Figure 1. Calculated potential curve for Mo_2; + indicates a calculated energy. The arrow on the abscissa is at the experimental internuclear distance (from ref 1).
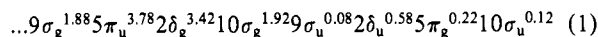
configurations contains those necessary for proper dissociation into neutral but not ^7S Mo atoms. Within the orbital optimization afforded by the GMO method, this is the equivalent of a perfect-pairing generalized valence bond (GVB) calculation.[13] Calculation B included the 151 double excitations from the sextuply bonded ground configuration. The resultant wave function will not dissociate correctly, but all of the excited configurations can interact directly with the ground configuration, leading to a greater recovery of correlation energy near the equilibrium internuclear distance than in A. Calculations C and D include all single and double excitations out of the 64 configurations of A, a total of 3212 spin-adapted configurations.

The potential energy curves resulting from a least-squares fit to a fourth-order polynomial of calculations at $R = 1.7, 1.8, 1.9, 2.0, 2.1,$ and 2.2 Å (calculations A–C) and the latter five of these points (calculation D) are shown in Figure 1. The polynomial functions were analyzed by using the method of Dunham.[14] The calculated molecular constants are listed in Table I. Calculation A minimizes at the unsatisfactorily long bond length of $R = 2.09$ Å, in agreement with a recent GVB calculation on Mo_2.[7] Calculation B achieves the best agreement with the experimental bond distance, most likely due to a fortuitous cancellation of errors due to basis set incompleteness and a rather limited CI. Indeed, an increase in the number of configurations without a change in basis set size (C) has the expected effect of lengthening the Mo–Mo bond and decreasing ω_e. Calculations C and D differ only in the addition of bond-centered functions in the latter, but the effect of the larger basis is quite dramatic; the calculated bond distance decreases by 0.09 Å, and the predicted stretching frequency increases by 83 cm^{-1}. The equilibrium bond distance, stretching frequency, and first anharmonicity constant as derived from calculation D are all in excellent accord with experiment.

It is evident from the above that caution must be used when choosing a set of configurations to correlate orbitals based upon a limited basis. Although calculation B is superior to A, it is expected that either an increase in basis set size or a better optimization of the orbitals used to construct the pair-correlated geminals would improve the results of the latter. Similarly, while calculation D achieves impressive agreement with the experimental results, it must be realized that the addition of more configurations or basis functions might change the results.[15] We expect, however, that any further changes would be small and that calculation D is a good representation of the potential near the equilibrium bond length.

The final electronic configuration for D at 2.0 Å is

$$...9\sigma_g^{1.88}5\pi_u^{3.78}2\delta_g^{3.42}10\sigma_g^{1.92}9\sigma_u^{0.08}2\delta_u^{0.58}5\pi_g^{0.22}10\sigma_u^{0.12} \quad (1)$$

The sextuply bonded leading configuration comprises 61% of the total wave function for both calculations C and D at 2.0 Å. It is interesting that this contribution is greater than that in A (46%) despite all of the configurations of A being included in C and D as well as excitations out of them. The source of this apparent anomaly will be discussed in a subsequent publication. Analysis of (1) indicates that the dominant excited configurations in the CI wave function are the result of excitations from the $2\delta_g$ to the $2\delta_u$ orbitals, representative of strong left–right correlation of the δ electrons. By comparison, configurations representing excitations from the $10\sigma_g$ to the $9\sigma_u$ orbital make only a small (<4%) contribution to the CI wave function. Clearly, the comparative insignificance of the $10\sigma_g$ to $9\sigma_u$ correlation relative to the $2\delta_g$ to $2\delta_u$ correlation implies stronger bonding in the $10\sigma_g$ MO than that found in the $2\delta_g$ MO, i.e., the one-electron orbital description is more valid for the former. In fact, the $10\sigma_g$ orbital, which is mostly 5s in character, correlates less than the $9\sigma_g$ orbital, which is mostly $4d_{z^2}$, further evidence for the strong metal–metal interaction in the $10\sigma_g$ orbital. This strongly supports our proposal[6] that the second σ bond in Mo_2 has a far more important role than the second δ bond in making the bond length of Mo_2 shorter than those of the quadrupole Mo–Mo bonds. The formation of this second σ bond by using the s orbitals is restricted to the naked metal clusters, as the addition of ligands, with concomitant increase in oxidation number, will force the 5s atomic orbitals, and hence this molecular orbital, to higher energy.

(15) Of probable importance will be the totally split quadruple excitations in which eight orbitals are singly occupied: Goddard, W. A., III, private communication.

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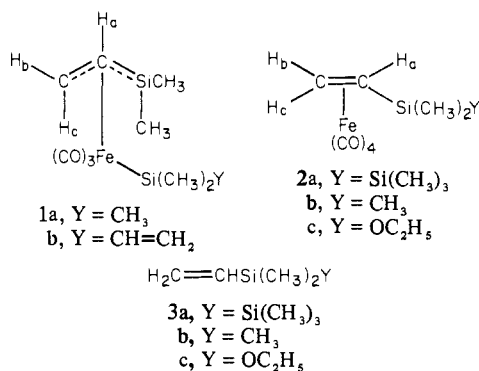## Evidence for the Nonexistence of a (η^3-1-Silapropenyl)tricarbonyliron Complex. A Correction

Sir:

The extraordinary stability of transition-metal complexes of highly reactive species such as cyclobutadiene and trimethylenemethane has been well documented.[1] Recently, the preparation of two tricarbonyliron complexes of the 1-silapropenyl radical (**1a** and **b**) was reported and heralded as representing the preparation of the first stable compounds of doubly bonded silicon.[2]

(13) Goddard, W. A., III; Dunning, T. H., Jr.; Hunt, W. J.; Hay, P. J. *Acc. Chem. Res.* **1973**, *6*, 368-376.
(14) Dunham, J. L. *Phys. Rev.* **1932**, *41*, 721-731.

(1) See, e.g.: Emerson, G. F.; Watts, L.; Pettit, R. *J. Am. Chem. Soc.* **1965**, *87*, 131. Pettit, R. *J. Organomet. Chem.* **1975**, *100*, 205. Emerson, G. F.; Ehrlich, K.; Giering, W. P.; Lauterbur, P. C. *J. Am. Chem. Soc.* **1966**, *88*, 3172.

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Communications to the Editor

1a, Y = CH₃
b, Y = CH=CH₂

2a, Y = Si(CH₃)₃
b, Y = CH₃
c, Y = OC₂H₅

$$H_2C{=\!=\!=}CHSi(CH_3)_2Y$$

3a, Y = Si(CH₃)₃
b, Y = CH₃
c, Y = OC₂H₅

Numerous attempts to prepare other stable transition-metal complexes of multiply bonded silicon species have been made, but to date, all have failed.[3] We now report evidence indicating that unfortunately the structure of the compound reported earlier as **1a** is in error, the correct structure being **2a**.

Recently, we have prepared a variety of η^2-tetracarbonyliron complexes of vinylsilanes, e.g., **2b,c**, by reaction of vinylsilanes, e.g., **3b,c**, with enneacarbonyldiiron [Fe₂(CO)₉] and have studied some of their chemistry.[4] Owing to the similarity of the spectroscopic data obtained for our η^2-tetracarbonyliron complexes and that reported for the (η^3-silapropenyl)tricarbonyliron complexes **1**, we have reexamined the reaction of vinylpentamethyldisilane (**3a**) with Fe₂(CO)₉, purported to give **1a**.

With the same reaction conditions (solvent, reaction time, workup, etc.) reported earlier,[2,5] a dark yellow liquid with a boiling point identical with that reported for **1a** was obtained. The ^{1}H and ^{13}C NMR spectra (Table I) of this liquid are, except for small differences in chemical shift, very similar to the analogous spectra obtained for the η^2-tetracarbonyliron complexes with the generalized structure **2**, e.g., **2b** and **2c**.[6] Consistent with the η^2-coordinated vinylsilane structure **2a**, the spectra exhibit the upfield shifts expected upon coordination of the double bond to iron,[7] two distinct absorptions (relative intensity, 1:1) expected for the diastereotopic methyl groups on the silicon atom adjacent to the coordinated vinyl group,[8] and a single absorption for Si(CH₃)₃.

The metal–carbonyl stretching frequencies in the infrared[4] spectrum of the yellow liquid (Table II) are consistent with either a tetra- or a tricarbonyliron complex.[9] Similarly, C, H elemental

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(2) (a) Sakurai, H.; Kamiyama, Y.; Nakadaira, Y. *J. Am. Chem. Soc.* **1976**, *98*, 7453. (b) *J. Organomet. Chem.* **1980**, *184*, 13.

(3) (a) The formation of such complexes as transient intermediates has, however, been suggested; e.g.: Curtis, D. M.; Greene, J. *J. Am. Chem. Soc.* **1978**, *100*, 6362. Tamao, K.; Yoshida, J.-I.; Okazaki, S.; Kumada, M. *Isr. J. Chem.* **1976**, *15*, 265. Nakadaira, Y.; Kobayashi, T.; Sukurai, H. *J. Organomet. Chem.* **1979**, *165*, 399. (b) For a discussion of such attempts, see: Gusel'nikov, L. E.; Nametkin, N. S. *Chem. Rev.* **1979**, *79*, 529.

(4) Radnia, P.; McKennis, J. S., unpublished work; manuscript in preparation.

(5) Change in solvent (ether) and reaction time (4 h) does not change the reaction product.

(6) Complex **2b**: ^{1}H NMR (CS₂) σ 3.04 (d, *J* = 10.7 Hz, Hᵦ), 2.58 (d, *J* = 14.6 Hz, Hᵤ), 2.14 (dd, *J* = 10.7, 14.6 Hz, Hₐ), 0.18 [s, 9, Si(CH₃)₃]; ^{13}C NMR (CDCl₃) σ 212 (CO), 45.1 (d, J_{CH_a} = 134 Hz, C$_\alpha$), 41.8 (t, $J_{CH_{b,c}}$ = 157 Hz, C$_\beta$), 0.26 [q, Si(CH₃)₃]. Complex **2c**: ^{1}H NMR (CS₂) σ 2.90 (d, *J* = 11.0 Hz, Hᵦ), 2.56 (d, *J* = 14.5 Hz, Hᵤ), 1.9 (dd, *J* = 11.0, 14.5 Hz, Hₐ), 0.22 [s, 3, Si(CH₃)CH₃], 0.20 [s, 3, Si(CH₃)CH₃]; ^{13}C NMR (CDCl₃) σ 212 (CO), 42.4 (d, J_{CH_a} = 136 Hz, C$_\alpha$), 40.7 (t, $J_{CH_{b,c}}$ = 158 Hz, C$_\beta$), –0.26 [q, Si(CH₃)CH₃], –0.62 [q, Si(CH₃)CH₃].

(7) Pettit, R.; Emerson, G. F. *Adv. Organomet. Chem.* **1964**, *1*, 1.

(8) The ^{1}H and ^{13}C NMR spectra of all seven of the tetracarbonyliron complexes of vinyldimethylsilanes which we have prepared exhibit two signals for the diastereotopic methyl groups (ref 4). The diastereotopic methyl groups in the bis(triphenylphosphine)platinum complex of **3c** are also easily discernible in the ^{1}H NMR spectrum: Fitch, J. W.; Chan, K. C.; Froelich, J. A. *J. Organomet. Chem.* **1978**, *160*, 477.

(9) (a) Thin-film (5–10 μm) spectra of **2a** obtained at 90 K by using a Digilab FTS-20C FT–IR spectrometer clearly indicated a shoulder at 2030 cm⁻¹ in addition to three other distinguishable metal carbonyl absorptions consistent with a tetracarbonyliron species. (b) Absorption for the M–SiR₃ stretching mode has been reported at ~300 cm⁻¹, e.g., [CH₂CH₂CH₂Si(CH₃)₂]Fe(CO₄), ν(Fe–Si) 302 cm⁻¹ (medium intensity): Cundy, C. S.; Lappert, M. F. *J. Chem. Soc., Chem. Commun.* **1972**, 445. The absence of any appreciable absorption for **2a** in the region 350–255 cm⁻¹, although negative evidence, does not support the tricarbonyliron structure **1a**.

Table I. NMR Spectral Data for Complexes "1a" and 2a

2a	"1a"[b]	Δ[c]
	^{1}H NMR[a]	
3.03 [d, *J* = 10.5 Hz, Hᵦ]	3.17 [d, *J* = 10.0 Hz, Hᵦ]	0.15
2.53 [d, *J* = 14.8 Hz, Hᵤ]	2.68 [d, *J* = 15.0 Hz, Hᵤ]	0.15
2.15 [dd, *J* = 10.5, 14.8 Hz, Hₐ]	2.29 [dd, *J* = 10.0, 15.0 Hz, Hₐ]	0.14
0.23 [s, Si(CH₃)CH₃]	0.38 [s, Si(CH₃)CH₃]	0.15
0.12 [s, Si(CH₃)₃]	0.27 [s, Si(CH₃)₃]	0.15
0.00 [s, Si(CH₃)CH₃]	0.15 [s, Si(CH₃)CH₃]	0.15
	^{13}C NMR	
211.7[d] [CO]	212[a] [CO]	0.3
44.7[d] [d, J_{CH} = 135 Hz, C$_\alpha$]	46.0[a] na	1.3
41.9[d] [t, J_{CH} = 155 Hz, C$_\beta$]	43.0[a] na	1.1
–1.4[d] [Si(CH₃)CH₃]	0.0[a] [Si(CH₃)CH₃]	1.4
–2.2[d] [Si(CH₃)₃]	–1.0[a] [Si(CH₃)₃]	1.2
–4.4[d] [Si(CH₃)CH₃]	–3.0[a] [Si(CH₃)CH₃]	1.4

[a] σ relative to Me₄Si; CS₂ as solvent. [b] Data from ref 2. [c] $\sigma_{1a} - \sigma_{2a} = \Delta$. [d] CDCl₃ as solvent, internal deuterium lock; chemical shift with lowest field absorption of CDCl₃ triplet at 1970.00 Hz. [e] na, no assignment made.

Table II. IR Spectral Data for Complexes "1a" and 2a (cm⁻¹)[a]

2a	"1a"[b]
2083 (w–m)	
2030 (s, sh)	2025 (s)
1997 (vs)	1995 (vs)
1973 (vs)	1975 (vs)
~1400 (w)	1400 (w)
1317 (m)	1315 (m)
1245 (s)	1245 (s)
1200 (m)	1195 (m)
835 (s)	830 (s)
800 (s)	795 (s)
635 (s)	

[a] Thin film. [b] From ref 2.

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analysis is unable to definitively distinguish between the two possibilities.[10] Mass spectroscopic examination, on the other hand, clearly indicated a Fe(CO)₄ species, with the spectrum exhibiting a parent mass peak at *m/e* 326, peaks for successive loss of four carbonyls, and, finally, the loss of Fe.[11,17b] The preceding data, coupled with the ability to recover the starting vinyldisilane (**3a**) by oxidation of the complex with ceric ion at 0 °C, albeit in low yield,[12] lead to the assignment of structure **2a** for the yellow liquid. Structure **1a**, with a tricarbonyliron moiety, clearly is inconsistent with the mass spectral data.[13]

Comparison of the physical constants and spectral data reported for the compound purported to have structure **1a** with the data obtained for **2a** (see Tables I and II) leaves little doubt as to the identity of the two compounds. Not only are the boiling points

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(10) (a) Anal. Calcd for C₁₀H₁₈Si₂O₃Fe: C, 40.27; H, 6.08. Calcd for C₁₁H₁₈Si₂O₄Fe: C, 40.49; H, 5.56. Found: C, 40.43; H, 5.86 (ref 2). (b) Elemental C, H analysis is also unable to definitively distinguish structure **1b** from its tetracarbonyliron counterpart to **2a**.

(11) (a) Spectra were obtained on a CEC 21-110B mass spectrometer at 70 eV with an ambient inlet temperature and an ion-source temperature of 100–110 °C. The samples were introduced directly into the ionization chamber through a molecular leak. At higher temperatures, the parent mass peak is either very weak or absent. (b) Parent mass peaks were observable for the complexes **2b** and **2c**, as well. (c) To our knowledge there are no examples of Fe(CO)₃ species giving rise to a mass peak (P + CO) corresponding to a Fe(CO)₄ species in the mass spectrometer.

(12) Although, as indicated by a referee, the liberation of **3a** by reductive elimination from **1a** cannot be ruled out, documentation of such a process for π-allyl tricarbonyliron complexes is lacking. In view of both the sensitivity of the complex toward even air oxidation, affording decomposition products, and the expected sensitivity of **3a** toward the acidic medium generated during the oxidation with ceric ammonium nitrate, we were, in fact, pleasantly surprised to detect any free ligand.

(13) Recently, both the thermolysis and photolysis reactions of Fe(CO)₅ with **3a** have been interpreted in view of the earlier report (ref 2a) in terms of the transient intermediacy of **1a** (ref 2b). Although a (η^3-1-silapropenyl)tricarbonyliron species may well be an intermediate in such reactions, at the present time there is no compelling evidence for such a species.

identical but the refractive indices, although not identical, are reasonably close, considering the air sensitivity of the compound and the sensitivity of such indices to impurities.[14]

Examination of the ¹H and ¹³C NMR data reveals only small differences between our data and that reported for "1a". These differences, as indicated in Table I, are uniform and represent a shift of the spectrum as a whole, a fact which strongly suggests either concentration, solvent (¹³C NMR), or instrumental differences, rather than gross structural differences, e.g., between 1a and 2a.

The infrared spectra of 2a and "1a" (Table II) are virtually identical, differing in only one respect. An additional metal carbonyl band at 2080 cm⁻¹ with weak intensity is observed by us, but is not reported by the Sakurai group for their compound. Whether such a band was in fact observed but simply not reported owing to its weak intensity is not clear.

The observation of an infrared absorption of medium to weak intensity at ∼1315 cm⁻¹ in the spectra of *all* of the (vinylsilane)tetracarbonyliron complexes prepared to date is interesting in view of the suggestion[2a] that this absorption band, observed in the spectrum of "1a", might be assigned to the stretching frequency of a coordinated Si=C bond. Clearly, with the present results, such an assignment is not warranted.[15] The agreement between our UV data and that reported for "1a" is good,[16] although we fail to detect the reported shoulder at 221 nm.[16c]

The key to the assignment of the correct structure to the complex obtained from 3a and Fe₂(CO)₉ resides in the mass spectral (MS) data. As mentioned earlier, our MS data with a parent peak at *m/e* 326 clearly indicate a *tetra*carbonyl rather than a *tri*carbonyliron complex, i.e., structure 2a rather than 1a. Additionally, the high relative intensity of a peak at *m/e* 158, corresponding to the free ligand 3a, is consistent with the preservation of the structural integrity of 3a upon complexation.[17b] Although Sakurai and co-workers observed the same fragmentation peaks,[2,17] they failed to detect the parent mass peak and assumed that the P-28(CO) peak was the parent peak and indicative of a parent Fe(CO)₃ species. Presumably, on the basis of this assumption, in part, and the belief that the cleavage of, or oxidative insertion in, a relatively weak Si–Si bond by a transition metal was quite reasonable,[18] the IR and NMR spectra, as well as the analytical data, were rationalized cogently (but clearly now speciously) in terms of the η³-silapropenyl complex 1a. The failure to observe the parent mass peak, however, is not too surprising in view of the propensity of carbonyl–metal complexes to readily fragment in the mass spectrometer. Such fragmentation, characterized by the successive loss of CO molecules, often results in weak, if not absent, parent mass peaks. All of the (vinylsilane)tetracarbonyliron complexes that we have prepared to date are especially sensitive and fragment or decompose extremely easily at elevated probe and ion-source temperatures. As a consequence, great care was necessary in order to obtain meaningful spectra exhibiting parent mass peaks.[11]

Despite their air sensitivity and thermal instability above ∼80 °C, the η²-vinylsilane complexes 2 are remarkably stable. (Ethylene)tetracarbonyliron, by comparison, is quite unstable and is reported to decompose slowly above –40 °C.[20] Although the inductive effect of the silicon, which raises the energies of the π and π* orbitals,[19] would be destabilizing, the resonance effect, involving p–d π bonding and the lowering of the π* orbital, presumably would be stabilizing by virtue of enhanced back-donation by the metal.

Attempts to prepare 1a from 2a thermally as well as photochemically have failed. In view of the evidence for the failure to prepare the η³-silapropenyl complex 1a, the validity of the structure 1b immediately becomes suspect. As a consequence, it would appear that the stabilization of a doubly bonded silicon species by a transition metal remains to be conclusively demonstrated.

(20) Murdoch, H. D.; Weiss, E. *Helv. Chim. Acta* **1963**, *46*, 1588.

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Total Synthesis of (±)-Hirsutene

Sir:

The recent years have experienced intense activity in the development of synthetic routes to terpenoids possessing tricyclo-[6.3.0.0²,⁶]undecane ring systems. Hirsutene (1), coriolin, and hirsutic acid present themselves as challenging targets in carbocyclic synthesis as well as an exercise in the efficient introduction of numerous oxygenated centers.

Considerable efforts have also been seen in the area of nonlinearly fused tricyclopentanoids such as isocomene and retigeranic acid, both containing the tricyclo[6.3.0.0⁴,⁸]undecane ring system.[1]

In addition to the synthetic interest elicited by these terpenoids, there exists an array of remarkable physiological properties associated especially with the coriolin-type sequiterpenes.[2] The antibiotic and antitumor activities of hirsutic acid and coriolin dictate an *efficient* synthetic approach to these compounds, particularly in view of their uncertain supply from natural sources.[3] Several interesting syntheses have appeared to date, describing the preparation of hirsutic acid,[4] coriolin,[5] and their biogenic precursor, hirsutene.[6]

(14) Complex 2a: n^{16}D 1.5269, n^{20}D 1.5254. Reported for "1a": n^{16}D 1.5065 (ref 2b).

(15) The infrared spectrum of (ethylene)tetracarbonyliron in the solid state (thin film) also exhibits a weak absorption at 1317 cm⁻¹: Andrews, D. C.; Davidson, G. *J. Organomet. Chem.* **1972**, *35*, 162.

(16) (a) Complex 2a (Beckman Model 25): λ_{max} (hexane) 270 (sh) (ε 8800), 236 (sh) (14700), 212 nm (23300). Reported for "1a": λ_{max} (hexane) 270 (sh) (ε 7000[7700]), 236 (sh) (12600[13800]), 221 (sh) (17400[19000]), 212 nm (20500[22400]). Values in brackets are corrected extinction coefficients, assuming a Fe(CO)₄ rather than a Fe(CO)₃ species; agreement is within 5%. (b) For comparison, complex 2c: λ_{max} (hexane) 262 (sh) (ε 8000), 215 nm (27000). (c) The absorbance at 221 nm corresponds to ε 18600, consistent with the notion that there is a similar absorption at 221 nm for 2a, but because of poor resolution, it is not observed as a shoulder. On the other hand, first-derivative analysis of the UV spectrum does not reveal a shoulder.

(17) (a) Reported for "1a" (ref 2): MS, *m/e* (rel intensity) 298 (2.9), 270 (4.9), 242 (6.8), 214 (21.6), 158 (23.9), 143 (21.0), 85 (54.4), 73 (100). Complex 2a: MS, *m/e* (rel intensity) 326 (12.8), 298 (9.9), 270 (36.1), 242 (48.9), 214 (73.0), 158 (30.5), 143 (39.7), 85 (87.2), 73 (100). (b) The observation of mass peaks at *m/e* 311 (3.5) and 253 (3.5), corresponding to P–CH₃ and P–Si(CH₃)₃, respectively, as well as peaks corresponding to the successive loss of CO from these fragments, also indicates a tetracarbonyliron species.

(18) Sakurai and co-workers had earlier demonstrated the interesting palladium-catalyzed addition of alkynes across a Si–Si bond: Sakurai, H.; Kamiyama, Y.; Nakadiara, Y. *J. Am. Chem. Soc.* **1975**, *97*, 931.

(19) West, R. *J. Organomet. Chem.* **1965**, *3*, 314.

(1) For recent synthetic efforts in this area, see: Paquette, L. A.; Han, Y. K. *J. Org. Chem.* **1979**, *44*, 4016. Pirrung, M. C. *J. Am. Chem. Soc.* **1979**, *101*, 7130. Oppolzer, W.; Bättig, K.; Hudlicky, T. *Helv. Chim. Acta* **1979**, *62*, 1493.

(2) For a concise list of references pertaining to the biological activities, see: Little, R. D.; Muller, G. W. *J. Am. Chem. Soc.* **1979**, *101*, 7129.

(3) Lansbury, P. T.; Wang, N. Y.; Rhodes, J. E. *Tetrahedron Lett.* **1979**, 1829. Comer, F. W.; McCapra, F.; Quershi, I. H.; Scott, A. I. *Tetrahedron* **1967**, *23*, 4761.

(4) Trost, B. M.; Shuey, C. D.; DiNimo, F., Jr.; McElvain, S. S. *J. Am. Chem. Soc.* **1979**, *101*, 1284.

(5) Danishefsky, S.; Zamboni, R.; Kahn, M.; Etheredge, S. J. *J. Am. Chem. Soc.* **1980**, *102*, 2097.