Molybdenum–π-Allyl Compounds for Chemoselective Synthesis of Tetrahydrofurans, *cis*-1,3-Dienes, *cis*-Hexatrienes, and Isoxazole Compounds

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The enolate $CpMo(CO)_2(\eta^3\text{-}1\text{-}syn\text{-}C_3H_4COCH_2Li)$ was utilized for stereoselective synthesis of (2*R**,5*S**)-3-oxo-5-R-2-vinyltetrahydrofurans (R = Ph, 3′-methoxyphenyl) and (2*R**,3*S**,5*S**)-3-hydroxy-5-R-2-vinyltetrahydrofurans (R = Ph, 3′-methoxyphenyl). Compounds of the type $CpMo(CO)_2(\eta^3\text{-}1\text{-}syn\text{-}C_3H_4COCH_2R)$ for chemoselective synthesis of *cis*-1,3-dienes and *cis*-1,3,5-hexatrienes were used with excellent selectivities and fair yields. The syntheses of compounds of the type $CpMo(CO)_2(\eta^3\text{-}1\text{-}anti\text{-}CH_2CH(OH)R\text{-}3\text{-}syn\text{-}R'\text{-allyl})$ are described; their reactions with excess nitrosonium tetrafluoroborate produced 3-(1′-R′CH_2=CH)-5-R-isoxazole, which involves a remarkable nitrosyl insertion into the π-allyl ligand as the key step.

Introduction

$CpMo(CO)_2(\eta^3\text{-allyl})$[1,2] has proved useful in organic synthesis. Similar to other useful organometallic complexes such as $(\eta^4\text{-diene})Fe(CO)_3$,[2,3] $(\eta^6\text{-benzene})Cr(CO)_3$,[4] and $(CO)_5Cr{=}C(OR)R'$,[5] these π-allyl compounds are inexpensive, easily prepared, and stable chemically in air. The most interesting feature of these compounds is the versatility of removal of the metal fragment. The dicarbonyl compounds are readily converted to the $CpMoCO(NO)(\eta^3\text{-allyl})^+$ cation[6] which in synthetic utility is equivalent to an allyl cation. Because of their highly electrophilic nature, cations of this type have been widely used as reactive intermediates for the synthesis of α-functionalized olefins.[6–8] Further treatment of this cation with LiCl gives $CpMoNO(Cl)(\eta^3\text{-allyl})$[9] which in synthesis functions equivalently to an allyl anion. Analogous to allylsilanes or boranes, this Mo–π-allyl complex condenses with aldehydes in excellent diastereoselectivity to give homoallylic alcohols.[10,11] One therefore expects that the major task in developing new chemistry of this field is to control the stereochemical course by functionalization of the dicarbonyl Mo–π-allyl compounds.

We previously reported[12] diastereofacial aldol condensation of the enolate $CpMo(CO)_2(\eta^3\text{-}1\text{-}syn\text{-}C_3H_4COCH_2Li)$ (I) with aldehydes; the major diastereomeric products have been utilized for stereoselective syntheses of 1,3-diol and 1,3,5-triol. In continuing development of the utilization of Mo–π-allyl complexes in organic synthesis, we report here a new molybdenum-mediated stereoselective synthesis of the derivatives of tetrahydrofurans, *cis*-1,3-diene, *cis*-1,3,5-hexatrienes, and isoxazole compounds; part of this work has appeared as a brief communication.[13]

Results and Discussion

Stereoselective Synthesis of Tetrahydrofurans. Following an analogous procedure,[12] the reaction of the enolate $CpMo(CO)_2(\eta^3\text{-}1\text{-}syn\text{-}C_3H_4COCH_2Li)$ (I) with 3-methoxybenzaldehyde gave the two diastereomers **2a** and **2b** in a 78:22 ratio which were separated on a SiO_2–gel column; the combined yield was 80%. Similar to that of its phenyl analogue **3a**[12] given in Scheme I, $NaBH_4$ reduction of **2a** gave the η^3-allyl 1,3-diol (**4**) as one single diastereomer. We utilized **2a–3a**, **4**, and **5** for stereoselective synthesis of tetrahydrofurans as depicted in Scheme I. Treatment of **2a** and **3a** with $NOBF_4$ (1.2 equimolar) in CH_3CN gave their η^3-allyl nitroso cations.[5] Stirring of these cations over Na_2CO_3 in ether for 3 h, followed by air oxidation produced (2*R**,5*S**)[14]-3-oxo-5-R-2-vinyltetrahydrofurans (R = 3-C_6H_4OMe (**6**), Ph (**7**)) in moderate yields (44–51%). Using this synthetic procedure, we obtained the (2*R**,3*S**,5*S**)-3-hydroxy-5-R-2-vinyltetrahydrofurans (R = 3′-C_6H_4OMe (**8**), Ph (**9**)) as single stereoisomers from **3** and **4** in fair yields (>50%). The specific proton positions on the ether ring of these compounds were determined by a proton NOE experiment. Irradiation of the H^5 signal (δ 4.25 ppm) of **9** caused an increase in the intensities of the H^1 (δ 4.25 ppm) and CH_2=C*H* (δ 5.89 ppm) protons by 2.2% and 1.8%, respectively, whereas the H^4 proton signal (δ 4.46 ppm) was unaffected. This information reveals that the ether ring formations of **6–9** were from the intramolecular $CHPhO^-$ attack at the C(3) allylic carbon opposite the $CpMo(CO)_2$ fragment.

† National Tsing-Hua University.
‡ National Taiwan University.

(1) Pearson, A. J. *Synlett* **1990**, 10.
(2) Pearson, A. J. In *Advances in Metal-Organic Chemistry*; Liebeskind, L. S., Ed.; JAI Press: London, 1989; Vol 1, Chapter 1.
(3) Gree, R. *Synthesis* **1989**, 341 and references cited therein.
(4) Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransition Metal Chemistry*; University Science Books; Mill Valley, CA, 1987; Chapter 20.
(5) Wulff, W. D. In *Advances in Metal-Organic Chemistry*; Liebeskind, L. S., Ed.; JAI Press: London, 1989; Vol. 1.
(6) Faller, J. W.; Rosan, A. M. *Ann. N.Y. Acad. Sci.* **1977**, *295*, 18.
(7) Hansson, S.; Miller, J. F.; Liebeskind, L. S. *J. Am. Chem. Soc.* **1990**, *112*, 9660.
(8) Pearson, A. J.; Mallik, S.; Penny, M. D.; Youngs, W. J. *J. Am. Chem. Soc.* **1990**, *112*, 8034.
(9) (a) Faller, J. W.; Rosan, A. M. *Ann. N.Y. Acad. Sci.* **1977**, *295*, 186. (b) Adams, R. D.; Chodosh, D. F.; Faller, J. W.; Rosan, A. M. *J. Am. Chem. Soc.* **1979**, *101*, 2570.
(10) Faller, J. W.; John, J. A.; Mazzieri, M. R. *Tetrahedron Lett.* **1989**, *30*, 1769.
(11) Faller, J. W.; Linebarrier, D. L. *J. Am. Chem. Soc.* **1989**, *111*, 1937.
(12) Vong, W.-J.; Peng, S.-M.; Lin, S.-H.; Lin, W.-J.; Liu, R.-S. *J. Am. Chem. Soc.* **1991**, *113*, 573.
(13) Lin, S.-H.; Peng, S.-M.; Liu, R.-S. *J. Chem. Soc., Chem. Commun.* **1992**, 615.
(14) The term (2*R**,5*S**) represents an enantiomeric mixture of (2*R*,5*S*) and (2*S*,5*R*). The picture is shown in one enantiomeric form (2*R*,5*S*) for clarity.

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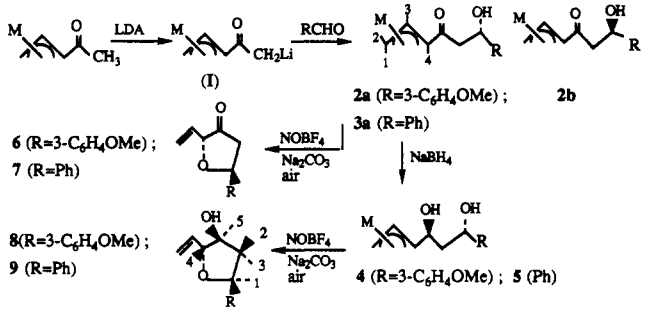
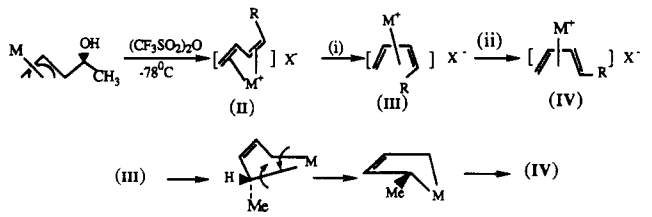
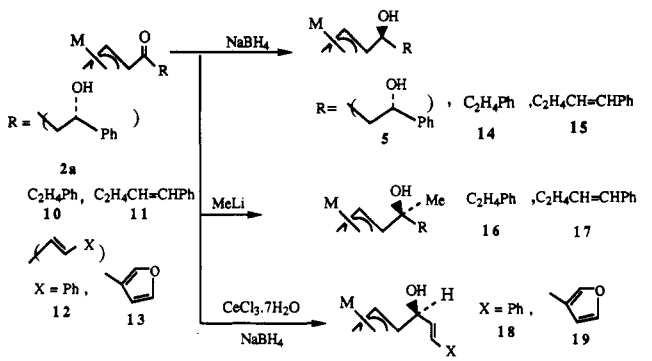
Scheme I. M = $CpMO(CO)_2$



Scheme II. M = $CpMo(CO)_2$, X = CF_3SO_3

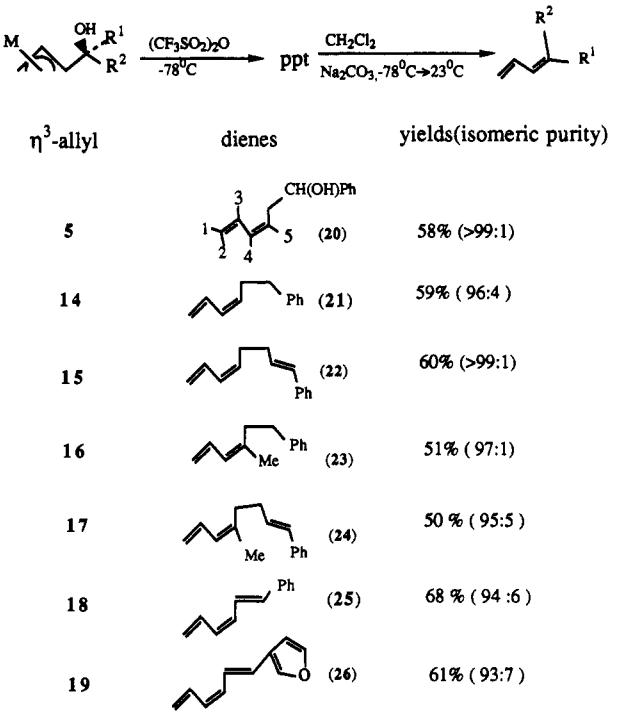


Scheme III. M = $CpMo(CO)_2$



Syntheses of *cis*-1,3-Dienes and *cis*-1,3,5-Hexatrienes. We previously reported[12] that treatment of α-hydroxyallyl complex (R = Me, Scheme II) in diethyl ether at −78 °C stereoselectively generated the η^4-*cis-s-trans*-pentadiene cation (R = Me) (II). Here, the $CpMo(CO)_2$ fragment acted as a base to displace $CF_3SO_3^-$ in an intramolecular S_N2 mechanism. Above −25 °C in CH_2Cl_2, this cation underwent an irreversible isomerization to the η^4-*cis-s-cis*-pentadiene cation (R = Me) (III) and finally to the η^4-*trans-s-cis*-pentadiene cation (IV). Although this reaction is potentially useful for synthesis of *cis*-1,3-diene through demetalation of the cations II and III, its feasibility is challenged by the particular isomerization step ii via a ring-flipping mechanism which involves metallocyclopent-3-ene as intermediate.[15] In order to circumvent this problem, we employed bulky substituent R, hoping that it would exert increasing interligand steric hindrance within the cation itself to slow down isomerization step i or ii. According to this approach, we transformed the functionalized η^3-ketones **3a** and **10–14** to the α-hydroxyallyl compounds **5** and **14–19** by $NaBH_4$ reduction, CH_3Li addition, and Ce^{III}–$NaBH_4$ reduction, respectively. In all cases, all isolated alcohols exist as only one diastereomer after workup, to which the (3*R**,4*S**) configuration is assigned like that given for **5**.[12] In a typical reaction, the η^3-allyl complex **5** was treated with 1.1 equimolar of $(CF_3SO_2)_2O$ in diethyl ether at −78 °C, immediately generating a dark red precipitate. At this temperature, the ether layer containing a small amount (5%) of the diene (cis: trans = 0.5–1) was decanted away. Among our attempts, the best method to acquire the most nearly pure *cis*-1,3-diene was to decompose this cation thermally. The precipitate above, after being washed twice with diethyl ether (−78 °C), was dissolved in CH_2Cl_2 and stirred over anhydrous Na_2CO_3 solid at the same temperature. On gradually increasing the temperature (−78 → 23 °C, 4 h), we found a single organic component spectrally analyzed as *cis*-1-hydroxy-1-phenylhexa-3,5-diene[16] (**20**) in good isomeric purity (>99%) according to ^1H NMR and glc analyses. Application of this method to other η^3-α-hydroxyallyl compounds was also successful, and selective synthesis of the cis form was achieved in reasonable purity (>93%) as were yields (>50%) including the *cis*-1,3,5-hexatriene derivatives **25–26**; the results are shown in Scheme IV. For all cases, Na_2CO_3 was indispensable in the reaction to neutralize the residual acid.

Scheme IV



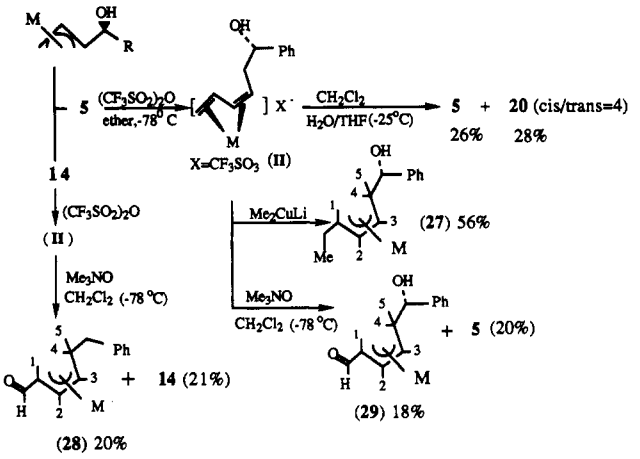
The cis configurations of **20–22**, **25**, and **26** are readily distinguished by the discrete coupling parameters between the two alkenyl protons, having the value J_{45} = 10 Hz; the trans isomer is expected to have J_{45} = 16–17 Hz. The cis configurations of **23** and **24** were clarified by means of a proton NOE experiment. Irradiation of the H^4 proton signals (δ 5.86 ppm) of **23** resulted in a 2.8% increase in intensity of the methyl resonance (δ 1.80 ppm) whereas the $=CMeCH_2$ proton signal (δ 2.45 ppm) was unaffected. The trans isomers of **21** and **23–26** in trace amount found by glc were further identified by GC–mass spectrometry. The *cis*-1,3,5-hexatrienes **25** and **26** can be kept at −30 °C in the dark for 2 days without degradation.

The cation responsible for liberation of *cis*-1,3-diene may be either η^4-*cis-s-trans*-diene (II) or η^4-*cis-s-cis*-diene (III) cation. To clarify the role of these two cations, we treated the precipitate derived from **5** and $(CF_3SO_2)_2O$ with H_2O/THF in CH_2Cl_2 at −25 °C, after which 1,3-diene was liberated abruptly. As depicted in Scheme V, the

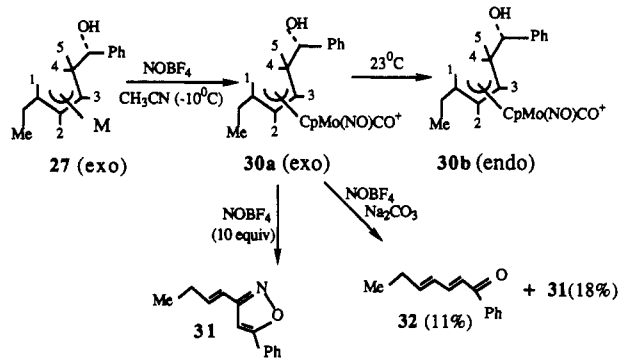
(15) (a) Faller, J. W.; Rosan, A. M. *J. Am. Chem. Soc.* **1977**, *99*, 4858. (b) Faller, J. W.; Incorvia, M. J. *Inorg. Chem.* **1968**, *7*, 840.

(16) The pure trans form of **20** was synthesized from BF_3-catalyzed condensation of PhCHO to a molybdenum–η^3-pentadienyl compound, see: Lin, S.-H.; Yang, Y.-J.; Liu, R.-S. *J. Chem. Soc., Chem. Commun.* **1991**, 1004.

Scheme V



Scheme VI. M = $CpMo(CO)_2$



products after workup were found as a mixture of **5** (26%) and 1,3-diene **20** (cis:trans = 4, 28%). This information indicates that no II → III transformation was observed before II was thermally decomposed to *cis*-1,3-diene. If the η^4-*cis-s-cis*-1,3-diene cation were present at −25 °C, its reaction with H_2O would be expected to give the anti isomer like that of its η^4-*cis*-pentadiene cation.[12]

Treatment of this salt with Me_2CuLi in THF at −78 °C afforded **27** in 54% yield. Demetalation of this salt with anhydrous Me_3NO gave the η^3-1-formyl compounds **28** and **29** in 18–20% yield, which provided interesting instances of oxidation of η^4-*s-trans*-diene (II). The η^3-1-*syn*-3-*anti*-allyl configurations of **27–29** were indicated by both the coupling parameter J_{12} = 9.0–12.5 Hz and J_{23} = 6.5–7.8 Hz and the chemical shift of the syn H^3 proton (δ 3.4–4.0 ppm) which is greater than that of the anti H^1 proton (δ 1.76–2.10 ppm).

It is interesting to note that *cis*-1,3-diene was liberated from only the *cis-s-trans*-1,3-diene (II). We believe that the presence of bulky R in the anti position of III exerts more severe steric hindrance on the proximate CH_2=CH group than the cation II because of its planar geometry. Although the structure of η^4-*s-cis*-diene is more favorable energetically than that of η^4-*s-trans*-diene,[17] the strong interligand steric hindrance of III may overwhelm this electronic superiority to retard the isomerization step i.

Synthesis of 3-(1′-RCH_2=CH)-5-R-isoxazole Derivatives. The allyl orientation of **27** with respect to the cyclopentadienyl group is assigned to be the exo conformation, indicated by the anti H^1 proton NMR resonance (δ 1.83 ppm), closer to that (δ 1.76 ppm) of the exo isomer

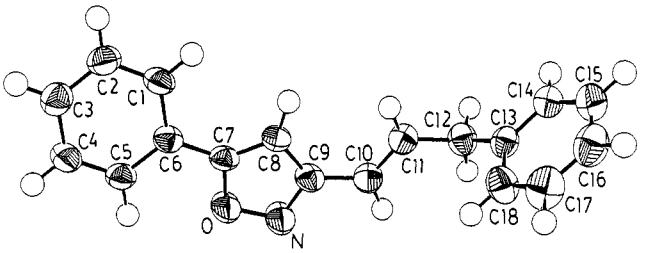


Figure 1. ORTEP drawing of compound **41**.

of $CpMo(CO)_2(\eta^3$-*syn*-$MeC_3H_4)$[18] than to the corresponding proton resonance of the endo isomer at δ 2.76 ppm. Treatment of **27** with nitrosonium tetrafluoroborate (1.20 equimolar) in CH_3CN (−10 °C), followed by precipitation with anhydrous diethyl ether, afforded the *exo*-η^3-*anti,syn*-allyl nitroso cation as a single stereoisomer (90%). Like its parent $CpMo(NO)(CO)(\eta^3$-allyl$)^+$ cation[9], the exo isomer **30a** underwent a slow and irreversible isomerization to the more stable endo isomer **30b**. An organic component was liberated in a trace amount during formation of **30a**; we managed to acquire this organic component **31** in significant proportion (37%) by treatment of **30a** with excess nitrosonium tetrafluoroborate (10-fold excess) in CH_3CN at 0 °C, to cause demetalation of metal complexes. The endo isomer **30b** likewise gave the same compound in 18% yield under the same conditions. The structure of **31** was identified as an isoxazole molecule on the basis of X-ray diffraction measurements on its phenyl analogue **41** (vide infra). The ORTEP drawing (Figure 1) reveals that the η^3-allyl ligand is capable of undergoing a rare nitrosyl insertion which adds regioselectively at the η^3-*anti*-allylic carbon.[19] NO insertion into the η^3-*syn*-allylic carbon is incompatible with our expectation, because such a process would give the energetically unfavorable cis olefin. During the course of isoxazole production from **30a**, an aqueous Na_2CO_3 solution was added to quench the reaction which gave the dienone **32** (11%) in addition to **31** (18%). The presence of **32** indicates that the C*H*(OH)Ph group of **30a** was oxidized to ketone by the NO^+ ion. In organic chemistry the nitrosonium ion NO^+ is known to act well both for electrophilic nitrosation and as an oxidizing reagent.[20]

As isoxazole belongs to a class of useful aromatic heterocyclic compounds,[21] it is important to examine the generalization of this reaction. The results are provided in Table I. The starting *anti,syn*-allyl compounds **33–40** were prepared via a procedure similar to synthesis of **27**. For convenience, the isoxazole synthesis was conducted as a one-pot reaction, i.e. direct treatment of the dicarbonyl allyl complexes with excess nitrosonium tetrafluoroborate in CH_3CN at 0 °C; the yields were moderate, 35–55%. No isoxazole formation was detected for the η^3-*syn,syn*-allyl isomer **40**[16] (entry 9) under the same conditions; the compound remained almost completely as the nitrosyl allyl cation, shown by IR spectra [ν(CO) 2083 (vs), ν(NO) 1711 (vs) cm^{-1}]. The fact that activity toward nitrosyl insertion is operable only for these π-*syn,anti*-allyl complexes is

(17) (a) Erker, G.; Wicker, J.; Engel, K.; Rosenfeldt, F.; Dietrich, W.; Kruger, C. *J. Am. Chem. Soc.* **1980**, *102*, 6344. (b) Nakamura, A.; Yasuda, H. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 723.

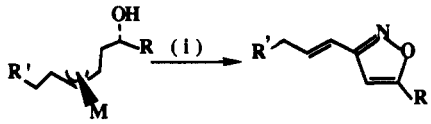
(18) Faller, J. W.; Chen, C. C.; Mattina, M. J.; Jakubowski, A. *J. Organomet. Chem.* **1973**, *52*, 361.

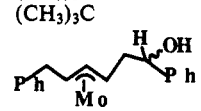
(19) One precedent is known for NO insertion into a π-allyl ligand, see: Schoonover, M. W.; Baker, E. C.; Eisenberg, R. *J. Am. Chem. Soc.* **1979**, *101*, 1880.

(20) (a) Olah, G. A. *Aldrichimica Acta* **1979**, *12*, 189. (b) Olah, G. A.; Salm, G.; Staral, J. S.; Ho, T. L. *J. Org. Chem.* **1978**, *43*, 173.

(21) Lang, S. A.; Lin, Y. I. In *Comprehensive Heterocyclic Chemistry; The Structure, Reaction, Synthesis and Uses of Heterocyclic Compounds*; Katritzky, A. R., Rees, C. W., Eds.; Pergamon Press: London, 1984; Vol. 6, p 416.

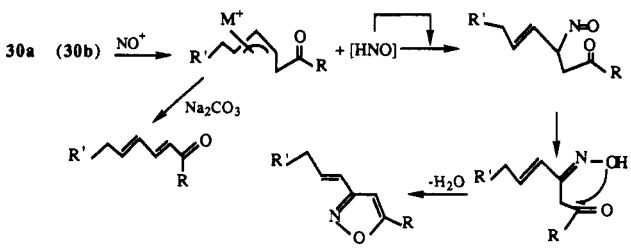
Table I. M = $CpMo(CO)_2$[a]



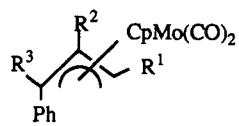
entry	R	R′	product no. (yields,[b] %)
1	Ph	CH_3 (27)	**31** (37)
2	Ph	Ph (33)	**41** (54)
3	Ph	n-C_4H_9 (34)	**42** (46)
4	m-C_6H_4OMe	n-C_4H_9 (35)	**43** (51)
5	m-C_6H_4OMe	CH_3 (36)	**44** (38)
6	$(CH_3)_2CH$	n-C_4H_9 (37)	**45** (39)
7	$(CH_3)_3C$	n-C_4H_9 (38)	**46** (44)
8	$(CH_3)_3C$	CH_3 (39)	**47** (35)
9		(40)[c]	NR

[a] (i) $NOBF_4$ (10.0 equiv), CH_3CN, 0 °C, 6 h. [b] Yields were calculated on the amount of the Mo–allyl compounds. [c] Consisting of 1:1 diastereomers.

Scheme VII. $M^+ = CpMo(CO)NO^+$



attributed to its stronger interligand steric hindrance relative to the η^3-*syn,syn*-allyl isomer. The bulky *anti*-$CH_2CH(OH)R$ is expected to exert steric repulsion on the CH^1–CH^2 bond because of its planarity, for which evidence is available from the X-ray structure of the related η^3-*syn,anti*-allyl compounds;[22] these contain a long Mo–C(Ph) bond (2.42 Å), significantly longer than those of the Mo–CR^1 (2.30 Å) and Mo–Cr^2 (2.30–2.33 Å) bonds. Such an



asymmetric metal–allyl bonding is expected to increase chemical reactivities of metal complexes toward the substrate, partly due to the intrinsic tendency toward $\eta^3 \rightarrow \eta^1$ slippage.[10,11] An additional effect of this steric hindrance is that the η^3-*anti,syn*-allyl compound became a more energetic species than the η^3-*syn,syn* isomer, and the former is superior to the latter in chemical reactions for this thermodynamic reason.

We propose the mechanism of formation of isoxazole compounds in Scheme VII. The role of the nitrosonium ion may be 2-fold: (i) to oxidize[23] secondary alcohols to ketones and (ii) to initiate nitrosyl insertion into the η^3-*anti*-allylic carbon.[24,25] Further hydrogen abstraction of the resulting allylic nitroso compound produced an oximine which is expected to give an isoxazole after intramolecular cyclization. The oxidation step i, well documented,[20,23] is further confirmed in our results by isolation of **32**, which is envisaged to be derived from deprotonation of the η^3-ketone compound by Na_2CO_3. The details of the insertion step ii are unclear at present. The cationic nature of **30a** and **30b** prohibits approach of the NO^+ ion, for which promotion of nitrosyl insertion by acting as an external ligand is hence eliminated. No NO gas was detectable by mass spectrometry during isoxazole formation. In a separate experiment, we treated **30a** with NO(g) in CH_3CN at 0 °C (6 h) which gave a complicated mixture of organic components. According to our proposal, the transient species $NO^-(HNO)$[20,23] was incorporated into the π-allyl group. The highly electrophilic nature[5-9] of the Mo–η^3-allyl nitroso cation strongly favors this reaction. The possibility that nitrosyl insertion occurs from the coordinated NO ligand during oxidative decomplexation of metal compounds cannot be excluded.

Experimental Section

All operations were carried out under argon in a Schlenk apparatus or glovebox. The solvents benzene, diethyl ether, tetrahydrofuran, and hexane were dried with sodium/benzophenone and distilled before use. Dichloromethane, acetonitrile, and chloroform were dried over calcium hydride and distilled. Anhydrous trimethylamine oxide was prepared by subliming its dihydrate (Fluka) at 110 °C without further purification. $Mo(CO)_6$, dicyclopentadiene, LiCl, $NOBF_4$, diisopropylamine, BuLi (1.6 M in hexane), CuI, and the aldehydes, RCHO (R = Ph, Me_2CH, Me_3C, 3-C_6H_4OMe, 2-furfuryl, and 3-furfuryl) were used without purification. Compounds **1**,[12] **3a**,[12] **5**,[12] and **40**[16] were synthesized according to the literature.

All ^1H and ^{13}C NMR spectra were measured on Bruker AM-400 and Varian Gemini-300 spectrometers; both ^1H and ^{13}C spectra were referenced to tetramethylsilane. Mass data for molybdenum–η^3-allyl compounds are reported according to the ^{98}Mo isotope (24%). High-resolution mass spectra were recorded on a JEOL HX-110 spectrometer. Glc analysis of compounds **20–26** was performed on a Shimadzu GC-8A PF with a capillary column (SE-52). Microanalyses were conducted at the Microanalytic Laboratory of National Cheng Kung University, Tainan, Taiwan.

(a) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)[(1,2,3-η)-3-*syn*-6-hydroxy-6-(*m*-methoxyphenyl)-4-oxo-2-hexen-1-yl]molybdenum (2a, 2b). Compound **1** (0.50 g, 1.67 mmol) in THF (20 mL) was treated with an ethereal solution (5 mL) of lithium diisopropylamide (1.70 mmol) at −78 °C. As the solution turned deep red, 3-methoxybenzaldehyde (0.40 g, 2.94 mmol) was added and the resultant mixture was stirred for 1 h at −78 °C. An aqueous NH_4Cl solution (20 mL) was added to quench the reaction, and the solution was concentrated to 20 mL. Diethyl ether (30 mL) was added, and the mixture was stirred rapidly. The organic layer was decanted, and the aqueous layer was twice extracted with 30 mL of ether. The combined extracts were evaporated to dryness to produce a yellow oil as a mixture of diastereomers **2a** and **2b**. Further elution through a long silica gel column (4.5 cm diameter × 26 cm height) with ether/hexane = 1:3 as the eluting solvent allowed separation of these two diastereomers. Complex **2a** ((3*R**,6*S**), orange solid, 0.46 g, 1.05 mmol): IR (Nujol, cm^{-1}) ν(CO) 1953 (s), 1874 (s), 1655 (m); ^1H NMR (400 MHz, $CDCl_3$): 7.24 (1 H, Ph, t, J = 8.0 Hz), 6.97 (1 H, Ph, s), 6.80 (1 H, Ph, d, J = 8.0 Hz), 5.17 (5 H, Cp, s), 5.07 (1 H, C*H*(OH), dd, J = 8.7, 4.1 Hz), 5.00 (1 H, H^3, td, J = 10.0, 7.6 Hz), 3.79 (3 H, OMe, s), 3.00 (1 H, H^2, d, J = 7.6 Hz), 2.97 (1 H, C*H*H′, dd, J = 16.6, 4.1 Hz), 2.82 (1 H, CH*H*′, dd, J = 16.6, 8.7 Hz), 1.83 (1 H, H^4, d, J = 10.0 Hz), 1.31 (1 H, H^1, d, J = 10.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) 239.6, 236.9, 207.5, 106.1, 145.0, 129.7, 118.4, 113.5, 111.3, 93.9, 71.2, 70.8, 55.4, 49.9, 40.3; mass

(22) Su, G.-M.; Lee, G.-H.; Peng, S.-M.; Liu, R.-S. *J. Chem. Soc., Chem. Commun.* **1992**, 215.
(23) Olah, G. A.; Ho, T. L. *Synthesis* **1991**, *1976*, 610.
(24) For reviews of a NO insertion into a metal–carbon bond, see: (a) Richter-Addo, G. B.; Legzdins, P. *Chem. Rev.* **1988**, *88*, 991. (b) Gladfelter, W. L. *Adv. Organomet. Chem.* **1985**, *24*, 41.
(25) External NO^+ ion promoting a nitrosyl insertion has been reported by Legzdins and co-workers; see: (a) Legzdins, P.; Wassink, B.; Einstein, F. W. B.; Willis, A. C. *J. Am. Chem. Soc.* **1986**, *108*, 317. (b) Legzdins, P.; Richter-Addo, G. B.; Einstein, F. W. B.; Jones, R. H. *Organometallics* **1987**, *6*, 1807.

(75 eV, *m/e*) 410 (M^+ – CO), 392 (M^+ – CO – OH – 1), 382 (M^+ – 2CO). Anal. Calcd for $C_{20}H_{20}MoO_5$: C, 55.05; H, 4.62. Found: C, 55.27; H, 4.38. Complex **2b** ((3*R**,6*R**), orange solid, 0.13 g, 0.30 mmol): IR (Nujol, cm^{-1}) ν(CO) 1953 (s), 1874 (s), 1655 (m); ^1H NMR (400 MHz, $CDCl_3$) 7.23 (1 H, Ph, t, *J* = 8.0 Hz), 6.95 (1 H, Ph, s), 6.93 (1 H, Ph, d, *J* = 8.0 Hz), 6.79 (1 H, Ph, d, *J* = 8.0 Hz), 5.10 (5 H, C_5H_5, s), 5.09 (1 H, C*H*(OH), dd, *J* = 10.0, 3.4 Hz), 5.00 (H^3, ddd, *J* = 10.8, 9.8, 7.2 Hz), 3.92 (1 H, OH), 3.78 (3 H, OMe, s), 3.05–2.98 (2 H, H^+ + C*H*H′, m), 2.89 (1 H, CH*H*′, dd, *J* = 17.5, 3.4 Hz), 1.82 (1 H, H^4, d, *J* = 9.8 Hz), 1.29 (1 H, H^1, d, *J* = 10.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 239.5, 237.2, 207.4, 160.1, 145.2, 129.7, 118.2, 113.2, 111.3, 94.0, 70.7, 70.2, 55.3, 55.2, 49.0, 40.6.

(b) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(3*R,4*S**,6*S**)-(1,2,3-η)-3-*syn*-4,6-dihydroxy-6-(*m*-methoxyphenyl)-2-hexen-1-yl]molybdenum (4).** To a THF/methanol (1:1, 20 mL) solution of **2a** (1.00 g, 2.28 mmol) was added $NaBH_4$ (0.70 g, 18.4 mmol) at 23 °C, and the resulting solution was stirred for 1 h. An aqueous HCl (1 M) solution was added to neutralize the solution. The solution was concentrated to half volume, diethyl ether (30 mL) was added, and the mixture was stirred rapidly. The organic layer was decanted, and the aqueous layer was twice extracted with diethyl ether (30 mL). The combined extracts were evaporated to dryness and eluted through a silica column with ether/hexane (1:1) as the eluting solvent. A yellow band was developed, collected, and evaporated to dryness to give a yellow oil (0.92 g, 2.12 mmol). IR (Nujol, cm^{-1}): ν(CO) 1939 (s), 1855 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 7.20 (1 H, Ph, t, *J* = 8.1 Hz), 6.89 (1 H, Ph, s), 6.87 (1 H, Ph, d, *J* = 8.1 Hz), 6.72 (1 H, Ph, d, *J* = 8.1 Hz), 5.24 (5 H, C_5H_5, s), 5.00 (1 H, C*H*Ph, dd, *J* = 7.8, 2.6 Hz), 3.94 (1 H, H^3, ddd, *J* = 10.6, 10.0, 7.4 Hz), 3.74 (3 H, OMe, s), 3.73 (1 H, C*H*(OH)CH³, m), 2.64 (1 H, H^2, d, *J* = 7.4 Hz), 2.17–2.99 (3 H, H^4 + C*H*H′, m), 0.72 (1 H, H^1, d, *J* = 10.6 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): 239.8, 237.3, 159.9, 146.4, 129.6, 117.9, 112.7, 111.3, 91.7, 71.5, 71.4, 71.2, 66.7, 55.0, 48.2, 35.6. Mass (75 eV, *m/e*): 412 (M^+ – CO), 384 (M^+ – 2CO). Anal. Calcd for $C_{20}H_{22}MoO_5$: C, 54.80; H, 5.05. Found: C, 54.92; H, 5.18.

(c) (i) Synthesis of (2*R,5*S**)-5-(*m*-Methoxyphenyl)-3-oxo-2-vinyltetrahydrofuran (6).** To a CH_3CN (5-mL) solution of **2a** (260 mg, 0.60 mmol) was added $NOBF_4$ (70 mg, 0.62 mmol), and the mixture was stirred at 0 °C for 10 min before addition of Na_2CO_3. The solution continued to stir for 6 h under air atmosphere, and a red precipitate gradually deposited during this period. After filtration, ether (10 mL) was added to the filtrate, and the organic layer was twice washed with H_2O (10 mL). After removal of the solvent, the residues were chromatographed with a preparative SiO_2 TLC plate with diethyl ether/hexane (1:3) as the eluting solvent, which produced an organic band of **6** (0.048 g, 0.22 mmol). IR (Nujol, cm^{-1}): ν(CO) 1757 (s), ν(C=C) 1650 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.28 (1 H, Ph, t, *J* = 8.0 Hz), 6.94 (1 H, Ph, s), 6.93 (1 H, Ph, t, *J* = 8.0 Hz), 6.84 (1 H, Ph, d, *J* = 8.0 Hz), 5.84 (1 H, CH_2=C*H*, ddd, *J* = 17.0, 10.5, 2.0 Hz), 5.49 (1 H, CH*H*′=, d, *J* = 17.0 Hz), 5.39 (1 H, H^1, dd, *J* = 8.6, 6.9 Hz), 5.31 (1 H, C*H*H′=, d, *J* = 10.5 Hz), 4.65 (1 H, d, H^4, *J* = 2.0 Hz), 3.80 (3 H, OMe, s), 2.88 (1 H, H^3, dd, *J* = 18.1, 6.9 Hz), 2.58 (1 H, H^2, dd, *J* = 18.1, 9.1 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 213.0, 160.2, 142.2, 131.4, 130.0, 118.1, 117.4, 111.5, 80.9, 76.7, 55.2, 43.7. HRMS: calcd for $C_{13}H_{14}O_3$, 218.0939; found, 218.0923.

(ii) Synthesis of (2*R,5*S**)-5-Phenyl-3-oxo-2-vinyltetrahydrofuran (7).** This compound was prepared from **3a**, $NOBF_4$, and Na_2CO_3 according to the procedure for synthesis of **6**; the yield was 51%. IR (Nujol, cm^{-1}): ν(CO) 1758 (s), ν(C=C) 1645 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.35–7.27 (5 H, Ph, m), 5.80 (1 H, CH_2=C*H*, ddd, *J* = 16.8, 10.6, 2.0 Hz), 5.50 (1 H, CH*H*′=, d, *J* = 16.8 Hz), 5.37 (1 H, H^1, dd, *J* = 8.6, 6.9 Hz), 5.28 (1 H, C*H*H′, d, *J* = 10.6 Hz), 4.61 (1 H, H^4, d, *J* = 2.0 Hz), 2.85 (1 H, H^3, dd, *J* = 18.2, 7.2 Hz), 2.56 (1 H, H^2, dd, *J* = 18.2, 8.2 Hz), ^{13}C NMR (75 MHz, $CDCl_3$): δ 213.1, 140.6, 131.4, 128.9, 128.4, 126.0, 117.4, 80.9, 76.9, 43.7. HRMS: calcd for $C_{12}H_{12}O_2$, 188.0837; found, 188.0845.

(iii) Synthesis of (2*R,3*R**,5*S**)-3-Hydroxy-5-(*m*-methoxyphenyl)-2-vinyltetrahydrofuran (8).** This compound was prepared similarly from **4**, $NOBF_4$, and Na_2CO_3 according to the procedure for synthesis of **6**, the yield was 55%. IR (Nujol, cm^{-1}): ν(OH) 3400 (br vs), ν(C=C) 1648 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.24 (1 H, Ph, t, *J* = 8.0 Hz), 6.96 (1 H, Ph, s), 6.78 (1 H, Ph, d, *J* = 8.0 Hz), 5.88 (=CH, ddd, *J* = 16.0, 10.7, 5.8 Hz), 5.39 (1 H, CH*H*′=, d, *J* = 16.8 Hz), 5.22 (1 H, C*H*H′=, d, *J* = 10.7 Hz), 5.13 (1 H, H^1, dd, *J* = 7.2, 6.8 Hz), 4.46 (1 H, H^4, dd, *J* = 5.8, 4.6 Hz), 4.24 (1 H, H^5, ddd, *J* = 6.2, 5.8, 4.6 Hz), 3.79 (3 H, OCH_3, s), 2.65 (1 H, H^2, ddd, *J* = 13.5, 7.2, 6.2 Hz), 2.01 (1 H, H^3, ddd, *J* = 13.5, 6.9, and 5.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): 159.8, 144.9, 136.3, 129.5, 117.7, 116.7, 112.8, 110.9, 86.8, 78.7, 76.7, 55.2, 42.4. HRMS: calcd for $C_{13}H_{16}O_3$, 220.1095; found, 220.1092.

(iv) Synthesis of (2*R,3*S**,5*S**)-3-hydroxy-5-phenyl-2-vinyltetrahydrofuran (9).** This compound was prepared similarly from **5**, $NOBF_4$, and Na_2CO_3; the yield was 51%. IR (Nujol, cm^{-1}): ν(OH) 3391 (br s), ν(C=C) 1643 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.40–7.24 (5 H, Ph, m), 5.89 (1 H, =CH, ddd, *J* = 16.9, 10.4, 5.7 Hz), 5.40 (1 H, CH*H*′=, d, *J* = 16.9 Hz), 5.22 (1 H, C*H*H′=, d, *J* = 10.4 Hz), 5.15 (1 H, H^1, t, *J* = 7.3 Hz), 4.46 (1 H, H^4, dd, *J* = 5.7, 4.6 Hz), 4.25 (1 H, H^5, m), 2.66 (1 H, H^2, ddd, *J* = 13.0, 7.3, 7.2 Hz), 2.01 (1 H, H^3, ddd, *J* = 13.0, 7.2, 5.8 Hz), 1.76 (1 H, OH, s). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.3, 136.5, 128.6, 127.5, 125.7, 116.8, 86.7, 78.9, 76.7, 42.4. HRMS: calcd for $C_{12}H_{14}O_2$, 190.0944; found, 190.0954.

(d) (i) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(1,2,3-η)-3-*syn*-6-phenyl-4-oxo-2-hexen-1-yl]molybdenum (10). The enolate I (1.00 mmol) prepared in section a was treated with benzyl iodide (0.142 g, 1.02 mmol) in THF at −78 °C, and the mixture was stirred for 0.5 h. The solution was warmed to 23 °C and evaporated to dryness. Elution through a silica column with hexane/ether (1:1) solvent produced a yellow band which gave an orange oil of **10** (0.37 g, 0.94 mmol) after concentration. IR (Nujol, cm^{-1}): ν(CO) 1951 (vs), 1871 (vs), 1663 (s). ^1H NMR (400 MHz, $CDCl_3$): 7.35–7.22 (5 H, Ph, m), 5.10 (5 H, C_5H_5, s), 5.05 (1 H, H^3, ddd, *J* = 11.2, 10.0, 7.2 Hz), 3.06–2.84 (5 H, H^+ + CH_2-CO + PhCH_2, m), 1.93 (1 H, H^4, d, *J* = 10.0 Hz), 1.31 (1 H, H^1, d, *J* = 11.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 239.7, 237.4, 206.0, 141.4, 128.9, 128.5, 126.2, 93.7, 70.8, 55.6, 43.0, 40.1, 30.0. Mass (12 eV, *m/e*): 390 (M^+). Anal. Calcd for $C_{19}H_{18}MoO_3$: C, 58.47; H, 4.65. Found: C, 58.67; H, 4.40.

(ii) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)[(1,2,3-η)-*trans,trans*-8-phenyl-4-oxo-2,7-octadien-1-yl]molybdenum (11). This compound was prepared similarly from the enolate I and 1-phenyl-3-iodopropene; the yield was 88%. IR (Nujol, cm^{-1}): 1953 (s), 1874 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 7.35–7.18 (5 H, Ph, m), 6.44 (1 H, C*H*Ph, d, *J* = 15.9 Hz), 6.20 (1 H, C*H*=CPh, dt, *J* = 15.9, 6.7 Hz), 5.17 (5 H, C_5H_5, s), 5.03 (1 H, H^3, ddd, *J* = 11.2, 9.8, 7.2 Hz), 3.01 (1 H, H^2, d, *J* = 7.2 Hz), 2.81–2.48 (4 H, CH_2CH_2, m), 1.92 (1 H, H^4, d, *J* = 9.8 Hz), 1.28 (1 H, H^1, d, *J* = 11.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): 239.8, 237.2, 206.3, 137.7, 130.9, 129.4, 128.7, 127.2, 126.1, 93.8, 70.9, 55.7, 41.3, 39.9, 27.3. Mass (12 eV, *m/e*): 418 (M^+). Anal. Calcd for $C_{21}H_{20}MoO_3$: C, 60.58; H, 4.84. Found: C, 60.72; H, 4.96.

(e) (i) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(1,2,3-η)-*trans,trans*-6-phenyl-4-oxo-hexa-2,5-dien-1-yl]molybdenum (12). The enolate I (3.34 mmol) prepared above was treated with PhCHO (0.56 g, 5.00 mmol) at −78 °C, and the mixture was stirred for 1 h before anhydrous Na_2CO_3 (0.50 g) was added. The solution was slowly warmed to 23 °C with rapid stirring. After 4 h, the solution was evaporated to dryness and extracted with diethyl ether (50 mL). The extract was washed twice with H_2O (20 mL) and further dried over $MgSO_4$. After removal of ether, the residues were chromatographed through a silica column with ether/hexane (1:2) as the eluting solvent. An orange band was developed, collected, and concentrated to yield **12** as an orange solid (1.04 g, 2.58 mmol). IR (Nujol, cm^{-1}): ν(CO) 1952 (s), 1872 (s); ν(C=C) 1655 (m), 1632 (w). ^1H NMR (400 MHz, $CDCl_3$): 7.60 (1 H, C*H*Ph, d, *J* = 16.0 Hz), 7.57–7.55 (2 H, Ph, m), 7.36 (3 H, Ph, m), 6.85 (1 H, C*H*=CPh, d, *J* = 16.0 Hz), 5.23 (5 H, C_5H_5, s), 5.17 (1 H, H^3, ddd, *J* = 11.3, 9.8, 7.2 Hz),

3.10 (1 H, H^2, d, J = 7.2 Hz), 2.28 (1 H, H^4, d, J = 9.8 Hz), 1.41 (1 H, H^3, d, J = 11.3 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): 239.8, 237.6, 195.7, 141.5, 135.1, 130.4, 129.1, 128.5, 126.4, 94.0, 71.5, 55.6, 40.9. Mass (75 eV, m/e): 390 (M^+). Anal. Calcd for $C_{19}H_{16}MoO_3$: C, 58.78; H, 4.15. Found: C, 58.62; H, 4.19.

(ii) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)[(1,2,3-η)-*trans,trans*-6-(3′-furfuryl)-4-oxo-hexa-2,5-dien-1-yl]molybdenum (13). This compound was prepared similarly from the enolate I and 3-furaldehyde and then dehydroxylated over Na_2CO_3; the yield was 90%. IR (Nujol, cm^{-1}): ν(CO) 1951 (s), 1871 (s), 1657 (s), 1604 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.66 (1 H, s), 7.49 (1 H, =CH, d, J = 15.8 Hz), 7.41 (1 H, s), 6.61 (1 H, s), 6.57 (1 H, CH=, d, J = 15.8 Hz), 5.23 (5 H, C_5H_5, s), 5.16 (1 H, H^3, ddd, J = 10.2, 9.8, 7.2 Hz), 3.09 (1 H, H^2, d, J = 7.2 Hz), 2.22 (1 H, H^4, d, J = 9.8 Hz), 1.38 (1 H, H^1, d, J = 10.2 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): 239.1, 237.0, 195.0, 144.9, 144.3, 131.2, 126.3, 123.1, 107.5, 93.9, 71.5, 55.6, 40.9. Mass (75 eV, m/e): 380 (M^+). Anal. Calcd for $C_{17}H_{14}MoO_4$: C, 53.98; H, 3.73. Found: C, 53.91; H, 3.78.

(f) (i) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(3*S,4*S**)-(1,2,3-η)-3-*syn*-4-hydroxy-6-phenyl-2-hexen-1-yl]molybdenum (14).** This compound was prepared from $NaBH_4$ reduction of **10** according to the procedure in (b); the yield was 83%. IR (Nujol, cm^{-1}): ν(CO) 1942 (vs), 1859 (vs). ^1H NMR (400 MHz, $CDCl_3$): δ 7.30–7.20 (5 H, Ph, m), 5.32 (5 H, Cp, s), 4.04 (1 H, H^3, ddd, J = 10.5, 9.8, 7.4 Hz), 3.64 (1 H, C*H*(OH), m), 2.88–2.68 (3 H, H^2 + CH_2Ph, m), 2.07 (2 H, CH_2, m), 1.85 (1 H, H^4, dd, J = 9.8, 7.7 Hz), 0.83 (1 H, H^1, d, J = 10.5 Hz). Mass (12 eV, m/e): 376 (M^+ − OH − 1). Anal. Calcd for $C_{19}H_{20}MoO_3$: C, 58.17; H, 5.14. Found: C, 58.81; H, 5.31.

(ii) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)[(1,2,3-η)-3-*syn*-4-hydroxy-8-phenyl-2,7-octadienyl-1-yl]molybdenum (15). This compound was similarly prepared from $NaBH_4$ reduction of **11**; the yield was 88%. IR (Nujol, cm^{-1}): ν(CO) 1942 (vs), 1859 (vs). ^1H NMR (300 MHz, $CDCl_3$): (exo isomer) δ 7.31–7.15 (5 H, Ph, m), 6.41 (1 H, =CHPh, d, J = 15.9 Hz), 6.21 (1 H, CH=, dt, J = 15.9, 6.8 Hz), 5.27 (5 H, Cp, s), 4.00 (1 H, H^3, td, J = 10.6, 7.3 Hz), 3.63 (1 H, CH(OH), m), 2.69 (1 H, H^2, d, J = 7.3 Hz), 2.38 (2 H, CH_2, m), 1.89–1.70 (3 H, CH_2, CH_2 + H^4), 0.78 (1 H, H^1, J = 10.6 Hz); (endo isomer) δ 7.31–7.15 (5 H, Ph, m), 6.39 (1 H, =CHPh, d, J = 15.8 Hz), 6.21 (1 H, =CH, dt, J = 15.9, 6.8 Hz), 5.24 (5 H, Cp, s), 3.72 (1 H, H^3, td, J = 10.4, 6.7 Hz), 3.61 (1 H, C*H*(OH), m), 2.62 (1 H, H^2, d, J = 6.7 Hz), 2.38 (2 H, CH_2, m), 1.89–1.67 (4 H, CH_2 + H^1 + H^4, m). Mass (12 eV, m/e): 402 (M^+ − OH − 1). Anal. Calcd for $C_{21}H_{22}MoO_3$: C, 60.29; H, 5.26. Found: C, 60.40; H, 5.41.

(iii) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)[(1,2,3-η)-3-*syn*-4-hydroxy-4-methyl-6-phenyl-2-hexen-1-yl]molybdenum (16). Compound **10** (0.30 g, 0.76 mmol) in THF (20 mL) at −78 °C was slowly added with CH_3Li (10.6 mL, 1.5 M, 0.90 mmol), which led to a rapid change of solution color from dark orange to light yellow. The solution was slowly warmed to 23 °C, and a saturated NH_4Cl solution (NH_4Cl) was added. The organic layer was extracted twice with ether (20 mL). After drying over $MgSO_4$, the extract was concentrated, and eluted through a silica gel column with ether/hexane (1:1) as the eluting solvent. A yellow band was developed, collected, and concentrated to give **16** as a yellow oil (0.26 g, 0.64 mmol). IR (Nujol): ν(OH) 3458 (vs); ν(CO) 1939 (s), 1853 (s). ^1H NMR (300 MHz, $CDCl_3$): δ 7.25–7.15 (5 H, Ph, m), 5.24 (5 H, C_5H_5, s), 4.08 (1 H, H^3, ddd, J = 10.3, 9.8, 7.4 Hz), 2.82–2.62 (3 H, H^2 + CH_2Ph, m), 2.08–1.08 (3 H, C(OH)CH_2 + H^4, m), 1.45 (3 H, Me, s), 0.70 (1 H, H^1, d, J = 10.3 Hz). Mass (12 eV, m/e): 408 (M^+). Anal. Calcd for $C_{20}H_{22}MoO_3$: C, 59.12; H, 5.46. Found: C, 59.38; H, 5.54.

(iv) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)[(1,2,3-η)-*trans,trans*-4-hydroxy-4-methyl-8-phenyl-2,7-octadien-1-yl]molybdenum (17). The compound was prepared similarly from MeLi addition to **11**; the yield was 84%. IR (Nujol, cm^{-1}): ν(OH) 3461 (br, s); ν(CO) 1940 (s), 1856 (s). ^1H NMR (400 MHz, $CDCl_3$): 7.25–7.15 (5 H, Ph, m) 6.43 (1 H, =C*H*Ph, d, J = 15.8 Hz), 6.26 (1 H, C*H*=, dt, J = 15.8, 6.8 Hz), 5.32 (5 H, C_5H_5, s), 4.14 (1 H, H^3, ddd, J = 10.6, 9.8, 7.2 Hz), 2.65 (1 H, H^2, d, J = 7.2 Hz), 2.37 (2 H, CH_2, m), 1.99–1.84 (3 H, H^4 + CH_2, m), 1.61 (3 H, Me, s), 0.75 (1 H, H^1, d, J = 10.6 Hz). Mass (12 eV, m/e): 434 (M^+). Anal. Calcd for $C_{22}H_{24}MoO_3$: C, 61.11; H, 5.59. Found: C, 61.32; H, 5.18.

(v) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(3*R,4*S**)-(1,2,3-η)-*trans,trans*-4-hydroxy-6-phenylhexa-2,5-dien-1-yl]molybdenum (18).** Compound **12** (0.50 g, 1.29 mmol) and $CeCl_3 \cdot 7H_2O$ (0.40 g, 1.34 mmol) in 15 mL of CH_3OH at −78 °C were added to $NaBH_4$ (0.15 g, 3.97 mmol), and the mixture was stirred for 1 h before it was warmed to 23 °C. The solvent was removed under reduced pressure, and the residues were added with diethyl ether (60 mL) and saturated NH_4Cl (20 mL) solution. The organic layer was separated, washed by H_2O (2 × 20 mL), and dried over $MgSO_4$ before being concentrated. The residues were chromatographed through a silica column with ether/hexane (1:1) as the eluting solvent. A yellow band was collected, concentrated, and evaporated to dryness to give **17** as a yellow oil (0.35 g, 0.90 mmol). IR (Nujol, cm^{-1}): ν(CO) 1940 (s), 1875 (s). ^1H NMR (400 MHz, $CDCl_3$): (exo isomer) δ 7.37–7.17 (5 H, Ph, m), 6.58 (1 H, =CHPh, d, J = 15.8 Hz), 6.33 (1 H, =CH, dd, J = 15.8, 6.4 Hz), 5.28 (1 H, Cp, s), 4.40 (1 H, CH(OH), dd, J = 6.9, 6.4 Hz), 4.10 (1 H, ddd, J = 10.2, 9.9, 7.3 Hz), 2.68 (1 H, H^2, J = 7.3 Hz), 1.82 (1 H, H^4, J = 9.9, 6.9 Hz), 0.81 (1 H, H^1, d, J = 10.4 Hz); (endo isomer) 7.37–7.17 (5 H, Ph, m), 6.60 (1 H, =C*H*Ph, d, J = 15.8 Hz), 6.35 (1 H, =C*H*, dd, J = 15.8, 6.2 Hz), 5.25 (5 H, Cp, s), 4.40 (1 H, C*H*(OH), dd, J = 6.9, 6.2 Hz), 3.69 (1 H, H^3, ddd, J = 10.4, 9.9, 6.2 Hz), 2.75 (1 H, H^4, d, J = 9.9 Hz), 2.64 (1 H, H^2, d, J = 6.4 Hz), 1.78 (1 H, H^1, d, J = 10.4 Hz). Mass (12 eV, m/e): 392 (M^+). Anal. Calcd for $C_{19}H_{18}MoO_3$: C, 58.47; H, 4.65. Found: C, 58.54; H, 4.72.

(vi) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(3*R,4*S**)-(1,2,3-η)-*trans,trans*-4-hydroxy-6-(3′-furfuryl)-2-hexa-2,5-dien-1-yl]molybdenum (19).** This compound was prepared from **14** according to the procedure above; the yield was 81%. IR (Nujol, cm^{-1}): ν(OH) 3380 (br, s); ν(CO) 1940 (s), 1857 (s); ν(C=C) 1625 (w). ^1H NMR (300 MHz, $CDCl_3$): (exo isomer) δ 7.42 (1 H, s), 7.52 (1 H, s), 6.52 (1 H, s), 6.47 (1 H, =C*H*, d, J = 15.7 Hz), 6.09 (1 H, C*H*=, dd, J = 15.7, 6.4 Hz), 5.30 (5 H, Cp, s), 4.35 (1 H, C*H*(OH), m), 4.10 (1 H, H^3, ddd, J = 10.5, 9.6, 6.4 Hz), 2.70 (1 H, H^1, d, J = 10.5 Hz), 1.81 (1 H, H^4, dd, J = 9.8, 3.4 Hz), 0.83 (1 H, H^1, d, J = 10.5 Hz); (endo isomer) δ 7.41 (1 H, s), 7.35 (1 H, s), 6.57 (1 H, s), 6.55 (1 H, =C*H*, d, J = 16.1 Hz), 6.11 (1 H, =C*H*, dd, J = 16.1, 6.3 Hz), 5.29 (5 H, Cp), 4.35 (1 H, C*H*(OH), m), 3.69 (1 H, H^3, ddd, J = 10.2, 9.8, 6.4 Hz), 2.76 (1 H, H^4, dd, J = 9.8, 9.1 Hz), 2.66 (1 H, H^2, d, J = 6.4 Hz), 1.81 (1 H, H^1, d, J = 10.2 Hz). Mass (75 eV, m/e): 382 (M^+). Anal. Calcd for $C_{17}H_{16}MoO_4$: C, 53.70; H, 4.24. Found: C, 53.84; H, 4.12.

(g) (i) Synthesis of *cis*-1-Phenylhexa-3,5-dien-1-ol (20). In a typical reaction, to **5** (0.53 g, 1.20 mmol) in ether (20 mL) was added $(CF_3SO_2)_2O$ (0.27 mL, 1.57 mmol) at −78 °C, from which a dark red precipitate immediately deposited. At this temperature, the ether layer was removed via a long syringe tube, and the remaining precipitate was washed twice with diethyl ether (30 mL). To this solid was added Na_2CO_3 (0.5 g) and CH_2Cl_2 (10 mL), and the mixture was stirred rapidly at −78 °C for 0.5 h. The mixture was slowly warmed to 23 °C within 4 h, and stirring was continued for an additional 36 h. After filtration, the filtrate was concentrated and eluted through a SiO_2 TLC plate with hexane as the eluant to yield **21** (121 mg, 0.70 mmol) as an oil. IR (neat, cm^{-1}): 3367 (br, s), 1640 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.37–7.24 (5 H, Ph, m), 6.61 (1 H, H^3, dt, J = 16.4, 10.8, 10.8 Hz), 6.14 (1 H, H^4, t, J = 10.8 Hz), 5.21 (1 H, H^2, d, J = 16.4 Hz), 5.12 (1 H, H^1, d, J = 10.2 Hz), 4.73 (1 H, C*H*(OH), dd, J = 5.6, 5.0 Hz), 2.74–2.55 (2 H, CH_2, m), 1.98 (H, OH, s). ^{13}C NMR (75 MHz, $CDCl_3$): δ 155.0, 132.4, 132.0, 128.6, 127.8, 127.5, 125.9, 118.4, 73.8, 37.4. HRMS: calcd for $C_{12}H_{14}O$, 174.1038; found, 174.1044. The following **21–26** were obtained similarly from their corresponding allyl compounds using the same procedure above.

(ii) *cis*-6-Phenyl-1,3-diene (21): 59% yield. IR (neat, cm^{-1}): ν(C=C) 1647 (m), 1600 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.34–7.15 (5 H, Ph, m), 6.61 (1 H, H^3, ddd, J = 16.6, 10.8, 10.1 Hz), 6.02 (1 H, H^4, t, J = 10.8 Hz), 5.49 (1 H, H^5, dt, J = 10.8,

7.6 Hz), 5.24 (1 H, H², d, J = 16.6 Hz), 5.08 (1 H, H¹, d, J = 10.1 Hz), 2.70 (2 H, CH_2Ph, t, J = 8.0 Hz), 2.51 (2 H, CH_2, dt, J = 8.0, 7.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.6, 132.0, 131.5, 129.5, 128.5, 128.3, 125.8, 117.1, 35.7, 29.5. HRMS: calcd for $C_{12}H_{14}$, 158.1096; found, 158.1086.

(iii) 3-*cis*-7-*trans*-8-Phenyl-1,3,7-octatriene (22): 60% yield. IR (neat, cm^{-1}): 1647 (m), 1598 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.15–7.30 (5 H, pH, m), 6.75 (1 H, H³, dt, J = 16.8, 10.6 Hz), 6.41 (1 H, =C*H*Ph, d, J = 16.0 Hz), 6.23 (dt, CH_2CH=, J = 16.0, 7.0 Hz), 6.05 (1 H, H⁴, t, J = 10.6 Hz), 5.52 (1 H, H⁵, dt, J = 10.6, 7.0 Hz), 5.22 (1 H, H², d, J = 16.8 Hz), 5.12 (1 H, H¹, d, J = 10.6 Hz), 2.27–2.37 (4 H, 2 CH_2, m). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.7, 132.2, 131.6, 130.4, 129.9, 129.7, 128.5, 126.9, 125.9, 117.2, 32.9, 17.7. HRMS: calcd for $C_{14}H_{16}$, 184.1252; found, 184.1260.

(iv) *cis*-4-Methyl-6-phenyl-1,3-hexadiene (23): 51% yield. IR (neat, cm^{-1}): 1601 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.30–7.17 (5 H, Ph, m), 6.50 (1 H, H³, ddd, J = 16.6, 10.8, 10.0 Hz), 5.86 (1 H, H⁴, d, J = 10.8 Hz), 5.06 (1 H, H², d, J = 16.6 Hz), 4.93 (1 H, H¹, d, J = 10.0 Hz), 2.69 (2 H, CH_2Ph, t, J = 7.0 Hz), 2.45 (2 H, CH_2, t, J = 7.0 Hz), 1.80 (3 H, Me, s). ^{13}C NMR (75 MHz, $CDCl_3$): δ 142.2, 139.2, 133.0, 128.5, 126.0, 114.9, 34.5, 34.3, 23.6. HRMS: calcd for $C_{13}H_{16}$, 172.1252; found, 172.1258.

(v) 3-*cis*-7-*trans*-4-Methyl-8-phenyl-1,3,7-octatriene (24): 50% yield. IR (neat, cm^{-1}): ν(C=C) 1647 (m), 1598 (m). ^1H NMR (300 MHz, $CDCl_3$): δ 7.40–7.20 (5 H, Ph, m), 6.59 (1 H, H_3, ddd, J = 16.7, 10.8, 9.7 Hz), 6.41 (1 H, =C*H*Ph, d, J = 15.8 Hz), 6.21 (1 H, CH_2CH=, dt, J = 15.8, 6.5 Hz), 5.90 (1 H, H⁴, d, J = 10.8 Hz), 5.20 (1 H, H², d, J = 16.7 Hz), 4.98 (1 H, H¹, d, J = 9.7 Hz), 2.40–2.30 (4 H, $2CH_2$, m), 1.81 (3 H, Me, s). ^{13}C NMR (100 MHz, $CDCl_3$): 139.1, 137.9, 133.1, 130.3, 130.2, 128.6, 127.9, 126.1, 114.9, 32.0, 31.5, 23.6. HRMS: calcd for $C_{15}H_{18}$, 198.1409; found, 198.1416.

(vi) 2-*trans*-3-*cis*-6-Phenylhexa-1,3,5-triene (25): 68% yield. IR (neat, cm^{-1}): ν(CO) 1600 (m). ^1H NMR (400 MHz, $CDCl_3$): δ 7.43–7.21 (5 H, Ph, m), 6.95 (1 H, H³, ddd, J = 16.7, 10.9, 10.0 Hz), 5.58 (1 H, =CHPh, d, J = 15.4 Hz), 6.17 (1 H, H⁵, t, J = 10.9 Hz), 6.08 (1 H, H⁴, t, J = 10.9 Hz), 5.28 (1 H, H², d, J = 16.7 Hz), 5.21 (1 H, H¹, d, J = 10.0 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): 137.1, 133.4, 132.0, 130.2, 129.9, 128.5, 127.6, 126.4, 123.9, 118.2. HRMS: calcd for $C_{12}H_{12}$, 156.0940; found, 156.0941.

(vii) 1-*trans*-3-*cis*-6-(3′-Furfuryl)hexa-1,3,5-hexatriene (26): 61% yield. IR (neat, cm^{-1}): ν(C=C) 1606 (w). ^1H NMR (400 MHz, $CDCl_3$): δ 7.44 (1 H, s), 7.36 (1 H, d, J = 1.1 Hz), 6.95–6.83 (2 H, H³ + H⁶, m), 6.56 (1 H, d, J = 1.1 Hz), 6.43 (1 H, H⁷, d, J = 15.4 Hz), 6.09 (1 H, H⁴, d, J = 10.8 Hz), 6.00 (1 H, H⁵, t, J = 10.8 Hz), 5.25 (1 H, H², d, J = 17.3 Hz), 5.16 (1 H, H¹, d, J = 10.0 Hz). ^{13}C NMR (400 MHz, $CDCl_3$): δ 143.7, 141.0, 132.1, 129.9, 129.4, 124.6, 123.2, 118.0, 107.4. HRMS: calcd for $C_{10}H_{10}O$, 146.0732; found, 146.0736.

(h) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(1*S,3*S**,5*S**)-(3,4,5-η)-3-*anti*-5-*syn*-1-hydroxy-1-phenyl-4-hepten-3-yl]molybdenum (27)**. To a diethyl ether solution (40 mL) of **5** (0.45 g, 1.1 mmol) was added $(CF_3SO_2)_2O$ (0.30 mL, 1.75 mmol) at −78 °C, and the mixture was stirred for 30 min. To this solution was added an ethereal solution (30 mL) of Me_2CuLi [CuI (1.0 g, 5.52 mmol), MeLi (7 mL, 1.5 M in hexane)] at −78 °C, and the suspension was stirred for 1 h before it was warmed to 23 °C. To this solution was added saturated NH_4Cl solution (10 mL), and the organic layer was extracted twice with diethyl ether (20 mL). The extract was dried over $MgSO_4$, concentrated, and finally eluted through a silica column (ether/hexane = 1:3) to give **27** as a yellow oil (0.24 g, 0.59 mmol). IR (Nujol, cm^{-1}): ν(CO) 1931 (s), 1847 (s). ^1H NMR (400 MHz, $CDCl_3$): 7.36–7.22 (5 H, Ph, m), 5.21 (5 H, Cp, s), 4.53 (1 H, C*H*(OH), dd, J = 8.2, 6.1 Hz), 3.87 (1 H, H², dd, J = 9.7, 7.4 Hz), 3.45 (1 H, H³, m), 2.34 (1 H, H⁴, m), 2.05 (1 H, OH), 1.99–1.86 (2 H, CH*H*′Me + H¹, m), 1.76 (1 H, C*H*H′Me, m), 1.04 (2 H, Me, t, J = 7.2 Hz), 0.46 (1 H, H⁵, m). ^{13}C NMR (75 MHz, $CDCl_3$): δ 239.8, 238.0, 144.4, 128.5, 127.6, 126.2, 92.1, 77.9, 68.9, 67.3, 48.4, 40.4, 29.0, 17.3. Mass (12 eV, *m/e*): 408 (M^+), 352 (M^+ − 2CO). Anal. Calcd for $C_{20}H_{22}MoO_3$: C, 59.11; H, 5.47. Found: C, 59.34; H, 5.54.

(i) (i) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(2,3,4-η)-2-*syn*-4-*anti*-6-phenyl-1-oxo-3-hexen-2-yl]molybdenum (28). To a diethyl ether (20 mL) solution of **14** (0.33 g, 0.84 mmol) was added $(CF_3SO_2)_2O$ (0.20 mL, 1.16 mmol), and a dark red precipitate immediately formed. At this temperature, the ether layer was removed by a syringe tube; the red precipitate was washed twice with diethyl ether (2 × 30 mL). To this solid was added a CH_2Cl_2 (20 mL) solution of Me_3NO (0.40 g, 5.33 mmol), and the solution was stirred at −78 °C for 2 h before it was warmed to 23 °C. To this solution was added H_2O (15 mL) and the organic layer was extracted with diethyl ether (2 × 20 mL). The extract was dried over $MgSO_4$, concentrated, and eluted through a silica column (ether/hexane = 1:1) to give two yellow bands—**14** (R_f = 0.52, 67 mg, 0.17 mmol) and **28** (R_f = 0.21, 68 mg, 0.17 mmol). IR (Nujol, cm^{-1}): 1953 (s), 1873 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 9.23 (CHO, d, J = 5.2 Hz), 7.26–7.10 (5 H, Ph, m), 5.29 (5 H, C_5H_5, s), 4.62 (1 H, H², J = 9.6, 7.3 Hz), 3.88 (1 H, H³, ddd, J = 11.7, 7.3, 3.6 Hz), 2.73–2.52 (2 H, $PhCH_2$, m), 2.40–2.32 (2 H, H¹ + H⁴, m), 0.53 (1 H, H⁵, m). ^{13}C NMR (75 MHz, $CDCl_3$): δ 238.0, 235.9, 194.6, 131.1, 128.8, 128.6, 126.2, 92.6, 70.0, 62.4, 55.3, 40.8, 33.5. Mass (12 eV, *m/e*): 392 (M^+). Anal. Calcd for $C_{19}H_{18}MoO_3$: C, 58.46; H, 4.66. Found: C, 58.54; H, 4.72.

(ii) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(2*R,4*S**,6*S**)-(2,3,4-η)-2-*syn*-4-*anti*-6-hydroxy-6-phenyl-1-oxo-3-hexen-2-yl]molybdenum (29)**. This compound was similarly prepared from the reaction between Me_3NO and the cation **II** derived from **5**, and the yields of **29** and **5** were 18% and 20%, respectively. IR (Nujol, cm^{-1}): ν(CO) 1955 (s), 1875 (s), 1665 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 9.25 (1 H, CHO, d, J = 5.2 Hz), 7.35–7.24 (5 H, Ph, m), 5.31 (5 H, Cp, s), 4.67 (1 H, H², dd, J = 9.7, 7.4 Hz), 4.55 (1 H, C*H*(OH), dd, J = 7.8, 4.2 Hz), 4.06 (1 H, H³, m), 2.50 (1 H, H⁴, ddd, J = 14.1, 7.8, 3.4 Hz), 2.33 (1 H, H¹, dd, J = 9.7, 5.2 Hz), 0.58 (1 H, H⁵, m). ^{13}C NMR (100 MHz, $CDCl_3$): δ 237.1, 233.0, 191.4, 143.8, 128.6, 127.9, 125.9, 92.7, 77.8, 70.6, 58.3, 56.1, 41.0. Mass (12 eV, *m/e*): 408 (M^+). Anal. Calcd for $C_{19}H_{18}MoO_4$: C, 56.16; H, 4.47. Found: C, 56.24; H, 4.49.

(j) Synthesis of 30a and 30b. To complex **27** (0.41 g, 1.00 mmol) in CH_3CN (5 mL) at 0 °C was added $NOBF_4$ (0.13 g, 1.0 mmol), and the solution was stirred for 15 min; diethyl ether (40 mL) was added to the solution to give an orange viscous solid (0.46 g, 0.90 mmol) of **30a**. This cation was completely converted to **30b** in CH_3CN at 23 °C within 12 h. Samples for elemental analysis were purified by recrystallization in CH_3CN/ether which gave a dark red viscous solid. IR (CH_3CN, cm^{-1}): ν(CO) 2100 (s), ν(NO) 1660 (s). ^1H NMR (400 MHz, CD_3CN, −20 °C): (**30a**, exo isomer) δ 1.16 (3 H, Me, t, J = 7.2 Hz), 1.36 (1 H, H⁵, ddd, J = 14.8, 7.8, 5.8 Hz), 2.12–2.20 (2 H, CH*H*′*Me* + H⁴, m), 2.45 (1 H, C*H*H′Me, m), 4.62 (1 H, C*H*(OH), dd, J = 6.4, 5.8 Hz), 4.94 (1 H, H¹, br m), 5.00 (1 H, H², dd, J = 11.6, 8.0 Hz), 6.12 (5 H, s, C_5H_5); (**30b**, endo isomer) δ 0.98 (3 H, Me, t, J = 7.0 Hz), 1.41 (1 H, H⁴, ddd, J = 12.2, 11.8, 6.8 Hz), 2.53–2.63 (2 H, CH*H*′*Me* + H⁵, m), 4.56 (1 H, H², dd, J = 12.4, 8.0 Hz), 4.71 (1 H, C*H*(OH), dd, J = 7.0, 6.8 Hz), 5.84 (1 H, H³, ddd, J = 12.8, 8.5, 5.6 Hz), 6.30 (5 H, s, C_5H_5). Anal. Calcd for $C_{19}H_{22}MoNO_3BF_4$: C, 46.09; H, 4.48. Found: C, 45.59; H, 4.80.

(k) Synthesis of 3-(*trans*-1′-Butenyl)-5-phenylisoxazole (31) and *trans*-1-Phenyl-2,4-heptadien-1-one (32). (i) To a CH_3CN solution (5 mL) of **27** (0.20 g, 0.49 mmol) was slowly added $NOBF_4$ (0.58 g, 4.96 mmol) at 0 °C during 20 min. After stirring for 10 h at 0 °C, the solution was extracted twice with diethyl ether (2 × 5 mL), dried over $MgSO_4$, and concentrated. The residues were chromatographed through a preparative SiO_2 TLC plate with hexane as the eluting solvent to afford **31** as a colorless oil (36 mg, 0.18 mmol). IR (Nujol, cm^{-1}): 1660 (m), 1643 (w), 1591 (m), 1573 (m). ^1H NMR (400 MHz, C_6D_6): δ 7.68 (2 H, Ph, d, J = 8.5 Hz), 7.16–7.10 (3 H, Ph, m), 6.60 (1 H, H³, d, J = 16.0 Hz), 6.27 (1 H, H⁴, s), 6.24 (1 H, H², dt, J = 16.0, 6.4 Hz), 2.00 (2 H, H¹, dq, J = 7.4, 6.4 Hz), 0.94 (3 H, Me, t, J = 7.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.4, 162.3, 140.1, 130.0, 128.9, 127.6, 125.8, 117.1, 96.4, 25.9, 12.9. HRMS: calcd for $C_{13}H_{13}NO$, 199.0997; found, 199.0991.

Table II. Crystal Data and Data Collection Parameters of 41

compd	**41**
empirical formula	$C_{18}H_{15}NO$
fw	261.32
cell dimens	
a, Å	10.351 (1)
b, Å	5.723(2)
c, Å	24.103(3)
β, deg	101.242(10)
vol, Å^3	1400.4(5)
cryst syst	monoclinic
space group	$P2_1/c$
Z	4
D_{calcd}, Mg/m^3	1.239
μ, cm^{-1}	0.7
cryst dimens, mm	0.05 × 0.25 × 0.50
collcn range ($2\theta_{max}$), deg	45, $\pm h,k,l$
scan mode and scan speed $\omega/2\theta$, deg min^{-1}	$\omega/2\theta$, 16.48/6–16.48/2
scan width, deg	0.80 + 0.35 tan θ
collcn T, K	298
decay corrcn	no (<2%)
abs corrcn	no
no. of ind reflns	1821
no. of obsd reflns (m)	1114 ($I > 2\sigma(I)$)
no. of params	182
R_F[a] (all reflns)	0.037 (0.079)
R_{wF}[a] (all reflns)	0.038 (0.042)
weighting scheme	$w^{-1} = \sigma^2(F)+0.0001F^2$
s[a]	
residual extrema in final diff map, e/Å^3	–0.41, +0.27

[a] $R = \sum\|F_o\|-|F_c\|/\sum|F_o|$. $R_w = [w(|F_o|-|F_c|)^2/\sum w|F_o|^2]^{1/2}$. $S = [\sum w(|F_o| - |F_c|)^2/(m-p)]^{1/2}$.

(ii) At the end of $NOBF_4$ addition to **27**, the solution was stirred at 0 °C for 1 h. Monitoring the solution by a preparative SiO_2 TLC plate showed appearance of **31**, to which a saturated Na_2CO_3 solution (3 mL) was added. The mixture was stirred for 30 min, extracted twice with diethyl ether (2 × 5 mL), and dried over $MgSO_4$ before removal of solvent under reduced pressure. The residues were chromatographed with a SiO_2 TLC plate with hexane as the eluting solvent to give two bands of **31** (R_f = 0.85, 18%) and **32** (R_f = 0.49, 11%), respectively. Spectroscopic data **32**: IR (Nujol, cm^{-1}) ν(C=C) 1661 (s), 1627 (m), 1591 (s); ^{1}H NMR (400 MHz, C_6D_6) δ 7.91 (2 H, Ph, m), 7.58 (1 H, =CH, dd, J = 15.0, 11.0 Hz), 7.13–7.02 (3 H, Ph, m), 6.72 (1 H, =CH, d, J = 15.0 Hz), 6.01 (1 H, =CH, dd, J = 15.0, 11.0 Hz), 5.75 (1 H, dt, J = 15.1, 6.5 Hz), 1.82 (2 H, CH_2Me, m), 0.77 (3 H, Me, t, J = 7.4 Hz); HRMS calcd for $C_{13}H_{14}O$ 186.1045, found 186.1039.

(l) (i) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(1*S,3*S**,5*S**)-(2,4,5-η)-3-*anti*-5-*syn*-1-hydroxy-1,6-diphenyl-4-hexen-3-yl]molybdenum (33).** This complex was similarly prepared from the reaction between excess Ph_2CuLi (5.0 equimolar) and the cation II derived from **5** according to the procedure for the synthesis of **27**; the yield was 56%. IR (Nujol, cm^{-1}): ν(CO) 1931 (s), 1850 (s). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18–6.99 (10 H, 2 Ph, m), 5.08 (5 H, Cp, s), 4.43 (1 H, C*H*(OH), dd, J = 8.2, 6.4 Hz), 3.91 (1 H, H^2, dd, J = 10.5, 7.6 Hz), 3.41–3.36 (2 H, PhCH*H*′ + H^3, m), 2.85 (1 H, PhC*H*H′, dd, J = 14.8, 10.7 Hz), 2.26 (1 H, H^4, m), 2.07 (1 H, H^1, ddd, J = 10.7, 10.5, 1.7 Hz), 0.43 (1 H, H^5, m). ^{13}C NMR (75 MHz, $CDCl_3$): δ 239.6, 237.3, 144.4, 141.9, 128.7, 128.6, 128.5, 127.7, 126.6, 126.1, 92.1, 78.1, 67.3, 66.4, 47.9, 40.8, 40.3. Mass (12 eV, m/e): 470 (M$^+$). Anal. Calcd for $C_{25}H_{24}MoO_3$: C, 64.11; H, 5.16. Found: C, 64.32; H, 5.18.

(ii) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(1*S,3*S**,5*S**)-(3,4,5-η)-3-*anti*-5-*syn*-1-hydroxy-1-phenyl-4-decen-3-yl]molybdenum (34).** This complex was prepared similarly from the reaction between excess Bu_2CuLi and the cation II derived from **5**; the yield was 42%. IR (Nujol, cm^{-1}): ν(CO) 1932 (s), 1850 (s). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28–7.18 (5 H, Ph, m), 5.15 (5 H, Cp, s), 4.45 (1 H, C*H*(OH), dd, J = 7.8, 5.8 Hz), 3.80 (1 H, H^2, dd, J = 10.3, 7.8 Hz), 3.41 (1 H, H^3, m), 2.44 (1 H, OH, s), 2.28 (1 H, H^4, m), 1.98–1.85 (2 H, C*H*^{1}CH*H*′, m), 1.52 (1 H, CH^{1}C*H*H′, m), 1.38–1.22 (6 H, $(CH_2)_3$, m), 0.89 (3 H, Me, t, J = 7.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 239.8, 237.9, 144.4, 128.4, 127.5, 126.1, 92.0, 77.9, 64.6, 64.0, 48.4, 40.3, 35.9, 32.8, 31.2, 22.2, 13.7. Mass (75 eV, m/e): 450 (M$^+$). Anal. Calcd for $C_{23}H_{28}MoO_3$: C, 61.61; H, 6.29. Found: C, 61.74; H, 6.40.

(iii) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(1*S,3*S**,5*S**)-(3,4,5-η)-3-*anti*-5-*syn*-1-hydroxy-1-(*m*-methoxyphenyl)-4-decen-3-yl]molybdenum (35).** This compound was prepared similarly from the reaction between excess Bu_2CuLi (6.0 equimolar) and the cation II derived from **4** in cold THF (–78 °C); the yield was 41%. IR (Nujol, cm^{-1}): ν(CO) 1931 (s), 1850 (s). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12–6.66 (4 H, Ph, m), 5.10 (5 H, Cp, s), 4.40 (1 H, C*H*(OH), dd, J = 8.0, 6.2 Hz), 3.77 (1 H, H^2, dd, J = 10.3, 7.8 Hz), 3.70 (3 H, OMe, s), 3.34 (1 H, H^3, m), 2.21 (1 H, H^4, m), 1.92–1.82 (2 H, C*H*^{1}CH*H*′, m), 1.44 (1 H, CH^{1}C*H*H′, m), 1.32–1.15 (6 H, $(CH_2)_3$, m), 0.80 (3 H, Me, t, J = 6.7 Hz), 0.35 (1 H, H^5, m). ^{13}C NMR (100 MHz, $CDCl_3$): 239.0, 237.2, 159.6, 145.9, 129.2, 118.3, 112.6, 111.6, 92.0, 77.8, 67.7, 67.1, 55.1, 48.5, 40.5, 36.1, 33.0, 31.5, 22.4, 14.0. Mass (75 eV, m/e): 480 (M$^+$). Anal. Calcd for $C_{24}H_{30}MoO_4$: C, 60.25; H, 6.32. Found: C, 60.42; H, 6.44.

(iv) Synthesis of Dicarbonyl(η^5-cyclopentadienyl)-[(1*S,3*S**,5*S**)-(3,4,5-η)-3-*anti*-5-*syn*-1-hydroxy-1-(*m*-methoxyphenyl)-4-hepten-3-yl]molybdenum (36).** This compound was prepared similarly from the reaction between excess Me_2-

Table III. Atomic Parameters x, y, and z of 41

	x	y	z	B_{iso},[a] Å^2
C(1)	0.8221(3)	0.2946(5)	0.30124(13)	4.22(16)
C(2)	0.9336(3)	0.3383(6)	0.34105(12)	4.73(17)
C(3)	1.0339(3)	0.1760(6)	0.35208(12)	4.74(17)
C(4)	1.0221(3)	–0.0320(6)	0.32254(13)	4.57(17)
C(5)	0.9102(3)	–0.0777(5)	0.28243(12)	4.10(16)
C(6)	0.8083(3)	0.0845(5)	0.27137(11)	3.63(14)
C(7)	0.6911(3)	0.0426(5)	0.22861(12)	3.78(16)
C(8)	0.5937(3)	0.1769(5)	0.20012(12)	3.92(14)
C(9)	0.5101(3)	0.0260(6)	0.16316(12)	4.02(16)
C(10)	0.3903(3)	0.0752(6)	0.12198(13)	4.33(15)
C(11)	0.3359(3)	0.2842(6)	0.11280(12)	4.25(16)
C(12)	0.2109(3)	0.3330(6)	0.07142(12)	4.59(16)
C(13)	0.2279(3)	0.4917(6)	0.02333(12)	4.04(15)
C(14)	0.1504(3)	0.6889(6)	0.01079(12)	4.62(16)
C(15)	0.1667(4)	0.8360(6)	–0.03294(14)	5.72(19)
C(16)	0.2589(4)	0.7883(7)	–0.06465(14)	5.90(20)
C(17)	0.3351(3)	0.5903(8)	–0.05388(15)	6.26(22)
C(18)	0.3198(3)	0.4424(6)	–0.00984(14)	5.37(18)
N	0.5531(3)	–0.1904(5)	0.16874(12)	5.62(15)
O	0.66939(20)	–0.1832(4)	0.21063(9)	5.36(12)

[a] B_{iso} is the mean of the principal axes of the thermal ellipsoid.

Table IV. Bond Distances and Bond Angles of 41

C(1)–C(2)	1.371(4)	C(9)–N	1.314(4)
C(1)–C(6)	1.393(4)	C(10)–C(11)	1.321(5)
C(2)–C(3)	1.377(5)	C(11)–C(12)	1.497(4)
C(3)–C(4)	1.379(5)	C(12)–C(13)	1.510(4)
C(4)–C(5)	1.380(4)	C(13)–C(14)	1.383(5)
C(5)–C(6)	1.393(4)	C(13)–C(18)	1.384(4)
C(6)–C(7)	1.450(4)	C(14)–C(15)	1.383(5)
C(7)–C(8)	1.342(4)	C(15)–C(16)	1.360(6)
C(7)–O	1.368(4)	C(16)–C(17)	1.374(6)
C(8)–C(9)	1.410(4)	C(17)–C(18)	1.390(5)
C(9)–C(10)	1.457(4)	O–N	1.412(3)
C(2)–C(1)–C(6)	120.5(3)	C(10)–C(9)–N	118.6(3)
C(1)–C(2)–C(3)	120.8(3)	C(9)–C(10)–C(11)	124.6(3)
C(2)–C(3)–C(4)	119.5(3)	C(10)–C(11)–C(12)	124.5(3)
C(3)–C(4)–C(5)	120.2(3)	C(11)–C(12)–C(13)	114.1(2)
C(4)–C(5)–C(6)	120.6(3)	C(12)–C(13)–C(14)	120.7(3)
C(1)–C(6)–C(5)	118.4(3)	C(12)–C(13)–C(18)	121.2(3)
C(1)–C(6)–C(7)	120.0(3)	C(14)–C(13)–C(18)	118.1(3)
C(5)–C(6)–C(7)	121.6(3)	C(13)–C(14)–C(15)	120.9(3)
C(6)–C(7)–C(8)	135.0(3)	C(14)–C(15)–C(16)	120.5(3)
C(6)–C(7)–O	116.4(3)	C(15)–C(16)–C(17)	119.8(3)
C(8)–C(7)–O	108.5(3)	C(16)–C(17)–C(18)	120.0(3)
C(7)–C(8)–C(9)	106.3(3)	C(13)–C(18)–C(17)	120.6(3)
C(8)–C(9)–C(10)	130.4(3)	C(7)–O–N	108.6(2)
C(8)–C(9)–N	110.9(3)	C(9)–N–O	105.7(2)

CuLi and the cation II derived from 4 in cold THF (−78 °C); the yield was 47%. IR (Nujol, cm^{-1}): ν(CO) 1931 (s), 1848 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 7.18–6.70 (4 H, Ph, m), 5.14 (5 H, Cp, s), 4.44 (1 H, C*H*(OH), dd, J = 8.4, 6.0 Hz), 3.80 (1 H, H^2, J = 9.7, 7.8 Hz), 3.72 (3 H, OMe, s), 3.37 (1 H, H^3, m), 2.26 (1 H, H^4, m), 1.91–1.84 (2 H, C*H*H′ + H^1, m), 1.68 (1 H, C*H*H′, m), 0.98 (3 H, Me, t, J = 7.2 Hz), 0.38 (1 H, H^5, m). ^{13}C NMR (75 MHz, $CDCl_3$): 239.7 (s), 237.9, 159.9, 146.1, 129.4, 118.5, 112.7, 111.7, 92.1, 77.7, 68.9, 67.2, 55.0, 48.3, 40.3, 29.0, 17.3. Mass (75 eV, m/e): 438 (M^+). Anal. Calcd for $C_{21}H_{24}MoO_4$: C, 57.80; H, 5.54. Found: C, 57.84; H, 5.61.

(v) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(3*S,5*S**,7*S**)-(5,6,7-η)-5-*anti*-7-*syn*-3-hydroxy-2-methyl-6-dodecen-5-yl]molybdenum (37).** This compound was similarly prepared from the reaction between excess $(C_4H_9)_2CuLi$ (10.0 equimolar) and the cation II derived from its corresponding η^3-allyl 1,3-diol;[12] the yield was 32%. IR (Nujol, cm^{-1}): ν(CO) 1934 (s), 1852 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 5.22 (5 H, Cp, s), 3.96 (1 H, H^2, dd, J = 10.0, 7.8 Hz), 3.60 (1 H, H^3, m), 3.14 (1 H, C*H*(OH), m), 2.10–2.01 (2 H, C*H*H′ + H^1 m), 1.87 (1 H, H^4, m), 1.68–1.58 (2 H, C*H*H′ + C*H*Me_2, m), 1.52 (2 H, CH_2, m), 1.37–1.28 (4 H, $(CH_2)_2$, m), 0.92–0.85 (4 H, Me + H^5, m), 0.82 (3 H, Me, d, J = 6.0 Hz), 0.81 (3 H, Me, d, J = 6.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): 240.0, 237.9, 92.1, 81.4, 74.6, 66.9, 50.0, 36.0, 35.2, 33.1, 33.0, 31.2, 22.3, 18.6, 16.9, 13.8. Mass (75 eV, m/e): 416 (M^+). Anal. Calcd for $C_{20}H_{30}MoO_3$: C, 57.96; H, 7.30. Found: C, 57.59; H, 7.13.

(vi) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(3*S,5*S**,7*S**)-(5,6,7-η)-5-*anti*-7-*syn*-3-hydroxy-2,2-dimethyl-6-dodecen-5-yl]molybdenum (38).** This compound was similarly prepared from the reaction between $(n\text{-}C_4H_9)_2CuLi$ and the cation II derived from its corresponding η^3-allyl 1,3-diol; the yield was 36%. IR (Nujol, cm^{-1}): ν(CO) 1935 (s), 1855 (s). ^1H NMR (300 MHz, $CDCl_3$): δ 5.95 (5 H, Cp, s), 4.75 (1 H, H^2, dd, J = 10.2, 8.0 Hz), 4.00 (1 H, H^3, m), 3.84 (1 H, C*H*(OH), dd, J = 7.2, 4.8 Hz), 2.87 (1 H, H^4, m), 2.80–2.70 (2 H, C*H*H′ + H^1, m), 2.42 (1 H, C*H*H′, m), 2.50 (2 H, CH^2, m), 2.00–1.80 (4 H, m), 1.72 (3 H, Me, t, J = 6.8 Hz), 1.53 (9 H, 3 Me, s). ^{13}C NMR (75 MHz, $CDCl_3$): 239.2, 237.9, 92.9, 81.0, 67.4, 66.6, 50.9, 35.8, 34.3, 32.8, 31.1, 25.2, 21.9, 13.4. Mass (75 eV, m/e): 430 (M^+). Anal. Calcd for $C_{21}H_{32}MoO_3$: C, 58.89; H, 7.53. Found: C, 58.90; H, 7.33.

(vii) Synthesis of Dicarbonyl(η⁵-cyclopentadienyl)-[(3*S,5*S**,7*S**)-(5,6,7-η)-5-*anti*-7-*syn*-3-hydroxy-2,2-dimethyl-6-nonen-5-yl]molybdenum (39).** This compound was prepared in a similar fashion from the reaction between Me_2CuLi (5.0 equimolar) and the cation II derived from its corresponding η^3-allyl 1,3-diol;[12] the yield was 40%. IR (Nujol, cm^{-1}): ν(CO) 1934 (s), 1852 (s). ^1H NMR (300 MHz, $CDCl_3$): δ 5.22 (5 H, Cp, s), 3.95 (1 H, H^2, dd, J = 10.0, 7.5 Hz), 3.75 (1 H, H^3, m), 2.98 (1 H, C*H*(OH), dd, J = 8.4, 4.2 Hz), 2.03–1.80 (2 H, C*H*H′ + H^1, m), 1.74 (1 H, H^4, m), 1.15 (3 H, Me, t, J = 7.2 Hz), 0.87 (3 H, Me, s), 0.77 (6 H, 2 Me, s), 0.23 (1 H, H^5, m). ^{13}C NMR (75 MHz, $CDCl_3$): δ 240.1, 237.9, 92.1, 85.9, 68.9, 66.8, 51.5, 35.0, 33.8, 29.2, 25.3 (3 Me), 17.6. Mass (75 eV, m/e): 388 (M^+). Anal. Calcd for $C_{18}H_{26}MoO_3$: C, 55.96; H, 6.78. Found: C, 60.03; H, 6.49.

(m) Synthesis of Isoxazole Derivatives 41–47. The following isoxazole compounds 41–47 were similarly prepared from the reaction between excess $NOBF_4$ (10 equimolar) and the η^3-allyl-3-*anti*-5-*syn* derivatives 33–39 according to the procedure described in section k.

(i) 3-(*trans*-3′-Phenyl-1′-propen-1′-yl)-5-phenylisoxazole (41): 54% yield. IR (Nujol, cm^{-1}): 1660 (m), 1591 (m), 1573 (m). ^1H NMR (400 MHz, C_6D_6): δ 7.62–7.05 (10 H, 2 Ph, m), 6.58 (1 H, H^3, d, J = 15.9 Hz), 6.24 (1 H, H^2, dt, J = 15.9, 6.9 Hz), 6.09 (1 H, H^4, s), 3.22 (2 H, CH_2, d, J = 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.5, 162.0, 138.7, 137.4, 132.6, 130.1, 128.9, 128.6, 126.5, 125.8, 119.2, 96.4, 39.3. HRMS: calcd for $C_{18}H_{15}NO$, 261.1154; found, 261.1167.

(ii) 3-(*trans*-1′-Hepten-1-yl)-5-phenylisoxazole (42): 46% yield. IR (Nujol, cm^{-1}): 1660 (m), 1591 (m), 1572 (m). ^1H NMR (400 MHz, C_6D_6): δ 7.60 (2 H, Ph, d, J = 8.0 Hz), 7.07–7.03 (3 H, Ph, m), 6.56 (1 H, H^3, d, J = 16.0 Hz), 6.20 (1 H, H^4, s), 6.18 (1 H, dt, J = 16.0, 6.9 Hz), 1.96 (2 H, H^1, q, J = 6.9 Hz), 1.32–1.09 (6 H, $(CH_2)_3$, m), 0.89 (3 H, Me, t, J = 7.0 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.3, 162.3, 139.4, 130.0, 128.9, 127.5, 117.9, 96.3, 32.8, 31.3, 28.3, 22.4. HRMS: calcd for $C_{16}H_{19}NO$, 241.1468; found, 241.1469.

(iii) 3-(*trans*-1′-Hepten-1′-yl)-5-(*m*-methoxyphenyl)isoxazole (43): 51% yield. IR (Nujol, cm^{-1}): 1661 (m), 1577 (m). ^1H NMR (400 MHz, C_6D_6): δ 7.35–6.74 (4 H, Ph, m), 6.57 (1 H, H^3, d, J = 16.0 Hz), 6.23 (1 H, H^4, s), 6.16 (1 H, H^2, dt, J = 16.0, 7.0 Hz), 3.21 (3 H, OMe, s), 1.96 (2 H, H^1, q, J = 7.0 Hz), 1.32–1.12 (6 H, $(CH_2)_3$, m), 0.86 (3 H, Me, t, J = 7.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.9, 162.0, 159.6, 139.1, 129.6, 128.0, 117.7, 117.4, 115.6, 110.3, 96.0, 54.7, 30.5, 27.5, 21.6, 13.1. HRMS: calcd for $C_{17}H_{21}NO_2$, 271.1567; found, 271.1574.

(iv) 3-(*trans*-1′-Buten-1′-yl)-5-(*m*-methoxyphenyl)isoxazole (44): 38% yield. IR (Nujol, cm^{-1}): 1661 (m), 1583 (m). ^1H NMR (400 MHz, C_6D_6): δ 7.34–6.73 (4 H, Ph, m), 6.52 (1 H, H^3, d, J = 16.0 Hz), 6.20 (1 H, H^4, s), 6.12 (1 H, H^2, dt, J = 16.0, 6.4 Hz), 3.24 (3 H, OMe, s), 1.90 (2 H, H^1, dt, J = 7.4, 6.4 Hz), 0.84 (3 H, Me, t, J = 7.4 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 162.6, 160.2, 140.9, 130.2, 128.9, 118.4, 117.2, 116.2, 110.9, 96.6, 55.3, 25.7, 12.6. HRMS: calcd for $C_{14}H_{15}NO_2$, 229.1099; found, 229.1108.

(v) 3-(*trans*-1′-Hepten-1′-yl)-5-isopropylisoxazole (45): 39% yield. IR (Nujol, cm^{-1}): 1661 (m), 1593 (m). ^1H NMR (400 MHz, C_6D_6): δ 6.56 (1 H, H^3, d, J = 16.0 Hz), 6.13 (1 H, H^2, dt, J = 16.0, 6.8 Hz), 5.74 (1 H, H^4, s), 2.67 (1 H, C*H*Me_2, hept, J = 7.0 Hz), 1.95 (2 H, H^1, q, J = 6.8 Hz), 1.30–0.98 (12 H, 2 Me + 2 $(CH_2)_3$, m), 0.84 (3 H, Me, t, J = 7.0 Hz). ^{13}C NMR (75 MHz, C_6D_6): δ 178.2, 161.7, 138.0, 119.3, 95.8, 32.6, 31.2, 28.3, 26.8, 22.4, 20.3, 13.7. HRMS: calcd for $C_{13}H_{21}NO$, 207.1623; found, 207.1630.

(vi) 3-(*trans*-1′-Hepten-1′-yl)-5-*tert*-butylisoxazole (46): 44% yield. IR (Nujol, cm^{-1}): 1661 (m), 1589 (m). ^1H NMR (300 MHz, C_6D_6): δ 6.56 (1 H, H^3, d, J = 16.0 Hz), 6.13 (1 H, H^2, dt, J = 16.0, 6.3 Hz), 5.79 (1 H, H^4, s), 1.94 (2 H, H^1, q, J = 6.3 Hz), 1.28–1.10 (6 H, $(CH_2)_3$, m), 1.12 (9 H, 3 Me, s), 0.84 (3 H, Me, t, J = 6.8 Hz). ^{13}C NMR (75 MHz, C_6D_6): δ 180.6, 161.6, 138.0, 119.3, 95.2, 32.6, 32.1, 31.2, 28.4, 22.4, 13.7. HRMS: calcd for $C_{14}H_{23}NO$, 221.1781; found, 221.1778.

(vii) 3-(*trans*-1′-Buten-1-yl)-5-*tert*-butylisoxazole (47): 35% yield. IR (Nujol, cm^{-1}): 1662 (m), 1587 (m). ^1H NMR (400 MHz, C_6D_6): δ 6.53 (1 H, H^3, d, J = 16.0 Hz), 6.09 (1 H, H^2, dt, J = 16.0, 6.4 Hz), 5.75 (1 H, H^4, s), 1.89 (2 H, H^1, t, J = 6.4 Hz), 1.12 (9 H, 3 Me, s), 0.83 (3 H, Me, d, J = 6.4 Hz). HRMS: calcd for $C_{11}H_{17}NO$, 179.1311; found, 179.1320.

(n). X-ray Structure Determination of Complex 41. Single crystals of 41 were obtained by recrystallization from hexane at −40 °C. The crystal data were measured on an Enraf-Nonius CAD-4 diffractometer using Mo Kα radiation (λ = 0.710 73 Å). The atomic scattering factors were taken from the *International Tables for X-ray Crystallographically*, the structures were solved by the direct method,[26,27] and all hydrogen atoms were fixed in the idealized positions. A summary of the crystal and data collection parameters is provided in Table II; position parameters are given in Table III, and bond distances and angles appear in Table IV.

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Supplementary Material Available: Tables of positional parameters and thermal parameters for compound 41 (2 pages). Ordering information is given on any current masthead page.

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(26) Gabe, E. J.; Lee, F. L. *Acta Crystallogr.* 1981, *A37*, 5339.
(27) Johnson, C. K. ORTEP. Report ORNL-3794; Oak Ridge National Laboratory: Oak Ridge, TN 1965.