Samarium Diiodide-Promoted Intramolecular Radical Cyclization of (η^4-Diene)Fe(CO)$_3$ Complexes Bearing Keto Side Chains

Ming-Chang P. Yeh,* Fu-Chun Wang, Jin-Juean Tu, Sun-Chi Chang, Cheng-Chiang Chou, and Jin-Wei Liao

Department of Chemistry, National Taiwan Normal University, 88 Sec 4, Ding-Jou Road, Taipei, Taiwan, 117 ROC

Received August 3, 1998

Reaction of samarium diiodide with (η^4-cyclohexadiene)Fe(CO)$_3$ complexes bearing keto side chains in THF/HMPA/*t*-BuOH gives fused bicyclo[4.3.0]nonenol derivatives, whereas (η^4-cycloheptadiene)Fe(CO)$_3$ analogues produce a bicyclo[5.3.0]decenol ring skeleton. The iron-mediated intramolecular radical addition allows for the direct stereocontrol of three contiguous stereogenic centers of these fused bicyclic skeletons. Under the same reaction conditions, intramolecular ketyl radical cyclization of acyclic (η^4-1,3-butadiene)Fe(CO)$_3$ complexes with keto side chains at the terminal position of the diene ligands furnishes disubstituted cyclopentanol and cyclohexanol derivatives with excellent diastereoselectivity.

The chemistry of diene−iron complexes is a subject of continuing interest. The general applications of the complexes are (i) electrophilic reactions with reactive carbon nucleophiles;[1] (ii) nucleophilic reactions with electrophiles;[2] and (iii) additions of nucleophiles to (η^5-pentadienyl)tricarbonyliron(0) and (η^5-cyclohexadienyl)-tricarbonyliron(0) cations.[3,4] Surprisingly, reports on the addition of free radical species to the diene ligand of (η^4-1,3-diene)tricarbonyliron(0) complexes are not found. Recently, our attention turned to the intramolecular radical cyclization of diene−iron complexes containing a primary iodide, for example **1**, using 1.1 equiv of tributyltin hydride and AIBN (cat.). However, only the reduced product **2** was isolated. Moreover, treatment of complex **1** with 1.2 molar equiv of SmI_2 and a catalytic amount of $FeCl_3$ using Molander's protocols also produced **2** in quantitative yield.[5] The primary radical might be formed under these two reaction conditions, however, the low nucleophilicity of the radical prevented its addition to the diene ligand. Inspired by recent successful examples of intramolecular addition of the relative nucleophilic ketyl radical[5c,d] to the dihydronaphthalenetricarbonylchromium(0) and tetralintricarbonylchromium(0) complexes developed by Schmalz,[6,7] we turned our efforts on radical cyclizations of this type to (η^4-1,3-diene)tricarbonyliron(0) complexes bearing keto groups. We here report on the first example of intramolecular cyclization of the ketyl radical to (η^4-1,3-diene)tricarbonyliron(0) complexes mediated by samarium(II) iodide.

Results and Discussion

The racemic starting complexes **3**−**9** required to test the intramolecular radical cyclization were prepared by addition of 2.5 molar equiv of methyllithium to the corresponding acid complexes following the literature procedure.[2c,8] Our first experiment began with **3**. Treatment of the keto complex **3** with 4.5 molar equiv of samarium(II) diiodide in THF with hexamethylphosphoric acid triamide (HMPA) as a cosolvent and *tert*-butyl alcohol as a proton source at −78 °C under nitrogen for 2 h provided a major product in 54% yield, identified as bicyclo[4.3.0]nonenol derivative **11** (entry 1, Table 1). It is important to note that three contiguous stereogenic centers of the racemic bicyclic compound **11** are created with high diastereoselectivity. The product of the relative stereochemistry as shown was isolated

(1) (a) Semmelhack, M. F.; Herndon, J. W. *Organometallics* **1983**, *2*, 363. (b) Semmelhack, M. F.; Herndon, J. W.; Liu, J. K. *Organometallics* **1983**, *2*, 1885. (c) Semmelhack, M. F.; Herndon, J. W.; Springer, J. P. *J. Am. Chem. Soc.* **1983**, *105*, 2497. (d) Semmelhack, M. F.; Le, H. T. M. *J. Am. Chem. Soc.* **1984**, *106*, 2715. (e) Semmelhack, M. F.; Herndon, J. W. *J. Organomet. Chem.* **1984**, *265*, C15. (f) Semmelhack, M. F.; Le, H. T. M. *J. Am. Chem. Soc.* **1985**, *107*, 1455. (g) Yeh, M. C. P.; Kang, K. K.; Hwu, C. C. *J. Chin. Chem. Soc.* **1991**, *38*, 475.

(2) (a) Graf, R. E.; Lillya, C. P. *J. Organomet. Chem.* **1976**, *122*, 377. (b) Birch, A. J.; Pearson, A. J. *J. Chem. Soc., Chem. Commun.* **1976**, 601. (c) Yeh, M. C. P.; Chuang, L. W.; Chang, S. C.; Lai, M. L.; Chou, C. C. *Organometallics* **1997**, *16*, 4435.

(3) (a) Birch, A. J.; Hass, M. A. *J. Chem. Soc. C* **1971**, 2465. (b) Pearson, A. J. In *Chemistry of Carbon-Metal Bond*; Hartley, F. R., Patai, S., Eds.; Wiley: Chichester, 1987; Vol. 14, Chapter 10. (c) Pearson, A. J.; Zettler, M. W. *J. Am. Chem. Soc.* **1989**, *111*, 3908.

(4) (a) Yeh, M. C. P.; Sheu, B. A.; Fu, H. W.; Tau, S. I.; Chuang, L. W. *J. Am. Chem. Soc.* **1993**, *115*, 5941. (b) Yeh, M. C. P.; Chuang, L. W.; Hwu, C. C.; Sheu, J. M.; Row: L. C. *Organometallics* **1995**, *14*, 3396.

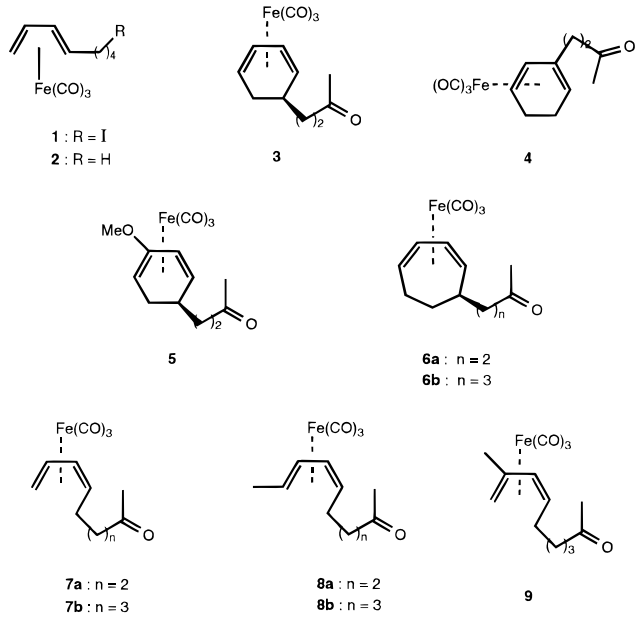
(5) (a) Molander, G. A.; Harris, C. R. *J. Org. Chem.* **1997**, *62*, 7418. (b) Molander, G. A.; Harris, C. R. *Chem Rev.* **1996**, *96*, 307. (c) Molander, G. A.; McKie, J. A. *J. Org. Chem.* **1992**, *57*, 3132. (d) Fukuzawa, S.; Nakanishi, A.; Fujinami, T.; Sakai, S. *J. Chem. Soc., Perkin Trans. 1* **1988**, 1669.

(6) (a) Schmalz, H.-G.; Siegel, S.; Bats, J. W. *Angew. Chem., Int. Ed. Engl.* **1995**, *34*, 2383. (b) Schmalz, H.-G.; Siegel, S.; Schwarz, A. *Tetrahedron Lett.* **1996**, *37*, 2947.

(7) In our preliminary examples, a simple (η^6-arene)tricarbonylchromium(0) complex, such as (η^6-4-phenyl-2-butanone)tricarbonylchromium(0) failed to undergo intramolecular radical cyclization using Schmalz's conditions. The reduced alcohol was isolated as the major product in 45% yield.

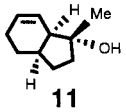
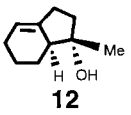
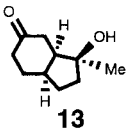
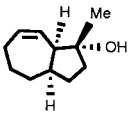
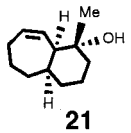
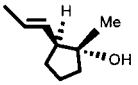
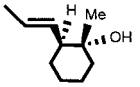
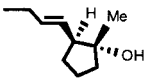
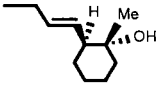
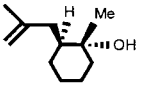
(8) Pearson, A. J. *J. Chem. Soc., Perkin Trans. 1* **1980**, 400.

10.1021/om980663d CCC: $15.00
Publication on Web 11/20/1998



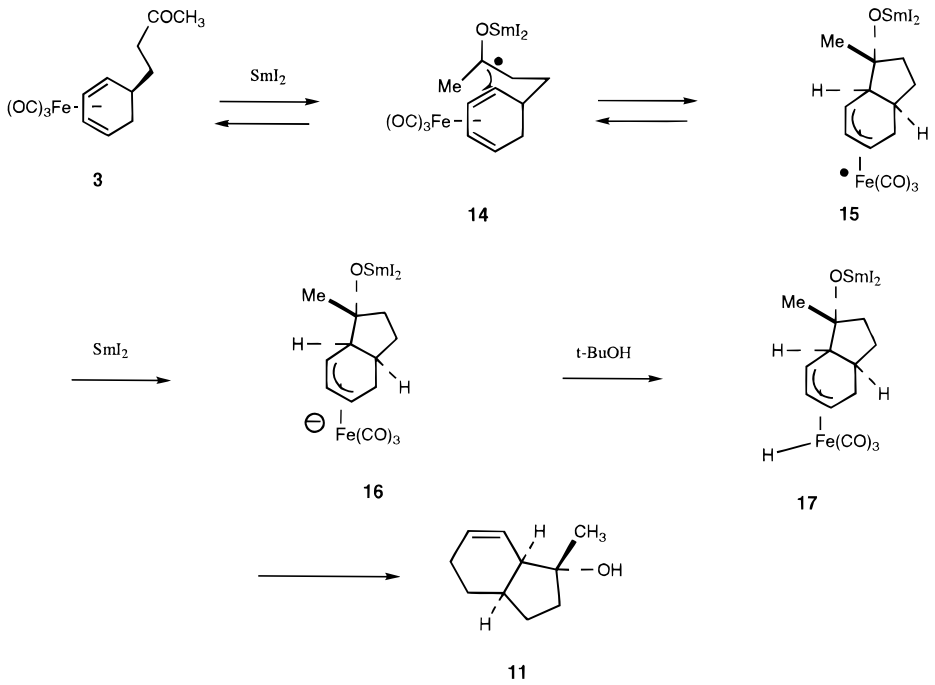
as a single diastereomer. Under the same reaction conditions, intramolecular radical cyclization of complex **4** afforded bicyclo[4.3.0]nonenol derivative **12** (39%) as the sole cyclized product (entry 2, Table 1). The relative stereochemistry of **11** and **12** was assigned on the basis of their close chemical shift values (81.8 and 79.5 ppm, respectively) of the tertiary alcohol carbon in their ^{13}C NMR spectra. The chemical shift values are consistent with the data of 1,2-*cis*-dialkyl-substituted cyclopentanol found in the literature.[5c,9] Interestingly, the complex with an electron-donating methoxy group, **5** (entry 3, Table 1), also underwent intramolecular radical cyclization to produce the bicyclo[4.3.0]nonanone derivative **13** in 89% yield as the only diastereomer isolated. Since a simple arene−chromium complex such as (η^6-4-phenylbutan-2-one)tricarbonylchromium does not underego intramolecular radical cyclization,[7] the result may indicate that iron−diene complexes undergo intramolecular ketyl radical addition easier than do arene−chromium complexes. However, the stereochemistry of the hydroxy group of **13** was assigned at the endo face on the basis of its ^{13}C NMR spectrum. The chemical shift of 69.6 ppm in **13** was assigned to the tertiary alcohol carbon. The observed upfield chemical shift of 69.6 ppm demands the cis relationship of the hydroxyl group and the adjacent alkyl substituents in the five-membered ring, and the assignment is consistent with the report found in the literature.[10] The origin of different stereochemical preferences observed for the formation of bicyclic compounds **11** and **12** and the bicyclo[4.3.0]nonanone derivative **13** was suggested as follows. Reaction of complex **3** with samarium(II) iodide in THF/HMPA/*t*-BuOH generated ketyl radical anion **14**. Due to the steric bulk of the ketyl radical bearing a samarium atom, the ketyl radical points away from the diene moiety in the transition state. Moreover, the anti relationship of the diene ligand and the ketyl oxygen is likely to be favorable in the transition state as noted previously (Scheme 1).[5b,11] Thus anti, *si*-face addition of the ketyl radical **14** at the terminal position (C-4) of the diene ligand gave the putative allyl radical intermediate **15**. Addition at the internal position (C-3) was not found. In constrast to the intramolecular carbanion additions, which occur exclusively at the internal position of the diene ligand, the ketyl radical species only attack at the terminal position of the diene ligand at −78 °C.[4] The allyl radical species **15** could further be reduced by samarium(II) iodide to provide

Table 1. Racemic Cyclic Tertiary Alcohols Obtained by Intramolecular Addition of Ketyl Radicals to (η^4-diene)Fe(CO)$_3$ Complexes in THF/HMPA/t-BuOH

No.	keto complex	product	yield[%]
1	**3**	 **11**	54
2	**4**	 **12**	39
3	**5**	 **13**	89
4	**6a**	 **20**	49
5	**6b**	 **21**	21
6	**7a**	 **22**	36
7	**7b**	 **23**	67
8	**8a**	 **24**	35
9	**8b**	 **25**	53
10	**9**	 **26**	63

(9) Cheney, B. V.; Grant, D. M. *J. Am. Chem. Soc.* **1967**, *89*, 5319.
(10) Yadav, V.; Fallis, A. G. *Can. J. Chem.* **1991**, *69*, 779.
(11) Beckwith, A. L. J. *Tetrahedron* **1981**, *37*, 3073.

Scheme 1

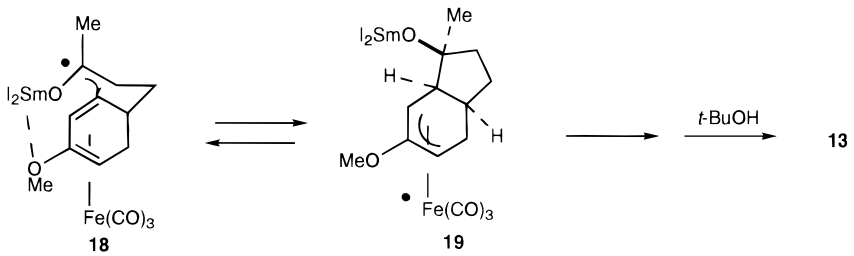


the allyl anionic complex **16**. Protonation of **16** with *t*-BuOH produced iron−hydride species **17**. Addition of the hydride at the less hindered site of the allyl ligand furnished 2-methylbicyclo[4.3.0]non-8-en-2-ol (**11**). The above reaction pathway involving single electron transfer of Sm(II) species and intramolecular ketyl radical cyclization was first proposed by Schmalz in the case of (η^6-arene)Cr(CO)$_3$ derivatives.[6] However, chelation control can be employed to alter the diastereoselectivity for complex **5**. As shown in Scheme 2, the methoxy group may provide a chelating center for the samarium species in **18**. Anti addition of the ketyl radical at the terminal C-4 position of the diene ligand gave allyl radical intermediate **19,** which led to the formation of **13** as the major product in 89% yield.

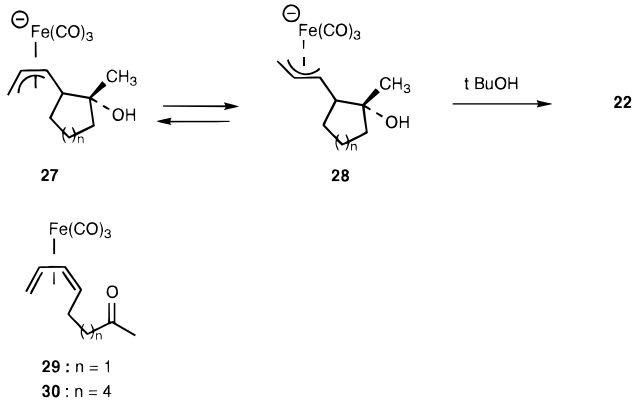
Under the same reaction conditions, intramolecular radical cyclizations of seven-membered ring substrates **6a** afforded bicyclo[5.3.0]decenol **20** (49%) as the only diastereomeric product (entry 4, Table 1). The observed chemical shift of 82.7 ppm for the tertiary alcohol carbon in **20** demands the *cis*-dialkyl relationship in a five-membered ring as stated previously. Increasing the tether length by 1 with complex **6b** (entry 5, Table 1) led to the bicyclo[5.4.0]decenol derivative **21** as the only diastereomer in 21% yield. The relative stereochemistry of **21** was determined by ^{13}C NMR spectroscopy. The chemical shift of 72.7 ppm assigned to the carbinol carbon is consistent with those of *cis*-1,2-disubstituted cyclohexanol found in the literature.[9]

Using the same approach, we are able to obtain 1,2-disubstituted cyclopentanol and cyclohexanol derivatives via intramolecular radical cyclization of acyclic (η^4-diene)Fe(CO)$_3$ complexes bearing a methyl ketone side chain at the terminal position of the diene ligand. Intramolecular cyclization of the ketyl radical anion generated by treating complex **7a** with 4.5 molar equiv of samarium(II) diiodide in THF/HMPA/*t*-BuOH afforded cyclopentanol derivative **22** as the only diastereomeric product in 36% yield after purification via flash column chromatography and short-path distillation of the residue. Several examples of radical cyclization of acyclic diene−iron complexes are summarized in Table 1 (entries 6−10). The stereochemical assignments of **22**−**26** were provided by comparison of their ^{13}C NMR chemical shifts of the tertiary alcohol carbon with the data of *cis*-1,2-dialkylcyclopentanol and -cyclohexanol derivatives formed in the literature.[9,10] The assignment of the stereochemistry of **22**−**26** is consistent with the reaction pathway proposed for the cyclic precursors (entries 1 and 2, Table 1). It is important to mention that the isolation of cyclohexanol derivative **26** (73.3 ppm for the tertiary carbinol center)[9] with the *cis*-1,2-dialkyl substituent is consistentent with the proposed reaction pathway stated in Scheme 1. Therefore, a methyl group presented at the C-2 position of the diene ligand, for example **9**, does not affect the relative stereochemistry of the cyclized product (entry 9, Table 1). The result may further explain the chelation effect (Scheme 2) caused by the methoxy group at the C-2 position of the diene ligand, which leads to the generation of **13** with an endo hydroxy group (entry 3, Table 1). Moreover, the stereochemistry of the double bonds in **22**−**25** (entries 6−9, Table 1) was assigned as trans on the basis of their ^{1}H NMR decoupling experiments. For example, the coupling constant of 15.2 Hz for the two vicinal vinyl protons of **22** suggested a trans orientation of the double bond. The allyl anion species **27** (Scheme 3) derived from the ketyl radical addition of **7a** followed by samarium diiodide reduction may undergo allyl syn−anti isomerization to give **28**.[12] Protonation of **28** with *t*-BuOH afforded **22.** Attempted intramolecular radical cyclization of complexes **29** and **30**, however, failed to produce cyclobutanol and cycloheptanol derivatives. The reduced secondary alcohols were isolated after allowing the reaction mixture to proceed for a longer period of time (14 h) at −78 °C. The difficulty in forming cyclobutanol and cycloheptanol

Scheme 2



Scheme 3



systems might be attributed to unfavorable formation of four- and seven-membered rings.

The reactions described herein demonstrate for the first time that intramolecular iron-mediated radical cyclization promoted by SmI_2 can be a convenient method for the formation of fused bicylic alcohols with excellent regio- and stereochemical control. The ability to achieve stereocontrol of three stereogenic centers in fused bicyclic compounds in a simple reaction may have further applications. This convenient synthetic strategy can also be applied for the diastereoselective synthesis of *cis*-1,2-diakylcyclopentanol and -cyclohexanol derivatives under very mild reaction conditions.

Experimental Section

All reactions were run under a nitrogen atmosphere in oven-dried glassware unless otherwise indicated. Anhydrous solvents or reaction mixtures were transferred via oven-dried syringe or cannula. Diethyl ether (ether) and tetrahydrofuran (THF) were distilled under nitrogen from a deep blue sodium benzophenone ketyl solution. Hexamethyl phosphoric acid triamide was distilled from calcium chloride. Flash column chromatography, following the method of Still,[13] employed E. Merck silica gel (Kieselgel 60, 230−400 mesh) using the indicated solvents. Analytical thin-layer chromatography was performed with silica gel 60 F_{254} plastic plates of 0.2 mm thickness from E. Merck. The term "concentration" refers to the removal of solvent with an aspirator pump (Yamato Instrument Company model WP-15) with a Buchi Rotovapor-R. The term "under nitrogen" implies that the apparatus was evacuated (oil pump) and then filled with nitrogen three times. The term "short-path distillation" refers to the process in which the entire distillation apparatus (a tube closed at one end, held horizontally), with the exception of the collection bulb, was slowly heated in an air bath from 25 to 150 °C under vacuum; the distillate was collected at −78 °C; and boiling points for fractions refers to the bath temperature range. Melting points were determined in open capillaries with a Thomas-Hoover apparatus and are uncorrected. ^{1}H nuclear magnetic resonance (NMR) spectra were obtained with JEOL-EX 400 (400 MHz) and Bruker AC-200 (200 MHz) spectrometers. The chemical shifts are reported in ppm with either tetramethylsilane (0.00 ppm) or $CHCl_3$ (7.26 ppm) as internal standards. ^{13}C NMR spectra were recorded with JEOL-EX 400 (100.4 MHz) and Bruker AC 200 (50.2 MHz) spectrometers with $CDCl_3$ (77.0 ppm) as the internal standard. Infrared (IR) spectra were recorded with a JASCO IR-700 spectrometer. Mass spectra were acquired on a JEOL JMS-D 100 spectrometer at an ionization potential of 70 eV and are reported as mass/charge (*m*/*e*) with percent relative abundance. High-resolution mass spectra were obtained with an AEI MS-9 double-focusing mass spectrometer and a JEOL JMS-HX 110 spectrometer in the Department of Chemistry, Central Instrument Center, Taichung.

General Procedure for Addition of Methyllithium to Acid Complexes. Synthesis of Iron Complexes Bearing a Methyl Ketone Side Chain.[8] Methyllithium (2.5 molor equiv) in hexane was added to a stirred solution of an acid complex in 10 mL of THF at 0 °C under nitrogen. The reaction was stirred at 0 °C for 30 min and then quenched with saturated aqueous ammonium chloride solution. The reaction mixture was diluted with 100 mL of 50% ethyl acetate/hexane. The resultant solution was washed with water (100 mL × 3) and brine (100 mL × 3), dried over anhydrous magnesium sulfate (10 g), and concentrated to give the crude mixtutre.

General Procedure for Intramolecular Radical Cyclization of (η^4-Diene)Fe(CO)$_3$ Complexes Bearing Keto Side Chains. In a typical procedure, to a solution of freshly prepared SmI_2[14] (4.5 mmol) and HMPA (20.0 mmol) in 20 mL of THF was added slowly a solution of a diene−iron complex (1.0 mmol) in 4.0 mL of THF followed by addition of 0.22 mL of *t*-BuOH under nitrogen at − 78 °C. The reaction mixture was allowed to stir at − 78 °C for 2 h. The reaction mixture was quenched with saturated aqueous ammonium chloride. The reaction mixture was diluted with ether (100 mL). The resultant solution was washed with water (100 mL × 3) and brine (100 mL × 3), dried over anhydrous magnesium sulfate (10 g), and concentrated to give the crude mixture.

[*exo*-4-[(1−4-η)-1,3-Cyclohexadien-5-yl]butan-2-one]-tricarbonyliron Complex (3). The crude mixture obtained from the addition of methyllithium (23.45 mmol) to the corresponding acid complex[2c] (2.74 g, 9.38 mmol) was purified via flash column chromatography (silica gel 1:5 ethyl acetate/hexanes) to give **3** (0.9 g, 3.1 mmol, 33%) as a yellow oil: IR (CH_2Cl_2) 3057, 3009, 2988, 2936, 2849, 2042, 1968, 1712, 1614, 1422, 1366, 1268, 1159, 951, 879, 820 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.36 (m, 1 H), 5.28 (m, 1 H,), 3.06 (m, 1 H), 3.02 (m, 1 H), 2.36 (t, J = 6.7 Hz, 2 H), 2.12 (s, 3 H), 2.09−1.96 (m, 2 H,), 1.61−1.41 (m, 2 H), 1.20 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 212.0, 208.4, 85.7, 84.5, 65.9, 59.5, 42.2, 37.5, 33.4, 30.6, 29.9; MS (70 eV) *m*/*e* (rel intensity) 290 (M^+, 20), 262 (60), 234 (58), 206 (48), 204 (100), 148(36), 134(94); HRMS (EI) *m*/*e* calcd for $C_{13}H_{14}FeO_4$ 290.0241, found 290.0239.

(12) (a) Chang, S.; White, P. S.; Brookhart, M. *Organometallics* **1993**, *12*, 3636. (b) Brookhart, M.; Yoon, J.; Noh, S. K. *J. Am. Chem. Soc.* **1989**, *111*, 4117.

(13) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

[*exo*-4-[(1−4-η)-1,3-Cyclohexadien-2-yl]butan-2-one]-tricarbonyliron Complex (4). The crude mixture obtained from the addition of methyllithium (7.57 mmol) to the corresponding acid complex[2c] (0.88 g, 3.03 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **4** (0.42 g, 1.45 mmol, 47%) as a yellow oil: IR (CH_2Cl_2) 3067, 3047, 2990, 2938, 2855, 2043, 1968, 1715, 1667, 1366, 1088, 797, 735 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.16 (d, J = 5.4 Hz, 1 H), 3.17 (t, J = 6.5 Hz, 1 H), 3.06 (m, 1 H), 2.74 (m, 2 H) 2.47 (m, 1 H), 2.43 (m, 1 H), 2.20 (s, 3 H), 1.68 (m, 2 H), 1.54 (m, 2 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 211.9, 207.1, 105.5, 84.8, 64.5, 59.2, 44.0, 30.0, 24.6, 23.7; MS (70 eV) *m/e* (rel intensity) 262 (8), 234 (33), 206 (20), 204 (100), 148 (46), 91 (38); HRMS (EI) *m/e* calcd for $C_{12}H_{14}FeO_3$ (M^+ − CO) 262.0292, found 262.0290.

[*exo*-4-[(1−4-η)-2-Methoxy-1,3-cyclohexadien-5-yl]butan-2-one]tricarbonyliron Complex (5). The crude mixture obtained from the addition of methyllithium (8.85 mmol) to the corresponding acid complex[2c] (1.14 g, 3.54 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **5** (0.46 g, 1.44 mmol, 41%) as a yellow oil: IR (CH_2Cl_2) 3684, 3402, 3063, 2991, 2984, 2937, 2042, 1963, 1712, 1608, 1485, 1452, 1429, 1413, 1363, 1255, 1228, 1172, 1155, 1093, 1020, 896, 879, 804, 754 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.07 (dd, J = 3.1 Hz, 1.2 Hz, 1 H), 3.63 (s, 3 H), 3.27 (dd, J = 3.1 Hz, 1.2 Hz, 1 H), 2.65 (t, J = 2.0 Hz, 1 H), 2.36 (t, J = 6.3 Hz, 2 H), 2.14 (s, 3 H), 2.06 (m, 1 H), 2.01 (m, 1 H), 1.58 (m, 1 H), 1.34 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 210.8, 208.5, 139.7, 66.3, 54.6, 54.3, 52.5, 42.5, 37.2, 33.5, 31.7, 29.9; MS (70 eV) *m/e* (rel intensity) 320 (18), 292 (30), 264 (48), 234 (100), 204 (17), 178 (19), 163 (97), 121 (28); HRMS (EI) *m/e* calcd for $C_{14}H_{16}FeO_5$ 320.1040, found 320.1042.

[*exo*-4-[(1−4-η)-1,3-Cycloheptadien-5-yl]butan-2-one]-tricarbonyliron Complex (6a). The crude mixture obtained from the addition of methyllithium (14.0 mmol) to the corresponding acid complex[15] (1.14 g, 5.6 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **6a** (0.62 g, 2.05 mmol, 37%) as a yellow oil: IR (CH_2Cl_2) 3071, 3061, 3057, 3055, 3042, 3020, 2996, 2976, 2961, 2930, 2873, 2670, 2440, 2306, 2038, 1948, 1715, 1607, 1559 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.26 (m, 2 H), 3.04 (m, 1 H), 2.77 (d, J = 7.3 Hz, 1 H), 2.42 (dd, J = 6.3 Hz, 2 H), 2.14 (s, 3 H), 2.07 (m, 1 H), 1.90 (m, 2 H), 1.59 (m, 1 H), 1.47 (m, 1 H), 1.33 (m, 1 H), 0.88 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 211, 208, 88, 87, 63, 59, 40, 38, 34, 30, 29.5, 28; MS (30 eV) *m/e* (rel intensity) 248 (M^+ − 2 CO, 27), 220 (100), 218 (36), 174 (4), 162 (73), 148 (85), 134 (45), 91 (96), 56 (37); HRMS (EI) *m/e* calcd for $C_{12}H_{16}FeO_2$ (M^+ − 2 CO) 248.0500, found 248.0503.

[*exo*-5-[(1−4-η)-1,3-Cycloheptadien-5-yl]pentan-2-one]-tricarbonyliron Complex (6b). The crude mixture obtained from the addition of methyllithium (10.5 mmol) to the corresponding acid complex[15] (1.4 g, 4.2 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **6b** (0.37 g, 1.20 mmol, 28%) as a yellow oil: IR (CH_2Cl_2) 3070, 3022, 2999, 2928, 2849, 2669, 2438, 2042, 1954, 1712, 1608, 1446, 1406, 1359, 1300, 1217, 1161, 1089, 951, 925, 910, 875, 846, 788, 754, 744, 736 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.25 (m, 2 H), 3.04 (m, 1 H), 2.82 (d, J = 7.3 Hz, 1 H), 2.39 (t, J = 7.3 Hz, 2 H), 2.13 (s, 3 H), 2.04 (m, 1 H), 1.89 (m, 2 H), 1.54 (m, 2 H), 1.37 (m, 1 H,), 1.25 (m, 1 H), 1.17 (m, 1 H), 0.87 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 211.7, 208.8, 88.3, 87.1, 63.9, 59.4, 43.7, 40.6, 38.4, 29.8, 28.3, 20.6; MS (30 eV) *m/e* (rel intensity) 262 (M^+ − 2 CO, 22), 234 (M^+ − 3 CO, 100), 232 (28), 176 (7), 162 (30), 148 (24), 105 (11), 91 (21), 56 (9); HRMS (EI) *m/e* calcd for $C_{13}H_{18}FeO_2$ (M^+ − 2 CO) 262.0656, found 262.0658.

[(6−9-η)-*cis*-6,8-Nonadien-2-one]tricarbonyliron Complex (7a). The crude mixture obtained from the addition of methyllithium (3.30 mmol) to the corresponding acid complex[2c] (0.37 g, 1.32 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **7a** (0.15 g, 0.50 mmol, 38%) as a yellow oil: IR (CH_2Cl_2) 3069, 3058, 3050, 3044, 2993, 2982, 2928, 2854, 2043, 1958, 1713, 1460, 1367, 1158, 807, 795 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.46 (m, 1 H), 5.30 (m, 1 H), 2.53 (m, 1 H), 2.37 (t, J = 6.4 Hz, 2 H), 2.12 (s, 3 H), 1.86 (m, 1 H), 1.80−1.40 (m, 4 H), 1.03 (m, 1 H); ^{13}C NMR (100.4 MHz, CDCl3) δ 211.4, 208.6, 90.8, 86.9, 59.4, 42.8, 40.9, 29.8, 28.2, 26.7; MS (70 eV) *m/e* (rel intensity) 278 (M^+, 2), 250 (5), 222 (34), 194 (100); HRMS (EI) *m/e* calcd for $C_{11}H_{14}FeO_3$ (M^+ − CO) 250.0292, found 250.0293.

[(7−10-η)-*cis*-7,9-Decadien-2-one]tricarbonyliron Complex (7b). The crude mixture obtained from the addition of methyllithium (3.05 mmol) to the corresponding acid complex[2c] (0.36 g, 1.22 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **7b** (0.15 g, 0.51 mmol, 42%) as a yellow oil: IR (CH_2Cl_2) 3083, 3007, 2940, 2047, 1971, 1711, 1609, 1460, 1362, 1092 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.41 (m, 1 H), 5.28 (m, 1 H), 2.54 (m, 1 H), 2.37 (t, J = 6.6 Hz, 2 H), 2.12 (s, 3 H), 1.84 (m, 1 H), 1.60−1.35 (m, 3 H), 1.25 (m, 3 H), 1.05 (m, 1 H); ^{13}C NMR (100.4 MHz, CDCl3) δ 211.3, 208.9, 90.6, 87.0, 60.0, 43.4, 40.8, 32.4, 29.9, 28.5 23.1; MS (70 eV) *m/e* (rel intensity) 292 (M^+, 5), 264 (6), 236 (24), 208 (100), 140 (46); HMRS (EI) *m/e* calcd for $C_{12}H_{16}FeO_3$ (M^+ − CO) 264.0449, found 264.0441.

[(6−9-η)-*cis*-6,*trans*-8-Decadien-2-one]tricarbonyliron Complex (8a). The crude mixture obtained from the addition of methyllithium (13.3 mmol) to the corresponding acid complex[4a] (1.55 g, 5.31 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **8a** (0.54 g, 1.86 mmol, 35%) as a yellow oil: IR (CH_2Cl_2) 3059, 3036, 2984, 2982, 2040, 1968, 1714, 1607, 1426, 1418, 1283 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.24 (dd, J = 9.0, 5.1 Hz, 1 H), 5.10 (dd, J = 7.8, 4.9 Hz, 1 H), 2.44 (m, 1 H), 2.36 (t, J = 6.1 Hz, 2 H), 2.30 (m, 1 H), 2.11 (s, 3 H), 1.61 (m, 1 H), 1.54 (m, 2 H) 1.43 (d, J = 5.9 Hz, 3 H), 1.08 (m, 1 H); ^{13}C NMR (100.4 MHz, CDCl3) δ 211.9, 208.5, 94.6, 82.2, 59.0, 57.8, 42.9, 29.9, 28.7, 26.9, 20.2; MS (70 eV) *m/e* (rel intensity) 292 (M^+, 32), 264 (18), 236 (44), 208 (100), 180 (10), 152 (7), 134 (29), 110 (6); HRMS (EI) *m/e* calcd for $C_{13}H_{16}FeO_4$ (M^+) 292.0398, found 292.0390.

[(7−10-η)-*cis*-7,*trans*-9-Undecadien-2-one]tricarbonyliron Complex (8b). The crude mixture obtained from the addition of methyllithium (16.3 mmol) to the corresponding acid complex[4a] (2.0 g, 6.53 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **8b** (0.76 g, 2.48 mmol, 35%) as a yellow oil: IR (CH_2Cl_2) 3059, 3017, 2955, 2039, 1966, 1713, 1607, 1426, 1420, 1092 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.23 (dd, J = 9.3, 4.9 Hz, 1 H), 5.11 (dd, J = 7.9, 4.9 Hz, 1 H), 2.43 (m, 1 H), 2.38 (t, J = 7.4 Hz, 1 H), 2.31 (m, 2 H), 2.12 (s, 3 H), 1.60−1.52 (m, 2 H), 1.44 (t, J = 5.9 Hz, 3 H) 1.37−1.25 (m, 3 H), 1.10 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 211.9, 208.5, 94.5, 82.3, 59.6, 57.6, 43.4, 33.0, 29.9, 29.0, 23.2, 20.2; MS (70 eV) *m/e* (rel intensity) 306 (M^+, 26), 278 (7), 250 (26), 222 (100), 194 (3), 166 (7), 140 (86); HRMS (EI) *m/e* calcd for $C_{11}H_{18}FeO_4$ (M^+ − 3 CO) 222.0707, found 222.0715.

[(6−9-η)-8-Methyl-*cis*-6,8-nonadien-2-one]tricarbonyliron Complex (9). The crude mixture obtained from the addition of methyllithium (26.5 mmol) to the corresponding acid complex[4a] (2.4 g, 10.6 mmol) was purified via flash column chromatography (silica gel, 1:5 ethyl acetate/hexanes) to give **9** (0.50 g, 1.71 mmol, 21%) as a yellow oil: IR (CH_2Cl_2) 3055, 3047, 2991, 2984, 2040, 1958, 1713, 1421, 1279, 1262, cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.21 (d, J = 7.3 Hz, 1 H), 2.40−2.34 (m, 3 H), 2.17(s, 3 H), 2.11(s, 3 H), 1.87 (m, 1 H), 1.67−

(14) Samarium(II) diiodide was prepared from samarium powder and 1,2-diiodoethane following the literature procedure. Kagan, H. B.; Girard, P.; Namy, J. L. *J. Am. Chem. Soc.* **1980**, *102*, 2693.

(15) Blankenfeldt, W.; Liao, J. W.; Lo, L. C.; Yeh, M. C. P. *Tetrahedron Lett.* **1996**, *37*, 7361.

1.46 (m, 4 H), 1.05 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 211.3, 208.6, 108, 86.7, 55.7, 43.4, 42.9, 30.0, 28.1, 27.0, 24.4; MS (70 eV) *m/e* (rel intensity) 292 (20, M^+), 264 (21), 236 (28), 208 (100), 179.9 (6), 148 (44), 96 (7); HRMS (EI) *m/e* calcd for $C_{13}H_{16}FeO_4$ 292.0398 (M^+), found 292.0402.

(1*S,2*R**,5*R**)-2-Methylbicyclo[4.3.0]non-8-en-2-ol (11).** The crude mixture obtained from the intramolecular radical addition of complex **3** (0.51 g, 1.72 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **11** (0.14 g, 0.93 mmol, 54%) as a colorless oil: IR (CH_2Cl_2) 3059, 3025, 2984, 2932, 2872, 1712, 1676, 1454, 1423, 1377, 1356, 1271, 1252, 1101, 922, 899, 870, 841 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (dd, J = 6.4, 2.5 Hz, 1 H), 5.65 (m, 1 H), 2.54 (m, 1 H), 2.30 (m, 1 H), 1.99 (m, 2 H), 1.92 (m, 2 H), 1.65 (m, 2 H), 1.41 (m, 2 H), 1.27 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$): δ 128.8, 126.7, 81.8, 50.3, 39.6, 35.0, 27.2, 25.6, 25.4, 21.8; MS (70 eV) *m/e* (rel intensity) 152 (M^+, 42), 135 (100), 119 (26), 109 (21), 94 (64), 92 (64), 83 (49); HRMS (EI) *m/e* calcd for $C_{10}H_{16}O$ (M^+) 152.1201, found 152.1209.

(1*S,2*S**)-2-Methylbicyclo[4.3.0]non-5-en-2-ol (12).** The crude mixture obtained from the intramolecular radical addition of complex **4** (0.53 g, 1.85 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **12** (82 mg, 0.55 mmol, 30%) as a colorless oil: IR (CH_2Cl_2) 3598, 3518, 3059, 2988, 2938, 2865, 1669, 1609, 1451, 1379, 1341, 1300, 1217, 1107 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.37 (t, J = 3.2 Hz, 1 H), 2.39 (m, 1 H), 2.26 (m, 1 H), 2.13 (m, 1 H), 1.99 (m, 2 H), 1.85−1.83 (m, 2 H), 1.78−1.72 (m, 2 H), 1.70−1.61 (m, 1 H), 1.45 (m, 1 H), 1.05 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 141.1, 119.3, 79.5, 51.4, 39.7, 29.1, 25.0, 22.5, 22.1, 22.0; MS (70 eV) *m/e* (rel intensity) 152 (30, M^+), 133 (8), 123 (7), 109 (33), 94 (52), 84 (64), 82 (100), 67 (18); HRMS (EI) *m/e* calcd for $C_{10}H_{16}O$ (M^+) 152.1201, found 152.1206.

(1*R,2*S**,5*R**)-2-Hydroxy-2-methylbicyclo[4.3.0]nonan-8-one (13).** The crude mixture obtained from the intramolecular radical addition of complex **5** (0.51 g, 1.58 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **13** (0.24 g, 1.40 mmol, 88%); IR (CH_2Cl_2) 3686, 3597, 3377, 3067, 3045, 2991, 2930, 2866, 2060, 1983, 1697, 1606, 1456, 1419, 1381, 1296, 1284, 1267, 1255, 1244, 1219, 1097, 1014, 976, 908, 879, 858 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.47 (d, J = 4.0 Hz, 2 H), 2.43 (m, 1 H), 2.18 (m, 1 H), 2.17 (m, 2 H), 1.75 (m, 2 H), 1.69 (m, 2 H), 1.26 (m, 2 H), 1.19 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 212.8, 69.6, 58.5, 38.7, 34.6, 29.8, 29.4, 28.7, 28.0, 25.7; MS (70 eV) *m/e* (rel intensity) 168 (M^+, 33), 153 (22), 132 (44), 122 (98), 149 (100), 106 (23), 93 (18); HRMS (EI) *m/e* calcd for $C_{10}H_{16}O_2$ (M^+) 168.1014, found 168.1156.

(1*S,2*R**,5*R**)-2-Methylbicyclo[5.3.0]deca-9-en-2-ol (20).** The crude mixture obtained from the intramolecular radical addition of complex **6a** (0.35 g, 1.16 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give ketone **20** (91 mg, 0.53 mmol, 49%) as a colorless oil: IR (CH_2Cl_2) 3694, 3404, 3168, 3072, 3006, 2966, 2670, 2474, 2418, 2316, 2292, 2254, 2106, 1905, 1556, 1439, 1431, 1410, 1363, 1307, 1222, 1092, 1036, 918, 905, 905 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.59 (m, 2 H), 2.57 (m, 1 H), 2.22 (m, 1 H), 2.13 (m, 2 H), 2.00 (m, 2 H), 1.66 (m, 4 H), 1.25 (m, 2 H), 1.23 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 131.2, 128.0, 83.5, 55.6, 41.2, 40.2, 30.4, 30.3, 29.6, 25.5, 24.0; MS (30 eV) *m/e* (rel intensity) 165 (M^+, 27), 149 (25), 147 (68), 123 (20), 120 (27), 119 (91), 105 (25), 93 (27), 91 (100), 79 (25), 67 (18), 51 (10); HRMS (EI) *m/e* calcd for $C_{11}H_{18}O$ (M^+) 166.1357, found 166.1445.

(1*S,2*R**,6*R**)-2-Methylbicyclo[5.4.0]undeca-10-en-2-ol (21).** The crude mixture obtained from the intramolecular radical addition of complex **6b** (0.20 g, 0.63 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **21** (23 mg, 0.13 mmol, 21%) as a colorless oil: IR (CH_2Cl_2) 3686, 3597, 3385, 3057, 3045, 3022, 2989, 2932, 2858, 2062, 1988, 1666, 1608, 1448, 1423, 1377, 1313, 1269, 1248, 1217, 1107, 933, 914, 879, 858, 842, 817, 810, 800, 788 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.71 (m, 1 H), 5.23(dd, J = 11.2 Hz, 1 H), 2.54 (m, 1 H), 2.13 (m, 3 H), 1.63 (m, 1 H), 1.59 (m, 3 H), 1.44 (m, 2 H), 1.30 (m, 2 H), 1.25 (m, 2 H), 1.21-(s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 130.7, 130.5, 72.7, 49.1, 35.4, 34.2, 34.0, 29.6, 29.4, 27.3, 21.9, 21.2; MS (30 eV) *m/e* (rel intensity) 180 (M^+, 19), 179 (1), 163 (10), 162 (74), 147 (52), 133 (57), 122 (69), 120 (63), 107 (41), 105 (56), 93 (56), 91 (80), 79 (100), 77 (51), 67 (46), 55 (52), 53 (32), 51 (11); HRMS (EI) *m/e* calcd for $C_{12}H_{20}O$ (M^+) 180.1514, found 180.1454.

(1*R,2*S**)-2-(*trans*-1-Propenyl)-1-methylcyclopentanol (22).** The crude mixture obtained from the intramolecular radical addition of complex **7a** (0.15 g, 0.54 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **22** (32 mg, 0.19 mmol, 36%): IR (CH_2Cl_2) 3680, 3597, 3458, 3076, 3057, 2997, 2962, 2876, 2048, 1975, 1834, 1639, 1448, 1379, 1302, 1194, 1122, 1001, 974, 937 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.50 (dt, J = 15.2, 6.4 Hz, 1 H), 5.33 (dd, J = 15.2, 8.3 Hz, 1 H), 1.93 (m, 1 H), 1.80−1.60 (m, 5 H), 1.50−1.25 (m, 4 H), 1.13 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 131.3, 126.4, 80.9, 54.4, 40.1, 29.8, 23.7, 20.6, 18.1.

(1*R,2*S**)-2-(*trans*-1-Propenyl)-1-methylcyclohexaneol (23).** The crude mixture obtained from the intramolecular radical addition of complex **7b** (0.15 g, 0.51 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **23** (50 mg, 0.34 mmol, 67%) as a colorless oil: IR (CH_2Cl_2) 3062, 3054, 3049, 2991, 2984, 2935, 1654, 1560, 1438, 1425, 1283, 1248, 1098, 911, 888, 847 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 (dt, J = 15.0, 6.3 Hz, 1 H), 5.37 (dd, J = 15.0, 8.8 Hz, 1 H), 2.04 (m, 1 H), 1.78−1.65 (m, 3 H), 1.71 (d, J = 6.2 Hz, 3 H), 1.40−1.20 (m, 6 H), 1.13 (s, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 125.7, 121.8, 66.2, 46.1, 34.2, 24.1, 19.4, 17.9, 16.1, 12.3; MS (70 eV) *m/e* (rel intensity) 154 (M^+, 94), 139 (43), 136 (54), 125 (55), 111 (93), 96 (100), 71 (50); HRMS (EI) *m/e* calcd for $C_{10}H_{18}O$ (M^+) 154.1358, found 154.1355.

(1*R,2*S**)-2-(*trans*-1-Butenyl)-1-methylcyclopentanol (24).** The crude mixture obtained from the intramolecular radical addition of complex **8a** (0.65 g, 2.23 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **24** (0.15 g, 0.78 mmol, 35%)**:** IR (CH_2Cl_2) 3380, 3073, 3039, 2997, 2992, 2974, 1988, 1608, 1437 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.44 (m, 2 H), 2.16 (m, 1 H), 1.90 (m, 2 H), 1.77−1.67 (m, 4 H), 1.64−1.58 (m, 2 H), 1.16 (s, 3 H), 0.79 (m, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 130.5, 125.8, 80.7, 50.2, 41.0, 33.6, 29.3, 22.9, 20.3, 17.9; MS (30 eV) *m/e* (rel intensity) 154 (M^+, 3), 153 (10), 136 (17), 123 (10), 107 (53), 89 (35), 77 (100), 57 (67); HRMS (EI) *m/e* calcd for $C_{10}H_{16}$ ($M^+ - H_2O$) 136.1252, found 132.1246.

(1*R,2*S**)-2-(*trans*-1-Butenyl)-1-methylcyclohexanol (25).** The crude mixture obtained from the intramolecular radical addition of complex **8b** (0.24 g, 0.78 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **25** (72 mg, 0.41 mmol, 53%) as a colorless oil: IR (CH_2Cl_2) 3391, 3063, 3050, 2986, 2936, 1620, 1460, 1421, 1258, 857 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.43 (m, 2 H), 2.37 (m, 1 H), 1.69−1.54 (m, 6 H), 1.39−1.13 (m, 5 H), 1.09 (s, 3 H), 0.94 (t, J = 11.2 Hz, 3 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 130.9, 126.2, 73.3, 48.0, 42.0, 33.4, 29.1, 25.6, 24.0, 21.0, 17.9; MS (30 eV) *m/e* (rel intensity) 168 (M^+, 15), 153 (17), 151 (100), 135 (5), 125 (16), 110 (17), 95 (22), 79 (25); HRMS (EI) *m/e* calcd for $C_{11}H_{20}O$ (M^+) 168.1514, found 168.1519.

(1*R,2*S**)-1-Methyl-2-(2-methylpropenyl)cyclohexanol (26).** The crude mixture obtained from the intramolecular radical addition of complex **9** (0.51 g, 1.67 mmol) was purified via flash column chromatography (silica gel, 1:20 ethyl acetate/hexanes) to give **26** (0.18 g, 1.05 mmol, 63%) as a

colorless oil: IR (CH_2Cl_2) 3596, 3401, 3067, 3050, 2990, 2975, 2935, 2859, 1645, 1445, 1424, 1379, 1157, 1128 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.77 (s, 1 H), 4.71 (s, 1 H), 2.39 (m, 1 H), 1.74 (s, 3 H), 1.76−1.19 (m, 9 H), 1.13 (s, 3 H), 0.94 (m, 1 H); ^{13}C NMR (100.4 MHz, $CDCl_3$) δ 145.4, 111.7, 73.3, 45.3, 42.0, 38.8, 29.1, 25.6, 24.0, 22.2, 21.0; MS (70 eV) *m*/*e* (rel intensity) 168 (M^+, 10), 153 (11), 151 (95), 150 (100), 153 (63), 125 (37), 108 (46), 97 (30), 95 (90), 81 (37), 69 (48); HRMS (EI) *m*/*e* calcd for $C_{11}H_{20}O$ (M^+) 168.1514, found 168.1521.

Acknowledgment. Support for this research from the National Science Council (87-2113-M-003-009) of the Republic of China is gratefully acknowledged.

OM980663D