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Structure and Bonding in Dialkyltin Tetracarbonyliron Dianions

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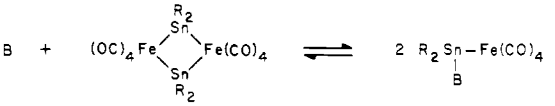
Molecular orbital calculations of the extended Hückel type were carried out on $(H_2Sn)Fe(CO)_4{}^{2-}$. The most stable geometry corresponds to a trigonal-bipyramidal structure with a highly pyramidal SnH_2 group at an axial position. The extra two electrons are essentially localized in a hybrid orbital on tin. Another stable geometry has a basically trigonal SnH_2 group with the two electrons in an a_2 equatorial-carbonyl-based orbital. There is a symmetry-imposed barrier on going from one geometry to another. In other words, there is a highly unusual double-well potential for pyramidalization at tin. Other geometries of this system and other complexes were studied in a similar vein.

It has been known for some time that Lewis bases cleave $(R_2Sn)Fe(CO)_4$ dimer (**1**) to form the monomeric adducts (**2**).[2,3] One of us recently reported[4] that when metal hydrides



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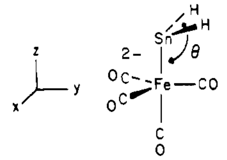
were used a reduction occurred, yielding a *monomeric* $(R_2Sn)Fe(CO)_4{}^{2-}$ complex and molecular hydrogen. The geometric and electronic structure of this novel dianion is the focus of this work. We had anticipated that the electropositive SnR_2 group would prefer an axial site over an equatorial one in a trigonal-bipyramidal structure. The theoretical basis for this is well-known.[5] Furthermore, we expected the SnR_2 group to be highly pyramidal with the extra two electrons in a hybrid orbital.[6] The structure would then be equivalent to a transition-metal carbanion for which there are no currently known examples. Molecular orbital calculations of the extended Hückel type, with details given in the Appendix, were carried out to check this prediction for $(H_2Sn)Fe(CO)_4{}^{2-}$. The pyramidality angle, defined in **3** as the angle between the Fe–Sn bond and the plane of the SnH_2 group, was varied along with the H–Sn–H angle so that when θ = 180°, for a trigonal structure, the H–Sn–H angle was 120°, and when θ = 90°, the H–Sn–H angle was 90°. The ground-state geometry was calculated to be one with θ = 98°, represented by **4**. But, to our surprise, another minimum appears on the potential surface for pyramidalization. The SnH_2 group is nearly trigonal (θ = 150°) in this structure, represented by **5**. There is a barrier of 8 kcal/mol for going from **4** to **5** with **4** about 3 kcal/mol more stable than **5**. The electronic structure of **5** around the SnH_2 group resembles that of a monomeric stannylene (or carbene) complex. When the SnH_2 is at or nearly at planarity, the p orbital at tin is formally empty with the extra two electrons confined to the $Fe(CO)_4$ portion.



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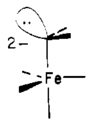

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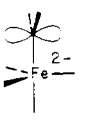

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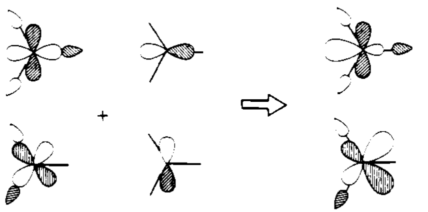
To understand how this comes about, let us first develop the important valence orbitals of **5** by interacting the valence orbitals of an $Fe(CO)_4$ fragment with those of SnH_2. This is done in Figure 1. On the right side are the orbitals of a C_{3v} $Fe(CO)_4$.[7] With use of the coordinate system in **3**, 1e is primarily xz and yz,[8] stabilized slightly by the carbonyl π^* orbitals. At higher energy is 2e, which is comprised primarily of $x^2 - y^2$ and xy mixed in an antibonding way with carbonyl σ. A top view is shown in **6**. Furthermore, some metal x and y is mixed in a bonding way to carbonyl σ, hybridizing 2e away from the equatorial carbonyls, **7**. At somewhat higher energy is a_1. This is an orbital, mainly of z^2, with some s and z mixed in to hybridize the orbital away from the carbonyls. Finally, at even higher energy is a carbonyl π^* orbital of a_2 symmetry. That orbital is well separated from the other carbonyl-based ones since there is no orbital at the metal of the same symmetry to destabilize it. The hybridized orbital labeled n and nonhybridized p level are the valence orbitals of SnH_2 displayed on the left side of Figure 1. One component of 1e, both orbitals of 2e, and a_2 are nonbonding. They give the molecular levels 1a″, 3a′, 2a″, and 3a″, respectively. The n orbital of SnH_2 interacts strongly with a_1 to produce a Fe–Sn σ level, 2a′.[9]



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(1) (a) University of Houston. (b) Rice University. (c) Camille and Henry Dreyfus Teacher-Scholar, 1979–1984.

(2) (a) Marks, T. J.; Newman, A. R. *J. Am. Chem. Soc.* **1973**, *95*, 769. Marks, T. J.; Grynkewich, G. W. *J. Organomet. Chem.* **1975**, *91*, C9. (b) Grynkewich, G. W. *Inorg. Chem.* **1976**, *15*, 1307. (c) Cornwell, A. B.; Harrison, P. G.; Richards, J. A. *J. Organomet. Chem.* **1976**, *108*, 47 and references therein. (d) Taylor, M. J. "Metal-to-Metal Bonded States of the Main Group Elements"; Academic Press: New York, 1975; pp 107–114.

(3) For structures of **1** see: (a) Gilmore, C. J.; Woodward, P. *J. Chem. Soc., Dalton Trans.* **1972**, 1387. (b) Sweet, R. M.; Fritchie, C. J.; Schunn, R. A. *Inorg. Chem.* **1967**, *6*, 749. For other related examples see: (c) Watkins, S. F. *J. Chem. Soc. A* **1969**, 1552. Neustadt, R. J.; Cymbaluk, T. H.; Ernst, R. D.; Cagle, F. W., Jr. *Inorg. Chem.* **1980**, *19*, 2375 and references therein.

(4) Sosinsky, B. A.; Shelly, J.; Shong, R. *Inorg. Chem.* **1981**, *20*, 1370.

(5) Rossi, A. R.; Hoffmann, R. *Inorg. Chem.* **1975**, *14*, 365.

(6) The sodium cations would probably be tightly associated with the lone pair at tin along with an ethereal solvent. For a review of analogous structures see: Jonas, K.; Krüger, C. *Angew. Chem.* **1980**, *92*, 513; *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 520.

(7) (a) Elian, M.; Hoffmann, R. *Inorg. Chem.* **1975**, *14*, 1058. (b) Burdett, J. K. *J. Chem. Soc., Faraday Trans. 2* **1974**, *70*, 1599. Böhm, M. C.; Daub, J.; Gleiter, R.; Hofmann, P.; Lappert, M. F.; Öfele, K. *Chem. Ber.* **1980**, *113*, 3629. (d) Hoffmann, R.; Chen, M. M. L.; Elian, M.; Rossi, A. R.; Mingos, D. M. P. *Inorg. Chem.* **1974**, *13*, 2666.

(8) Here xz, yz, xy, $x^2 - y^2$, and z^2 stand for the nd orbitals and x, y, and z for the $(n + 1)$p orbitals.

(9) One might think that 2a′ lies quite high in energy for a σ level. This is due to a very electropositive tin atom, which means that n is very high in energy to begin with. The ionization potential for an R_2Sn compound (R = $(Me_3Si)_2CH$) was found to be 7.42 eV.[10] Putting 2a′ just below the 2e set is, therefore, not unreasonable. That corresponding level for $(R_2C)Fe(CO)_4$ complexes lies just below the 1e set; see ref 7c for the PE spectra.

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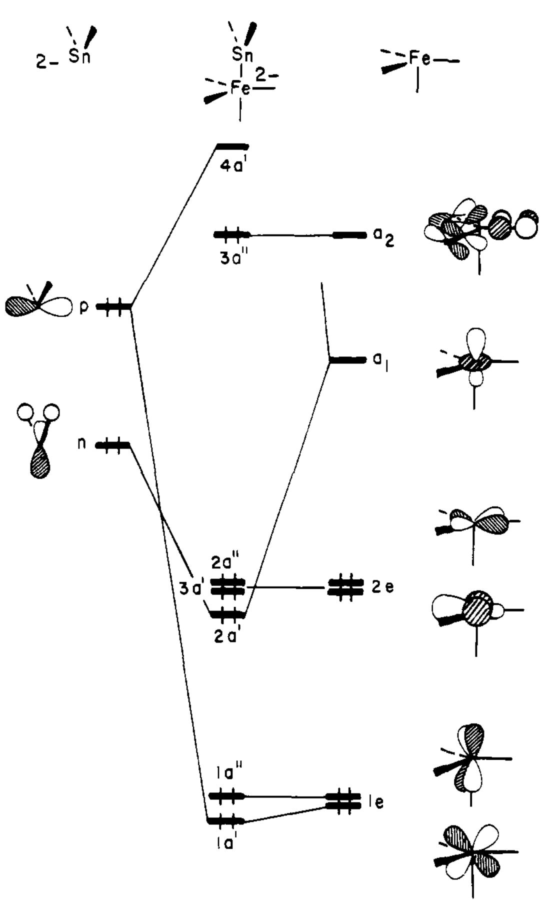
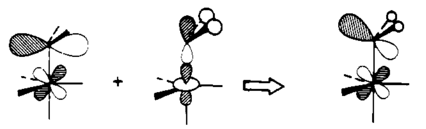


Figure 1. Orbital interaction diagram for a trigonal $(H_2Sn)Fe(CO)_4^{2-}$ complex.

The other component of 1e interacts with the SnH_2 p orbital, giving a bonding (1a′) and antibonding (4a′) level. The important point is that the p level starts off so high in energy (the ionization potential for a 5p level at atomic tin is 7.32 eV)[10] that the antibonding combination, 4a′, lies higher than 3a″ for a trigonal geometry at SnH_2. The two extra electrons then go into 3a″ rather than 4a′.

Figure 2 shows the evolution for each of the valence orbitals in $(H_2Sn)Fe(CO)_4^{2-}$ as a function of θ, defined in **3**. As θ decreases, the antibonding analogue of 2a′ mixes into 4a′, **8**,



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sending the latter to lower energy. Levin and others have treated this sort of pyramidal inversion problem in some depth.[11] The a_2 $Fe(CO)_4$ level, 3a″, is unaffected by the distortion. Therefore, a crossing between 4a′ and 3a″ occurs; we calculate that to be at $\theta = 121°$. Pyramidalization of the SnH_2 group is a symmetry-forbidden process. Thus, a barrier is realized in going from **5** to **4**. The other levels remain relatively constant in energy on variation of θ except 3a′. The 3a′ level goes up in energy primarily because some SnH_2 p is mixed into one component of 2e in a bonding manner. Recall that 2e contains metal x and y (**7**). The Fe–Sn σ bond is also slightly weakened on decreasing θ. Instead of 2a′ going up in energy, it transmits the effect to 3a′. A few of the levels lower in energy than those displayed in Figure 2 go down in energy very slightly on pyramidalization. This gives a minimum for **5** at a value of θ less than 180° (150°). This is not unlike the pyramidization seen for the dimer of $[(Me_3Si)_2CH]_2Si$.[12] The orbitals are primarily metal carbonyl σ in origin. The surface for **5** (with 3a″ filled) is very soft and will be sensitive to steric factors. With larger, more realistic R groups at tin, the structure of **5** will become more trigonal.

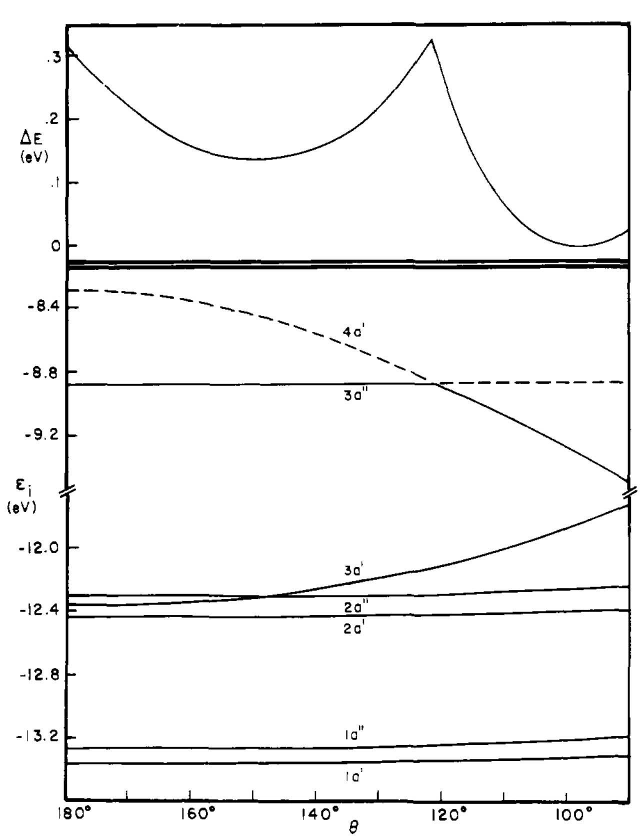

Figure 2. Top: Variation of the relative total energy vs. θ, defined in **3**. Bottom: Plot of the energy variation for each of the valence orbitals in Figure 1. The dashed lines indicate those levels that are empty.

Before turning to alternative geometries, let us further pursue the electronic ramifications of **4** and **5**. In **4**, with 4a′ filled and 3a″ empty, the $Fe(CO)_4$ portion of the molecule is relatively normal. Iron gains ~0.11 e by donation from the hybrid orbital on tin. With 4a′ filled and 3a″ empty in **5**, there are a number of substantial electronic changes. Tin loses 1.49 e in charge. That is redistributed entirely on the equatorial carbonyls. Population of 3a″ also means that the C–O bond length should become longer. The average C–O overlap population for the equatorial carbonyls in **4** is 1.139. That decreases to 0.968 on going to optimized **5**. Note that **5** is not a 20-electron complex. The two additional electrons are in an orbital that is nonbonding with respect to the metal. There are a number of other complexes where one symmetry-adapted combination of ligand donor orbitals does not match any metal atomic function, most notably $(Ph_2C_2)_3W(CO)$ and Cp_3ZrR, and are reminiscent of the situation here.[13]

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(10) Harris, D. H.; Lappert, M. F.; Pedley, J. B.; Sharp, G. J. *J. Chem. Soc., Dalton Trans.* **1976**, 945.

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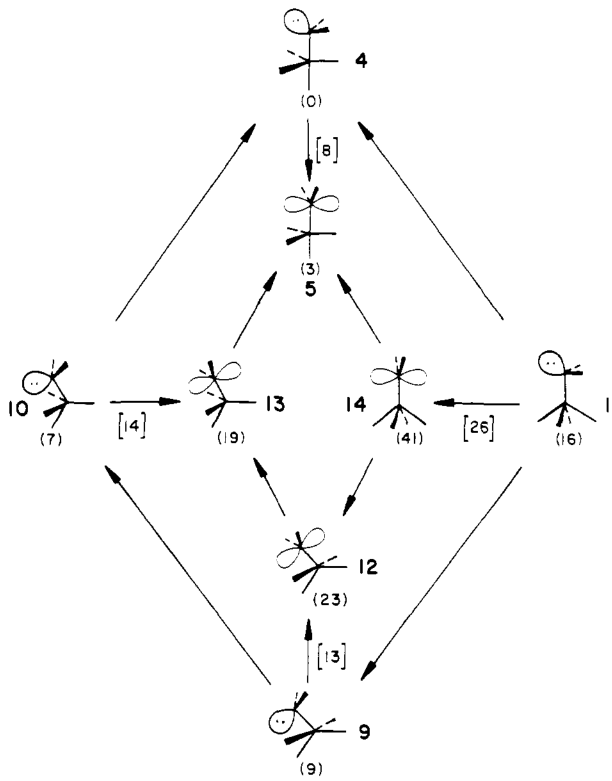
Chart I $(H_2Sn)Fe(CO)_4{}^{2-}$ Isomers



That donor orbital would lie at low energy, of course. It has been proposed on the basis of ESR and electron spectroscopy data for D_3 $M(bpy)_3$[14] and $M(quinone)_3$[15] complexes that an a_2 orbital lies not far in energy to the e + a_1 (t_{2g} in an octahedron) set. This a_2 orbital is a ligand π^* one, which does not find a symmetry match with the metal orbitals. In our case putting 4a′ above 3a″ is sensitive to the parameters used. We will return to this point.

We also investigated other possible geometries for $(H_2Sn)Fe(CO)_4{}^{2-}$. These are diagrammed in Chart I. The numbers in parentheses under each structure refer to the total energies in kcal/mol relative to the most stable structure, **4**. The outer set of four structures are the two trigonal-bipyramidal (TBP), **4** and **9**, and two square-pyramidal (SP) isomers, **10** and **11**, for an optimized pyramidal SnH_2 unit. For them θ (see **3**) ranges from 96–98°. The inner quadrant corresponds to optimized trigonal isomers with θ ranging from 149 to 160°. Nothing particularly unusual is found in this set of Berry pseudorotations. The relative stabilities are a reflection of the fact that the SnH_2 group is very electropositive. We refer the reader to an excellent theoretical treatment of substituent effects in TBP and SP isomers by Rossi and Hoffmann.[5] A set of ab initio calculations[16] on $CH_3Co(CO)_4$, which should mimic our pyramidal isomers, also give the axially substituted TBP isomer as the most stable one. The SP isomer analogous to **10** and the TBP isomer analogous to **9** were 8 and 10 kcal/mol, respectively, less stable. The relative stabilities of the trigonal isomers, **5** and **12–14**, again follow typical site preferences in pentacoordinate transition-metal complexes. In each case there is a barrier for going from the pyramidal to the trigonal isomer. That barrier (kcal/mol) is given in brackets in Chart I. In three cases (**4** to **5**, **7** to **13**, and **11** to **14**) the barrier is rigorously symmetry imposed. The HOMO for each of the trigonal isomers (and in **12**) is carbonyl based. They are equivalent to the 3a″ level in **5** in that there is essentially no contribution from the metal or SnH_2 and they are antisymmetric to the mirror plane of the molecule. In the **12** to **9** conversion the reaction is technically symmetry allowed but the crossing is weakly avoided and so there is again a barrier.[16] According to our calculations, bending one or all three carbonyls in the axial plane for the trigonal isomer, **5**, is destabilizing.

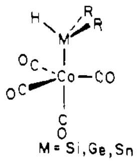
Table I. Parameters for the Extended Hückel Calculations

orbital	H_{ii}, eV	ζ_1	ζ_2	$C_1{}^a$	$C_2{}^a$
Fe 3d	−12.70	5.35	1.80	0.5366	0.6678
4s	−9.17	1.90			
4p	−5.37	1.90			
Cr 3d	−11.22	4.95	1.60	0.4876	0.7205
4s	−8.66	1.70			
4p	−5.24	1.70			

orbital	H_{ii}, eV	ζ_1	orbital	H_{ii}, eV	ζ_1
Sn 5s	−16.16	2.12	N 2s	−26.00	1.95
5p	−8.32	1.82	2p	−13.40	1.95
C 2s	−21.40	1.625	O 2s	−32.30	2.275
2p	−11.40	1.625	2p	−14.80	2.275

[a] Contraction coefficients used in the double ζ expansion.

Monomeric stannylene complexes with two electrons less follow the pattern exhibited by the trigonal complexes in the inner quadrant of Chart I. Optimum values of θ range from 147 to 150°. The most stable structure is an axial TBP one analogous to **5**. It may be possible to prepare monomeric stannylene–$Fe(CO)_4$ complexes where the substituents at tin are extraordinarily bulky to prevent dimerization. Low-temperature electrochemical reduction would generate a dianion of type **5**. Reduction of the dimer, **1**, would populate a Fe–Sn antibonding σ orbital, probably of b_{3u} symmetry.[18] The dimer should decompose to reduced monomeric units; their entrance onto the surface outlined in Chart I would then correspond to the pyramidal form, **9**, and facile pseudorotation to **4** should occur. Deprotonation of **15**[19] should also lead to complexes



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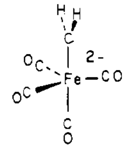

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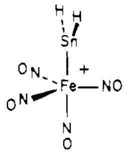

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related to **4**. A calculation on trigonal $(CH_2)Fe(CO)_4{}^{2-}$ (**16**) comes out differently in one important respect to the tin analogue. When $\theta = 180°$, 4a′ lies lower in energy than 3a″ (see Figure 1). The p orbital on CH_2 starts off at a considerably lower energy than that in SnH_2. Variation of θ in **16** will show only one minimum at a pyramidal geometry. Therefore, the ordering of 4a′ and 3a″ is parameter dependent and we encourage higher level calculations to be carried out on **3** and **16**. Cowley, Kemp, and Wilburn have recently prepared a complex with one electron less than our system,

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(14) Hanazak, I.; Nagakura, S. *Inorg. Chem.* **1969**, *8*, 648, 654; *Bull Chem. Soc. Jpn.* **1971**, *44*, 2312.

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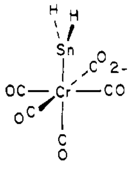
(16) Demuynck, J.; Strich, A.; Veillard, A. *Nouv. J. Chim.* **1977**, *1*, 217.

(17) Equatorially substituted TBP isomers where the SnH_2 was rotated by 90° from that in **9** and **12** were also investigated. The relative energies and barrier are very similar to those for **9** and **12** in Chart I.

(18) For a discussion of the bonding in complexes related to **1** see: Shaik, S.; Hoffmann, R.; Fisel, C. R.; Summerville, R. H. *J. Am. Chem. Soc.* **1980**, *102*, 4555.

(19) For the Si compounds see, for example: Haggen, A. P.; McDiarmid, A. G. *Inorg. Chem.* **1967**, *6*, 686. There appear to be no examples of Ge or Sn hydrides; however, general routes to such complexes are well-known: Patmore, D. J.; Graham, W. A. G. *Ibid.* **1967**, *6*, 981; **1968**, *7*, 771. Lichtenberger, D. L.; Brown, T. L. *J. Am. Chem. Soc.* **1977**, *99*, 8187.

namely, $[(Me_3Si)_2CH]_2PFe(CO)_4$.[20] The alkyl groups at phosphorus are very bulky. Their experimental results nicely agree with our contention that in a trigonal isomer 3a″ may lie lower than 4a′. The ESR spectrum of their compound shows no observable hyperfine coupling to ^{31}P or ^{1}H. Furthermore, narrow lines are observed in the ^{31}P, ^{13}C, and ^{1}H NMR spectra for the ligand portion of their molecule. This is not consistent with placing the odd electron in 4a′—an orbital that would be heavily weighted on phosphorus. The Mössbauer spectrum suggests that the extra electron does not reside at the metal. Finally, the CO stretching frequencies are lowered by ~100 cm^{-1} from analogues. This is compatible with 3a″ being singly occupied. As we pointed out earlier, since 3a″ is carbonyl π^* based, one would expect the equatorial bond length and stretching frequencies to be increased upon occupation. An optimal case for the occupation of 3a″ rather than 4a′ would be one where the equatorial carbonyls of **3** were replaced by isoelectronic NO^+ ligands. The more electronegative nitrogen lowers the energy of π^* significantly. This means that a_2 (see Figure 1) will go down in energy greatly. Our calculations on **17** confirm this. In fact, at all reasonable values of θ, 3a″ lies lower than 4a′. One minimum is then found for pyramidalization (θ = 147°). Another complex where we have found a similar pattern is $(H_2Sn)Cr(CO)_5^{2-}$ (**18**). **19** is found to lie lower in energy than **20**. Several complexes with two electrons less are known.[21] Low-temperature electrochemical reduction studies on them would be very interesting. We again calculate that in the carbon analogue of **18** there is a reversal of level ordering. The level **20** is calculated to lie lower than **19**. One-electron reduction of $(R_2C)M(CO)_5$ (M = Cr, Mo, W) complexes yields a species that has been investigated by ESR.[22] In agreement with our results the unpaired electron resides in **20** rather than **19**.



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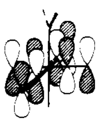

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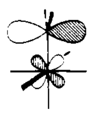

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Appendix

All calculations were performed with use of the extended Hückel method.[23] The H_{ii}'s and orbital exponents listed in Table I were taken from other work.[24] The modified Wolfsberg–Helmholz formula[25] was used. All Fe–C, Cr–C, C–O, M–Sn, Fe–N, N–O, Sn–H, and C–H distances were set at 1.78, 1.84, 1.14, 2.54, 1.70, 1.17, 1.70, and 1.09 Å, respectively. The M–C–O and Fe–N–O angles were held at 180°.

Registry No. $(H_2Sn)Fe(CO)_4^{2-}$, 78939-73-8.

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(23) Hoffmann, R. *J. Chem. Phys.* **1963**, *39*, 1397. Hoffmann, R.; Lipscomb, W. N. *Ibid.* **1962**, *36*, 3179; **1962**, *37*, 2872.

(24) Albright, T. A.; Hofmann, P.; Hoffmann, R. *J. Am. Chem. Soc.* **1977**, *99*, 7546. Corran, P. J. Ph.D. Thesis, University of Oklahoma, 1979.

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Preparation and Properties of Some Low-Spin Octahedral [*o*-Phenylenebis(dimethylarsine)]iron(II) Alkyl and η^1-Acyl Complexes. Carbonyl Insertion/Extrusion Reactions

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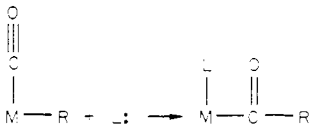
Reaction of CH_3OSO_2F with $(DIARS)Fe(CO)_3$ (DIARS = *o*-phenylenebis(dimethylarsine)) results in simple oxidative addition to give $[(DIARS)Fe(CO)_3CH_3]^+$. With CH_3I or CF_3I initial oxidative addition is followed by iodide-promoted migratory carbonyl insertion to form iodo η^1-acyl or η^1-perfluoroacyl complexes. The acyl $(DIARS)Fe(CO)_2(COCH_3)I$ does not readily decarbonylate even under forcing conditions. Halide abstraction of the η^1-acyl produces a coordinatively unsaturated intermediate, which extrudes CO to give *fac*-$[(DIARS)Fe(CO)_3CH_3]^+$. The cationic CO extrusion product readily undergoes migratory CO insertion in the presence of both neutral and anionic Lewis bases.

Introduction

An understanding of the factors determining stability/reactivity patterns of metal–carbon σ bonds is fundamental for meaningful extensions of most catalytic cycles involving transition metals. Carbonyl insertion,[1,2] eq 1, wherein a coordinated CO molecule formally "inserts" into a metal–carbon σ bond provides for the formation of value added oxygenated products in the hydroformylation process,[3a] the catalytic carbonylation of methanol,[3b] and the chain propagation steps



$$\overset{\overset{O}{|||}}{\underset{|}{C}}\!\!-\!M{-}R + L\!: \longrightarrow M(L){-}C(=O){-}R \qquad (1)$$

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