electron. All six $Ni(CO)_2$ vertices provide the usual three internal orbitals, and thus, each is a donor of two skeletal electrons. This leads to the following electron counting scheme:

6 $Ni(CO)_2$ vertices: $6 \times 2 =$	12 electrons
3 equatorial (flattened) $Rh(CO)_2$ vertices: $3 \times 3 =$	9 electrons
2 axial $Rh(CO)_2$ vertices: $2 \times 1 =$	2 electrons
deficiency of one CO group:	−2 electrons
−3 charge	3 electrons
total available skeletal electrons:	24 electrons

Thus, the cluster $Rh_5Ni_6(CO)_{21}^{3-}$, like the isostructural clusters E_{11}^{7-} (E = Ga, In, Tl), has the 24 skeletal electrons corresponding to $2n + 2$ for an 11-vertex deltahedron with globally delocalized bonding.

3.2. Another 11-Vertex Cluster, $RhNi_{10}(CO)_{19}^{3-}$. The 11-vertex cluster[39] $RhNi_{10}(CO)_{19}^{3-}$ can be considered to be a η^6-rhodium complex of an Ni_9 polyhedral network with an open hexagonal face (Figures 3 and 4). The tenth nickel atom caps one of the faces of the Ni_9 network. The $RhNi_{10}(CO)_{19}^{3-}$ structure is thus derived from a 10-vertex *isocloso* deltahedron of ideal C_{3v} symmetry. However, this 3-fold symmetry is broken by the additional nickel cap. Furthermore, the unique degree 6 rhodium vertex in this structure is pushed toward the center of the polyhedron so that it can be considered to be a flattened vertex and thus provide four rather than three internal orbitals and hence an

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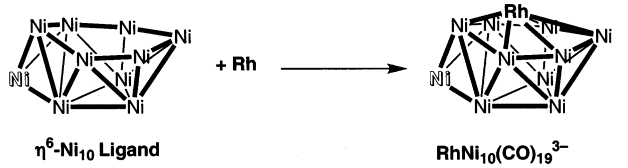

Figure 4. Considering $RhNi_{10}(CO)_{19}^{3-}$ as a rhodium complex of a η^6-Ni_{10} ligand. The unique nickel atom capping one of the triangular faces of the nickel polyhedron is indicated in shadow type (Ni).

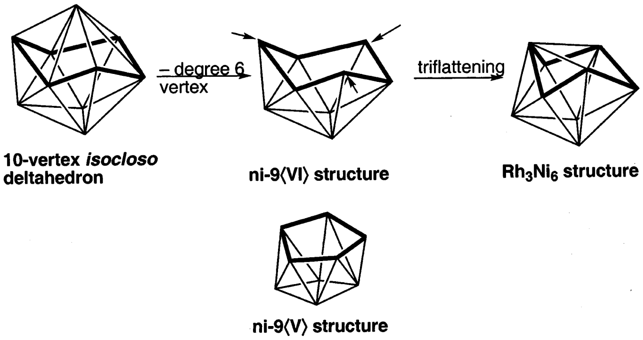

Figure 5. (a) Removal of the degree 6 vertex from the 10-vertex *isocloso* deltahedron to give a ni-9⟨VI⟩ structure followed by flattening alternate vertices in the open hexagonal face to give the Rh_3Ni_6 structure observed in $Rh_3Ni_6(CO)_{17}^{3-}$. (b) ni-9⟨V⟩ structure found in $B_9H_{12}^-$. Edges forming the "open" faces in the ni-9⟨VI⟩ and ni-9⟨V⟩ structures are indicated by bold lines.

extra electron pair to the skeletal bonding. The flattening of the rhodium vertex distorts the hexagonal face of the underlying Ni_9 polyhedron to which the rhodium is bonded.

The *isocloso* deltahedra of metallaboranes with *n* vertices have $2n$ rather than the $2n + 2$ skeletal electrons required by Wade−Mingos's rules. This has been attributed to more localized skeletal bonding consisting of *n* three-center two-electron bonds in *n* of the $2n - 4$ faces of the deltahedron.[40] In $RhNi_{10}(CO)_{19}^{3-}$ the required 20 ($=2n$ for $n = 10$) skeletal electrons can arise from the following electron counting scheme:

10 normal $Ni(CO)_2$ vertices (3 internal orbitals): $10 \times 2 =$	20 electrons
1 flattened $Rh(CO)_2$ vertex (4 internal orbitals): $1 \times 3 =$	3 electrons
deficiency of 3 CO groups: $-3 \times 2 =$	−6 electrons
−3 charge on anion	3 electrons
total skeletal electrons:	20 electrons

3.3. Nine-Vertex Cluster $Rh_3Ni_6(CO)_{17}^{3-}$. The structure of the nine-vertex cluster $Rh_3Ni_6(CO)_{17}^{3-}$ (Figure 3) can be derived from a *nido* polyhedron having a hexagonal open face with alternating nickel and rhodium vertices.[39] This nine-vertex *nido* polyhedron is obtained by the following sequence of operations:

(1) A triangular face in a regular icosahedron is first replaced by a single vertex.

(2) Enough edges are then added to this new vertex to make all new faces triangles while retaining the 3-fold symmetry. This gives the 10-vertex *isocloso* deltahedron.

(3) The degree 6 vertex is removed from this 10-vertex *isocloso* deltahedron (Figure 5a).

Flattening of the three rhodium vertices in this open hexagonal face of this nine-vertex *nido* polyhedron divides this face into four triangular faces by forming three new Rh−Rh edges. The electron counting in $Rh_3Ni_6(CO)_{17}^{3-}$ can then proceed as follows:

6 normal $Ni(CO)_2$ vertices (3 internal orbitals): $6 \times 2 =$	12 electrons
3 flattened $Rh(CO)_2$ vertices (4 internal orbitals): $3 \times 3 =$	9 electrons
deficiency of one CO group: $-1 \times 2 =$	−2 electrons
−3 charge on anion	3 electrons
total skeletal electrons:	22 electrons

The resulting 22 skeletal electrons correspond to the $2n + 4$ skeletal electrons required for a nine-vertex *nido* structure (i.e., $n = 9$). The 3-fold symmetry of the triflattened *nido* structure of $Rh_3Ni_6(CO)_{17}^{3-}$ derives from a ni-9⟨VI⟩ structure in the nomenclature of Williams[41] and thus differs from the ni-9⟨V⟩ structure with a pentagonal open face in the known[42] nine-vertex borane $B_9H_{12}^-$ (Figure 5b).

3.4. Triflattened Icosahedron $Rh_3Ni_9(CO)_{22}^{3-}$. The structure of $Rh_3Ni_9(CO)_{22}^{3-}$ [39] can be derived from a Ni_9-Rh_3 icosahedron with the three rhodium vertices related by C_3 symmetry so that the 5-fold axis of a regular icosahedron is lost but a C_3 axis is retained so that the point group of the Ni_9Rh_3 framework is D_{3h} (Figure 3). Furthermore, the three rhodium vertices are flattened so that the Rh−Rh distances through the icosahedron (2.92 Å) fall within the range of the lengths of the 30 icosahedral edges (Rh−Ni and Ni−Ni distances from 2.44 to 3.06 Å). The electron counting in $Rh_3Ni_9(CO)_{22}^{3-}$ can proceed as follows:

9 normal $Ni(CO)_2$ vertices (3 internal orbitals): $9 \times 2 =$	18 electrons
3 flattened $Rh(CO)_2$ vertices (4 internal orbitals): $3 \times 3 =$	9 electrons
deficiency of two CO groups: $-2 \times 2 =$	−4 electrons
−3 charge on anion	3 electrons
total skeletal electrons:	26 electrons

The resulting 26 skeletal electrons correspond to the $2n + 2$ skeletal electrons required for a *closo* icosahedral structure (i.e., $n = 12$).

The triflattened icosahedral structure of $Rh_3Ni_9(CO)_{22}^{3-}$ retains the 3-fold but destroys the 5-fold symmetry of the underlying regular icosahedron. Losing the 5-fold symmetry allows the Rh_3Ni_9 unit of the $Rh_3Ni_9(CO)_{22}^{3-}$ structure to form an infinite lattice. In this sense this Rh_3Ni_9 unit corresponds to a piece of the hexagonal close-packed metal lattice as suggested by the discoverers of the $Rh_3Ni_9(CO)_{22}^{3-}$ structure.[39] Also the fact that a mixed icosahedral cluster of rhodium and nickel can undergo flattening whereas no examples of flattened group 13 icosahedral clusters are known indicates the greater flexibility of transition metal carbonyl clusters. This may relate to the presence of several external lone pairs on most transition metal carbonyl vertices using nine-orbital sp^3d^5 manifolds in contrast to bare post-transition metal vertices, such as indium and thallium, using only four-orbital sp^3 manifolds with only a single external lone pair.

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4. Summary

The mixed rhodium−nickel carbonyl clusters discussed in this paper all exhibit the following structural features: (1) a polyhedron with 3-fold symmetry as the underlying structural unit; (2) flattening of all or at least most of the rhodium vertices in this polyhedron thereby leading to an "extra" skeletal electron pair from each of the flattened vertices; (3) skeletal electron counts corresponding to those expected from the Wade−Mingos rules after the "extra" electron pairs from the flattened vertices are considered. Furthermore, these rhodium−nickel carbonyl clusters appear to be closely related to clusters of boron and its heavier congeners such as indium and thallium. More generally, the novel flattened polyhedra found in the currently known mixed rhodium−nickel carbonyl clusters suggests that mixed metal carbonyl clusters might be prolific sources of structures based on unusual polyhedra of various types.

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