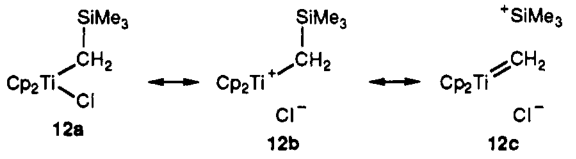
(1) and ((trimethylsilyl)methyl)aluminum dichloride (**9**; ^1H NMR 6.64, -0.07, -0.61 ppm; ^{13}C NMR 120.7, 0.1 ppm).[16]

In order to learn whether the Me_3Si group in **6** might be exerting a σ-hyperconjugative stabilizing influence on the positively polarized titanium center in **6** itself, the X-ray crystal structure of **6** was determined. Two interesting features emerge from this analysis (Figure 1): (1) the trimethylsilyl group adopts a conformation exclusively anti to the Ti-Cl bond[17] and (2) the Ti-CH_2 bond distance at 2.166 (3) Å is significantly shorter than comparable bonds between titanium and sp^3-hybridized carbon, such as the Ti-CH_2 bond at 2.22 Å in $(Cp_2Ti\text{-}CH_2CH_3)_2O$.[18] Both the conformational preference and the shortening of the Ti-CH_2 bond in **6** are suggestive of the operation of σ-bond hyperconjugation depicted in resonance structures **12a-c**.



12a **12b** **12c**

The importance of polar contributors **12b** and **12c** is further reflected in the effect of polar or donor solvents on the methylene signal in the ^1H NMR spectrum of **6**. (1) Polar solvents shift the CH_2 signal downfield: 2.02 (CCl_4) → 2.16 (CH_2Cl_2) → 2.25 ppm ($CDCl_3$).[19] (2) Coordinating solvents (:D) shift the CH_2 signal upfield: 2.02 (CCl_4) → 1.96 (CD_3SOCD_3) → 1.83 ppm ($OP(NMe_2)_3$).[20]

Finally, combining **6** with **7** or with **2** in 1,2-dichloroethane yields a homogeneous catalyst effective for the polymerization of ethylene at 0 °C. The resulting polyethylene melts up to 139 °C and displays infrared absorption bands characteristic of those of high-density linear polyethylene.[21] Furthermore, a mass spectrum of the HDPE thus obtained displayed fragments containing trimethylsilyl end groups and between 22 and 25 ethylene units.[22] This demonstrates that ion pair **8** is the active site in these polymerizations and that of necessity the Me_3Si group therefore becomes incorporated into the final polymer.

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Supplementary Material Available: Tables of crystal data, atomic coordinates, anisotropic thermal parameters, and bond distances and angles for **6** (5 pages); a listing of observed and calculated structure factors (11 pages). Ordering information is given on any current masthead page.

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[16] The ultimate formation of **1** and **9** from **6** and **7** accords well with the general observation that the following equilibrium lies much to the left:[5]

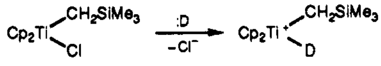
$$Cp_2TiCl_2 + RAlCl_2 \rightleftharpoons Cp_2TiRCl + AlCl_3$$

[17] Force field calculations (SYBYL 5.3, Tripos Associates, Inc., St. Louis, MO; force field, Tripos (Ver. 5.2) with extensions) on the related system $Cp_2Zr(CH_2SiMe_3)Cl$ lead to the suggestion that the syn conformer should be actually more stable than the anti conformer by 1.5 kcal/mol. Of course, such a calculation does not take into account any effect of electron delocalization such as σ-bond hyperconjugation (Angermund, K.; Krüger, C.; Nolte, M. Unpublished studies, MPI für Kohlenforschung, Mülheim an der Ruhr, FRG).

[18] Alt, H. G.; Schwind, K. H.; Rausch, M. D.; Thewalt, U. *J. Organomet. Chem.* **1988**, *349*, C7. The Ti-CH_2 bond length in **6** rather resembles those between titanium and sp^2-hybridized carbon, as in **5** (2.13 Å) or in dicyclopentadienyltitanacyclopentadiene derivatives (2.15 Å).

[19] The solvent shifts with the halomethanes do not correlate strictly with dipole moment. Part of their effect may stem from hydrogen-bonding solvation: $Cp_2Ti(CH_2SiMe_3)Cl\cdots HCH_nCl_{3-n}$.

[20] The solvent shifts with such polar coordinating solvents (:D) could be reconciled with pentacoordination of titanium: on the flank side bearing the Ti-Cl bond (cf. crystal structure in Figure 1). However, given the stabilizing effect of the Me_3Si group on the titanocenium cation, displacement of the chloride ion and formation of the solvated titanocenium cation (**13**) is more likely:



6 **13**

[21] Infrared bands for the polyethylene were observed at (cm^{-1}) 2920, 2860, 1477, 1466, 1370, 1248, 728, and 717.

[22] The mass spectrum was measured at 70 eV on a deep-insertion sample.

Thermolysis of a Siladigermirane: Evidence for the Formation of a Germasilene

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Summary: Thermolysis of hexamesitylsiladigermirane in the presence of methanol gives two products: dimesityl(dimesitylmethoxysilyl)germane (**4**) and dimesitylmethoxygermane (**5**).

The synthesis of stable derivatives of both silenes[1] and disilenes[2] was first reported a decade ago. In the intervening years, a wealth of information has been obtained on the physical properties and the chemistry of these and similar species.[3] Not surprisingly, these compounds have proven to be invaluable as starting materials for the

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[1] Brook, A. G.; Abdesaken, F.; Gutekunst, B.; Gutekunst, G.; Kallury, R. K. *J. Chem. Soc., Chem. Commun.* **1981**, 191. Brook, A. G.; Nyburg, S. C.; Abdesaken, F.; Gutekunst, B.; Gutekunst, G.; Kallury, R. K. M. R.; Poon, Y. C.; Chang, Y.-M.; Wong-Ng, W. *J. Am. Chem. Soc.* **1982**, *104*, 5667.

[2] West, R.; Fink, M. J.; Michl, J. *Science (Washington, D.C.)* **1981**, *214*, 1343.

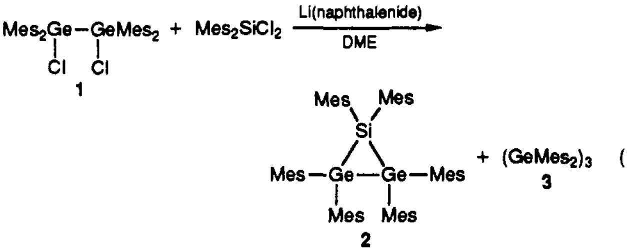
[3] For reviews on M=M systems see the following studies. Si=C: Brook, A. G.; Baines, K. M. *Adv. Organomet. Chem.* **1986**, *25*, 1. Wiberg, N. *J. Organomet. Chem.* **1984**, *273*, 141. Si=Si: West, R. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 1201. Si=M: Raabe, G.; Michl, J. In *The Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1989; p 1015. Ge=M: Barrau, J.; Escudié, J.; Satgé, J. *Chem. Rev.* **1990**, *90*, 283.

0276-7333/91/2310-3419$02.50/0

preparation of a variety of novel cyclic species.[4] Despite the increasing interest in mixed silicon/germanium systems,[5] there have been no reports on the synthesis of a germasilene (a compound containing a double bond between two *different* heavy group 14 atoms) either as an intermediate or as a stable compound. We now report the first evidence of the formation of a germasilene.

The photolysis of cyclotrisilanes and cyclotrigermanes is a well-known method for the synthesis of disilenes and digermenes.[6] By the same methodology, both a siladigermirane and a disilagermirane initially appeared to be possible precursors of a germasilene. However, because of the time and cost required to synthesize such compounds, a judicious choice of the precursor was required. Hexamesitylsiladigermirane (2; mesityl = Mes = 2,4,6-trimethylphenyl) was chosen for the following reasons: (i) analysis of the relative thermodynamic stabilities of the possible fragmentation products suggested that a siladigermirane would be the best candidate as a precursor of the desired germasilene[7] and (ii) bulky aryl substituents are known to stabilize and, hence, permit the easy isolation not only of heavy group 14 three-membered-ring compounds but also of doubly bonded heavy group 14 compounds.[3]

1,1,2,2-Tetramesityldigermane[8] is readily synthesized by coupling of chlorodimesitylgermane[9,10] using magnesium amalgam in refluxing THF. Dimesitylgermane also is formed in significant amounts. The germane/digermane mixture was chlorinated in refluxing THF with an excess of *N*-chlorosuccinimide, and the resulting dichlorinated compounds were separated by dry-column flash chromatography[11] to give 1,2-dichlorotetramesityldigermane (1) in an overall isolated yield (from chlorodimesitylgermane) of 43%. A solution of 1,2-dichlorotetramesityldigermane (0.50 g, 0.70 mmol) and dichlorodimesitylsilane[12] (0.24 g, 0.70 mmol) in dry 1,2-dimethoxyethane (DME) (20 mL) was added to a cold (dry ice/acetone), dark green suspension of lithium naphthalenide in DME (4 mL; made by sonication of a mixture of 25% lithium dispersion in oil (0.01 g, 1.4 mmol) and naphthalene (0.21 g, 1.6 mmol) in DME). The reaction mixture was stirred at −78 °C for 1 h and then warmed to room temperature over 24 h. The color of the solution changed from dark green to rust orange. Workup under acidic conditions (1 N HCl followed by extraction with diethyl ether) yielded a light brown, oily residue, which was then dissolved in hexane. A pale yellow, crystalline material slowly precipitated from the solution (0.34 g). This material consisted of two components: the minor component (5–20% of the precipitated material as measured by ^{1}H NMR spectroscopy) was identified as hexamesitylcyclotrigermane (3) by comparison with an authentic sample.[13] The major component of the mixture was identified as hexamesitylsiladigermirane (2) on the basis of spectroscopic evidence[14] and the chemical behavior of this compound, although we were not able to separate 2 and 3 by conventional methods. Crystal structure determinations of compounds 2 and 3 are underway.[15] The mass spectrum of the product mixture had a cluster of peaks from *m/e* 882 to 897 consistent with the molecular ion of the siladigermirane. The ^{13}C NMR spectrum of the mixture clearly showed the presence of two different aromatic rings (besides that of 3), and its ^{29}Si NMR spectrum consisted of one signal at −35.7 ppm, consistent with a structure containing a silicon atom in a three-membered ring.[16] There were no M–H (M = Si, Ge) stretching bands in the infrared spectrum of the material, indicating the absence of reduced products. As has been observed in the ^{1}H NMR spectra of similar 2,6-dialkylaryl-substituted cyclotrisilanes and cyclotrigermanes,[17] only a broad signal is observed for the *o*-methyl groups of the mesityl substituents, indicating hindered rotation of the mesityl groups.



(1)

The formation of the cyclotrigermane during this reaction is also very interesting. Attempts to prepare this compound by addition of a solution of 1 in DME to a suspension of lithium naphthalenide under the same reaction conditions used for the preparation of the siladigermirane/cyclotrigermane mixture were unsuccessful. At this point, we do not know the mechanism for the formation of compound 3.

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(4) See ref 3 and: Brook, A. G.; Kong, Y. K.; Saxena, A. K.; Sawyer, J. F. *Organometallics* **1988**, 7, 2245. Kabeta, K.; Powell, D. R.; Hanson, J.; West, R. *Organometallics* **1991**, *10*, 827. Tan, R. P.-K.; Gillette, G. R.; Powell, D. R.; West, R. *Organometallics* **1991**, *10*, 546. Gillette, G. R.; West, R. *J. Organomet. Chem.* **1990**, *394*, 45. Gillette, G. R.; Maxka, J.; West, R. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 54. Driess, M.; Fanta, A. D.; Powell, D. R.; West, R. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 1038. West, R.; Gillette, G. R.; Yokelson, H. B.; Millevolte, A. J. *Phosphorus, Sulfur Silicon Relat. Elem.* **1989**, *41*, 3. Weidenbruch, M.; Lesch, A.; Peters, K.; von Schnering, H. G. *Chem. Ber.* **1990**, *123*, 1795. Weidenbruch, M.; Flintjer, B.; Pohl, S.; Saak, W. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 95. Weidenbruch, M.; Schäfer, A.; Grimm, T.; Thom, K.-L. *Phosphorus, Sulfur Silicon Relat. Elem.* **1989**, *41*, 229.

(5) Takeda, K.; Shiraishi, K.; Matsumoto, N. *J. Am. Chem. Soc.* **1990**, *112*, 5043. Schmidbauer, H.; Rott, J. *Z. Naturforsch.* **1990**, *45B*, 961. Pannell, K. H.; Kapoor, R. N.; Raptis, R.; Párkányi, L.; Fülöp, V. *J. Organomet. Chem.* **1990**, *384*, 41.

(6) Masamune, S. in *Silicon Chemistry*; Corey, E. R., Corey, J. Y., Gaspar, P. P., Eds.; Ellis Horwood: Chichester, U.K., 1988; p 257.

(7) Grev, R. S.; Schaefer, H. F., III; Baines, K. M. *J. Am. Chem. Soc.* **1990**, *112*, 9458.

(8) 1,1,2,2-Tetramesityldigermane and 1 have been previously synthesized from 1-chloro-1,1,2,2-tetramesityldigermane by reduction using lithium aluminum hydride or chlorination using carbon tetrachloride, respectively: Rivière, P.; Castel, A.; Guyot, D.; Satgé, J. *J. Organomet. Chem.* **1985**, *290*, C15.

(9) Rivière, P.; Satgé, J. *Synth. React. Inorg. Met.-Org. Chem.* **1987**, *17*, 539.

(10) We have been able to obtain yields of up to 75% of Mes_2GeH_2 (required for the synthesis of Mes_2GeHCl) using the following procedure with minor modifications (lit. yield 45%): Rivière, P.; Rivière-Baudet, M.; Satgé, J. In *Organometallic Syntheses*; King, R. B., Eisch, J. J., Eds.; Elsevier: New York, 1988; Vol. 4, p 545.

(11) Harwood, L. M. *Aldrichim. Acta* **1985**, *18*, 25.

(12) Fink, M. J.; Michalczyk, M. J.; Haller, K. J.; West, R.; Michl, J. *Organometallics* **1984**, *3*, 798.

(13) Ando, W.; Tsumuraya, T. *J. Chem. Soc., Chem. Commun.* **1987**, 1514. **3**: ^{1}H NMR (C_6D_6, δ) 2.08 (s), 2.27 (bs), 6.66 (s).

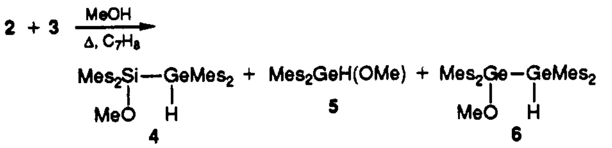
(14) Characterization data for **2** and **3**: ^{1}H NMR (C_6D_6, δ) 2.06 (s), 2.08 (s), 2.29 (bs), 6.64 (s), 6.67 (s); ^{13}C{^{1}H} NMR ($CDCl_3$, δ) 20.63, 20.71, 25.08 (bs), 128.28 (bs), 136.59, 137.07, 137.34 (**3**), 138.23, 142.64 (**3**), 143.23, 143.18 (**3**), 143.60, 144.53; ^{29}Si NMR (C_6D_6, δ) −35.7 (s); MS (CI, isobutane, *m/e* (%)) 888 (1, M^+, $^{72}Ge^{74}Ge$), 624 (4, Mes_4Ge_2, seen in the MS of **3**[13]), 576 (24, Mes_4GeSi), 456 (26, Mes_3GeSi), 385 (30, Mes_3Si), 312 (56, Mes_2Ge), 193 (44, MesGe), 120 (100, MesH); high-resolution MS (*m/e*) calcd for $^{74}Ge^{72}GeSiC_{54}H_{66}$ 888.33662, found 888.33132. N.B. When they stood in solution (C_6D_6) for extended periods of time, compounds **2** and **3** decomposed. We are currently investigating the nature of the decomposition products.

(15) Baines, K. M.; Cooke, J. A.; Payne, N. C.; Vittal, J. J. To be submitted for publication.

(16) Tan, R. P.-K.; Gillette, G. R.; Powell, D. R.; West, R. *Organometallics* **1991**, *10*, 546. Kuroda, M.; Kabe, Y.; Hashimoto, M.; Masamune, S. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1727.

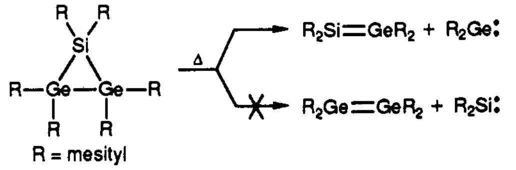
(17) Masamune, S.; Murakami, S.; Snow, J. T.; Tobita, H.; Williams, D. J. *Organometallics* **1984**, *3*, 333. Masamune, S.; Hanzawa, Y.; Murakami, S.; Bally, T.; Blount, J. F. *J. Am. Chem. Soc.* **1982**, *104*, 1150. Masamune, S.; Hanzawa, Y.; Williams, D. J. *J. Am. Chem. Soc.* **1982**, *104*, 6136.

Thermolysis (110 °C) of the siladigermirane/cyclotrigermane mixture (90 mg) in dry toluene (1 mL) for 4 h in the presence of an excess of methanol yielded a mixture of three major compounds: dimesityl(dimesitylmethoxysilyl)germane[18] (**4**), dimesitylmethoxygermane[19] (**5**), and 1,1,2,2-tetramesitylmethoxydigermane[18] (**6**). There was no evidence for the formation of dimesitylmethoxysilane.



$$\mathbf{2} + \mathbf{3} \xrightarrow[\Delta,\ C_7H_8]{MeOH} \underset{\mathbf{4}}{\underset{MeO\quad H}{Mes_2Si{-}GeMes_2}} + \underset{\mathbf{5}}{Mes_2GeH(OMe)} + \underset{\mathbf{6}}{\underset{MeO\quad H}{Mes_2Ge{-}GeMes_2}} \qquad (2)$$

Compound **3** is known to decompose at 80 °C to give dimesitylgermylene and tetramesityldigermene.[20] Thermolysis of **3** in the presence of methanol yields compounds **5** and **6**, apparently from trapping of the germylene and the digermene.[21] Compound **4**, on the other hand, appears to be derived from the addition of methanol to tetramesitylgermasilene, resulting from the thermal decomposition of the siladigermirane. Furthermore, the absence of any trapped *silylene* indicates selective decomposition of the siladigermirane to a germasilene and a germylene and not to a digermene and a silylene:



(3)

In order to confirm the selective decomposition, it is necessary to demonstrate that the sole source of compound **6** is the cyclotrigermane **3**. If the thermolysis temperature is lowered to 60 °C, compound **4** is formed in trace amounts, indicating that the cyclotrigermane is more thermally labile than the siladigermirane. This provides a means of separating **2** from **3**. Thus, thermolysis of the siladigermirane/cyclotrigermane mixture at 80 °C (to obtain a reasonable rate of reaction) in toluene in the presence of methanol for 4 h gave a mixture of compounds **2**, **4**, **5**, and **6**. Compounds **5** and **6** are readily separated from **2** by chromatography, but compound **4** elutes with **2**. Thermolysis of the recovered siladigermirane (containing <10% of **4** as determined by ^{1}H NMR spectroscopy) in toluene with excess methanol yielded only *two* major compounds, **4** and **5**, confirming that the decomposition of **2** occurs regioselectively. Furthermore, the addition of methanol to tetramesitylgermasilene is regioselective, with the alkoxy moiety adding to the silicon end of the double bond and the hydrogen becoming attached to the germanium.

In summary, all the available evidence suggests that we have generated tetramesitylgermasilene by the regioselective thermolysis of hexamesitylsiladigermirane. The germasilene was trapped with methanol in a regioselective manner to give the (methoxysilyl)germane. We are continuing to investigate this novel system.

Acknowledgment. We thank the NSERC (Canada) and the University of Western Ontario for financial support. The assistance of C. Dixon and J. A. Kohls in the preparation of the starting materials is gratefully acknowledged.

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(18) Characterization data for **4**: white solid; IR (thin film) 2024 cm^{-1} (Ge–H); ^{1}H NMR (C_6D_6, δ) 2.07 (s, 6 H, *p*-CH_3), 2.12 (s, 6 H, *p*-CH_3), 2.29 (s, 12 H, *o*-CH_3), 2.38 (s, 12 H, *o*-CH_3), 3.40 (s, 3 H, –OCH_3), 5.57 (s, 1 H, GeH), 6.65 (s, 4 H, Mes H), 6.76 (s, 4 H, Mes H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, δ) 20.96, 21.01, 23.30, 24.16 (CH_3), 51.63 (OCH_3), 127.96, 129.00 (Mes CH), 132.07, 135.84, 137.17, 138.84, 143.81, 144.04 (Mes C); ^{29}Si NMR ($CDCl_3$, δ) 0.87 (d of m, *J* = 16 Hz); MS (EI, 70 eV, *m/e* (%)) 610 (<1, M$^+$), 312 (8, Mes_2Ge), 297 (100, Mes_2SiOCH_3), 192 (6, MesGe – 1); high-resolution MS (*m/e*) calcd for $^{74}Ge^{28}SiC_{37}H_{48}O$ 610.26862, found 610.26733. When it stood in solution (C_6D_6) for extended periods of time, compound **4** decomposed. Characterization data for **6**: IR (thin film) 2027 cm^{-1} (Ge–H); ^{1}H NMR (C_6D_6, δ) 2.07 (s, 6 H, *p*-CH_3), 2.10 (s, 6 H, *p*-CH_3), 2.33 (s, 12 H, *o*-CH_3), 2.42 (s, 12 H, *o*-CH_3), 3.57 (s, 3 H, –OCH_3), 5.83 (s, 1 H, GeH), 6.68 (s, 4 H, Mes H), 6.76 (s, 4 H, Mes H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, δ) 20.97, 23.12, 24.11 (CH_3), 52.98 (OCH_3), 128.18, 128.82 (Mes CH), 134.68, 135.78, 137.77, 138.44, 143.26, 143.76 (Mes C); MS (EI, *m/e* (%)) 654 (6, M$^+$, $^{74}Ge^{72}Ge$), 431 (5, Mes_3Ge), 343 (100, Mes_2GeOCH_3), 312 (24, Mes_2Ge), 192 (33, MesGe – H), 119 (24, Mes); high-resolution MS (*m/e*) calcd for $^{74}Ge^{72}GeC_{37}H_{48}O$ 654.21376, found 654.21410.

(19) Lazraq, M.; Escudié, J.; Couret, C.; Satgé, J.; Soufiaoui, M. *J. Organomet. Chem.* **1990**, *387*, 1. Characterization data for **5**: ^{1}H NMR (C_6D_6, δ) 2.07 (s, 6 H, *p*-CH_3), 2.45 (s, 12 H, *o*-CH_3), 3.56 (s, 3 H, –OCH_3), 6.67 (s, 1 H, GeH), 6.70 (s, 4 H, Mes H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, δ) 21.07, 22.53, 54.54, 128.83, 131.65, 139.52, 143.58. Compound **5** could not be isolated in a pure state from the reaction mixture.

(20) Ando, W.; Tsumuraya, T. *J. Chem. Soc., Chem. Commun.* **1989**, 770.

(21) Thermolysis of **3** (prepared as described in ref 13) in the presence of methanol, in xylene at 125 °C, yielded compounds **5** and **6**, as determined by ^{1}H NMR spectroscopy.

Synthesis and Structure of a Tungsten η^3-Enynyl Complex Resulting from Facile Alkynyl–Vinylidene Coupling

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Summary: Deprotonation of the bis(alkyne) complex [W(CO)(η^2-HC≡CCMe$_3$)$_2$(Cp*)][BF$_4$] (**1**) with KH gives the alkynyl/alkyne complex [W(C≡CCMe$_3$)(CO)(η^2-HC≡CCMe$_3$)(Cp*)] (**2**), methylation of which gives the cationic alkyne/vinylidene complex [W(C=CMeCMe$_3$)(CO)(η^2-HC≡CCMe$_3$)(Cp*)][BF$_4$] (**3**). Deprotonation of **3** with sodium bis(trimethylsilyl)amide under CO gives the crystallographically characterized η^3-enynyl complex [W(CO)$_2$(η^3-Me$_3$CC≡CC=CMeCMe$_3$)(Cp*)] (**4**) via the facile insertion of a vinylidene ligand into a metal–alkynyl σ-bond.

We are interested in preparing complexes with two or more vinylidene ligands bonded to a single transition-metal center, anticipating that the vinylidenes will undergo reductive coupling even more easily than isoelectronic isonitrile[1] and carbonyl ligands.[2] One approach is to start with a bis(1-alkyne) complex, converting each alkyne to

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(1) Caravana, C.; Giandomenico, C. M.; Lippard, S. J. *Inorg. Chem.* **1982**, *21*, 1860 and references cited therein.

(2) (a) Hoffmann, R.; Wilker, C. N.; Lippard, S. J.; Templeton, J. L.; Brower, D. C. *J. Am. Chem. Soc.* **1983**, *105*, 146. (b) Review: Vrtis, R. N.; Lippard, S. J. *Isr. J. Chem.* **1990**, *30*, 331–341. (c) Vrtis, R. N.; Bott, S. G.; Rardin, R. L.; Lippard, S. J. *Organometallics* **1991**, *10*, 1364–1373.