a singlet assigned to cyclopentadienyl protons at 4.96 ppm in addition to Ph multiplets.

The X-ray diffraction study of a single crystal of **2a**·CH_2Cl_2[7] shows that a molecule of the cluster bears two η-C_5Me_5 and two diphenyldithiolene ligands on the four iron atoms, and its Fe_4S_4 core is highly distorted (Figure 1): Three Fe–Fe distances (Fe(1)–Fe(3), Fe(3)–Fe(4), and Fe(4)–Fe(2)) are much shorter (2.711 (1)–2.789 (1) Å) than the other three (3.255 (1)–3.400 (1) Å). The shorter Fe–Fe distances are somewhat longer than Fe–Fe single bond distances found in $[(\eta\text{-}C_5H_5)_4Fe_4S_4]$[4] and $[Fe_4(NO)_4S_4]$[8] (average 2.65 Å), but shorter than those with a bond order of $^3/_4$ in $[(\eta\text{-}C_5H_5)_4Fe_4S_4]^{2+}$ (2.83 Å).[9] The longer Fe–Fe distances correspond to the absence of interiron bonds. This localization of Fe–Fe bonds renders the top four-membered ring of Fe_2S_2 and the bottom one twisted with respect to each other by ca. 18°. Interestingly, the top and bottom four-membered rings of Fe_2S_2 are nearly planar[10] in contrast to those of other iron–chalcogen cubane clusters.[4,8,9,11-13]

The core structure of **2a** with three Fe–Fe single bonds is consistent with the metal fragment orbital model,[3,14] which predicts that the total bond order becomes 3 for this type of cubane cluster with 18 metal electrons: 12 electrons occupy the six metal–metal bonding orbitals, and the remaining six electrons occupy the low-lying metal–metal antibonding orbitals. There are two other iron–sulfur cubane clusters known to have 18 metal electrons and a total M–M bond order of 3, i.e., $[(\eta\text{-}C_5H_5)_4Fe_4S_4]^{2+}$[9] and $[Fe_4(S_2C_2(CF_3)_2)_4S_4]^{2-}$,[11] but, unlike **2a**, these clusters have four equivalent Fe–Fe bonds with a bond order of $^3/_4$ probably due to the higher symmetry of the cluster molecule.

Each of the cyclic voltammograms of **2a** and **2b** in a 0.1 M $(n\text{-}Bu)_4NBF_4/CH_2Cl_2$ solution exhibits four reversible redox waves, indicating the existence of five discrete species $[Cp^*_2(Ph_2C_2S_2)_2Fe_4S_4]^n$ for which $n = -2, -1, 0, +1$, and $+2$. The formal potentials for the redox waves of $Cp^*_2(Ph_2C_2S_2)_2Fe_4S_4$ ($E_{1/2}$) are −1.13, −0.55, +0.20, and +0.64 ($Cp^* = \eta\text{-}C_5Me_5$) and −0.92, −0.40, +0.41, and +0.84 ($Cp^* = \eta\text{-}C_5H_5$) V vs SCE, respectively. The $E_{1/2}$'s of **2a** are about 200 mV more negative than those of **2b** and are probably due to the electron-donating effect of 10 methyl groups on the Cp rings of **2a**.

Bulk electrolysis of **2a** was performed at +0.20 V vs SCE on a platinum working electrode in a 1:1 mixture of CH_2Cl_2 and CH_3CN in the presence of 0.1 M NH_4PF_6 as supporting electrolyte. Nearly 1 equiv of charge required for the one-electron oxidation of **2a** was passed, and the salt of the monocationic cluster $[(\eta\text{-}C_5Me_5)_2(Ph_2C_2S_2)_2Fe_4S_4](PF_6)$ (**3**)[15] was obtained as dark purple crystals in 92% yield. The same product was obtained in high yield by the oxidation with ferricinium hexafluorophosphate. The ^1H NMR spectrum of **3** exhibits typical paramagnetic shifts and line broadening.

We have little information so far on the mechanism for the formation of **2**. However, in the early stage of the reaction of **1a**, S_8, and PhC≡CPh, TLC analysis showed a strong spot of $[(\eta\text{-}C_5Me_5)_2Fe_2S_4]$ (**4**), which is known to be formed in high yield by the reaction of **1a** and S_8 under milder conditions (toluene reflux).[16] Furthermore, we observed that the reaction of **4** with PhC≡CPh in a refluxing xylene solution provided **2a**. These results are consistent with a mechanism involving **4** as one of the intermediates.

Supplementary Material Available: Tables of atomic positional and thermal parameters and bond distances and angles for **2a** (17 pages); listing of observed and calculated structure factors for **2a** (43 pages). Odering information is given on any current masthead page.

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(7) Single crystals of **2a**·CH_2Cl_2 were grown by layering ether on the CH_2Cl_2 solution of **2a** at room temperature: space group $P2_1/a$, $a = 20.679$ (3) Å, $b = 20.130$ (3) Å, $c = 12.742$ (2) Å, $\beta = 104.97$ (2)°, $V = 5124$ (1) Å^3, $Z = 4$, $d_{measd} = 1.54$ g cm^{-3}. Diffraction data were collected at 21 °C by the ω–2θ scan technique ($3° < 2\theta < 60°$) using a Rigaku AFC-6A diffractometer with graphite-monochromated Mo Kα radiation. The structure was solved by direct methods (MULTAN) and refined via block-diagonal least-squares methods using anisotropic thermal parameters for non-hydrogen atoms of the cluster molecule and isotropic thermal parameters for hydrogen atoms and for atoms of CH_2Cl_2. Molecules of CH_2Cl_2 were disordered. For 9193 unique reflections with $|F_o| > 3\sigma(F_o)$, final $R = 0.069$ ($R_w = 0.102$).

(8) Chu, C. T.-W.; Lo, F. Y.-K.; Dahl, L. F. *J. Am. Chem. Soc.* **1982**, *104*, 3409–3422.

(9) Trinh-Toan; Teo, B. K.; Ferguson, J. A.; Meyer, T. J.; Dahl, L. F. *J. Am. Chem. Soc.* **1977**, *99*, 408–417.

(10) The maximum deviation is 0.11 Å (S(3)) from the least-squares plane for Fe(1)–S(3)–Fe(2)–S(4) and 0.05 Å (Fe(4)) from the least-squares plane for Fe(3)–S(1)–Fe(4)–S(2).

(11) Lemmen, T. H.; Kocal, J. A.; Lo, F. Y.-K.; Chen, M. W.; Dahl, L. F. *J. Am. Chem. Soc.* **1981**, *103*, 1932–1941.

(12) Trinh-Toan; Fehlhammer, W. P.; Dahl, L. F. *J. Am. Chem. Soc.* **1977**, *99*, 402–407.

(13) Ogino, H.; Tobita, H.; Yanagisawa, K.; Shimoi, M.; Kabuto, C. *J. Am. Chem. Soc.* **1987**, *109*, 5847–5848.

(14) Harris, S. *Inorg. Chem.* **1987**, *26*, 4278–4285.

(15) For **3**: Anal. Calcd for $C_{48}H_{50}F_6Fe_4PS_8·CH_2Cl_2$: C, 44.03; H, 3.92. Found: C, 43.77; H, 3.83.

(16) Brunner, H.; Janietz, N.; Meier, W.; Sergeson, G.; Wachter, J.; Zahn, T.; Ziegler, M. L. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 1060–1061.

Direct Conversion of Vinylic Organocopper Compounds to Allylic Zinc and Copper Organometallics

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Nucleophilic allylic organometallics have proven to be very efficient reagents for the formation of new carbon–carbon bonds with a variety of organic electrophiles.[1] Unfortunately, the most reactive allylic reagents (metal = Li, K, Mg, Zn) are often difficult to prepare due to their high reactivity and to side reactions during their preparation such as Wurtz coupling. Alternative syntheses such as oxidative additions or metalations of allylic halides using activated metals,[2] transmetalations,[3] homologation of vinyl organometallics,[4] and hydrometalation of dienes[5] have been successfully developed.

We report herein a new method allowing a very efficient direct conversion of alkenylcopper[6] compounds **1** to allylic zinc and copper reagents **2** mediated by (iodomethyl)zinc iodide[7] (**3**) (Scheme I). Thus the addition of ICH_2ZnI (**3**) (1.7 equiv) to an alkenylcopper **1** in THF at −30 °C leads to a fast methylene homologation reaction.[8] The resulting highly reactive allylic

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(1) (a) Miginiac, L. In *The Chemistry of the Metal–Carbon Bond*; Hartley, F. R., Patai, S., Eds.; John Wiley and Sons: New York, 1985; Vol. 3, p 99. (b) Courtois, G.; Miginiac, L. *J. Organomet. Chem.* **1974**, *69*, 1. (c) Hoffmann, R. W. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 555. (d) Yamamoto, Y. *Acc. Chem. Res.* **1987**, *20*, 243.

(2) (a) Oppolzer, W.; Schneider, P. *Tetrahedron Lett.* **1984**, *25*, 3305. (b) Bogdanovic, B. *Acc. Chem. Res.* **1988**, *21*, 261. (c) Hartmann, J.; Schlosser, M. *Helv. Chim. Acta* **1976**, *59*, 453. (d) Hartmann, J.; Schlosser, M. *J. Am. Chem. Soc.* **1976**, *98*, 4674. (e) Guo, B.-S.; Cohen, T. *Tetrahedron* **1986**, *42*, 2803. (f) Guo, B.-S.; Doubleday, W.; Cohen, T. *J. Am. Chem. Soc.* **1987**, *109*, 4710.

(3) (a) Seyferth, D.; Jula, T. F. *J. Organomet. Chem.* **1974**, *66*, 195. (b) Seyferth, D.; Mammarella, R. E. *J. Organomet. Chem.* **1979**, *177*, 53. (c) Doucoure, A.; Mauzé, B.; Miginiac, L. *J. Organomet. Chem.* **1982**, *236*, 139. (d) Seyferth, D.; Wursthorn, K. R. *J. Organomet. Chem.* **1977**, *137*, C17. (e) Clarembeau, M.; Krief, A. *Tetrahedron Lett.* **1984**, *25*, 3629. (f) Lipshutz, B. H.; Ellsworth, E. L.; Dimock, S. H.; Smith, R. A. J. *J. Org. Chem.* **1989**, *54*, 4977. (g) Lipshutz, B. H.; Elworthy, T. R. *Tetrahedron Lett.* **1990**, *31*, 477. (h) Lipshutz, B. H.; Elworthy, T. R. *J. Org. Chem.* **1990**, *55*, 1695. (i) Lipshutz, B. H.; Crow, R.; Dimock, S. H.; Ellsworth, E. L.; Smith, R. A. J.; Behling, J. A. *J. Am. Chem. Soc.* **1990**, *112*, 4063.

(4) (a) Villieras, J.; Reliquet, A.; Normant, J. F. *Synthesis* **1978**, 27. (b) Villieras, J.; Reliquet, A.; Normant, J. F. *J. Organomet. Chem.* **1978**, *144*, 263. (c) Hoffmann, R. W.; Landmann, B. *Tetrahedron Lett.* **1983**, *24*, 3209. (d) Wuts, P. G. M.; Thompson, P. A.; Callen, G. R. *J. Org. Chem.* **1983**, *48*, 5398. (e) Negishi, E.; Akiyoshi, K. *J. Am. Chem. Soc.* **1988**, *110*, 646.

(5) Sato, F. *J. Organomet. Chem.* **1985**, *285*, 53.

(6) The insertion reaction using alkenylmagnesium or -lithium compounds instead of the corresponding organocopper does not proceed well; it is not selective and not complete.

(7) (a) Seyferth, D.; Andrews, S. B. *J. Organomet. Chem.* **1971**, *30*, 151. (b) Seyferth, D.; Dertouzos, H.; Todd, L. J. *J. Organomet. Chem.* **1965**, *4*, 18.

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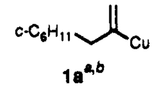
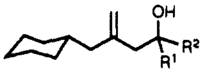
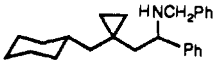
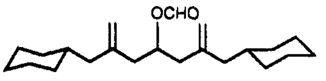
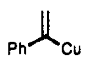
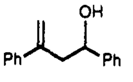
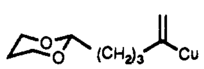
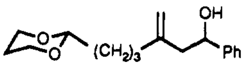
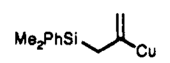
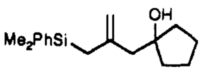
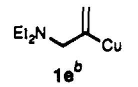
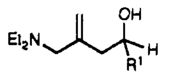
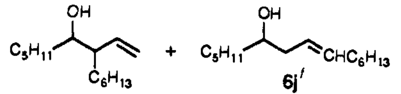
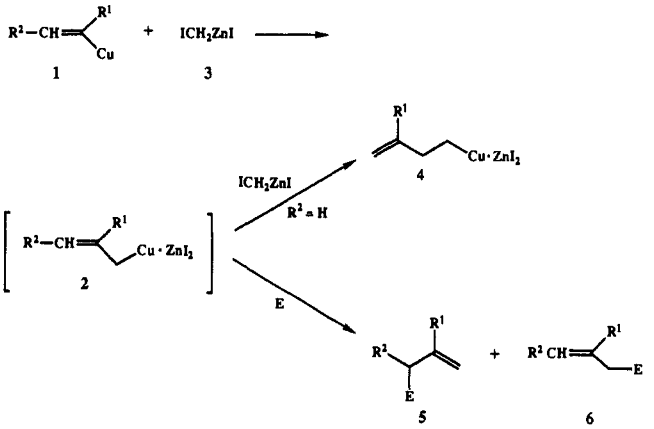
Table I. Preparation of Allylic Organometallics of Type **2** by the Insertion of (Iodomethyl)zinc Iodide (**3**) to the Alkenylcopper Compounds **1a–f** and Their In Situ Trapping by Electrophiles Leading to Products of Type **5** and **6**

entry	alkenylcopper of type **1**	electrophile	product(s)	yield,[d] %
1	 **1a**[a,b]	$C_5H_{11}CHO$	 **5a**: $R^1 = C_5H_{11}$, $R^2 = H$	86
2	**1a**	cyclohexanone	**5b**: $R^1,R^2 = (CH_2)_5$–	91
3	**1a**	$PhCH{=}NCH_2Ph$	 **5c**	76[e]
4	**1a**	HCO_2Et[c]	 **5d**	94
5	 **1b**[b]	PhCHO	 **5e**	93
6	 **1c**[b]	PhCHO	 **5f**	96
7	 **1d**[b]	cyclopentanone	 **5g**	71
8	 **1e**[b]	PhCHO	 **5h**: R^1 = Ph	90
	1e	$NC(CH_2)_2C(Et)_2CHO$[b]	**5i**: $R^1 = C(Et)_2(CH_2)_2CN$	75
10	$C_6H_{13}CH{=}CHCu$ **1f**	$C_5H_{11}CHO$	 **5j**[f] + **6j**[f] 94:6	79

[a] The copper reagent has been prepared in three steps from 2,3-dibromopropene; (i) c-HexCu(CN)ZnI, THF, 0 °C, 5 min; 80%; (ii) Mg, THF, 35 °C, 1 h; >90%; (iii) CuI·2LiI, THF, −25 °C, 10 min. [b] See the Supplementary Material for a detailed preparation. [c] A (1:1) ratio of 2a and ethyl formate has been used. [d] Isolated yields of analytically pure products. [e] An excess of ICH_2ZnI has been used. [f] Mixture of stereoisomers.

Scheme I



organometallic **2**, in the absence of any electrophile, readily inserts a further methylene unit, leading to a homoallylic copper reagent of type **4**.[9] However, if the allylic reagent **2** is generated in the presence of an electrophile (0.6–0.7 equiv), such as an aldehyde, ketone, ethyl formate, or imine, then its trapping by the electrophile was found to be faster than further methylene homologation to **4**, and excellent yields (71–96%) of allylated products of type **5** and **6** could be obtained[10] (Table I). The addition to an imine leads to an intermediate homoallylic amine, which is converted to the cyclopropane derivative **5c** in the presence of an excess of ICH_2ZnI (entry 3), and the addition to ethyl formate affords under our reaction conditions the formylated secondary alcohol **5d** (entry 4). The exact nature of the allylic intermediate **2** is not known, but it should be considered as being a mixed zinc–copper cluster.[11] Of special interest is the use of this method for the preparation of new *functionalized* allylic reagents containing an acetal, allylic silane,[12] or allylic amine (entries 6–9 of Table I). The allylic

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(8) For other homologations using the reagent **3**, see: (a) Knochel, P.; Jeong, N.; Rozema, M. J.; Yeh, M. C. P. *J. Am. Chem. Soc.* **1989**, *111*, 6474. (b) Knochel, P.; Chou, T.-S.; Chen, H. G.; Rozema, M. J. *J. Org. Chem.* **1989**, *54*, 5202.

(9) The conversion of alkynyl and allylic copper–zinc organometallics to methylene-homologated reagents will be reported in a separate paper: Rozema, M. J.; Knochel, P., manuscript in preparation.

(10) Typical procedure: A solution of vinylic Grignard reagent (6 mmol, 11.0 mL) in THF was slowly added to a solution of copper iodide (6 mmol, 1.15 g) and lithium iodide (10 mmol, 1.33 g) in 25 mL of THF at −50 °C. The reaction mixture was warmed to −25 °C, leading to an orange suspension, and was cooled back to −50 °C. A solution of an aldehyde, ketone, imine, or ethyl formate (3.5–4 mmol) in 2 mL of THF was added, and the reaction mixture was warmed to −30 °C. A solution of (iodomethyl)zinc iodide[7] (10 mmol), freshly prepared from diiodomethane (10 mmol, 2.72 g) and zinc foil (11 mmol, 0.72 g) in 5 mL of THF, was added at −50 °C. The reaction mixture was warmed to −20 °C and stirred for 15 min at this temperature. After this time, the reaction was completed as indicated by GLC analysis, and the reaction mixture was worked up as usual, affording after flash chromatography the pure compounds **5** in 71–96% yield.

(11) No reaction has been observed between the allylic reagents **2** and benzyl bromide or butyl iodide (−30 to −20 °C, 0.5 h) and benzonitrile, suggesting that **2** is neither an allylic organocopper (compare with refs 3f,g,i) nor an allylic zinc reagent, but may rather have the structure of a mixed zinc–copper cluster. Also the reaction of **2a** with 3-methyl-2-cyclohexenone in the presence of Me_3SiCl afforded only the 1,2-adduct.

(12) (a) Trost, B. M. *Angew. Chem., Int. Ed. Engl.* **1986**, *25*, 1. (b) Molander, G. A.; Shubert, D. C. *J. Am. Chem. Soc.* **1987**, *109*, 6877.

copper-zinc reagents **2** also display an interesting chemoselectivity, and the reaction of **2e** with 4-ethyl-4-formylhexanenitrile gives as the sole product the cyano amino alcohol **5i** (entry 9). Substituted reagents like **2f** react regioselectively with aldehydes, giving mostly the "branched" allylated product **5** (branched/linear: 94/6; entry 10).

In conclusion the methylene homologation of vinylic copper compounds by (iodomethyl)zinc iodide allows the preparation of various new allylic zinc and copper reagents which were found to react efficiently with several classes of electrophiles. The extension of this methodology is currently underway in our laboratories.

Acknowledgment. We thank the National Science Foundation (Grant CHE-8818704) and the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

Registry No. 5a, 127998-28-1; **5b**, 127998-29-2; **5c**, 127998-30-5; **5d**, 127998-31-6; **5e**, 125315-69-7; **5f**, 127998-32-7; **5g**, 127998-33-8; **5h**, 127998-34-9; **5i**, 127998-35-0; **5j** (isomer 1), 127998-36-1; **5j** (isomer 2), 127998-38-3; (*E*)-**6j**, 127998-37-2; (*Z*)-**6j**, 127998-39-4; $H_2C{=}C(Br)CH_2\text{-}c\text{-}C_6H_{11}$, 53608-85-8; $PhSi(Me)_2CH_2C(Br){=}CH_2$, 122244-66-0; $H_2C{=}C(Br)CH_2N(Et)_2$, 13249-59-7; $C_5H_{11}CHO$, 66-25-1; $PhCH{=}NCH_2Ph$, 780-25-6; HCO_2Et, 109-94-4; PhCHO, 100-52-7; $NC(CH_2)_2C(Et)_2CHO$, 2938-69-4; $PhC(Br){=}CH_2$, 98-81-7; $C_6H_{13}CH{=}CHBr$, 1119-88-6; $H_2C{=}C(Br)CH_2Br$, 513-31-5; iodocyclohexane, 626-62-0; 2-(2-bromoethyl)-1,3-dioxane, 33884-43-4; phenyldimethylchlorosilane, 768-33-2; diiodomethane, 75-11-6; cyclohexanone, 108-94-1; cyclopentanone, 120-92-3; 2-(4-bromo-4-pentenyl)-1,3-dioxane, 127998-40-7.

Supplementary Material Available: Typical procedure and spectral data for new compounds (6 pages). Ordering information is given on any current masthead page.

Protonation of Alkylidyne(carbaborane)tungsten Complexes: Framework Rearrangement of an Icosahedral Cage at an Unprecedentedly Low Temperature

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Alkylidyne-metal complexes[1] and carbametallaboranes[2] have played important roles in the development of organometallic and metal cluster chemistry, respectively. These two areas of research have converged as a result of the discovery of the salts [X][*closo*-$1,2\text{-}Me_2\text{-}3\text{-}({\equiv}CR)\text{-}3,3\text{-}(CO)_2\text{-}3,1,2\text{-}WC_2B_9H_9$] (**1**, X = PPh_4, NEt_4, or $N(PPh_3)_2$; R = alkyl or aryl) in which a metal atom is ligated by an alkylidyne group while simultaneously functioning as a vertex of an icosahedral cluster.[3] The reagents **1** display novel chemistry due to the presence of the reactive CR fragment on the periphery of the closo cage. Herein we report protonation studies resulting in migration and hydroboration of the alkylidyne group. This process is accompanied by a framework rearrangement, which occurs at an unprecedentedly low temperature for an icosahedral carbametallaborane structure. Hawthorne and co-workers[4] have previously observed similar polytopal rearrangements of carbametallaborane cages. Thus treatment of [*exo-nido*-$4,9\text{-}\{(Ph_3P)_2Rh\}\text{-}4,9\text{-}\mu\text{-}(H)_2\text{-}7\text{-}Me\text{-}8\text{-}Ph\text{-}7,8\text{-}C_2B_9H_8$] with excess PEt_3 affords [*closo*-$1\text{-}Me\text{-}2,2\text{-}(PEt_3)_2\text{-}2\text{-}H\text{-}8\text{-}Ph\text{-}2,1,8\text{-}RhC_2B_9H_9$]. However, this process, itself uncommonly facile, requires heating the reagents in tetrahydrofuran.[4b]

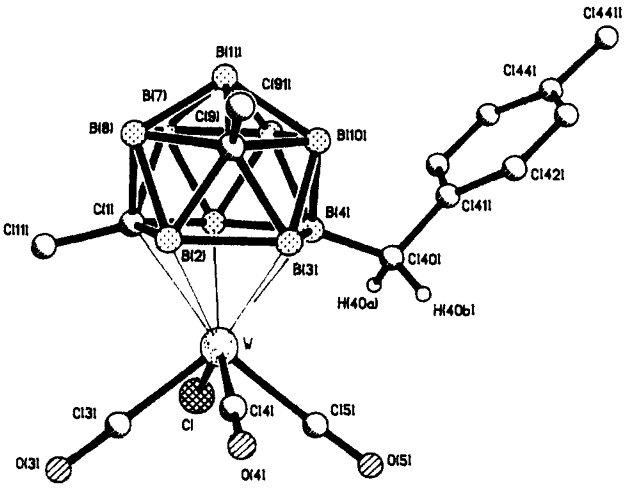
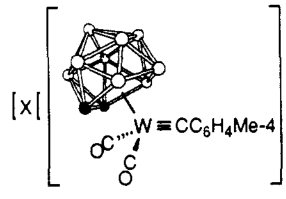
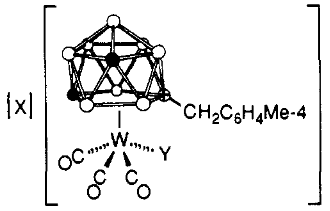


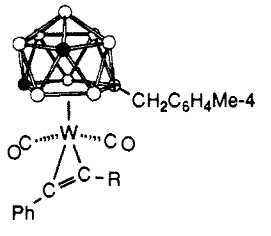
Figure 1. Structure of the anion [*closo*-$1,8\text{-}Me_2\text{-}11\text{-}(CH_2C_6H_4Me\text{-}4)\text{-}2\text{-}Cl\text{-}2,2,2\text{-}(CO)_3\text{-}2,1,8\text{-}WC_2B_9H_8$]$^-$ of **2b** showing the crystallographic numbering scheme. Selected distances (Å): W–Cl, 2.549 (2); W–C(1), 2.395 (7); W–B(2), 2.310 (7); W–B(3), 2.320 (7); W–B(4), 2.439 (6); W–B(5), 2.391 (7); B(4)–C(40), 1.62 (1); C(1)–B(2), 1.70 (1); C(1)–B(5), 1.72 (1); C(1)–B(7), 1.73 (1); C(1)–B(8), 1.73 (1); C(9)–B(2), 1.70 (1); C(9)–B(3), 1.72 (1); C(9)–B(8), 1.69 (1); C(9)–B(10), 1.70 (1); C(9)–B(11), 1.72 (1).



	X
1a	PPh_4
1b	NEt_4
1c	$N(PPh_3)_2$



	Y
2a	I
2b	Cl



	R
3a	Me
3b	Ph

● CMe ○ BH ⊕ B

A CH_2Cl_2 (20 mL) solution of **1a** (X = PPh_4, R = $C_6H_4Me\text{-}4$)[3a] (1.00 g, 1.19 mmol) was stirred rapidly at −78 °C and treated dropwise with 1 equiv of HI (57% aqueous solution). Successive IR measurements progressively indicated the course of the reaction, which was complete within 10 min.[5] Warming to ambient temperature and removal of solvent in vacuo to ca. 3 mL, followed

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[5] At ambient temperatures the reaction proceeds to completion before IR measurements can be made by standard techniques.

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