Table I. Azo Compound Photolyses[a]

		prod ratio ± 0.04[b]		
compd	prod	350 nm	254 nm	[3]sens
7	1/2	1:1.06	1:1.04	1:1.02
8	1/2	1:1.20	1:1.01	1:1.05
9	3/4	1:1.10	1:1.34	1:1.33
10	3/4	1:1.77	1:1.32	1:1.33

[a] 254-nm irradiations in cyclohexane with Rayonet photochemical reactor. 350-nm irradiations in cyclohexane and sensitized irradiations in benzene with 300-W Xe lamp and cutoff filter ($\lambda > 340$ nm). Triplet sensitizer is 4,4-bis(dimethylamino)benzophenone, at concentrations calculated to absorb greater than 99% of the light. No other products were detected by capillary GC or ^{1}H NMR. [b] All numbers represent the average of at least eight separate analyses at several different irradiation times short enough to ensure negligible product rearrangement. The error limit represents two standard deviations, which was approximately the same for all the reported numbers.

itrogenations. Photolyses of the azo compounds, in contrast, are more informative. Results of direct and sensitized irradiations are listed in Table I.[9] The photoproducts are stable to direct irradiation at 350 nm, but the product ratios change slowly under sensitized conditions or at 254 nm. The reported ratios were obtained by GC at short irradiation times where the photoproducts were independently shown to be stable.

Under all irradiation conditions the denitrogenations approach the ring-closure ratios exhibited in the corresponding meta photoadditions. Direct 350-nm irradiation shows some memory effects, albeit smaller than in the analogous thermolyses. The sensitized and direct 254-nm irradiations are more clear-cut. Under these excitation conditions ring-closure ratios are independent of azo position (i.e., 7 and 8 give the same product ratio, as do 9 and 10). Moreover, the ratios of photoproducts obtained on triplet sensitization and on 254-nm irradiation are identical, within experimental error. Most dramatically, the ratio of 1:2 from 7 and 8 and the ratio of 3:4 from 9 and 10 are identical within experimental error to the ratios obtained in the corresponding arene photoadditions.

We believe that the above results provide direct evidence for the intermediacy of biradicals such as 5 and 6 in meta photoadditions. The ground-state multiplicity of such biradicals is unknown. Although meta photoadditions have been shown to be singlet reactions,[1] the same regioselectivity is exhibited in the triplet denitrogenations. Triplet pyrazolines have been shown to generate triplet 1,3-biradicals, whose chemistry can be correlated with triplet biradicals from other sources.[10] Triplet 1,3-biradicals, however, often give different product mixtures than the corresponding singlets.[11] In our case the intermediates are rigidly constrained, and major differences between singlet and triplet geometries are not possible. Our results suggest that triplet biradicals 5 and 6, generated from the triplet azo compounds, intersystem cross to the same singlet biradicals as generated in the photoadditions.

It seems likely that the 350-nm fragmentations proceed mainly via the biradical route. The exhibition of a small memory effect is puzzling, however. A minor [π2s + σ2s + σ2s] component would fit these results, although simple-minded analysis suggests such a process to be orbital symmetry forbidden photochemically. Epiotis and co-workers[12] have discussed various schemes by which such orbital symmetry constraints may be overridden in photochemical reactions, and certain of these suggestions may have relevance here. In the absence of additional data, however, we hesitate to speculate further. It is of note that Berson and Olin have also observed apparent forbidden six-electron concerted cycloreversions from azo $n\pi^*$ states.[13]

Most 1,2-diazenes show only $n\pi^*$ absorption (ca. 360 nm) in the near-UV.[14] Interestingly, 7–10 exhibit an additional transition at ca. 235 nm ($\epsilon \sim 3100$ M^{-1} cm^{-1}). The assignment of this band is not known, although $n_+\pi^*$, $\pi\pi^*$, and $n_-\sigma^*$ are possibilities.[14] Selective excitation at 254 nm gives ring closure ratios identical with those observed on triplet sensitization (Table I). Adam and co-workers[15] have shown that S$_2$ azo excitation (at 185 or 193 nm) in general leads to more efficient biradical formation than S$_1$. Consistent with this, our results suggest that S$_2$ azo gives biradical chemistry without competing concerted cycloreversion. Intersystem crossing to a triplet azo state, e.g., $\pi\pi^*$, cannot be excluded, however.

The reasons behind the substituent effects on ring closure are not known. The greater perturbation of bridgehead substitution suggests that steric interactions may dominate over electronics. Interestingly, similar bridgehead substitution effects are observed in di-π-methane rearrangements.[16] The effects of other substituents are currently being explored.

Supplementary Material Available: Spectral data for 9, 10, and corresponding urazoles (1 page). Ordering information is given on any current masthead page.

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Tetrahydroborate Complexes of Tantalum: Synthesis, Characterization, and Reactivity of Ta(BH$_4$)H$_2$(PMe$_3$)$_4$

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Received January 28, 1985

Covalent tetrahydroborate complexes of the group 5 metals are very rare and the only well-characterized examples are Cp$_2$V-(BH$_4$),[2] [V(μ-Cl)(μ-dppm)(BH$_4$)]$_2$,[3] Cp$_2$Nb(BH$_4$),[4] and CpNbH(BH$_4$)(dmpe).[5,6] Here we report the synthesis and characterization of an unusual 8-coordinate tetrahydroborate complex of tantalum, Ta(BH$_4$)H$_2$(PMe$_3$)$_4$ (1), and describe our

(1) (a) The University of Michigan. (b) Dow Britton Fellow 1983–1984. (c) Indiana University. (d) Present address: Isotope and Nuclear Chemistry Division, Los Alamos National Laboratory, Los Alamos, NM 87545.
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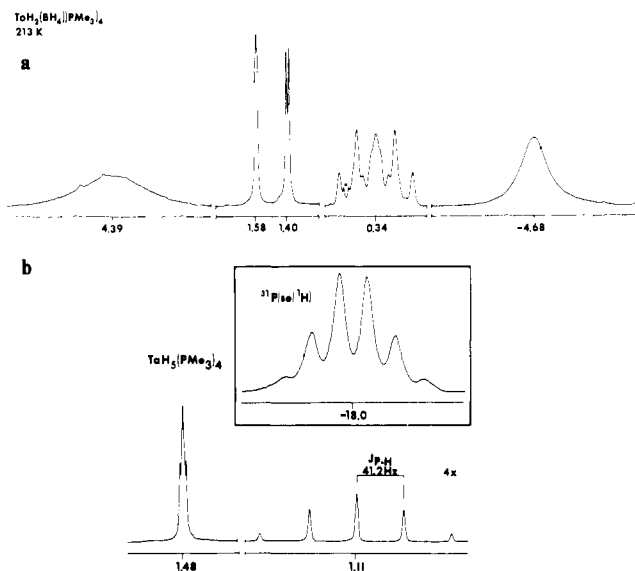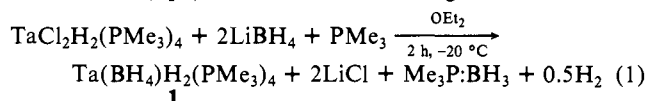(12) Epiotis, N. D.; Yates, R. L. J. Org. Chem. 1974, 39, 3150. Epiotis, N. D. Angew. Chem., Int. Ed. Engl. 1974, 13, 751.

Figure 1. (a) ^{1}H NMR, 360 MHz, spectrum of **1** recorded at -80 °C in toluene-d_8 (* indicates Me$_3$P:BH$_3$ impurity). (b) ^{1}H NMR, 360 MHz, spectrum of **2** recorded at 25 °C in benzene-d_6. The insert shows the selectivity ^{1}H-decoupled (from the proton resonance at δ 1.48) 145.8-MHz ^{31}P NMR spectrum of **2**.

preliminary studies of its reactivity.

Ether solutions of TaCl$_2$H$_2$(PMe$_3$)$_4$[7] react smoothly with 2 equiv of LiBH$_4$ in the presence of PMe$_3$ at -20 °C to provide dark green solutions of **1** (eq 1). Air-sensitive, emerald-green **1**[8] was isolated

$$\text{TaCl}_2\text{H}_2(\text{PMe}_3)_4 + 2\text{LiBH}_4 + \text{PMe}_3 \xrightarrow[\text{2 h, -20 °C}]{\text{OEt}_2}$$
$$\underset{\textbf{1}}{\text{Ta(BH}_4)\text{H}_2(\text{PMe}_3)_4} + 2\text{LiCl} + \text{Me}_3\text{P:BH}_3 + 0.5\text{H}_2 \quad (1)$$

in 80% yield after filtration, solvent removal, sublimation (10^{-5} torr, 25 °C) of volatile Me$_3$P:BH$_3$,[9] and recrystallization from hexane. The IR spectrum of **1** (Nujol mull) has a strong terminal Ta–H stretch at 1699 cm^{-1} and a –BH$_2$ deformation mode at 1170 cm^{-1}. The solution (benzene) infrared bands at (2460, 2440) and 1980 cm^{-1} are assigned as the B–H stretching modes for the terminal and bridging hydrides, respectively.[10] The -80 °C proton NMR spectrum of **1** in toluene-d_8 (Figure 1) consists of two phosphine methyl resonances at δ 1.58 (18 H) and 1.40 (18 H), a complex hydride multiplet at δ 0.34 (2 H), and two broad borohydride signals at δ 4.39 (2 H, $\Delta\nu_{1/2}$ = 150 Hz, TaH$_2$BH_2) and –4.68 (2 H, $\Delta\nu_{1/2}$ = 60 Hz, TaH_2BH$_2$).[11] Raising the temperature to 25 °C resulted in significant broadening of both BH$_2$ signals and collapse of the hydride resonance to a quintet ($^2J_{PH}$ = 38.7 Hz). A proposed structure (B) for **1** is shown below.

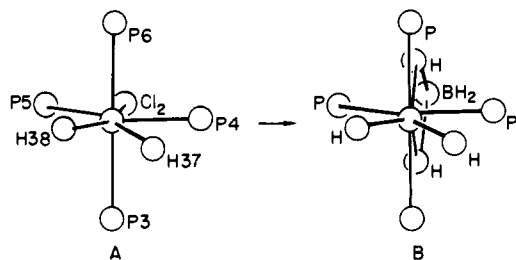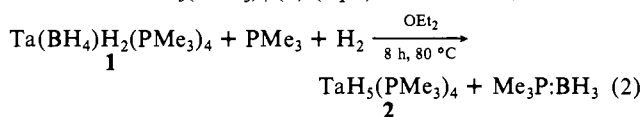(7) Luetkens, M. L., Jr.; Elcesser, W. L.; Huffman, J. C.; Sattelberger, A. P. *Inorg. Chem.* **1984**, *23*, 1718.

(8) Anal. Calcd for Ta(BH$_4$)H$_2$(PMe$_3$)$_4$: C, 28.70; H, 8.43; M_r 502. Found: C, 27.59; H, 7.87; M_r 490. ^{31}P NMR (ppm, C$_7$D$_8$, 145.8 MHz, {^{1}H}, -80 °C) δ 2.2 (br s, 1), 13.4 (br s, 1).

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(11) Homonuclear decoupling of the BH$_2$ resonances had no apparent effect on the TaH$_2$ resonance at δ 0.34. Our assignments are based on those proposed for Cp$_2$V(BH$_4$)^2 and IrH$_2$(BH$_4$)(P-t-Bu$_3$)$_2$.[12]

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Figure 2. ORTEP drawings (50% probability ellipsoids) of [Ta(CO)$_3$-(PMe$_3$)$_4$]$^+$ (top) and [Ta(CO)$_5$PMe$_3$]$^-$ showing the atom labeling schemes.

It is consistent with the low-temperature NMR data and it seems quite reasonable when we consider that **1** can be formed directly from TaClH$_2$(PMe$_3$)$_4$,[13] a complex whose neutron diffraction structure (A) has recently been completed.[14]

Ether solutions of **1** in the presence of excess (10 equiv) PMe$_3$ react slowly with hydrogen (40 psi) at 80 °C to provide red solutions of TaH$_5$(PMe$_3$)$_4$ (**2**) (eq 2). Red-brown, air-sensitive

$$\underset{\textbf{1}}{\text{Ta(BH}_4)\text{H}_2(\text{PMe}_3)_4} + \text{PMe}_3 + \text{H}_2 \xrightarrow[\text{8 h, 80 °C}]{\text{OEt}_2}$$
$$\underset{\textbf{2}}{\text{TaH}_5(\text{PMe}_3)_4} + \text{Me}_3\text{P:BH}_3 \quad (2)$$

2[15,16] was isolated in 60% yield after filtration, solvent removal, sublimation of Me$_3$P:BH$_3$, and recrystallization from hexane/PMe$_3$ at -78 °C. **2** was characterized by ^{1}H and ^{31}P NMR (Figure 1) and IR spectroscopy. The 1:4:6:4:1 hydride resonance at δ 1.11 in the proton NMR spectrum and the 1:5:10:10:5:1 sextet at δ -18.0 in the selectivity ^{1}H-decoupled (from the PMe$_3$ protons) ^{31}P NMR spectrum confirm the TaH$_5$(PMe$_3$)$_4$ stoichiometry and indicate that **2** is stereochemically nonrigid in solution.[17] The IR spectrum of **2** (Nujol mull) shows a strong terminal Ta–H stretch at 1593 cm^{-1}.

Hexane solutions of **1** react rapidly with CO (40 psi) at 25 °C. The initial product of the reaction, yellow-orange Ta(BH$_4$)-(CO)$_3$(PMe$_3$)$_3$ (**3**)[18] precipitates from solution (70% yield) within 15 min (eq 3). The solution infrared spectrum of **3** shows bands
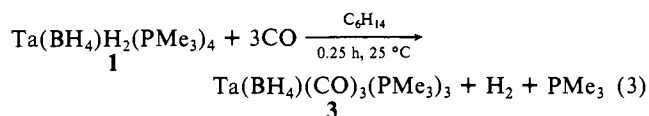
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(14) Schultz, A. J.; Williams, J. M., personal communication.

(15) (a) High-pressure hydrogenolysis (200 atm of H$_2$) of [Li(THF)$_4$]-TaPh$_6$ in benzene/PMe$_3$ at 50 °C has been claimed as a route to **2** (in unspecified yield) in the patent literature.[15b] (b) Tebbe, F. N. U.S. Patent 3 933 876, Jan 20, 1976.

(16) **2** decomposes slowly (days) at 25 °C to a hexane-insoluble black solid. It can be stored for several months at -40 °C with minimal decomposition.
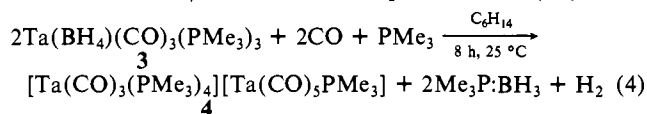
(17) The dmpe analogue of **2**, TaH$_5$(dmpe)$_2$, is stereochemically nonrigid from 25 to -140 °C. See: Tebbe, F. N. *J. Am. Chem. Soc.* **1973**, *95*, 5823.

$$Ta(BH_4)H_2(PMe_3)_4 + 3CO \xrightarrow[\text{0.25 h, 25 °C}]{C_6H_{14}}$$
$$\underset{\mathbf{1}}{}$$

$$\underset{\mathbf{3}}{Ta(BH_4)(CO)_3(PMe_3)_3} + H_2 + PMe_3 \quad (3)$$

at 2420, 2400, and 2140 cm^{-1} (benzene) and 1950, 1850, and 1830 cm^{-1} (hexane) which were assigned as B–H and C–O stretching frequencies, respectively. In addition, we were able to locate a –BH$_3$ deformation mode at 1115 cm^{-1} in the Nujol mull spectrum. The frequencies identified with the BH$_4$ moiety are indicative of monodentate coordination.[10] The room temperature ^{1}H NMR spectrum of **3** in toluene-d_8 shows a broad phosphine methyl resonance at δ 1.11 and a broad quartet ($^1J_{^{11}BH}$ = 82.8 Hz) at δ 0.05. The latter is typical of a fluxional tetrahydroborate ligand.[10] When the sample is cooled to –80 °C, the phosphine methyl resonance splits into two signals and the BH$_4$ resonance collapses to a broad hump.[18] The C–O stretching frequencies and low-temperature ^{1}H, ^{13}C, and ^{31}P NMR spectra of **3** are quite similar to those observed for the structurally characterized capped trig-onal-prismatic complex TaCl(CO)$_3$(PMe$_3$)$_3$.[19] As such, we assign structure C to **3**.

C
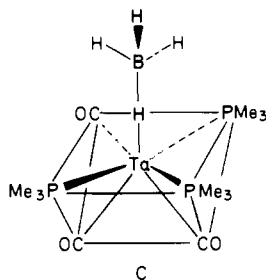
If **3** is not isolated in reaction 3, it slowly converts into an orange crystalline solid over the course of ca. 8 h. This product has been identified (vide infra) as the double salt [Ta(CO)$_3$(PMe$_3$)$_4$][Ta-(CO)$_5$PMe$_3$] (**4**)[20] and the yield (based on **1**), after recrystallization from THF/hexane, is 85% (eq 4). The ^{31}P{^{1}H} NMR

$$2\underset{\mathbf{3}}{Ta(BH_4)(CO)_3(PMe_3)_3} + 2CO + PMe_3 \xrightarrow[\text{8 h, 25 °C}]{C_6H_{14}}$$
$$\underset{\mathbf{4}}{[Ta(CO)_3(PMe_3)_4][Ta(CO)_5PMe_3]} + 2Me_3P{:}BH_3 + H_2 \quad (4)$$

spectrum of **4** (THF-d_8, 25 °C) exhibits three resonances. The singlet at δ –30 and the AX$_3$ pattern (δ_A +10.4, δ_X –33.5, J_{AX} = 24.4 Hz) are assigned to the anion and cation, respectively. The IR spectrum of **3** in THF shows the expected five-band pattern in the C–O stretching region: 1980, 1965, 1870, 1850, and 1825 cm^{-1}.

Single crystals of **3** were grown by slow diffusion of hexane into THF solutions at –40 °C and the structure was determined from X-ray diffraction data collected at –160 °C.[21] In the solid state, the compound is composed of discrete [Ta(CO)$_3$(PMe$_3$)$_4$]$^+$ cations and [Ta(CO)$_5$PMe$_3$]$^-$ anions and there are no unusual interion contacts. An ORTEP drawing of the cation, a capped octahedral complex, is shown in Figure 2. The view is down the approximate

3-fold axis and the methyl groups on P(25), which caps the trigonal Ta(CO)$_3$ face, have been omitted for clarity.[22] Selected structural data: Ta–C(33,35,37)$_{av}$, 2.07 [1] Å; Ta–P(17,21,29)$_{av}$, 2.642 [5] Å; Ta–P(25), 2.555 (3) Å; P(25)–Ta–C(33,35,37)$_{av}$, 72.0 [4]°; P(25)–Ta–P(17,21,29), 123.0 (1)°, 114.6 (1)°, and 131.9 (1)°.

An ORTEP drawing of the octahedral [Ta(CO)$_5$PMe$_3$]$^-$ anion is also shown in Figure 2. The equatorial Ta–C bond lengths, which average 2.10 [1] Å, are comparable to those found[23] in [PPN][Ta(CO)$_6$], i.e., 2.083 (6) Å. The axial Ta–C(14) bond length, 2.03 (1) Å, is significantly shorter, consistent with the expected trans effect.[24] Additional structural data: Ta–P(2), 2.568 (3) Å; P(2)–Ta–C(6,8,10,12,14), 91.9 (3)°, 87.2 (3)°, 84.8 (3)°, 94.6 (3)°, and 174.3(3)°.

We have shown here that tetrahydroborate complexes are useful precursors in organotantalum chemistry. Future papers in this series will expand on this theme and provide further details on the reactions and compounds described above.

Supplementary Material Available: Atomic positional and thermal parameters for compound **4** (2 pages). Ordering information is given on any current masthead page.

(22) (a) The sites occupied by the CO and PMe$_3$ ligands as well as the angles subtended from the capping phosphorus atom are in excellent agreement with the theoretical predictions of Hoffmann et al.[22b] (b) Hoffmann, R.; Beier, B. F.; Muetterties, E. L.; Rossi, A. R. *Inorg. Chem.* **1977**, *16*, 511.
(23) Calderazzo, F.; Englert, U.; Pampaloni, G.; Pelizzi, G.; Zamboni, R. *Inorg. Chem.* **1983**, *22*, 1865.
(24) A structural trans effect of this magnitude is not seen in the isoelectronic tungsten analogue. See: Cotton, F. A.; Darensbourg, D. J.; Kolthammer, B. W. S. *Inorg. Chem.* **1981**, *20*, 4440.

Thermocontrol of Ion Permeation through Ternary Composite Membranes Composed of Polymer/Liquid Crystal/Amphiphilic Crown Ethers

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Biological membranes are composed of various kinds of phospholipids, cholesterols, and proteins and the fundamental functions such as permeation and selectivity are frequently associated with the gel–liquid crystal phase transition. Recently, totally synthetic amphiphiles which contain a molecule with both a hydrophobic group of two alkyl chains and an appropriate hydrophilic group have been shown to also form oriented aggregates in water with a phase diagram very similar to that of natural phospholipids.[3,4] Therefore, they may be regarded as a new class of biomembrane models. To apply these new materials to practical systems without losing their membrane mimetic functions, one has to develop methods to polymerize or immobilize them in polymer matrices.[5-10] Composite membranes in which the ar-

(18) Anal. Calcd for Ta(BH$_4$)(CO)$_3$(PMe$_3$)$_3$: C, 28.37; H, 6.15. Found: C, 28.15; H, 6.20. ^{1}H NMR (ppm, C$_7$D$_8$, 360 MHz, –80 °C) δ 1.19 (d, 9, J_{PH} = 7.93 Hz), 1.00 (d, 18, J_{PH} = 3.66 Hz), –0.11 (v br, 4, BH$_4$); ^{13}C NMR (ppm, C$_7$D$_8$, 90.56 MHz, {^{1}H}, –80 °C) δ 259.3 (br s, 1, CO) 248.8 (br s, 2, CO), 16.6 (t, 6, J_{PC} = 10.8 Hz, P(CH$_3$)$_3$), 16.2 (d, 3, J_{PC} = 25.3 Hz, P'-(CH$_3$)$_3$); ^{31}P NMR (ppm, C$_7$D$_8$, 36.2 MHz, {^{1}H}, –80 °C) δ –13.9 (t, 1, J_{PP} = 72.8 Hz), –29.2 (d, 2, J_{PP} = 72.8 Hz).
(19) Luetkens, M. L., Jr.; Santure, D. J.; Huffman, J. C.; Sattelberger, A. P. *J. Chem. Soc., Chem. Commun.*, in press.
(20) Anal. Calcd for [Ta(CO)$_3$(PMe$_3$)$_4$][Ta(CO)$_5$(PMe$_3$)]: C, 28.59; H, 4.69. Found: C, 27.85; H, 4.84. ^{1}H NMR (ppm, C$_4$D$_8$O, 360 MHz, 25 °C) δ 2.15 (d, 9, J_{PH} = 9.16 Hz), 2.01 (br t, 27, J_{PH} = 3.05 Hz), 1.81 (d, 9, J_{PH} = 5.80 Hz).
(21) **4** crystallizes in the monoclinic space group $P2_1/c$ with a = 9.404 (9) Å, b = 17.007 (7) Å, c = 22.494 (11) Å, β = 91.78 (3)°; V = 7191.5 Å^3, and ρ(calcd) = 1.785 g cm^{-3} for M_r = 966.37 and Z = 4. The structure was solved by Patterson and Fourier techniques and refined by full-matrix least squares. Final discrepancy indices were R_F = 0.044 and R_{wF} = 0.046 for those 4045 reflections with $F_o \geq 3\sigma(F_o)$. The limits of data collection were $6 \leq 2\theta \leq 45°$ (Mo Kα).

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(3) Fendler, J. H. *Acc. Chem. Res.* **1980**, *13*, 7.
(4) Kunitake, T. *J. Macromol. Sci., Chem.* **1979**, *A13*, 587.
(5) Regen, S. L.; Czech, B.; Singh, A. *J. Am. Chem. Soc.* **1980**, *102*, 6638.
(6) Regen, S. L.; Shin, J.-S.; Yamaguchi, K. *J. Am. Chem. Soc.* **1984**, *106*, 2446.