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TRANSITION METALS IN ORGANIC SYNTHESIS
ANNUAL SURVEY COVERING THE YEAR 1990*

Louis S Hegedus
Department of Chemistry, Colorado State University, Fort Collins, CO 80523
USA

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* **Reprints for this review are not available.**

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I. General Comments

This annual survey covers the literature for 1990 dealing with the use of transition metal intermediates for organic synthetic transformations. It is not a comprehensive review but is limited to reports of discrete systems that lead to at least moderate yields of organic compounds, or that allow unique organic transformations, even if low yields are obtained. Catalytic reactions that lead cleanly to a major product and do not involve extreme conditions are also included This is not a critical review, but rather a listing of the papers published in the title area

The papers in this survey are grouped primarily by reaction type rather than by organometallic reagent, since the reader is likely to be more interested in the organic transformation effected than the metal causing it Oxidation, reduction, and hydroformylation reactions are specifically excluded, and will be covered in a different annual survey. Also excluded are structural and mechanistic studies of organometallic systems unless they present data useful for synthetic application. Finally, reports from the patent literature have not been surveyed since patents are rarely sufficiently detailed to allow reproduction of the reported results.

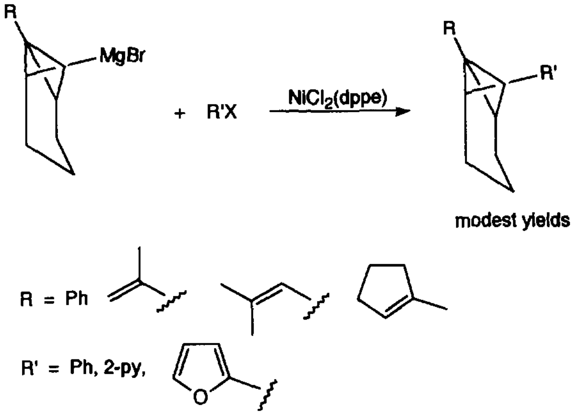
II. Carbon-Carbon Bond-Forming Reactions

A Alkylations

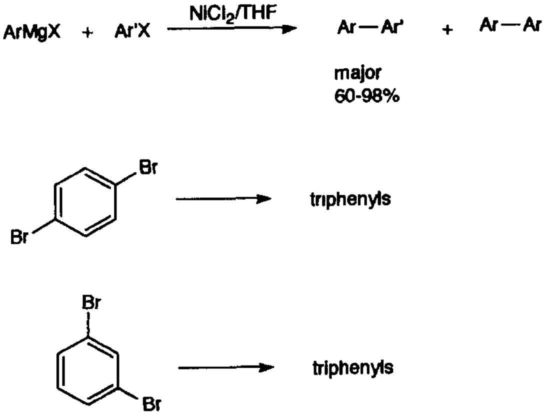
1 Alkylation of Organic Halides, Tosylates, Triflates, Acetates and Epoxides

Transition metal catalyzed reactions of Grignard reagents continue to be extensively used for synthesis. Substituted styrenes were synthesized by the cross-coupling of substituted phenylmagnesium bromides with vinyl bromide in the presence of nickel(II) phosphine complexes [1] Chiral ferrocenylamine sulfide and selenide (of Pd, Pt and Ni complexes) were used to catalyze the enantioselective cross-coupling of allyl magnesium chloride with 1-chloro-1-phenylethane [2] Nickel phosphine complexes catalyzed the alkylation of halides by bridge-head Grignard reagents (equation 1) [3]. Nickel phosphine complexes were also used to catalyze the cross-coupling of Grignard reagents with di-, tri-, and polychlorobiphenyls [4] Unsymmetrical biaryls were synthesized by the nickel(II) chloride catalyzed coupling of aryl halides with aryl Grignard reagents (equation 2) [5] Orthothioesters were converted to olefins by reaction with Grignard reagents in the presence of nickel(II) phosphine complexes (equation 3) [6]. Arylpalladium(II) halides coupled with vinyl magnesium bromide to produce styrenes [7] Nickel(II) and palladium(II) complexes were used to catalyze the coupling of aryl iodides with vinylmagnesium bromides [8]

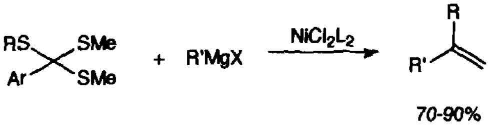
(Equation 1)



(Equation 2)



(Equation 3)

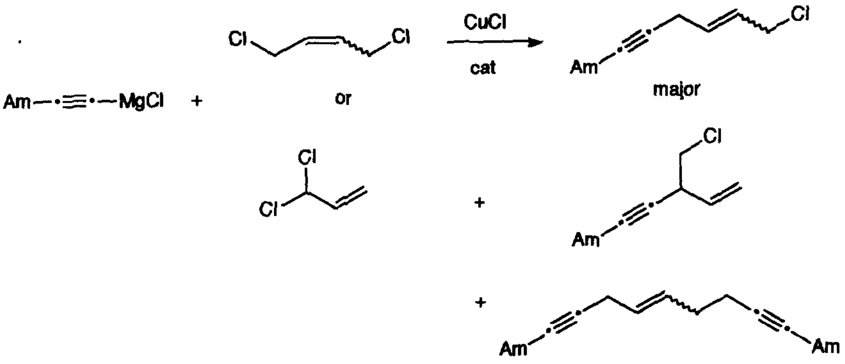


Ar = 1-Naphth, 2-Naphth, pMePh, pMeOPh, 2,4-$(MeO)_2Ph$, 2,5-$(MeO)_2Ph$, Ph

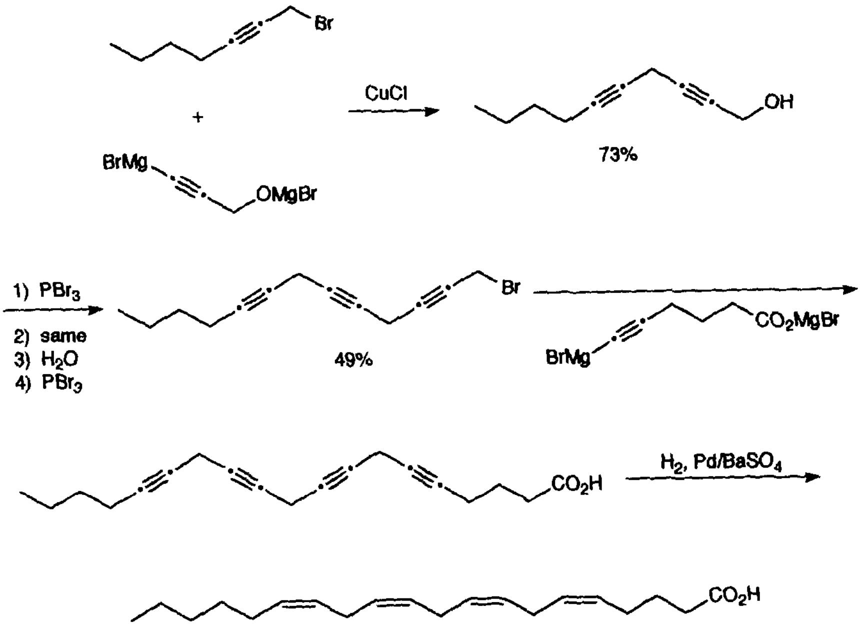
R' = Me, $TMSCH_2$

Copper(I) chloride catalyzed the coupling of bisallylic halides with alkynyl Grignard reagents (equation 4) [9], (equation 5) [10] α,ω-bishaloalkanes were monoalkylated by Grignard reagents in the presence of a copper catalyst. Arylmanganese halides coupled to thirteen different alkenyl halides, while Li_2MnCl_4 catalyzed the alkylation of allylic halides by arylmagnesium halides [12].

(Equation 4)



(Equation 5



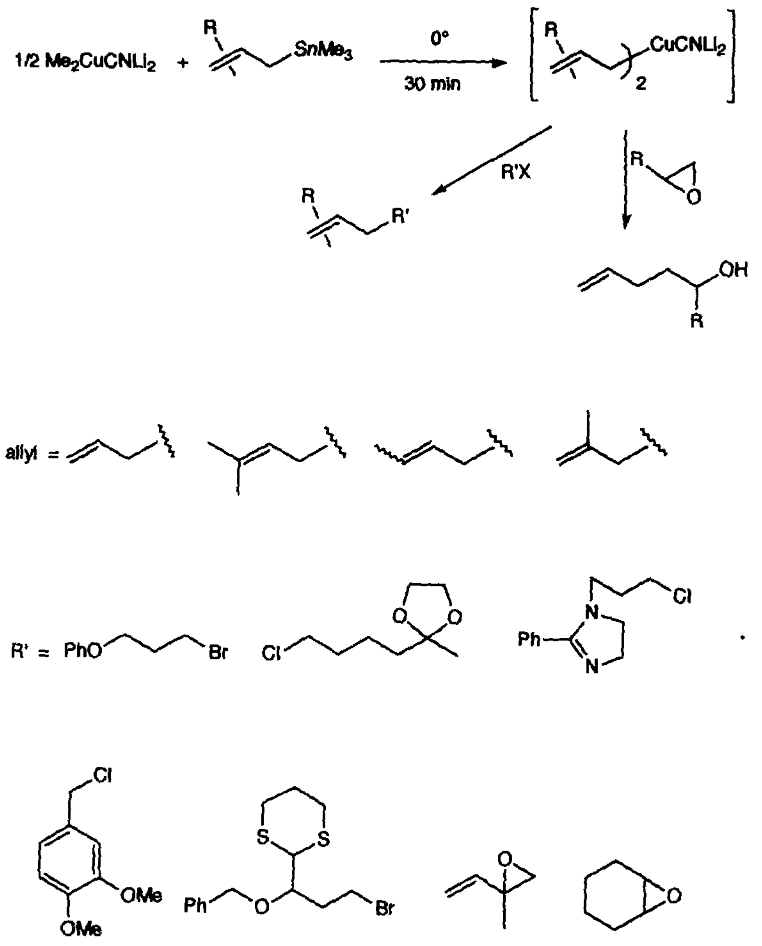
(Equation 6)

$$RMgBr + Br(CH_2)_nBr \xrightarrow{5\%\ Li_2CuCl_4} R(CH_2)_n{-}Br$$

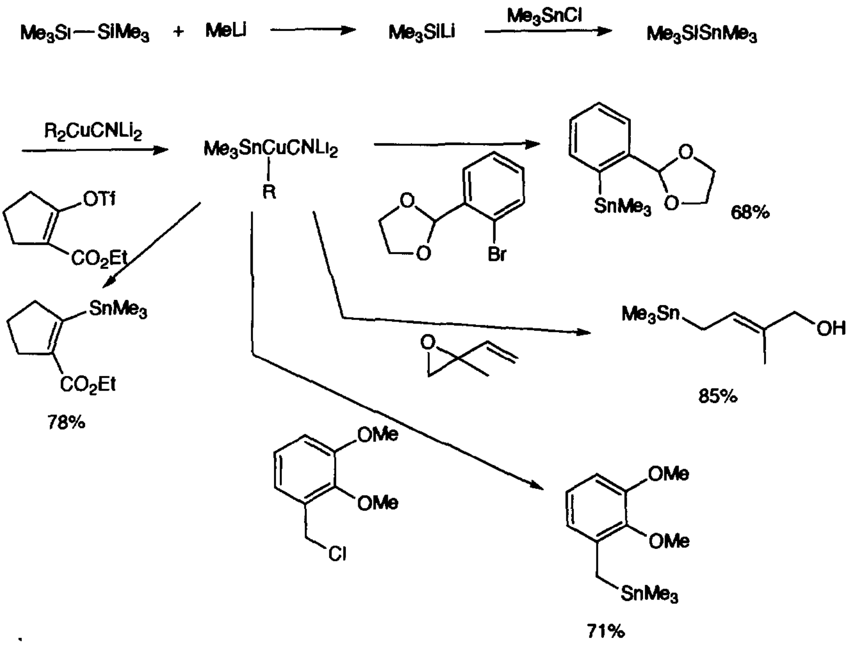
70-80%

The evolution of higher-order cuprates has been reviewed (56 references) [13]. A paper dealing with in situ cuprate formation via transmetallation between vinyl stannanes and higher-order cuprates was published [14]. Allylcuprates were prepared from allyl stannanes and higher-order (methyl)cyanocuprates, and these efficiently coupled with halides and epoxides (equation 7) [15] Stannyl cuprates were prepared, and they transferred their tin group to a variety of substrates (equation 8) [16]

(Equation 7)

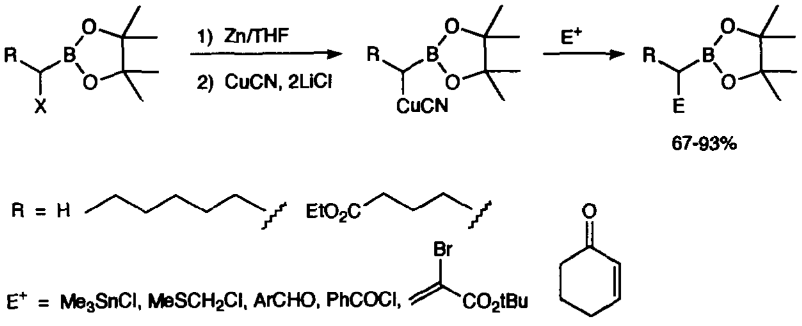

1/2 Me2CuCNLi2
+
R
SnMe3
0°
30 min
R
CuCNLi2
2
R'X
R
R'
R
O
OH
R
allyl =
R' = PhO
Br
Cl
O
O
N
Cl
Ph
N
Cl
OMe
OMe
S
S
Ph
O
Br
O
O

(Equation 8)

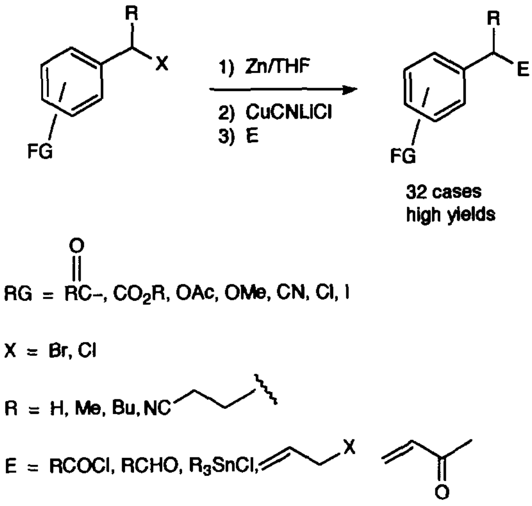


Functionalized organocopper species were readily prepared from organozinc compounds, were used in a variety of useful processes (equation 9) [17], (equation 10) [18], (equation 11) [19], (equation 12) [20], (equation 13) [21] Cuprates alkylated alkynyl iodides (equation 14) [22] Heterocyclic organocuprates introduced these functional groups into amino acids (equation 15) [23]

(Equation 9)



(Equation 10)



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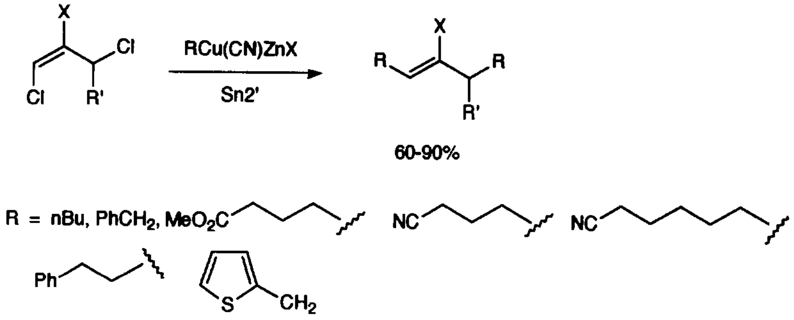
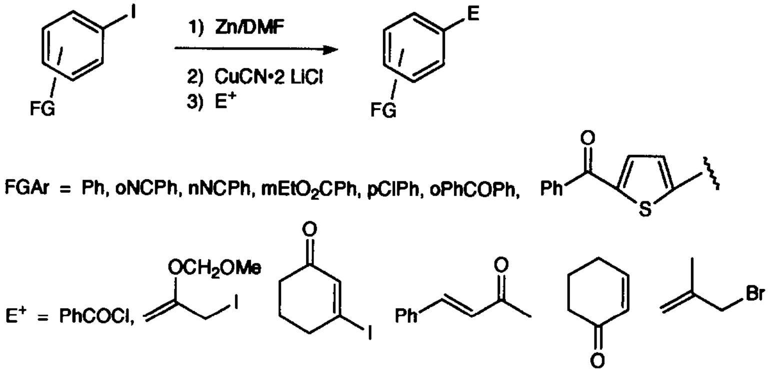
(Equation 11)



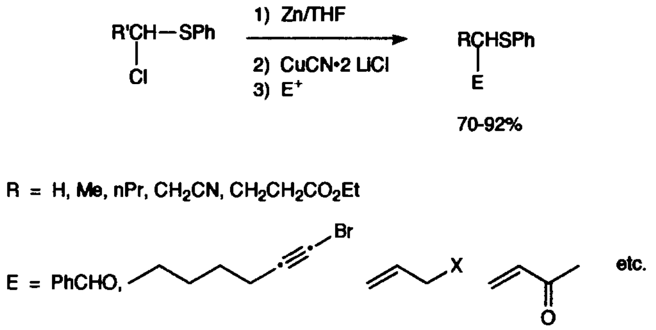
R' = Me, H

Y = SPh, SePh

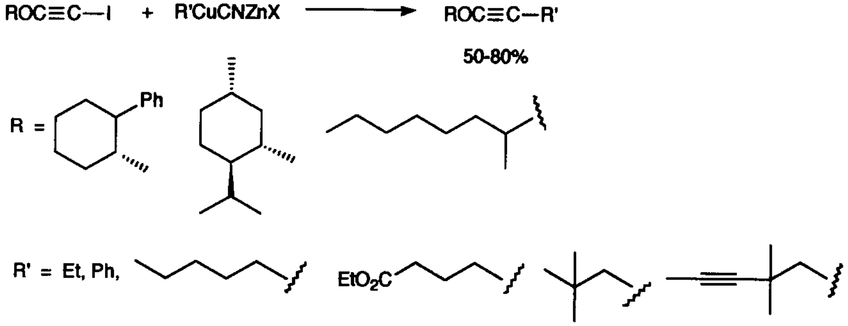
(Equation 12)



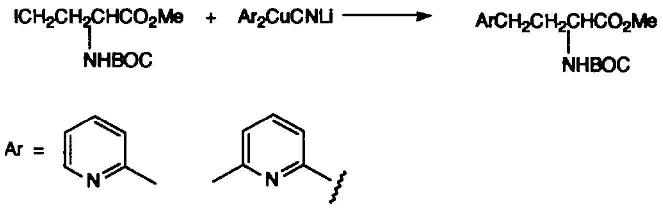
(Equation 13)



(Equation 14)

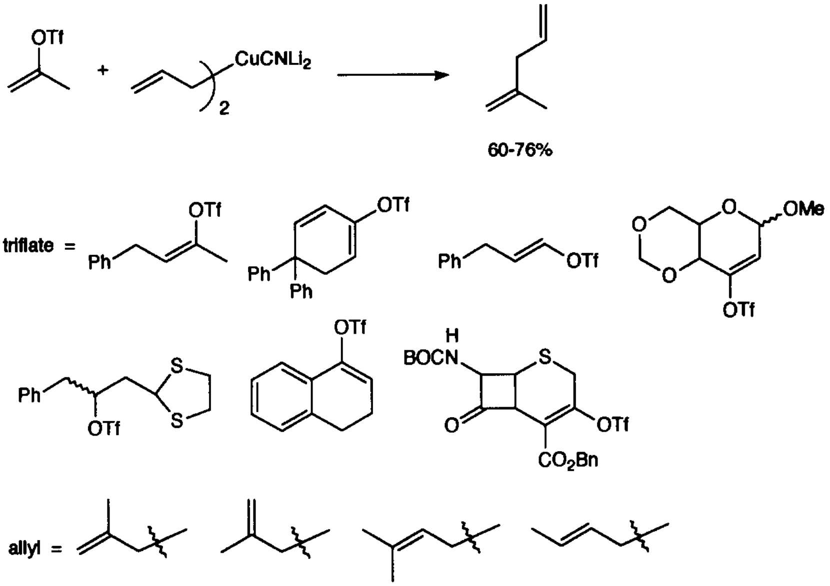


(Equation 15)

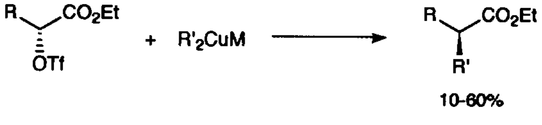


Allyl cuprates coupled to vinyl triflates (equation 16) [24] Triflates of optically active lactic acids were alkylated by cuprates with inversion (equation 17) [25] Triflates of cephams (equation 18) [26] and penems (equation 19) [27] were alkylated by cuprates. Enol triflates of lactones were similarly alkylated (equation 20) [28]

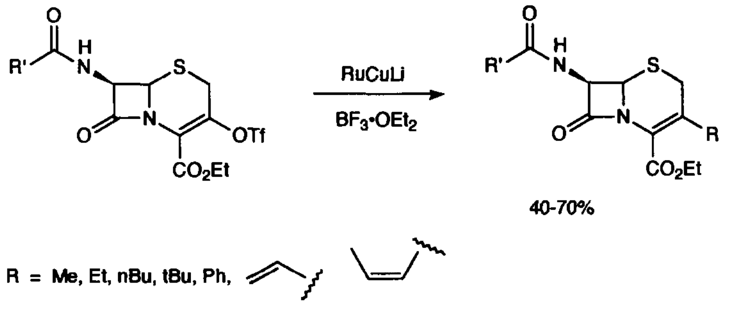
(Equation 16)



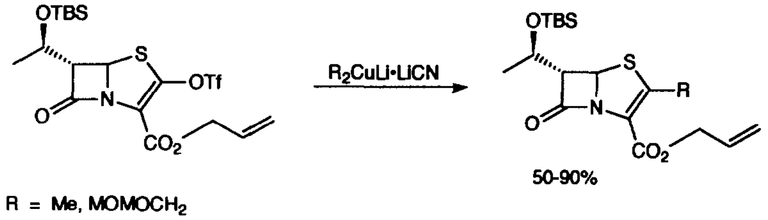
(Equation 17)



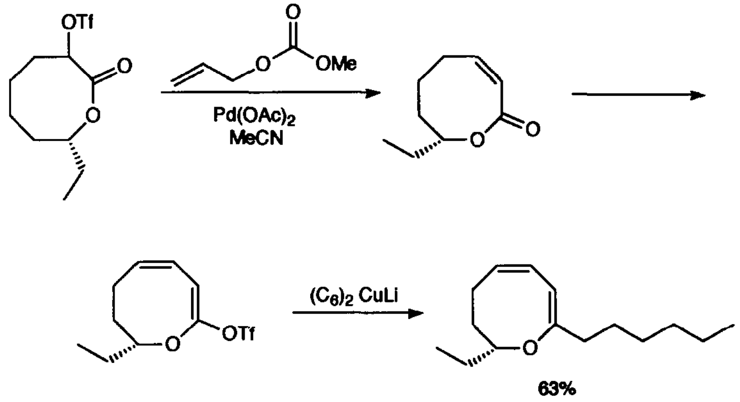
(Equation 18)



(Equation 19)

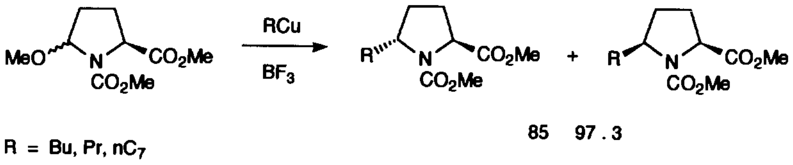


(Equation 20)

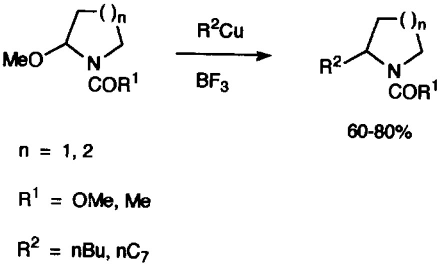


Cuprates alkylated α-methoxy amines (equation 21) [29], (equation 22) [30], and α-mesyloxy epoxides (equation 23) [31]; and ring opened trifluoromethyl epoxides (equation 24) [32] and N-tosylazirines (equation 25) [33]

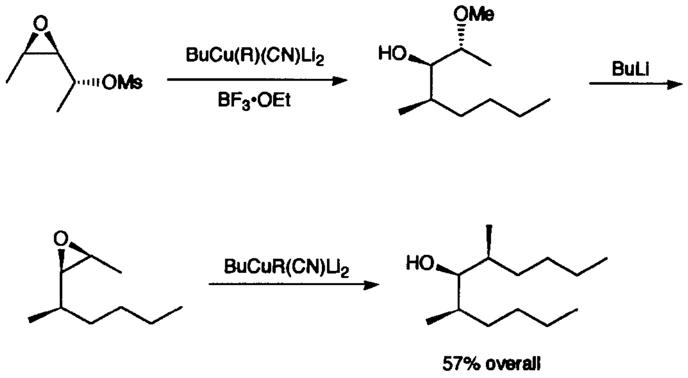
(Equation 21)



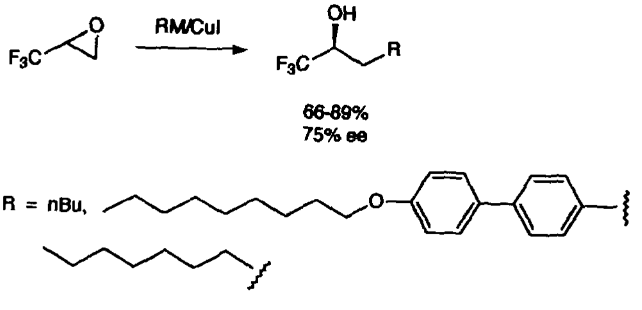
(Equation 22)



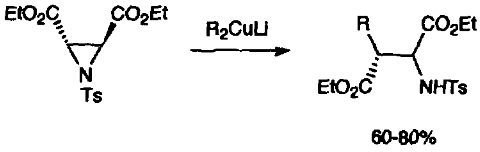
(Equation 23)



(Equation 24)

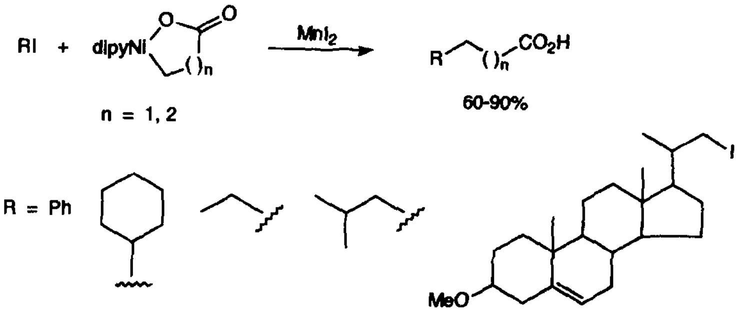


(Equation 25)



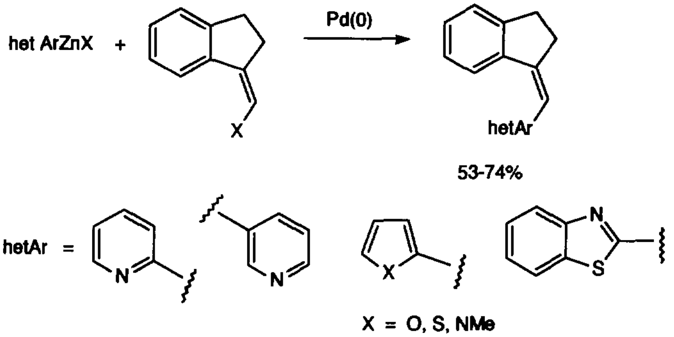
Manganese iodide promoted the alkylation of organic halides by a nickelacyclic propionic acid equivalent (equation 26) [34][35]

(Equation 26)

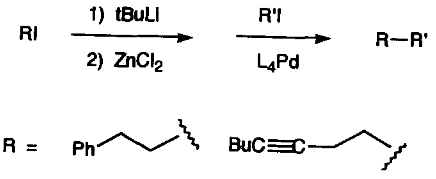


Alkylation of organic halides and triflates by palladium(0) catalyzed oxidative addition/transmetallation/reductive elimination process has become a growth industry, and many, many papers dealing with this process have recently appeared Aryl ethylenes (styrenes) were prepared by the palladium catalyzed cross-coupling of vinyl zinc chloride and vinyl trimethyl tin with aryl halides [36] Heteroaryl zinc reagents alkylated vinyl halides in the presence of palladium(0) catalysts (equation 27) [37]. Organic iodides were cross-coupled via transmetallation processes (equation 28) [38] Palladium catalyzed the alkylation of highly substituted furyl zinc halides by organic halides (equation 29) [39]. Aryl halides were cross-coupled to organozinc chlorides using palladium catalysis (equation 30) [40]

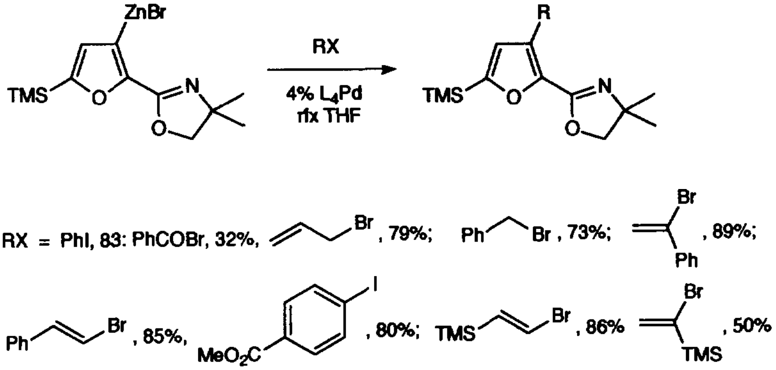
(Equation 27)



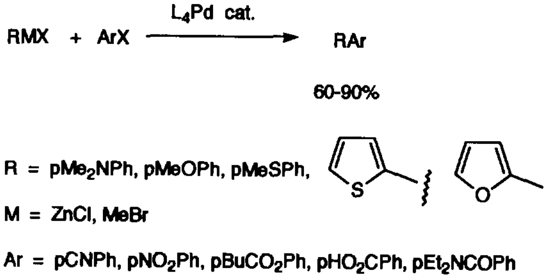
(Equation 28)



(Equation 29)

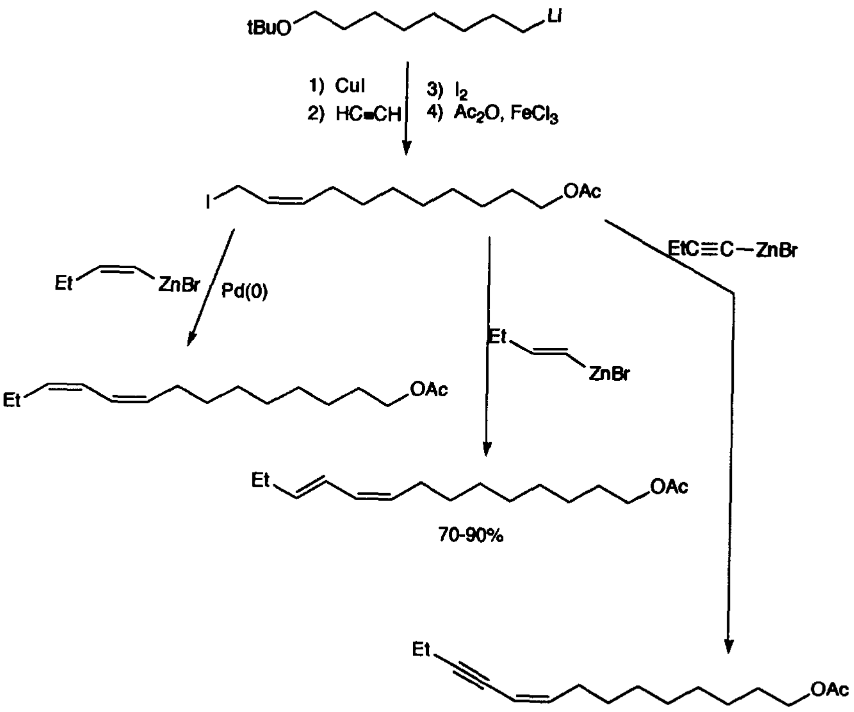


(Equation 30)

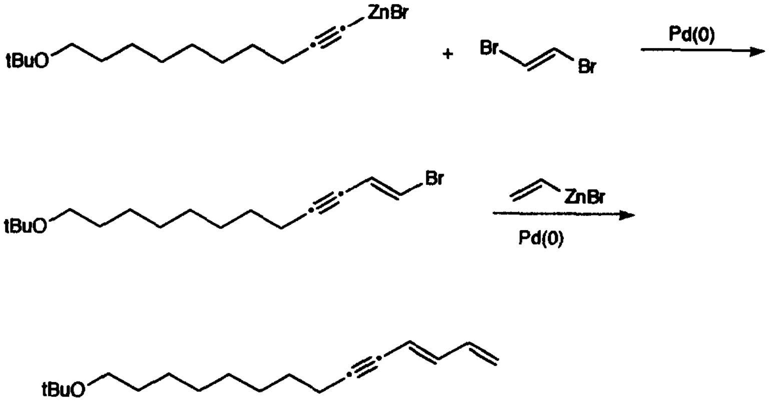


Palladium(0) catalyzed the coupling of long chain vinyl iodides with organozinc reagents (equation 31) [41], (equation 32) [42]. Aryl and vinyl triflates coupled to heteroaryl zinc halides in the presence of palladium catalysts (equation 33) [43]. α-Lithiated enol carbamates coupled to organic halides in the presence of zinc chloride and palladium(0) catalysts (equation 34) [44].

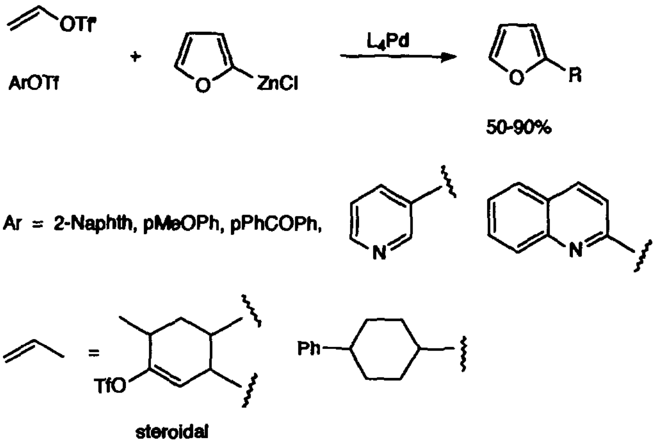
(Equation 31)



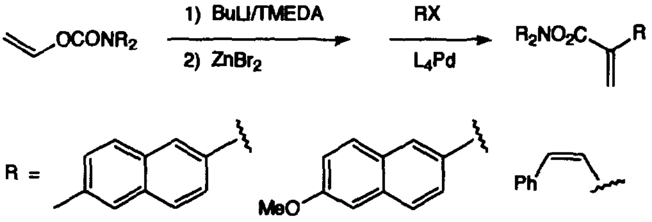
(Equation 32)



(Equation 33)

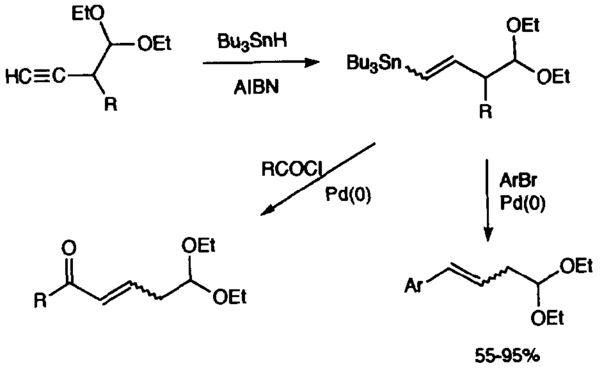


(Equation 34)

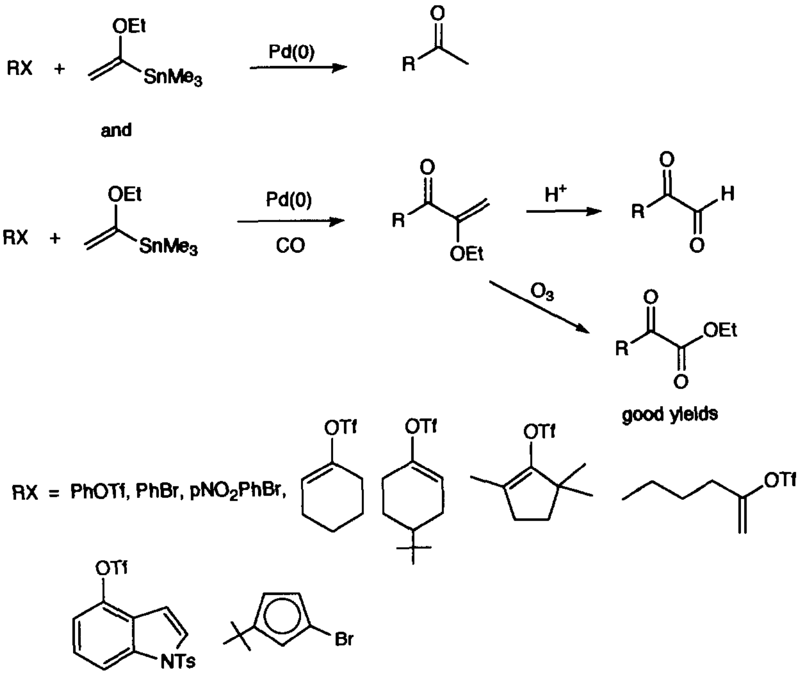


Transmetallation from tin to palladium has also been extensively investigated. "Synthesis of natural products using the palladium-catalyzed coupling of vinyltins and vinylhalides" was the title of a dissertation [45]. Palladium(0) catalyzed the coupling of functionalized vinyltins with halides (equation 35) [46] and triflates (equation 36) [47].

(Equation 35)

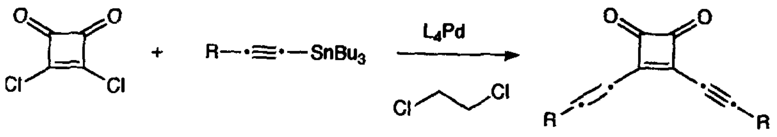


(Equation 36)



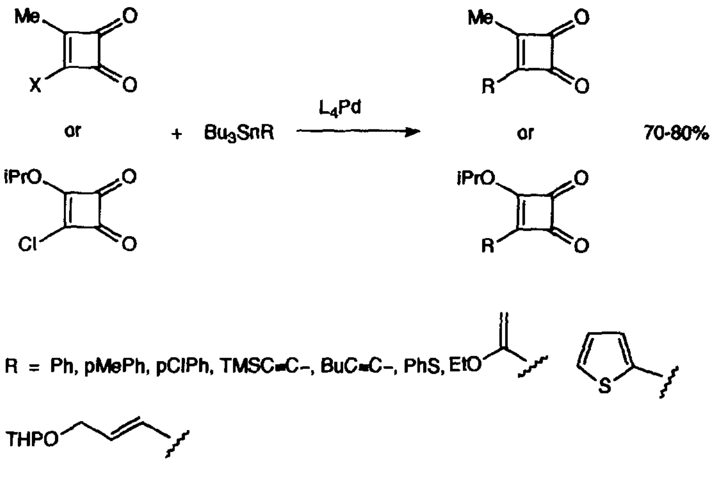
Cyclobutene diones were alkylated using palladium/tin chemistry (equation 37) [48], (equation 38) [49][50], as were α-stannylated dihydrofurans and dihydropyranes (equation 39) [51]

(Equation 37)

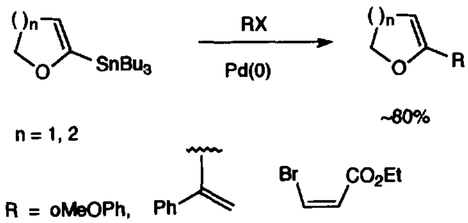


R = Ph, 70%; PhC≡C, 11%; nPr, 51%; TMS, 30%

(Equation 38)

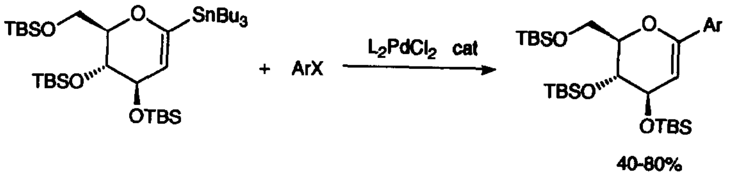


(Equation 39)



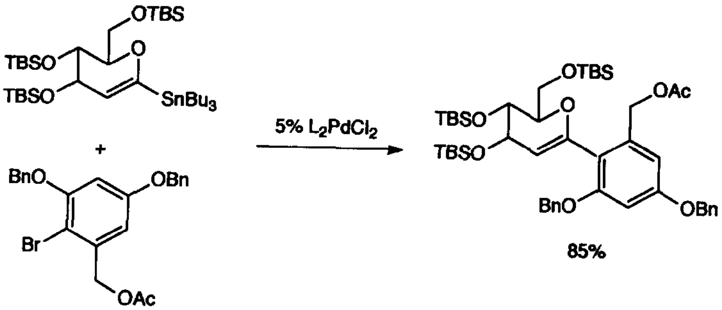
This chemistry has been extensively used to prepare alkylated dehydro sugars (equation 40) [52], (equation 41) [53], (equation 42) [54], (equation 43) [55], and alkylated nucleosides (equation 44) [56]

(Equation 40)

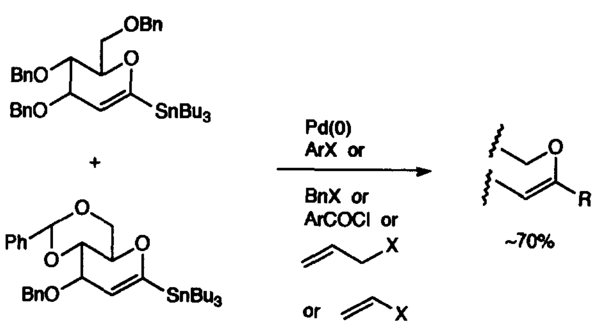


Ar = Ph, pNO_2Ph, pCNPh, 1-naphth, $pMeO_2CPh$, pClPh, oAcOPh, oBnOPh, 2,5-$(MeO)_2Ph$

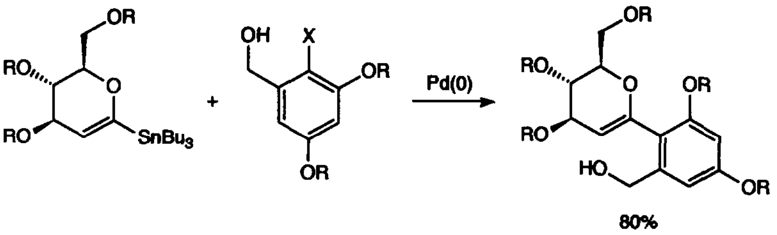
(Equation 41)



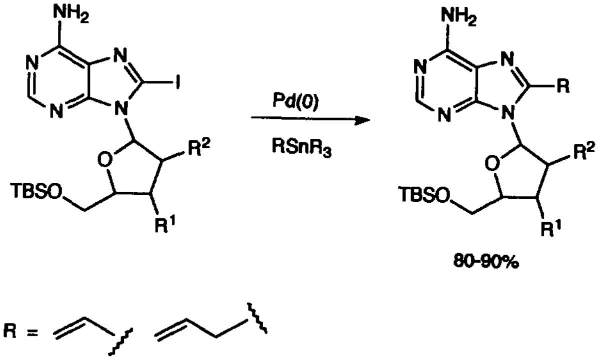
(Equation 42)



(Equation 43)

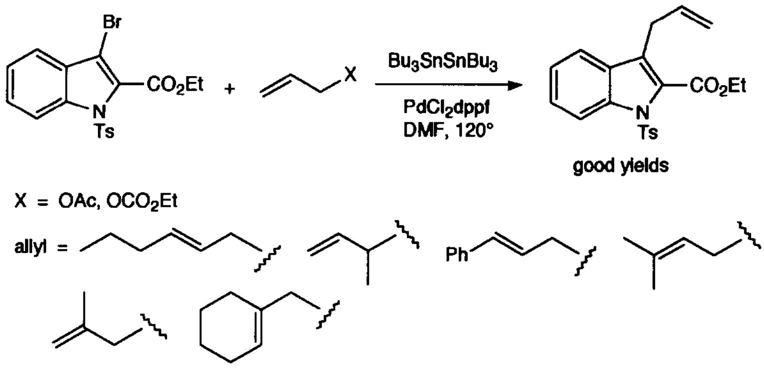


(Equation 44)

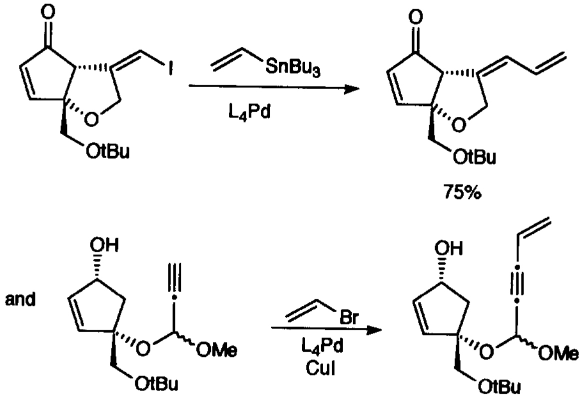


Palladium catalyzed coupling of halides to organostannanes has also been used to functionalize indoles (equation 45) [57], cyclopentenols (equation 46) [58], quinolines (equation 47) [59], and to make medium ring ene diynes (equation 48) [60]

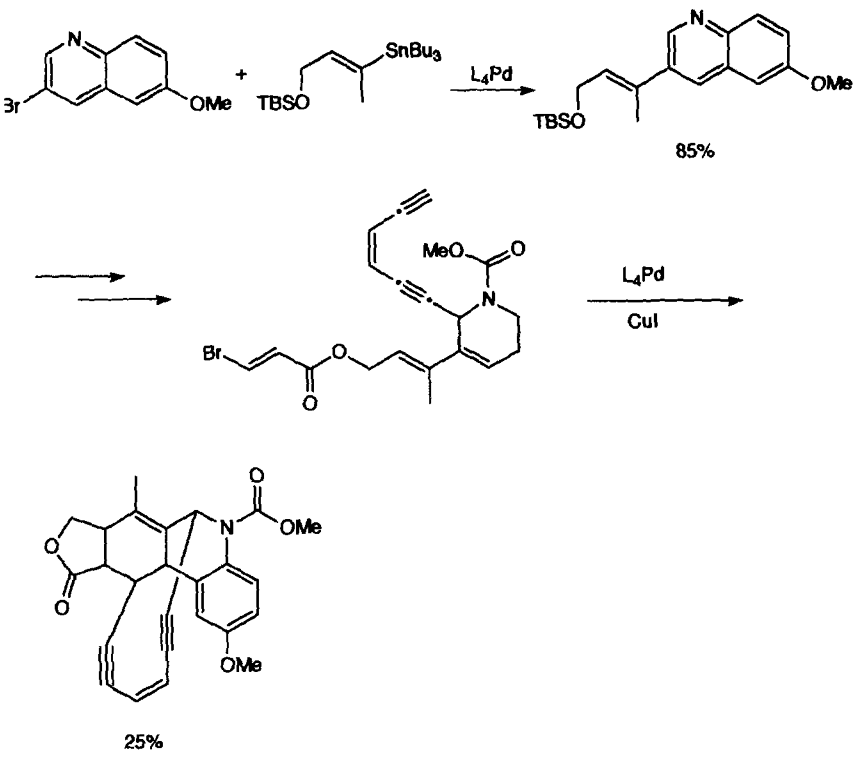
(Equation 45)



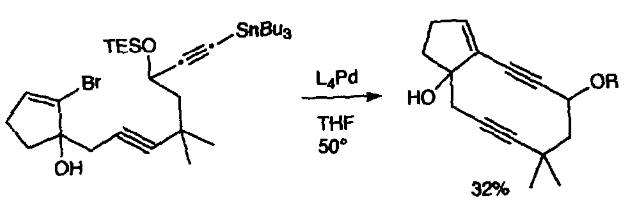
(Equation 46)



(Equation 47)



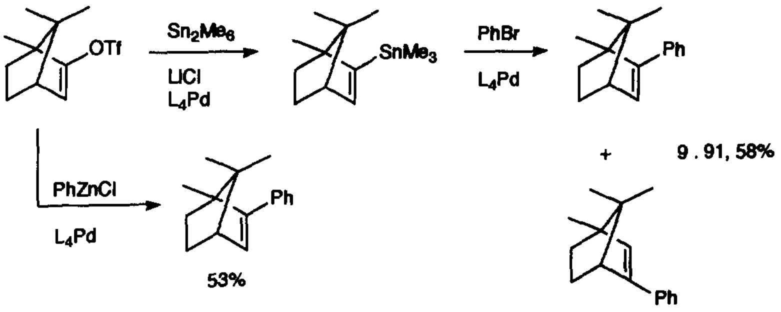
(Equation 48)



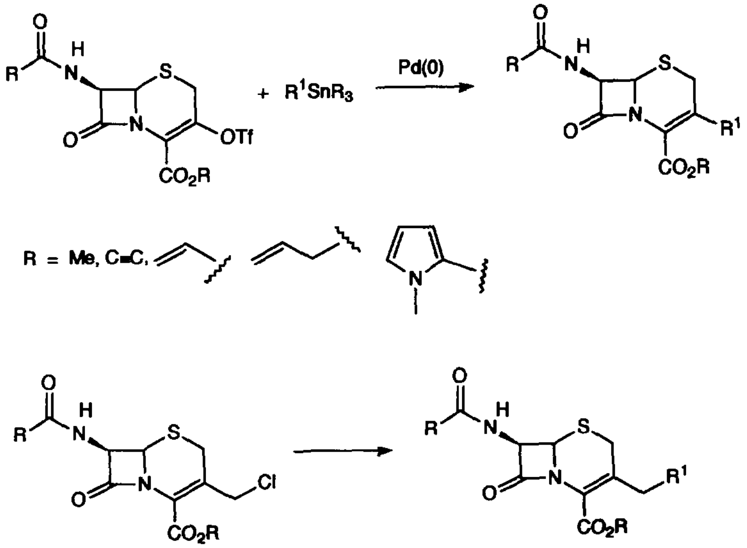
Palladium(0) complexes also catalyzed the coupling of organotriflates with stannanes (equation 49) [61] This has been extensively used in functionalizing β-lactams (equation 50) [62], (equation 51) [63], (equation

52) [64], prostanoid chemistry [65], steroid functionalization (equation 53) [66] and nucleoside chemistry (equation 54) [67] Highly functionalized aryl triflates were also alkylated utilizing this reaction (equation 55) [68], (equation 56) [69], (equation 57) [70], (equation 58) [71]

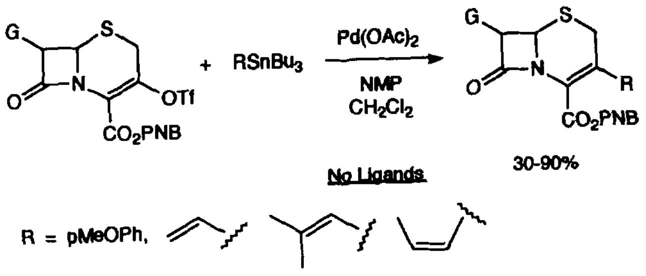
(Equation 49)



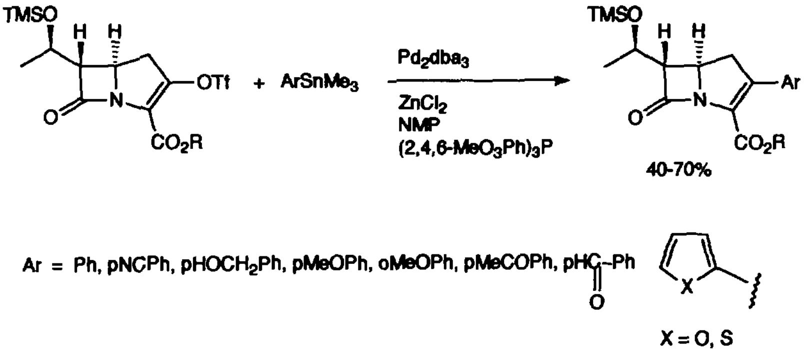
(Equation 50)



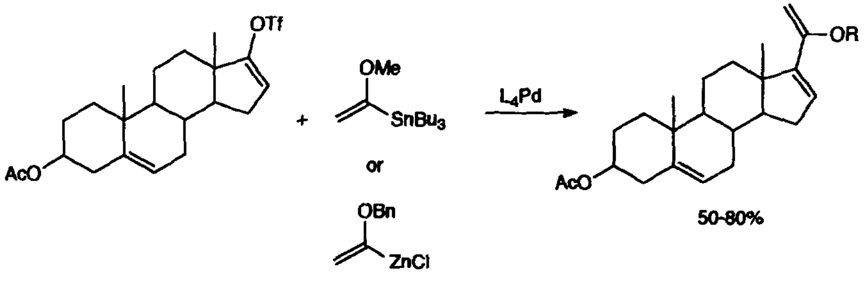
(Equation 51)



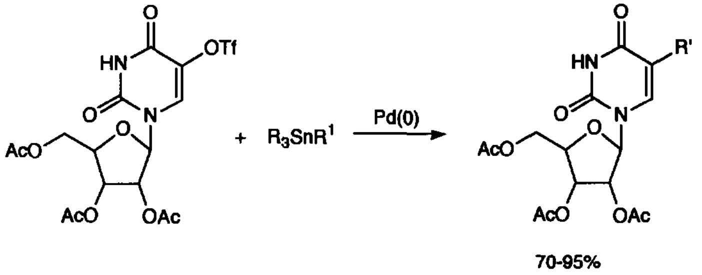
(Equation 52)



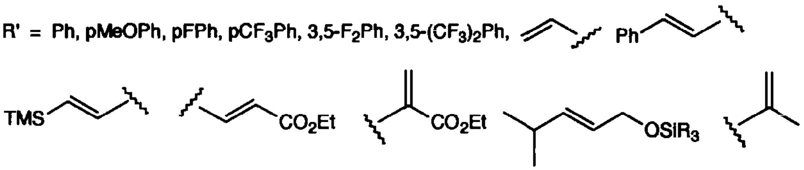
(Equation 53)



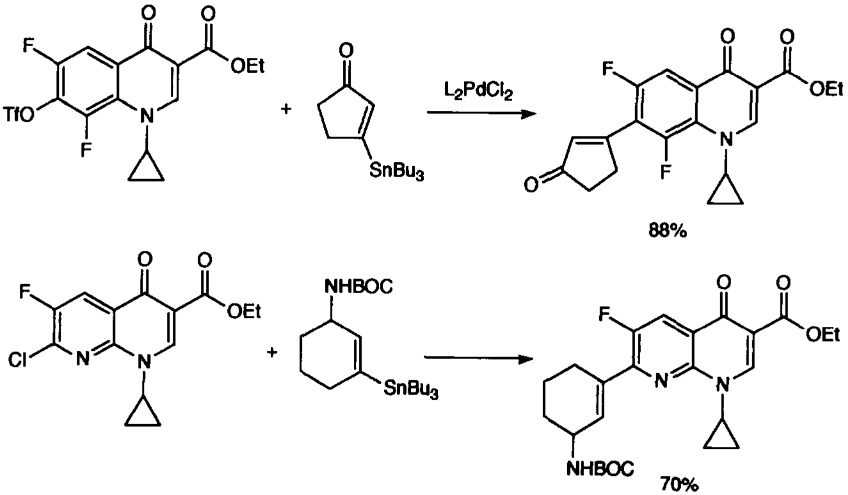
(Equation 54)



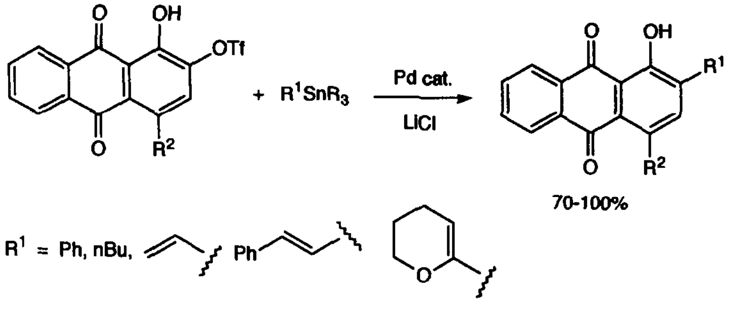
R' = Ph, pMeOPh, pFPh, pCF_3Ph, 3,5-F_2Ph, 3,5-$(CF_3)_2Ph$,



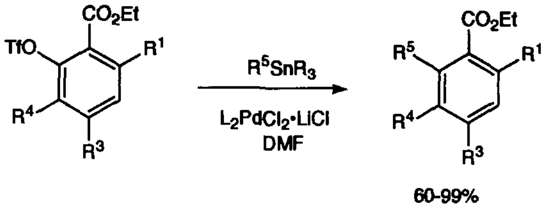
(Equation 55)



(Equation 56)

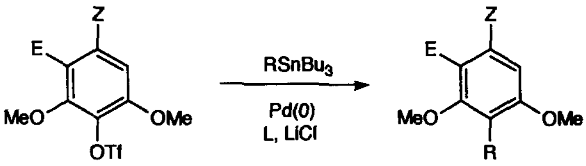


(Equation 57)



R^1 = iPr, 4-FPh; R^3 = Ph, Me, tBu; R^4 = H, Me; R^5 = pFPh, 3-Me-4-FPh, Et

(Equation 58)

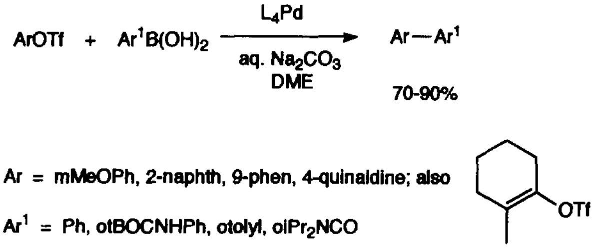


E, Z = H, Z = OMe; E = Br, SMe, Cl

R = H, alkyl, alkenyl, aryl, allyl, alkynyl

Palladium(0) complexes also catalyze the reactions between organic halides or triflates and organoboron compounds (transmetallations from B to Pd). Biaryls were synthesized by the palladium catalyzed coupling of aryl triflates with aryl borates (equation 59) [72] and aryl halides (equation 60) [73] Organic halides were methylated by methyl boron compounds (equation 61) [74] and arylated by aryl borates (equation 62) [75], (equation 63) [76], (equation 64) [77] Vinyl boranes coupled to vinyl halides in the presence of palladium(0) catalysts (equation 65) [78], (equation 66) [79].

(Equation 59)

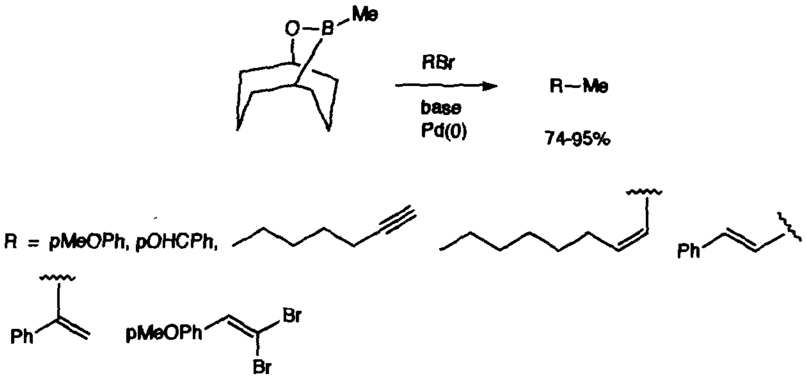


(Equation 60)

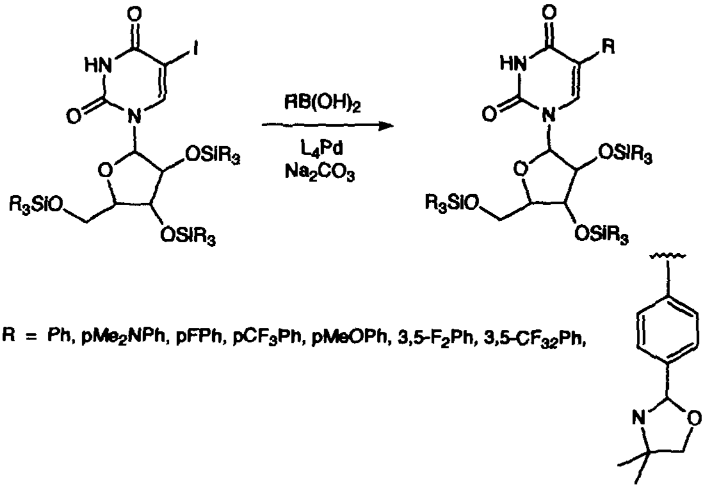
$$\mathrm{ArBr} + [\mathrm{Ar^1{}_4B}]\mathrm{Na} \xrightarrow[\substack{80^\circ \\ \text{anisole}}]{\text{Pd(0) cat.}} \underset{\text{major}}{\mathrm{ArAr^1}} + \underset{\text{minor}}{\mathrm{Ar^1Ar^1}}$$

$$60\text{-}90\%$$

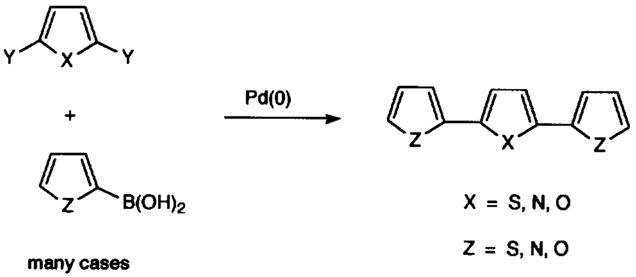
(Equation 61)



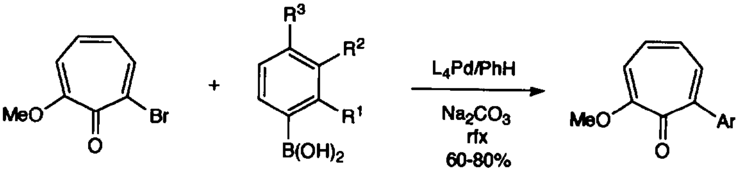
(Equation 62)



(Equation 63)



(Equation 64)

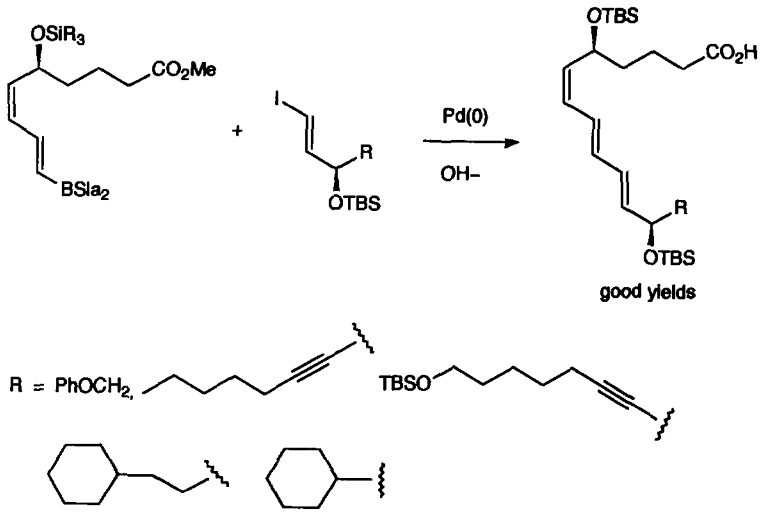


R^1 = H, OMe

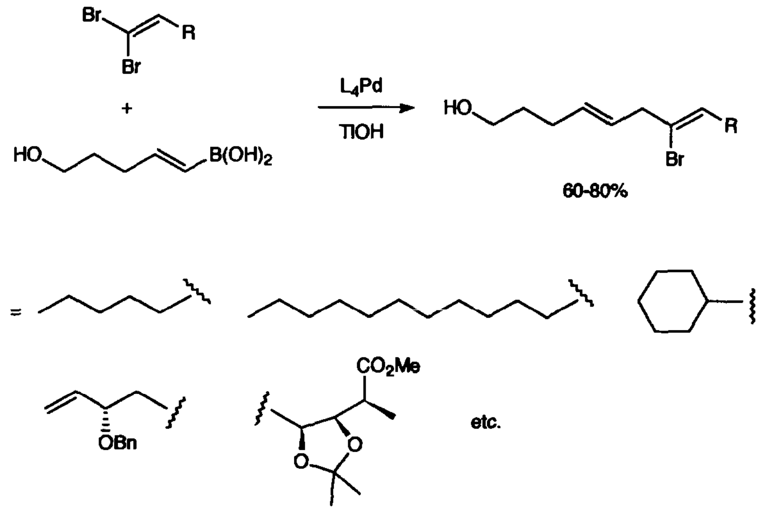
R^2 = H, OMe, NO_2

R^3 = H, OMe, Br

(Equation 65)



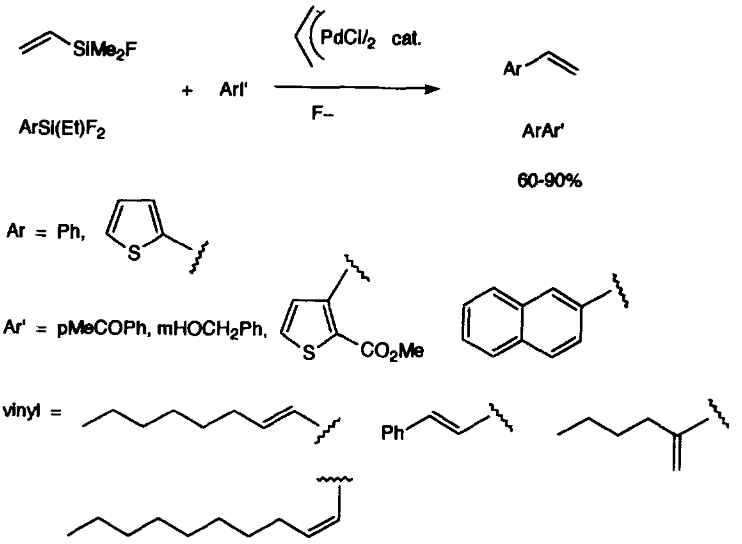
(Equation 66)



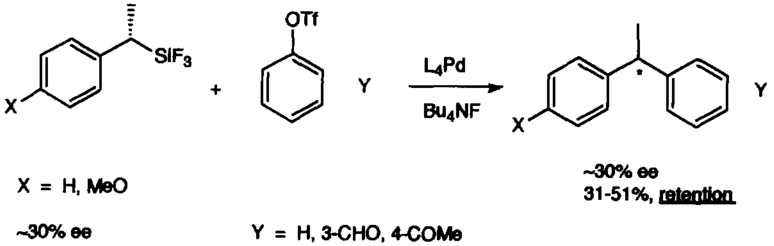
p 643

Transmetallation from silicon to palladium, mediated by fluoride ion has been achieved, and was recently reviewed (42 references) [80]. Aryl iodides (equation 67) [81] and aryl (equation 68) [82] and vinyl triflates (equation 69) [83] coupled to fluorosilanes. Palladium(0) catalyzed the coupling of vinyl germanes to aryl diazonium salts (equation 70) [84]

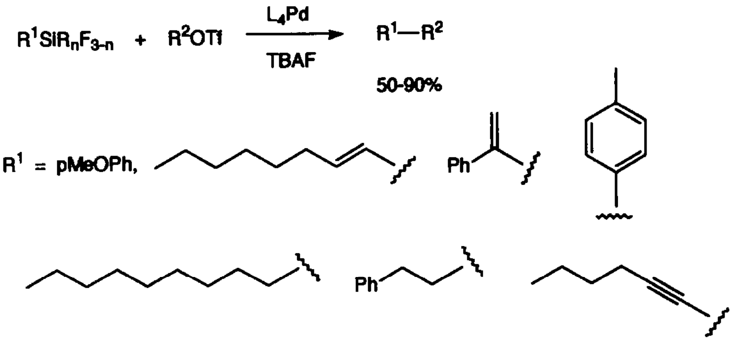
(Equation 67)



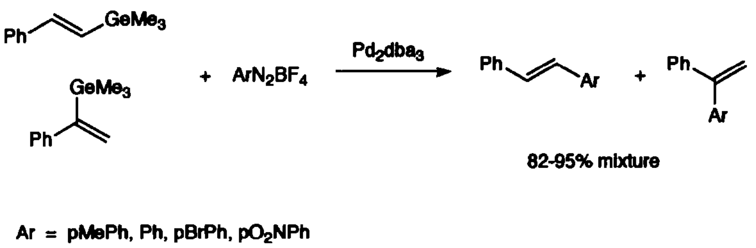
(Equation 68)



(Equation 69)

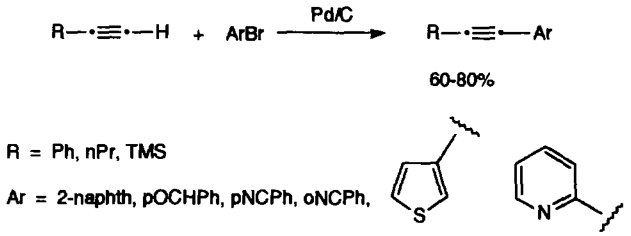


(Equation 70)

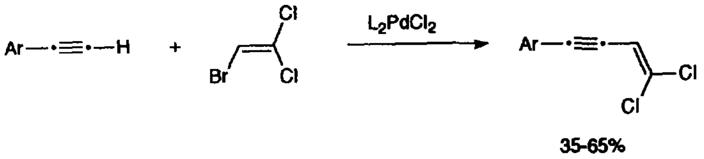


The long-known palladium-catalyzed coupling of terminal alkynes to aryl and vinyl halides is enjoying a resurgence with the advent of enyne natural products. Palladium on carbon catalyzed the coupling of terminal alkynes to aryl bromides (equation 71) [85]. Palladium(II) phosphine complexes catalyzed the coupling of terminal alkynes to vinyl halides (equation 72) [86]. The conventional catalyst system consisting of palladium complexes, copper salts and amines coupled alkynes to vinyl halides (equation 73) [87], (equation 74) [88], fluoro vinyl halides (equation 75) [89], (equation 76) [90], iodopurines (equation 77) [91], (equation 78) [92], and bromothiophenes (equation 79) [93].

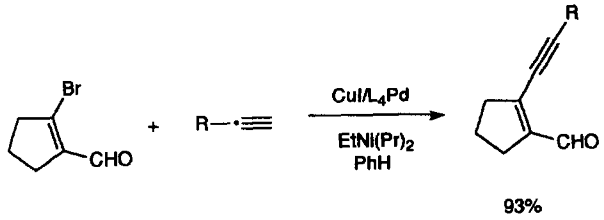
(Equation 71)



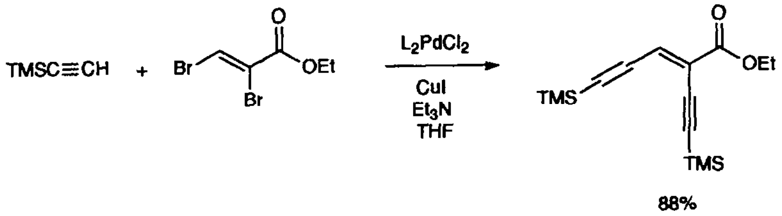
(Equation 72)



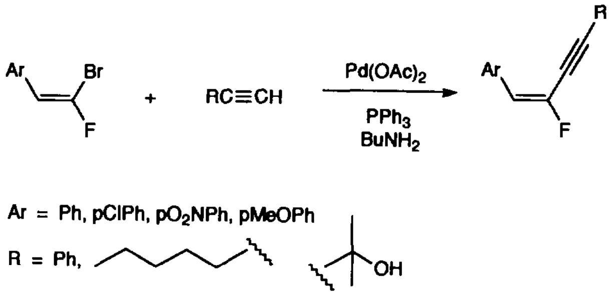
(Equation 73)



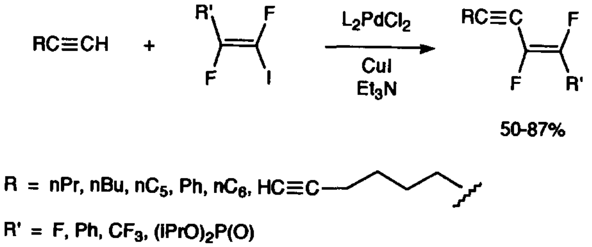
(Equation 74)



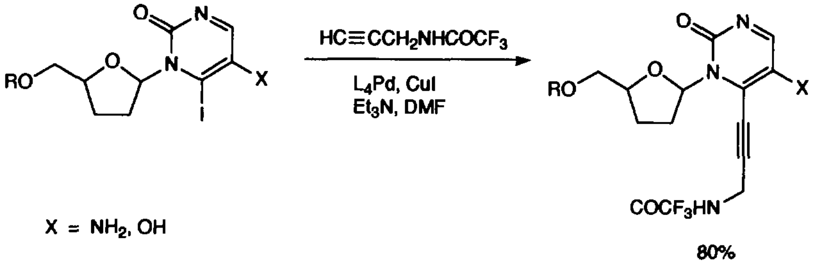
(Equation 75)



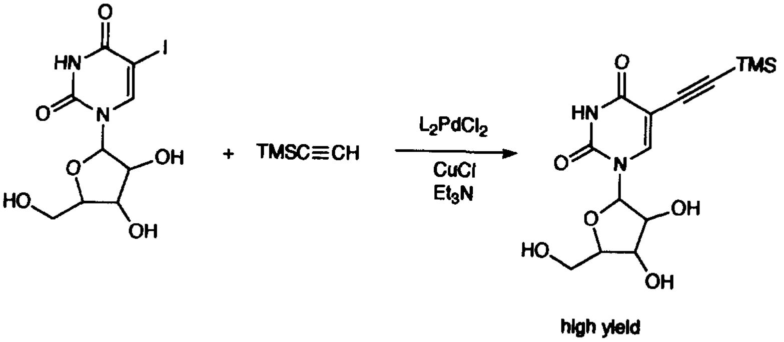
(Equation 76)



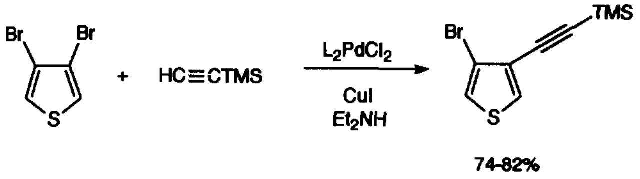
(Equation 77)



(Equation 78)

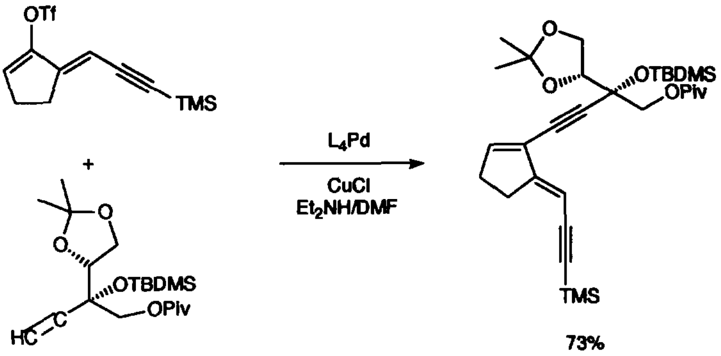


(Equation 79)

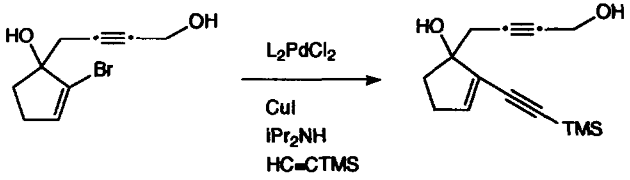


Palladium(0) mediated synthesis of polyacetylenic compounds - calicheamicin/esperamicin, was the topic of a dissertation [94] Polyacetylenes were prepared by the palladium/copper catalyzed alkylation of vinyl triflates (equation 80) [95], vinyl halides (equation 81) [96], (equation 82) [97], (equation 83) [98], and aryl halides (equation 84) [99], (equation 85) [100] Quite complex systems have been made by this procedure (equation 86) [101], (equation 87)([102]

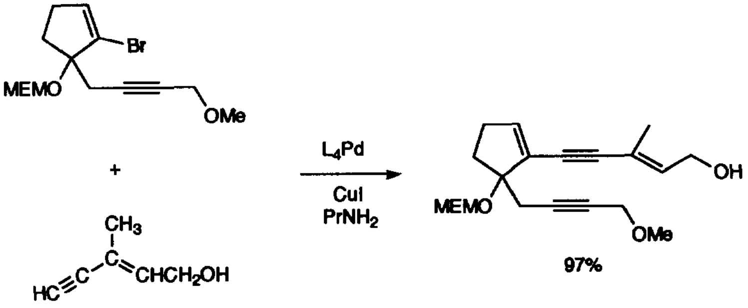
(Equation 80)



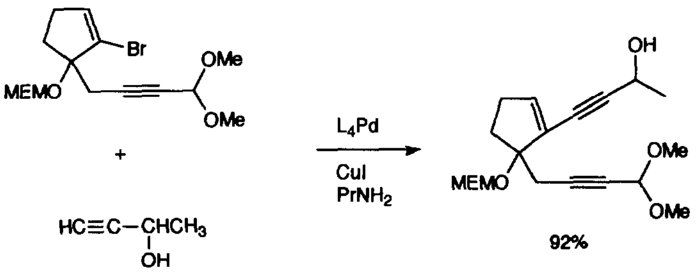
(Equation 81)



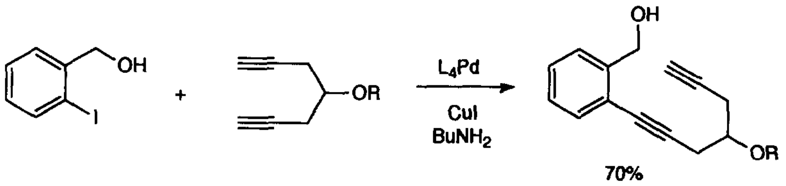
(Equation 82)



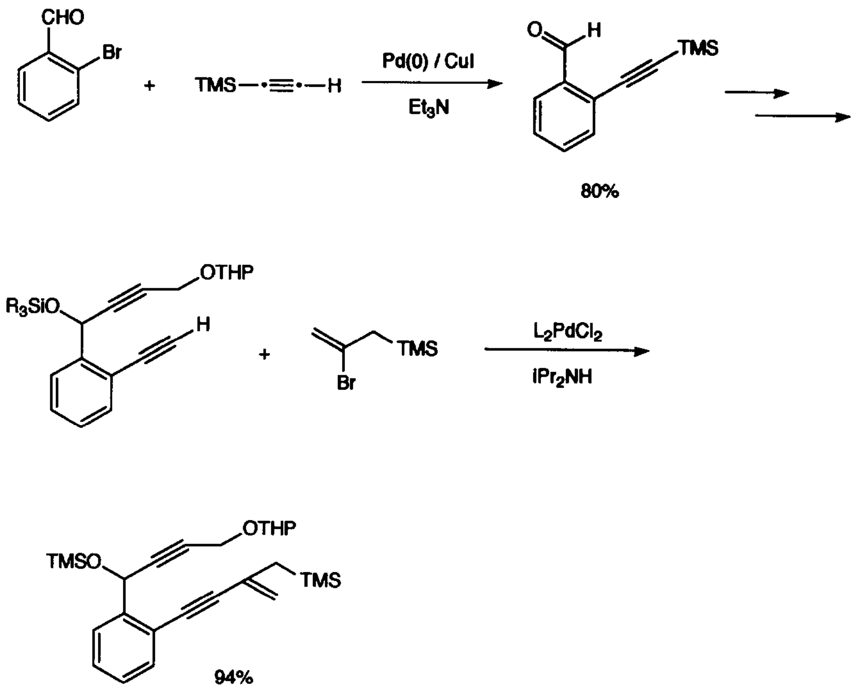
(Equation 83)



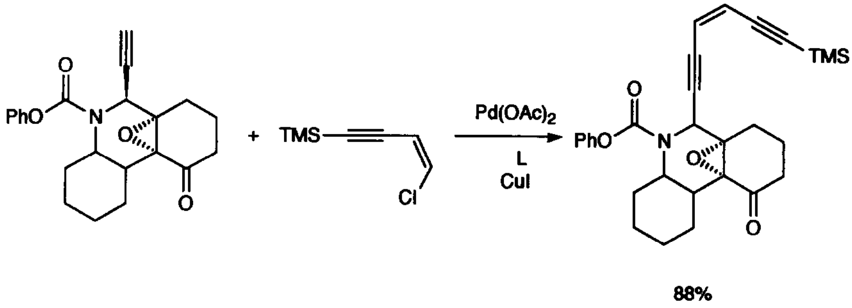
(Equation 84)



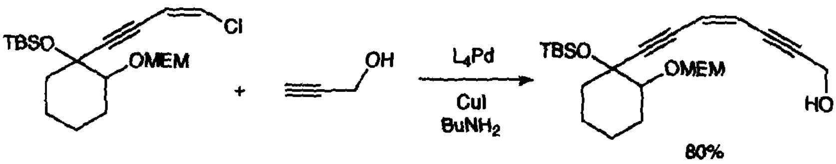
(Equation 85)



(Equation 86)

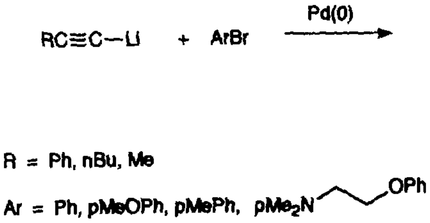


(Equation 87)

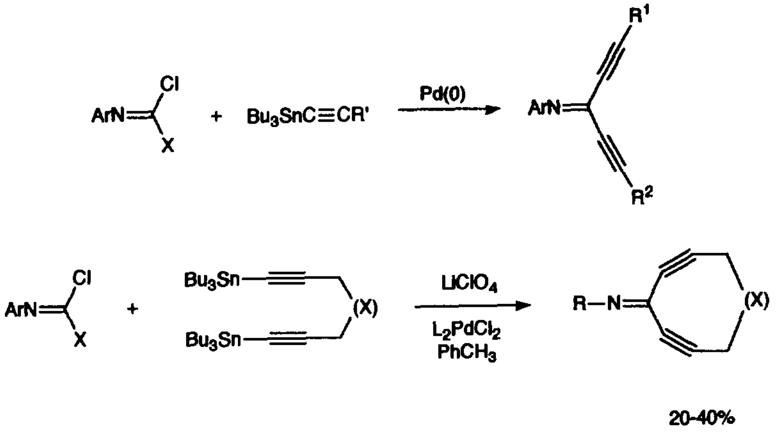


Palladium(0) catalyzed the coupling of lithium acetylides to aryl bromides (equation 88) [103], and acetylenic tin reagents to chloroimines (equation 89) [104]. Copper catalyzed the coupling of alkynes to allylic halides (equation 90) [105] and 5-iodofurfural (equation 91) [106]

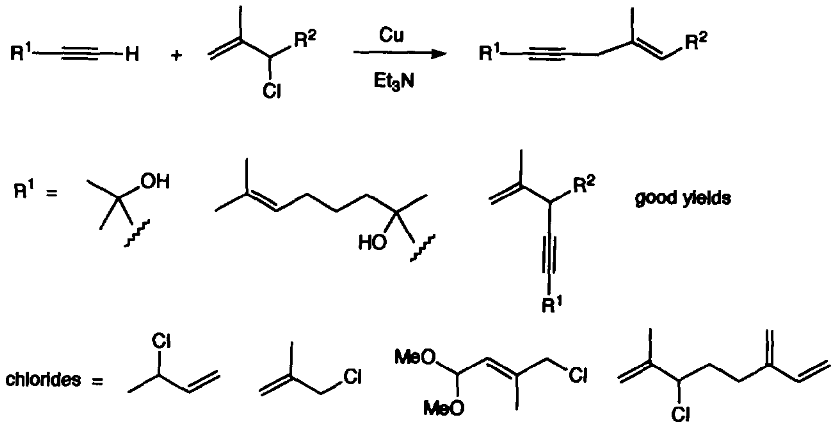
(Equation 88)



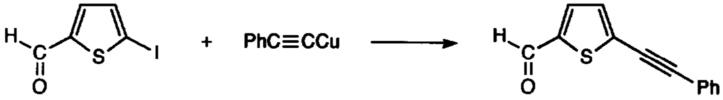
(Equation 89)



(Equation 90)

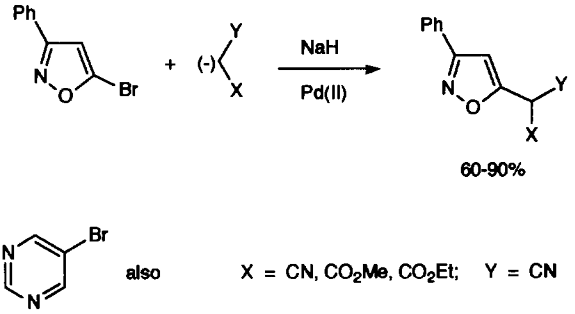


(Equation 91)



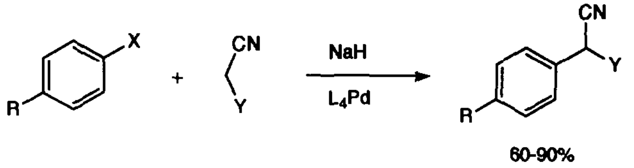
Palladium complexes catalyzed the alkylation of aryl halides by stabilized carbanions (equation 92) [107], (equation 93) [108] Complexation of diketoesters to metals permitted the control of regioselectivity of alkylation (equation 94) [109] Vinyl halides were dialkylated by stabilized carbanions in the presence of copper catalysts (equation 95) [110]

(Equation 92)



X = CN, CO_2Me, CO_2Et; Y = CN

(Equation 93)

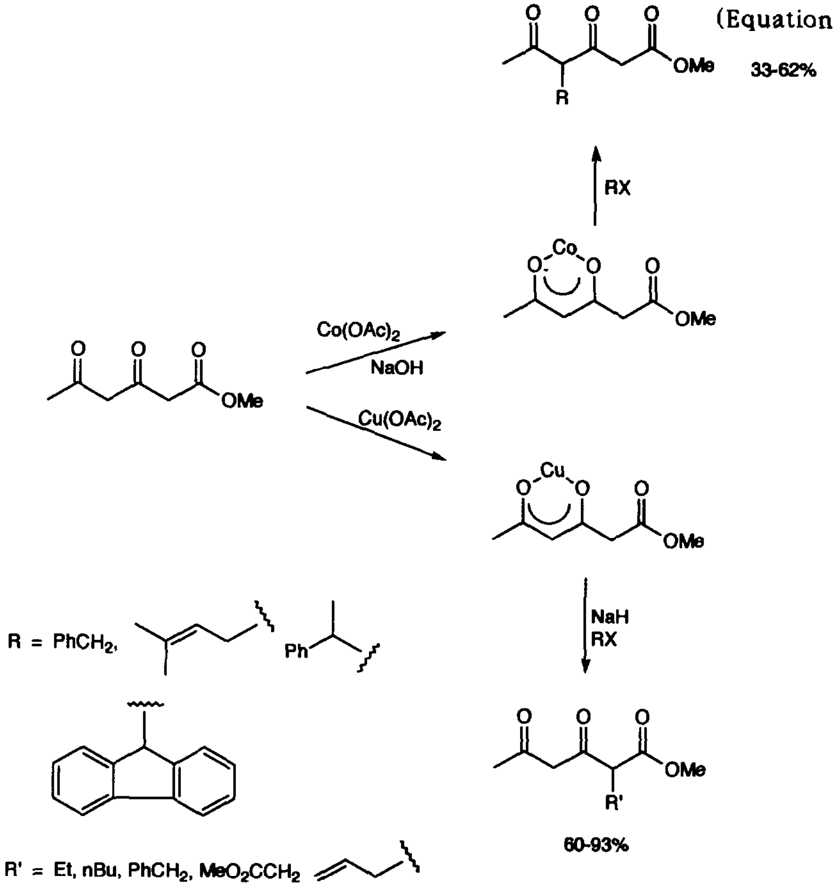


X = Br, I

Y = SO_2Ph, $P(O)(OEt)_2$

R = H, Me, MeO, CN, NO_2

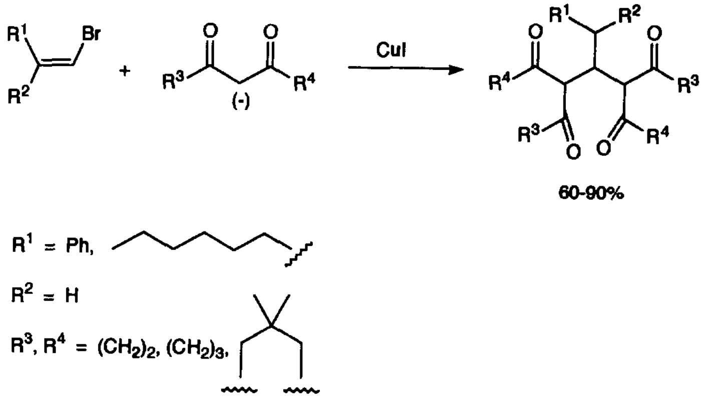
(Equation 94)



R = $PhCH_2$,

R' = Et, nBu, $PhCH_2$, MeO_2CCH_2

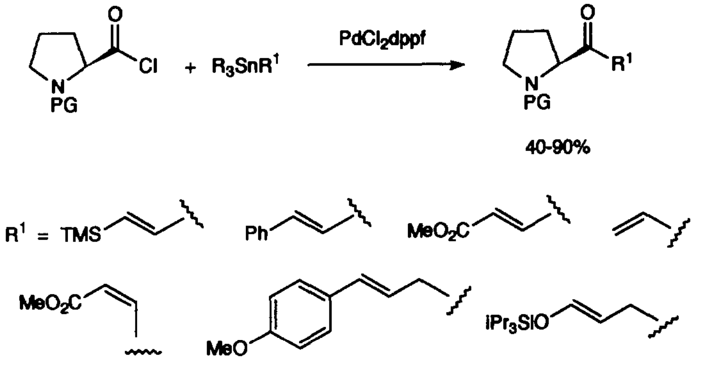
(Equation 95)



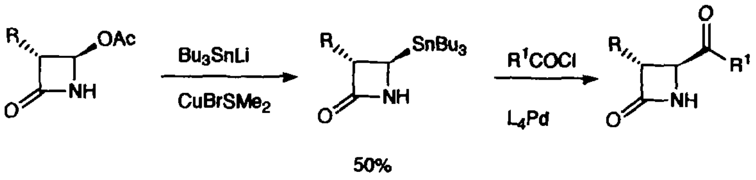
2. Alkylation of Acid Derivatives

Palladium catalyzed the coupling of L-proline acid chloride with vinyl stannanes (equation 96) [111], azetidinone stannanes with acid chlorides (equation 97) [112], and cyclization reactions (equation 98) [113]

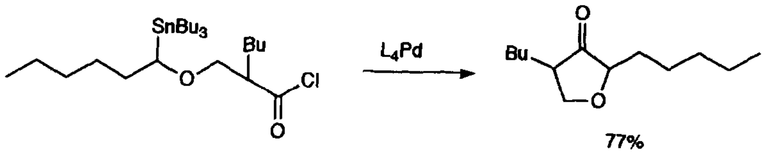
(Equation 96)



(Equation 97)



(Equation 98)



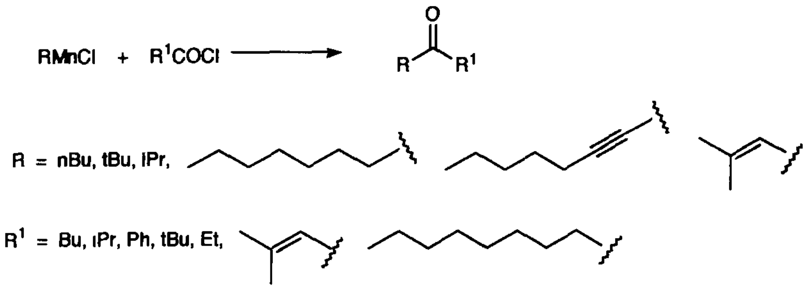
Acid chlorides were alkylated by butyliron complexes (equation 99) [114] and alkylmanganese complexes (equation 100) [115] Acid derivatives were methylenated Cp_2TiMe_2 (equation 101) [116] Ruthenium complexes catalyzed the alkylation of arenes by fluorinated sulfonyl halides (equation 102) [117]

(Equation 99)

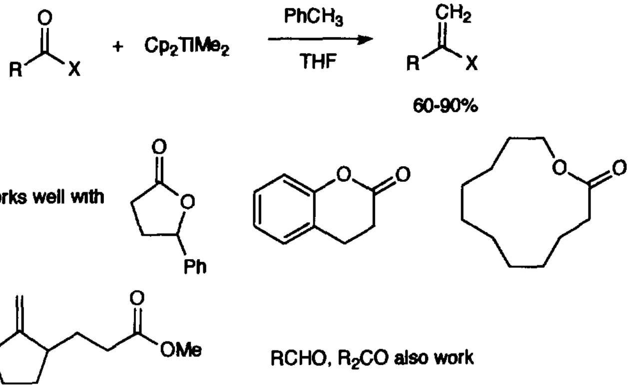
$$\mathrm{PhCOCl} + \mathrm{Bu_2Fe{\cdot}3MgBrCl} \longrightarrow \mathrm{PhCOBu}$$

94%

(Equation 100)



(Equation 101)



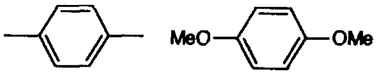
(Equation 102)

$$\mathrm{ArH} + \mathrm{R_fSO_2Cl} \xrightarrow[100°]{\mathrm{RuCl_2L_3}} \mathrm{ArR_f} + \mathrm{SO_2}$$

mixed isomers 36-54%

R_f = CF_3, C_6F_3

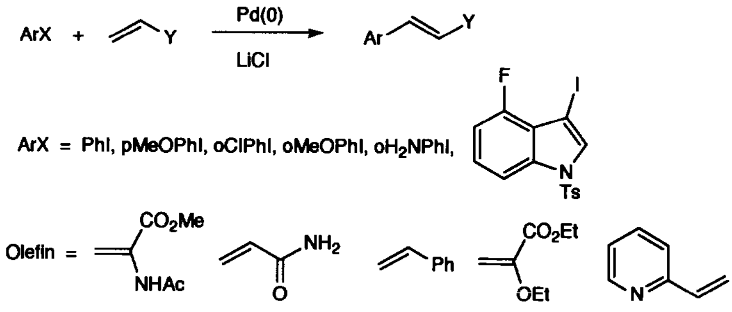
ArH = PhH, $PhCH_3$, MeOPh,



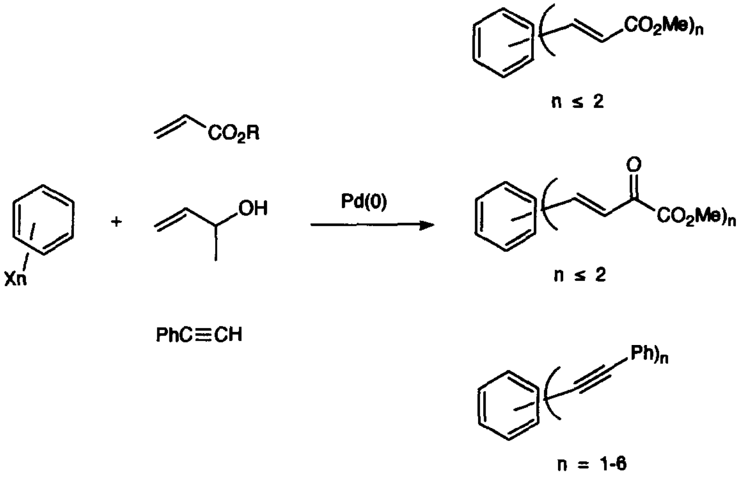
3. Alkylation of Olefins

By far the most popular way to alkylate olefins has been the "Heck" arylation procedure, involving palladium(0) catalyzed oxidative addition/olefin insertion/β-hydrogen elimination processes Many improvements and modifications have been made Lithium chloride was found to improve the yields of the Heck arylation of olefins (equation 103) [118] Sulfonated phenyl phosphines - $Ph_2P(m\text{-}O_3SPh)$ were efficient ligands for carrying out Heck arylation in aqueous solutions [119]. Acrylic acid was arylated by aryl iodides or bromides (o and pMePh, pMeOPh, pClPh, mCF_3Ph, pO_2NPh, pHOPh, mHO_2CPh) in aqueous solution using palladium(II) acetate as catalyst [120] Palladium catalysts supported on perfluorosulfonated resins also catalyzed the arylation of acrylate derivatives [121] Palladium chloride supported on montmorillonite-silica-phosphines catalyzed the arylation of methyl acrylate by p-iodoanisole but not o-iodoanisole [122] Polyhaloarenes (halogen = Br, I) were polyethylated by olefins under palladium catalysis(equation 104) [123] A range of palladium(II) salts catalyzed the arylation of acrylonitrile by aryl halides [124] Substituted aryl halides were also alkylated by olefins under Heck conditions (equation 105) [125]

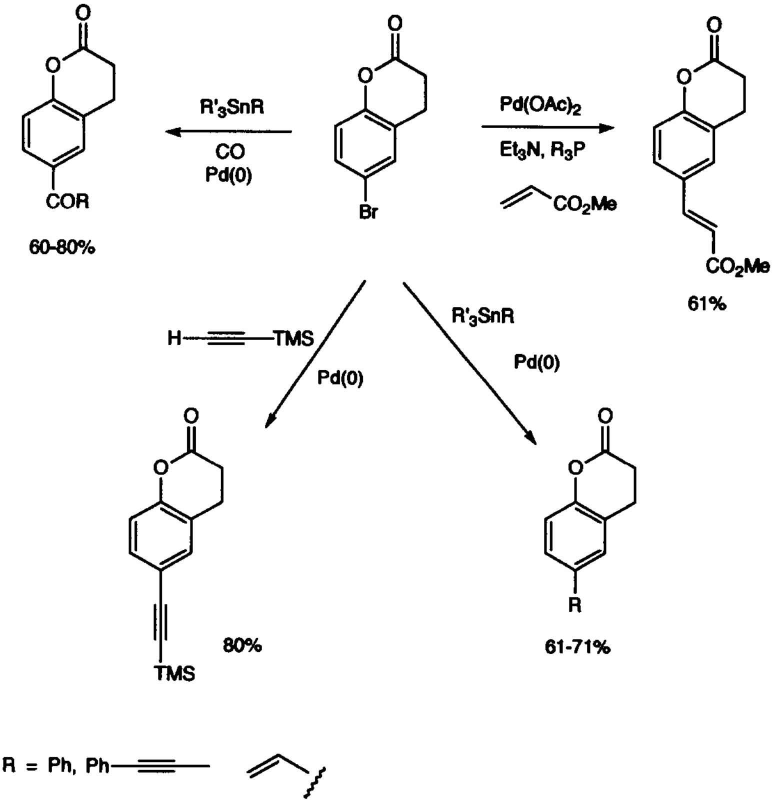
(Equation 103)



(Equation 104)

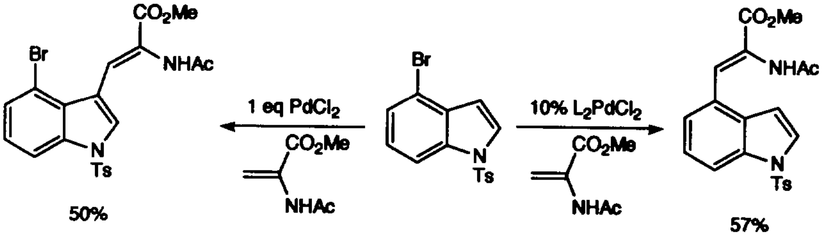


(Equation 105)

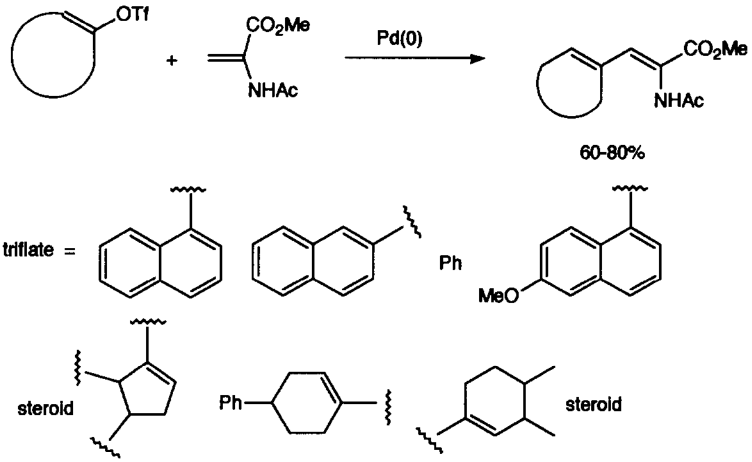


Aryl bromides (equation 106) [126] and triflates (equation 107) [127] cleanly alkylated acetamidoacrylates

(Equation 106)

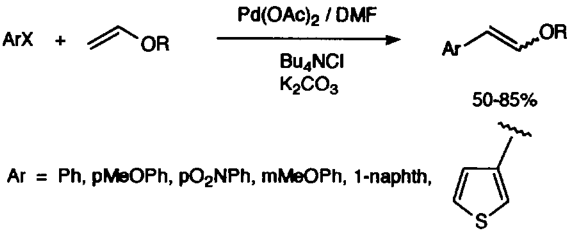


(Equation 107)

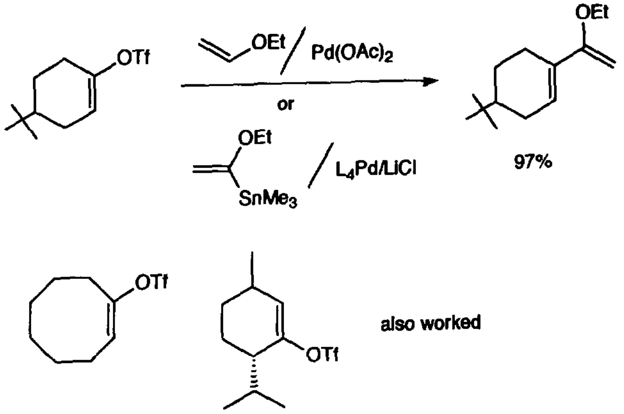


Electron rich olefins also underwent Heck arylation (equation 108) [128], (equation 109) [129] The regioselectivity depended on the substrate and conditions (equation 110) [130] Vinyl acetate underwent a double arylation, indicating that β-acetate elimination rather than β-hydride elimination had occurred (equation 111) [131] Dihydrofuran was also doubly arylated (equation 112) [132]

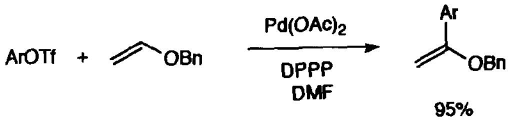
(Equation 108)



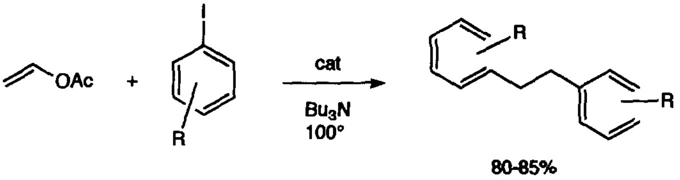
(Equation 109)



(Equation 110)



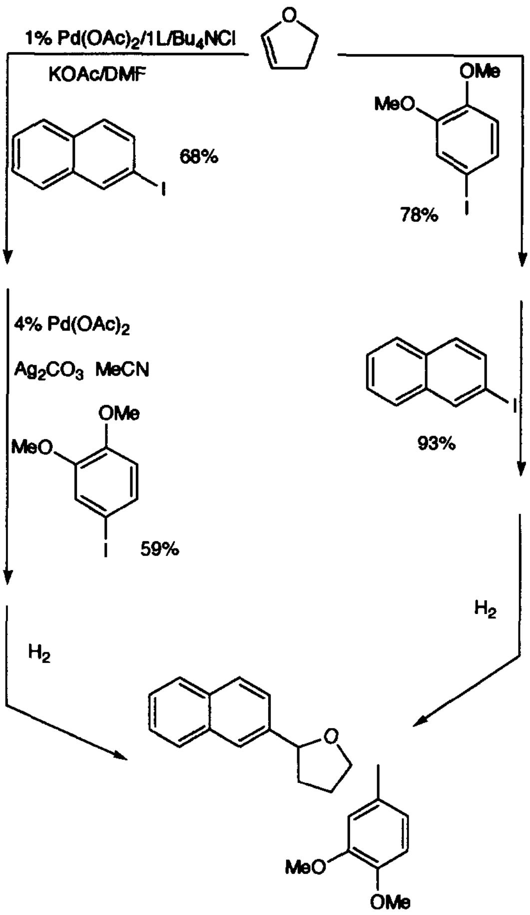
(Equation 111)



Ar = Ph, pMePh, oMePh, oMeOPh, pMeOPh, iPrPh

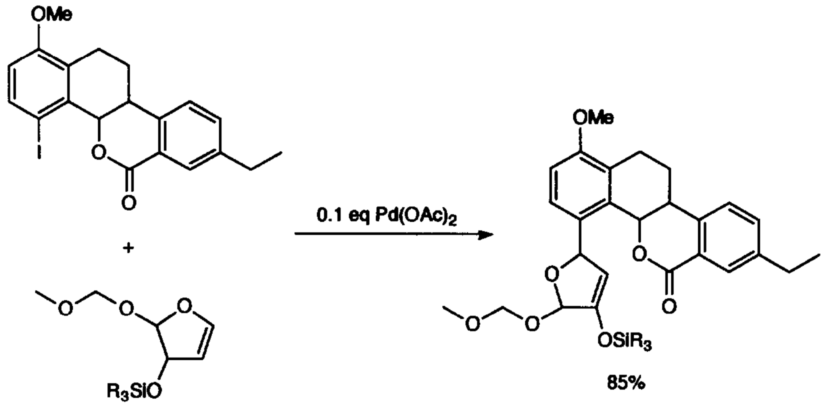
cat = L-$PdCl_2$ on interlamellar montmorillonite

(Equation 112)

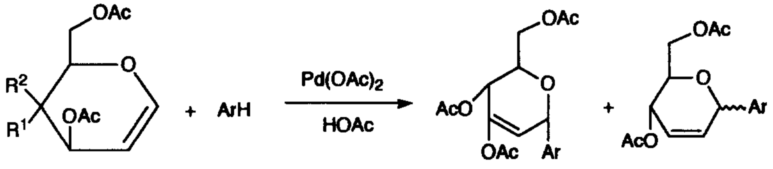


Protected glycals were excellent substrates for Heck arylation (equation 113) [133], (equation 114) [134]. Allyl alcohols (equation 115) [135] olefinic epoxides (equation 116) [136], cyclic enamides (equation 117) [137], and protected allyl amines (equation 118) [138] all were alkylated under Heck conditions, as were vinyl silanes (equation 119) [139], (equation 120) [140]

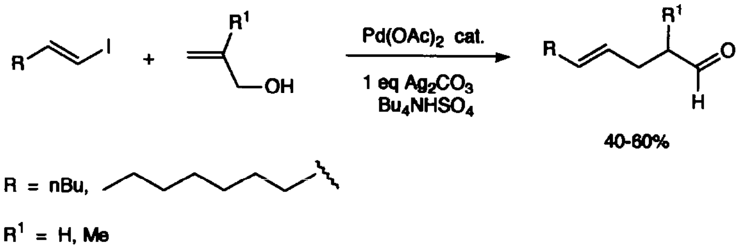
(Equation 113)



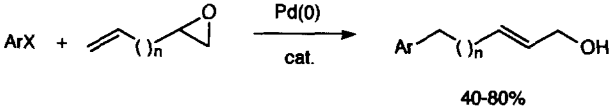
(Equation 114)



(Equation 115)



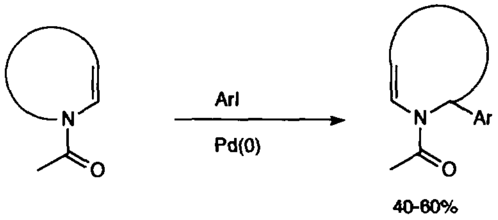
(Equation 116)



Ar = Ph, pMePh, pMeOPh, pMeCOPh, pEtO$_2$CPh

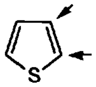
n = 1, 2, 4, 10

(Equation 117)

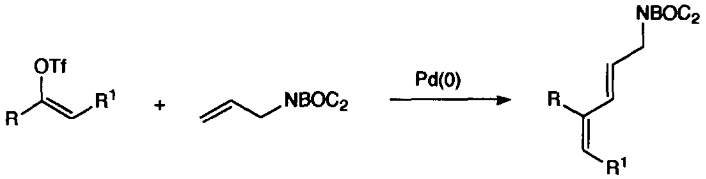


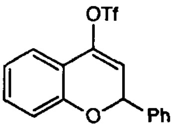
Ar = Ph, 1-naphth, pMeOPh, mMeOPh, oMeOPh, pMePh, pMeO$_2$CPh

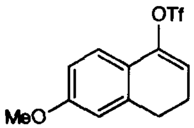
5-30% with oHOPh, pBrPh, pO$_2$NPh,

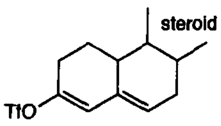


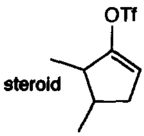
(Equation 118)



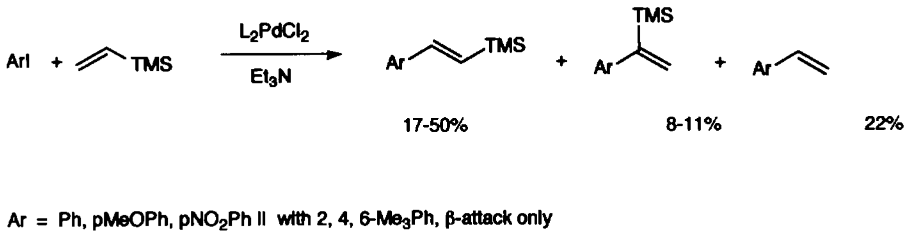




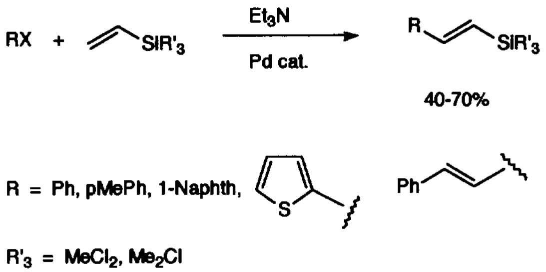




(Equation 119)

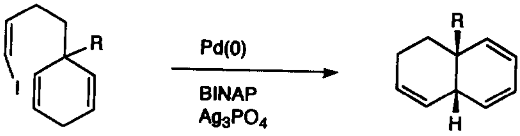


(Equation 120)



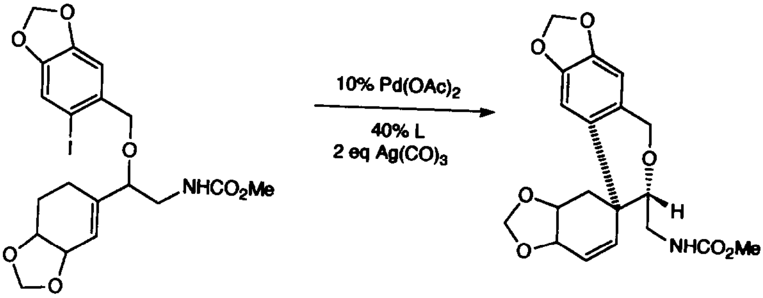
Intramolecular arylpalladation was promoted by ultrasound [141] and was used to synthesize polycyclic systems (equation 121) [142], (equation 122) [143], (equation 123) [144]. An annulation procedure involving Heck arylation followed by alkylation of a π-allylpalladium intermediate has been developed (equation 124) [145] The intermediate σ-alkylpalladium(II) complexes has been intercepted by cyanide (equation 125) [146] With σ-iodoketones, the cyclization occurs by atom transfer not redox (equation 126) [147].

(Equation 121)



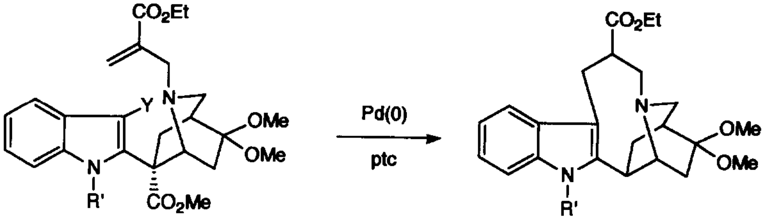
R = CO_2Me, $CH_2OTBDMS$
67%, up to 80% ee

(Equation 122)



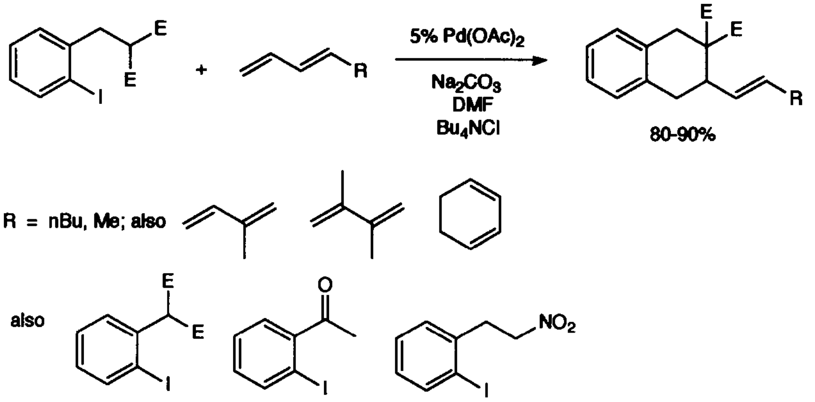
70%

(Equation 123)

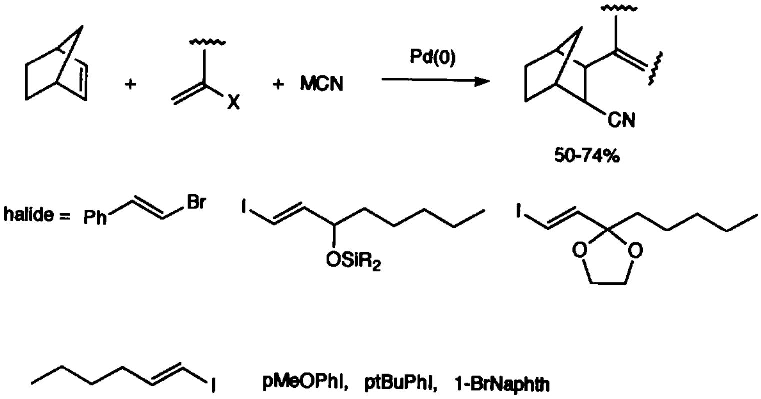


89%

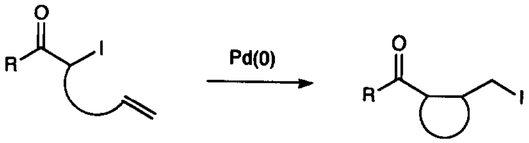
(Equation 124)



(Equation 125)

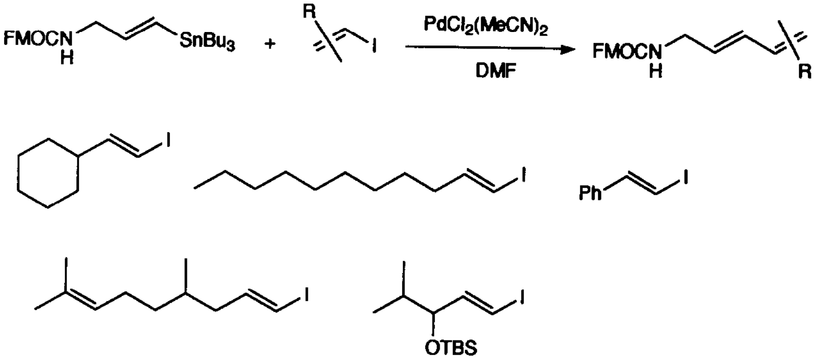


(Equation 126)

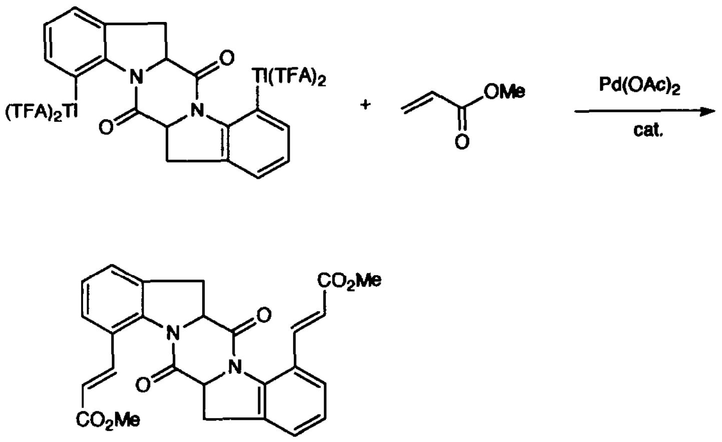


Olefins were also alkylated by palladium-catalyzed transmetallation/ insertion reactions from tin (equation 127) [148], thallium (equation 128) [149], and mercury (equation 129) [150], (equation 130) [151], (equation 131) [152]

(Equation 127)

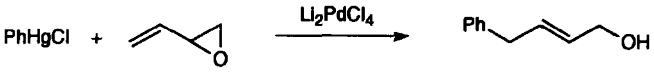


(Equation 128)

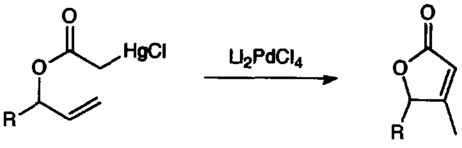


30%

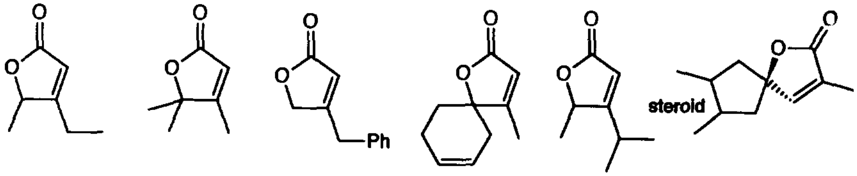
(Equation 129)



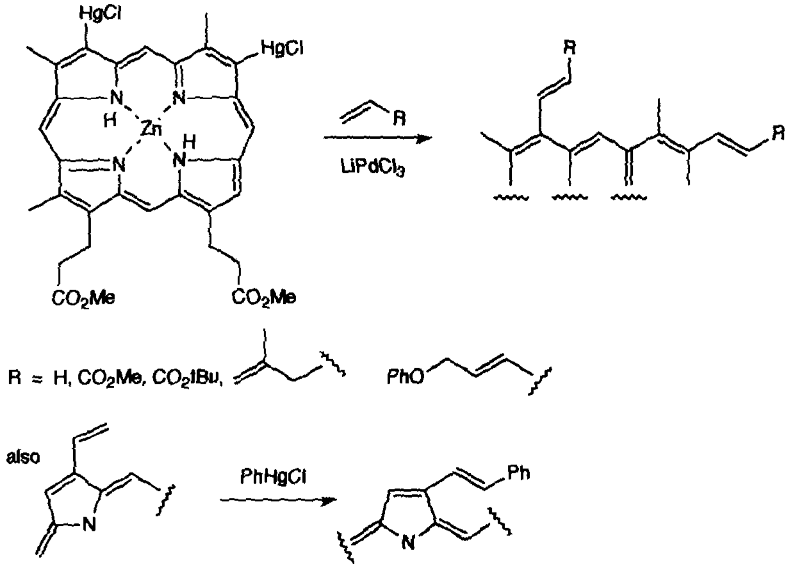
(Equation 130)



50-70%

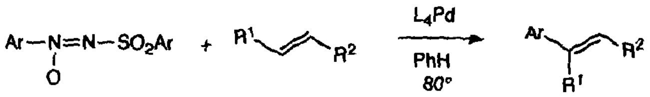


(Equation 131)



Olefins were arylated by arylazoalkyl sulfones (equation 132) [153] [154] [155] and arylsulfonyl chlorides (equation 133) [156] under palladium catalysis Palladium catalyzed hydroarylation and hydrovinylation of olefins was reviewed (36 references) [157].

(Equation 132)

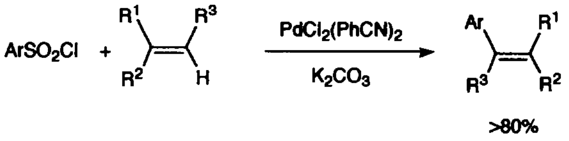


Ar = Ph, pMePh, pClPh

R^1 = H, Me, Ph

R^2 = CO_2Et, CN

(Equation 133)



R^1 = CO_2Bu, CO_2Me, Ph, H

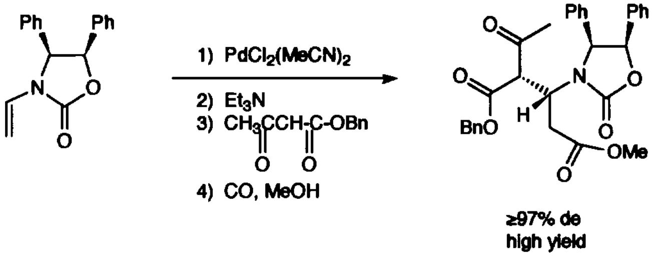
R^2 = H, Me

R^3 = H, CO_2Me

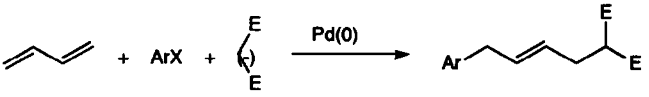
Ar = Ph, pMeOPh, pClPh, pFPh, mClPh, pO_2NPh, 1-naphth, 2-naphth

The palladium(II) assisted alkylation of olefins was used to synthesize a relay to (+)-thienamycin, using optically active ene carbamates as the olefin (equation 134) [158]. Olefins were dialkylated by combined insertion, nucleophile attack chemistry (equation 135) [159], (equation 136) [160]

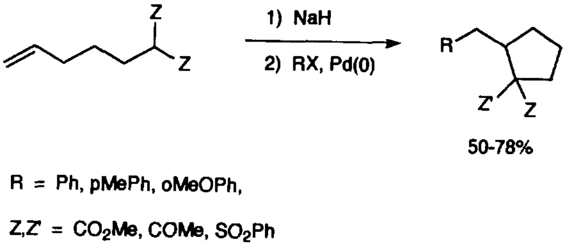
(Equation 134)



(Equation 135)

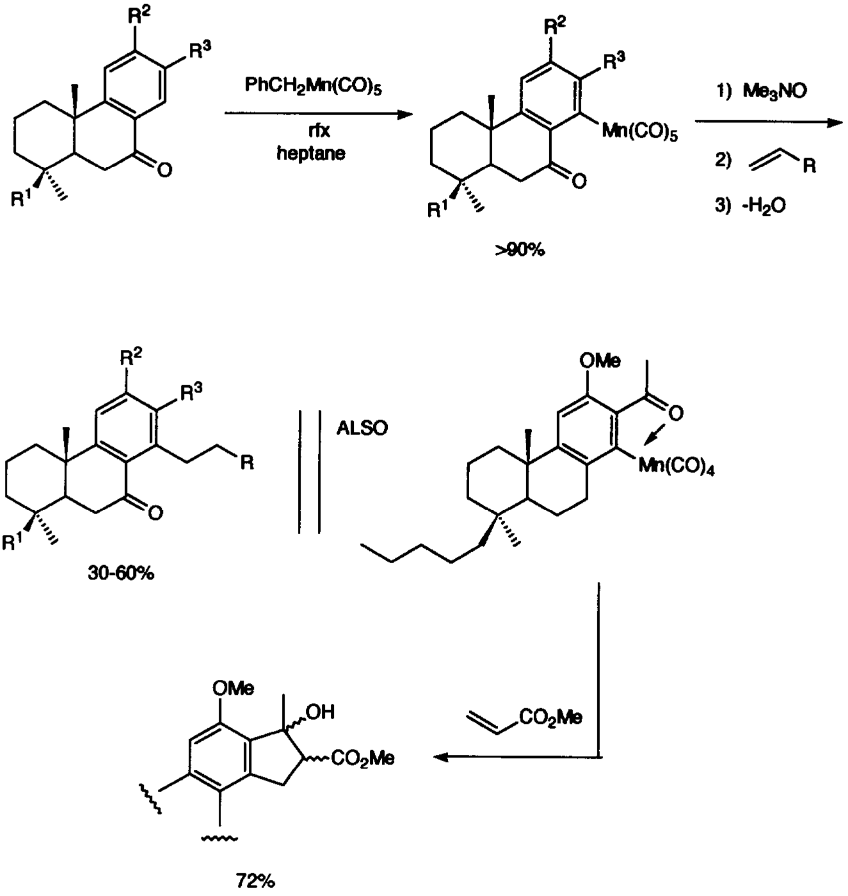


(Equation 136)



The C-14 position of diterpenoids was functionalized by cyclomanganation/olefin insertion (equation 137) [161] Manganese(III) acetate cyclized olefinic β-dicarbonyl compounds (equation 138) [162] Iron carbonyls [163] and ruthenium complexes (equation 139) [164], (equation 140) [165] catalyzed the addition of halocarbons to olefins

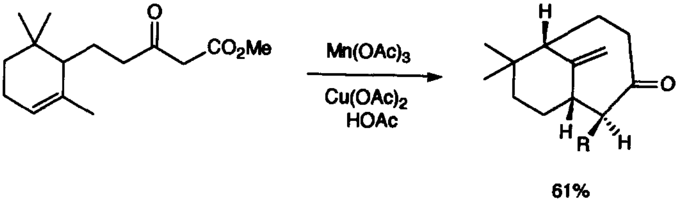
(Equation 137)



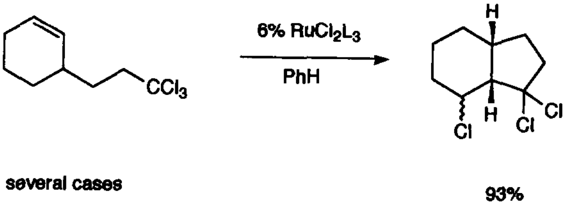
R^1 = CO_2Me, CH_2OMe, R^2 = H, OMe; R^3 = H, CO_2Me

R = CO_2Me, CN, CHO, CH_2OAc

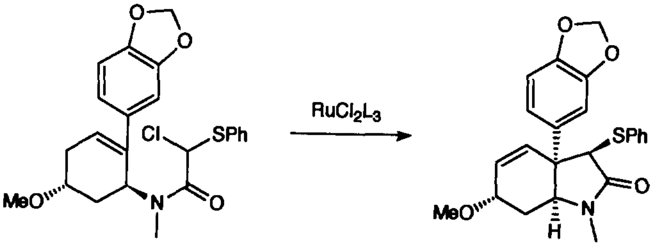
(Equation 138)



(Equation 139)

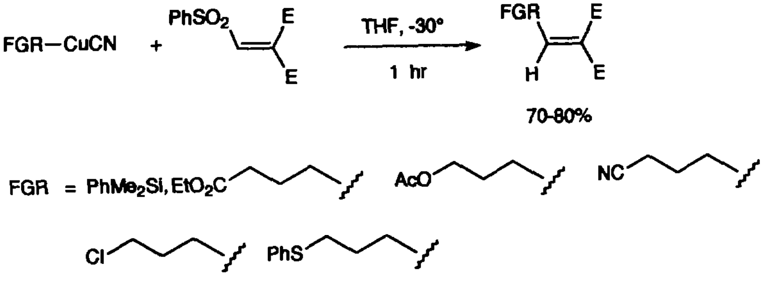


(Equation 140)

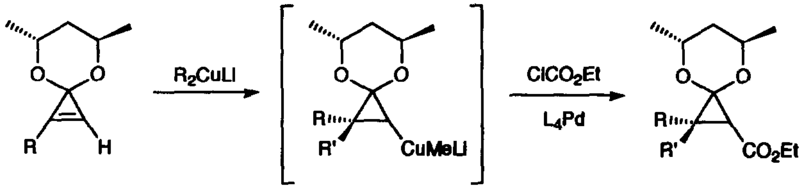


Copper powder catalyzed the addition of iododifluoroacetates to alkenes [166] and the addition of perfluoroalkyl iodides to bromo heterocyclic compounds [167]. Vinyl sulfones were alkylated by organocopper complexes (equation 141) [168]. Cyclopropenone ketals were alkylated by organocuprates (equation 142) [169] as were cyclic olefinic ethers (equation 143) [170], (equation 144) [171]

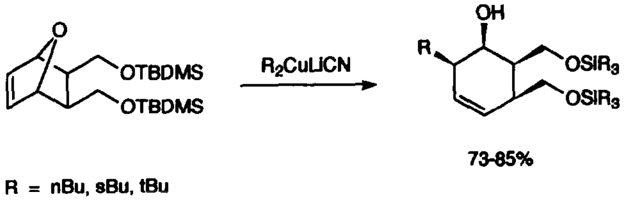
(Equation 141)



(Equation 142)

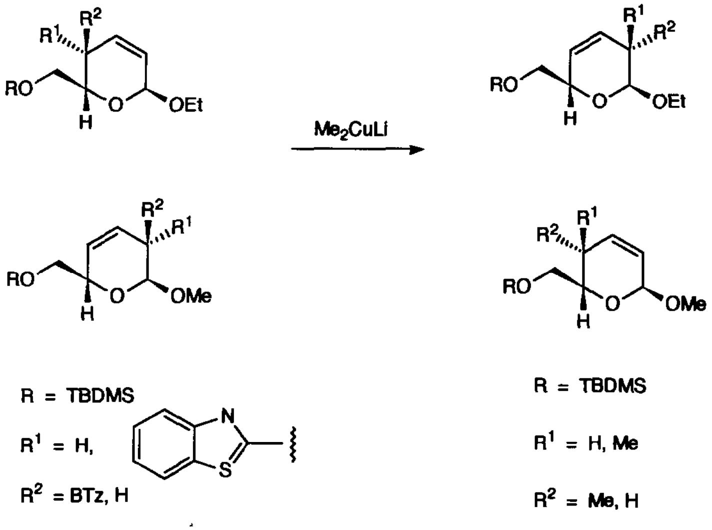


(Equation 143)



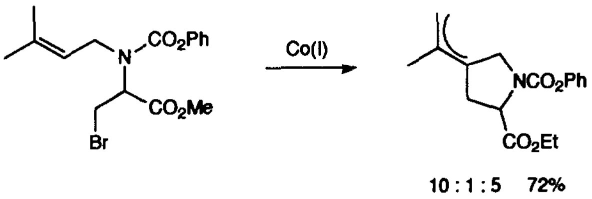
; p 643

(Equation 144)

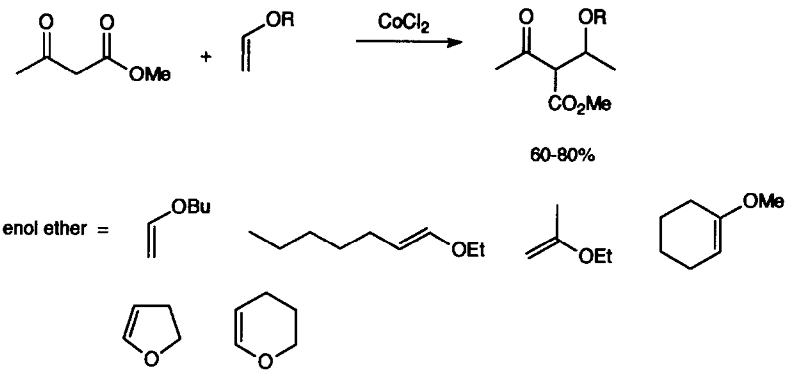


Manganese(III) difunctionalization of alkenes was the topic of a dissertation [172] Cobalt(I) complexes catalyzed the alkylation of olefins by halides (equation 145) [173] while cobalt(II) catalyzed the alkylation of enol ethers by stabilized carbanions (equation 146) [174].

(Equation 145)

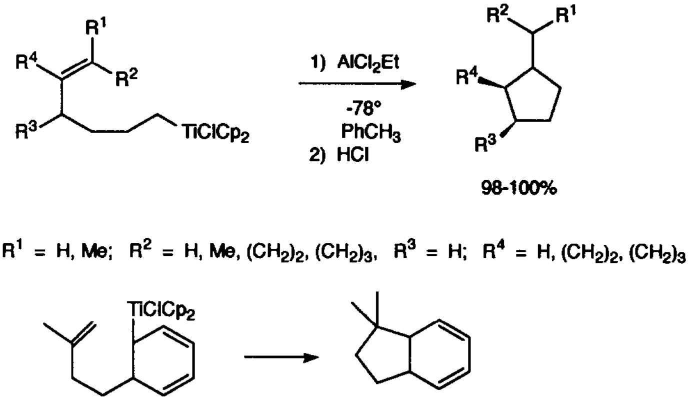


(Equation 146)



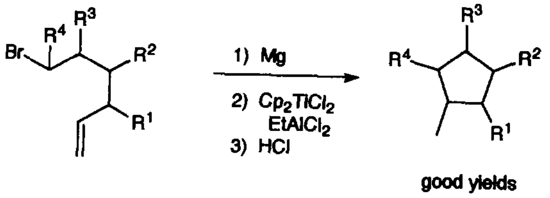
Zirconium catalyzed coupling of olefins with heteroaromatics was the subject of a lecture (15 references) [175] Olefins were alkylated by alkyltitanium(IV) species in the presence of aluminum alkyls (equation 147) [176], (equation 148) [177]. Zirconocene alkylated olefins with nitriles (equation 149) [178].

(Equation 147)

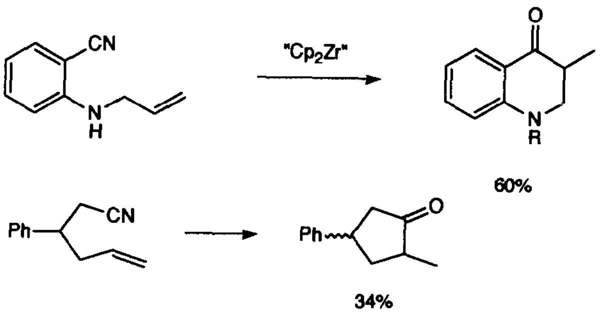


R^1 = H, Me; R^2 = H, Me, (CH$_2$)$_2$, (CH$_2$)$_3$, R^3 = H; R^4 = H, (CH$_2$)$_2$, (CH$_2$)$_3$

(Equation 148)

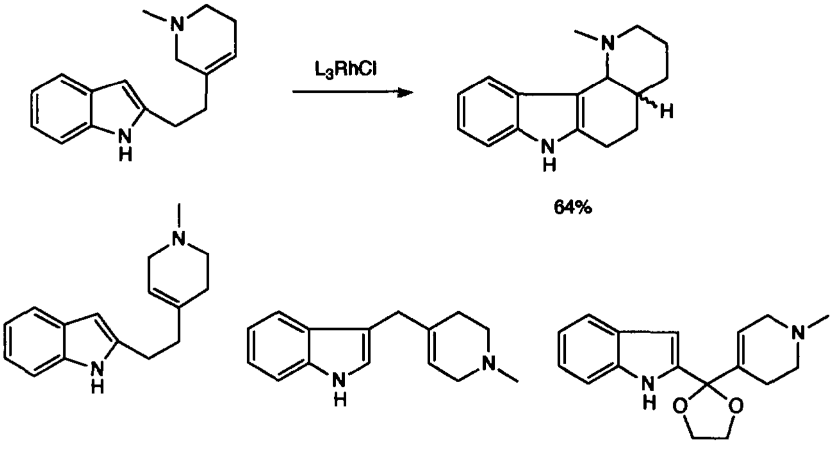


(Equation 149)



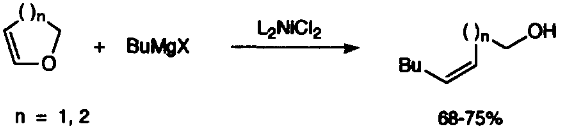
Rhodium(I) complexes catalyzed the C-alkylation of phenols with myrcene [179] and the alkylation of olefins by indoles (equation 150) [180]. Cyclic enol ethers were alkylatively ring opened in nickel catalyzed Grignard reactions (equation 151) [181] Catalytic iron(0)-mediated carbocyclizations of triene esters and triene-ethers was the subject of a disseration [182].

(Equation 150)



also cyclize

(Equation 151)

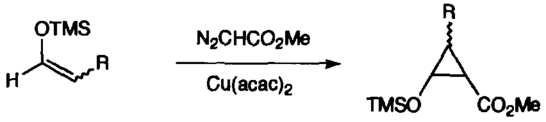


4 Decomposition of Diazoalkanes and Other Cyclopropanations

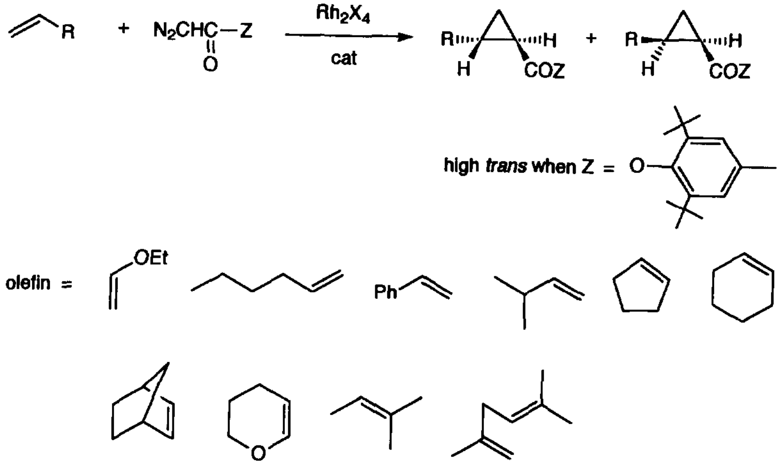
Metal-catalyzed decomposition of diazoalkanes continues to be popular for cyclopropanation of olefins. A variety of transition metal complexes was screened for reactivity in the cyclopropanation of norbornene by diazomethane, with Pd(acac)$_2$ being the best [183]. Palladium chloride catalyzed the cyclopropanation of 1-alkoxy butadienes by diazomethane [184], as well as allyl alcohol and allyl amine [185][186]. Rhodium(II) salts catalyzed the cyclopropanation of olefins by σ-nitro diazo acetate [187]. Silylenol ethers were cyclopropanated by diazoacetate in the presence of copper(II) acetylacetonate (equation 152) [188]. The *cis/trans* ratio in the cyclopropanation of olefins by σ-diazoesters using Rh(II) catalysts could be affected by the rhodium salt and by the alcohol group on the ester (equation

153) [189] as well as by secondary interactions between the carbenoid complex and the substrate (equation 154) [190]. Furan (equation 155) [191] and propargyl ethers (equation 156) [192] gave mixtures of products when treated with diazo compounds and rhodium(II) catalysts

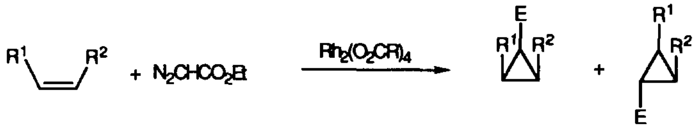
(Equation 152)



(Equation 153)

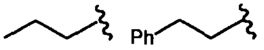


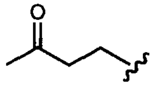
(Equation 154)



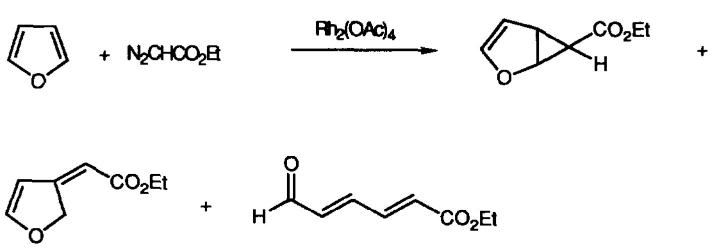
R = H, CH_3, nC_{17}, Ph, $PhCH_2$, Ph_3CCH_2, oHOPh, oPhCOPh, oCF_3Ph, mCF_3Ph,

2,4,6-Me_3Ph, CF_3, PhCO,

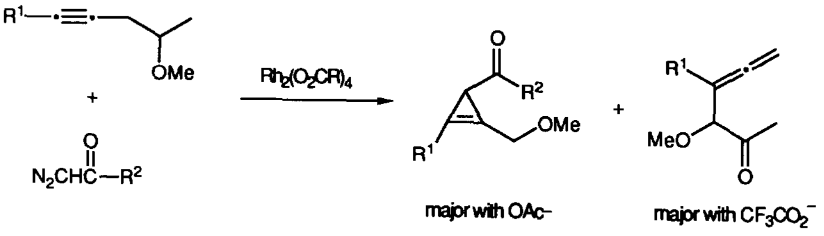




(Equation 155)

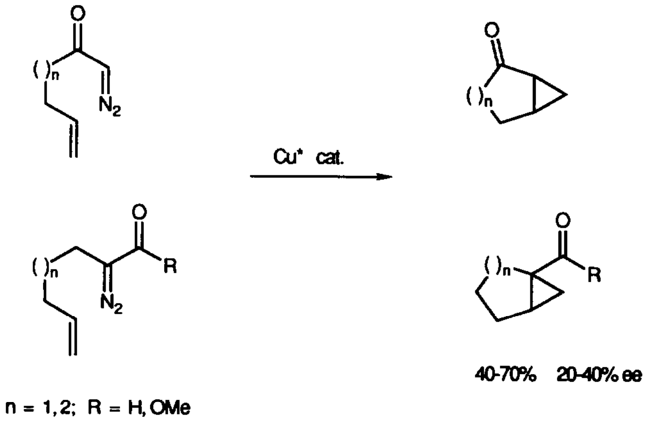


(Equation 156)



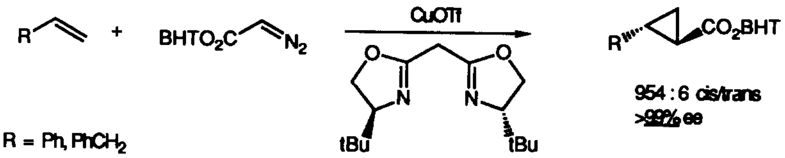
Intramolecular cyclopropanations with optically active catalysts have also been developed (equation 157) [193] A mechanistic study of the rhodium(II) carboxylate catalyzed reactions of diazo esters indicated that an equilibrium between free carbene and a metal-carbene complex existed [194]

(Equation 157)

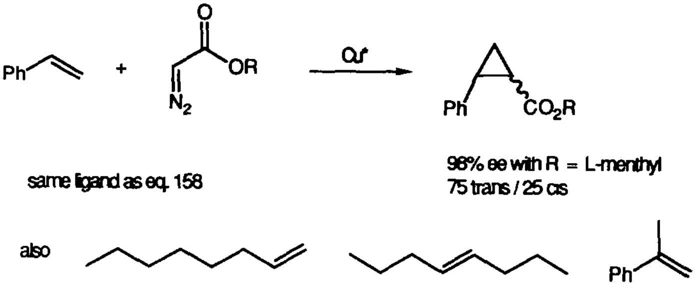


Several other approaches to asymmetric induction in this process have been studied. They are summarized in equation 158 [195], equation 159 [196], equation 160 [197], and equation 161 [198]

(Equation 158)



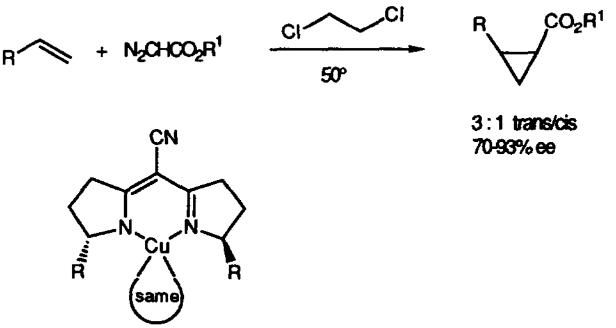
(Equation 159)



same ligand as eq. 158

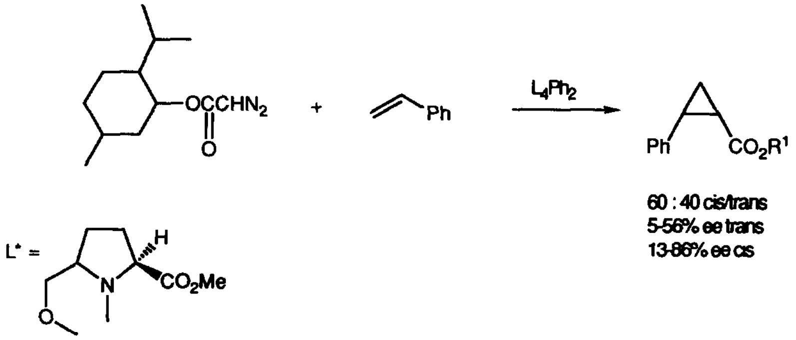
98% ee with R = L-menthyl
75 trans / 25 cis

(Equation 160)



3 : 1 trans/cis
70-93% ee

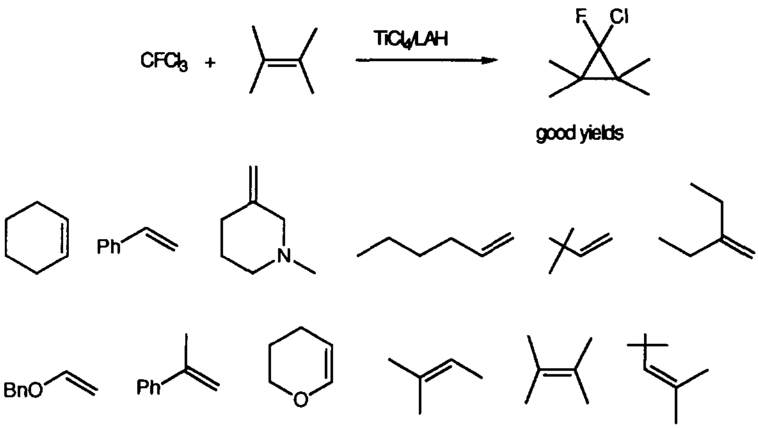
(Equation 161)



60 : 40 cis/trans
5-56% ee trans
13-86% ee cis

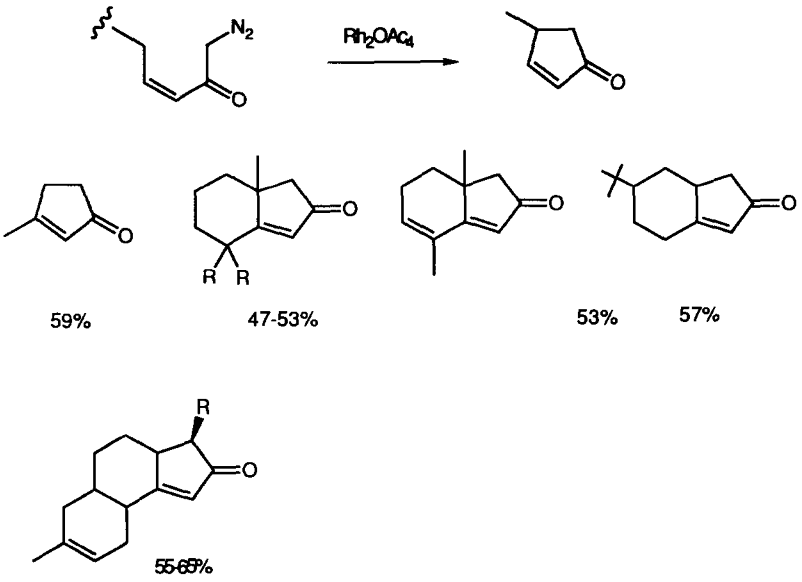
Low valent titanium cyclopropanated olefins with $CFCl_3$ (equation 162) [199]

(Equation 162)

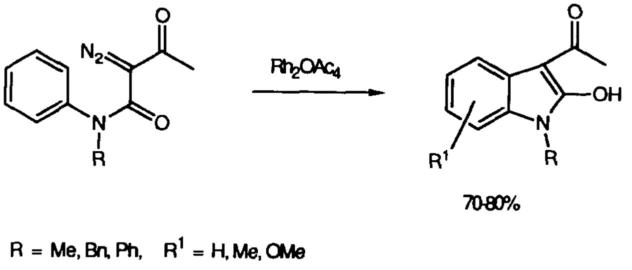


Transition metals catalyzed a variety of other reactions of diazo species, in addition to cyclopropanations The rhodium(II) catalyzed decomposition of optically active α-amino diazoketones to give optically active products resulting from C-H insertion, aromatic cycloaddition, cyclopropanation, α,α'-substitutions and β-diketone formation have been studied [200] Rhodium(II) catalyzed decomposition resulting in C-H insertions have been used to produce cyclic enones (equation 163) [201], hydroxyindoles (equation 164) [202], cyclic β-ketoesters (equation 165) [203], alkenes (equation 166) [204], furanones (equation 167) [205], lactones (equation 168) [206], lactams (equation 169) [207], and other cyclic systems (equation 170) [208][209] Unsaturated diazoketones cycloadded to cyclopentadiene (equation 171) [210]

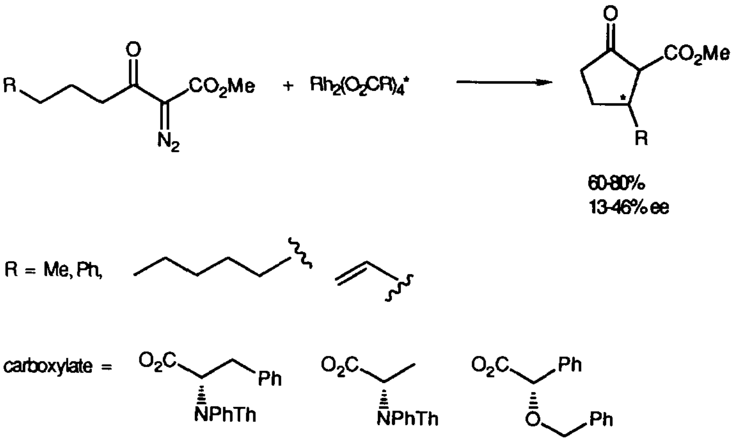
(Equation 163)



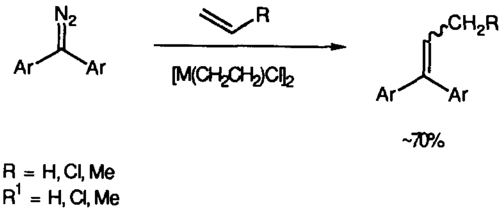
(Equation 164)



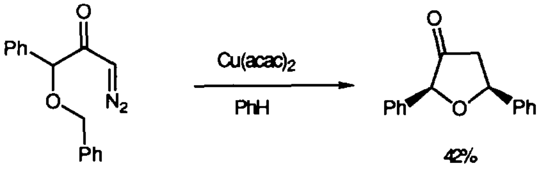
(Equation 165)



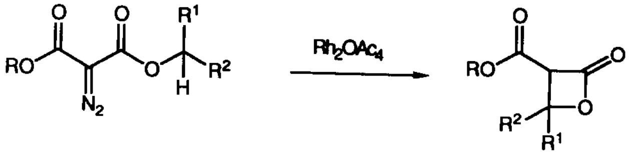
(Equation 166)



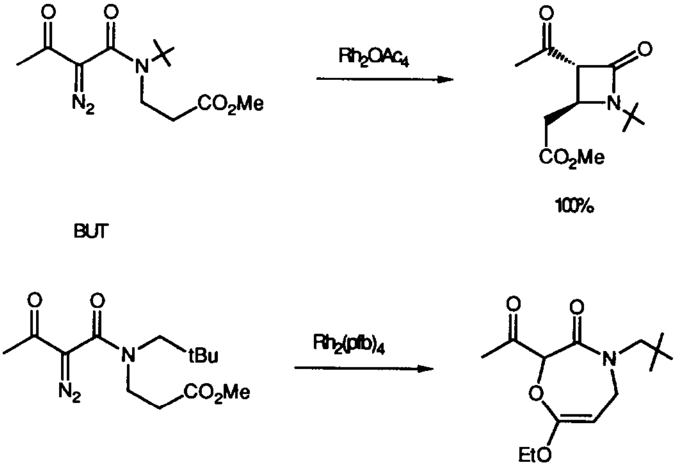
(Equation 167)



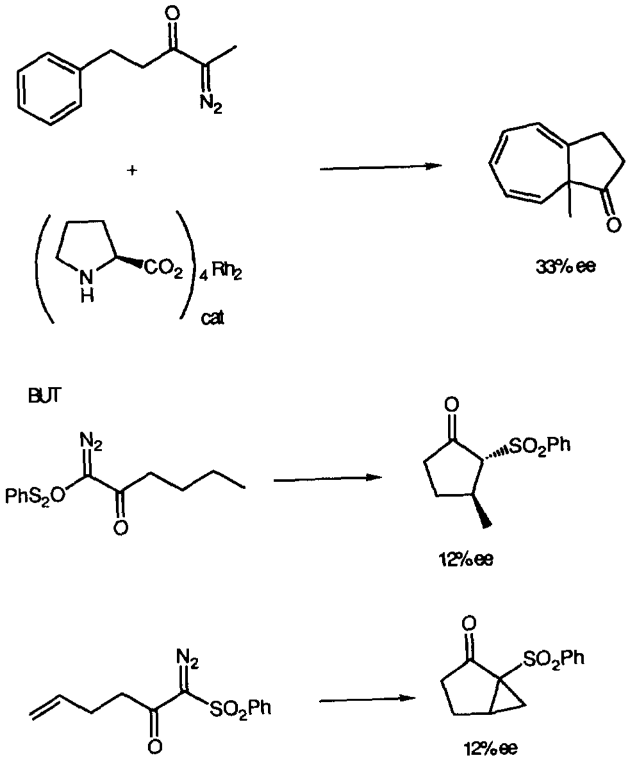
(Equation 168)



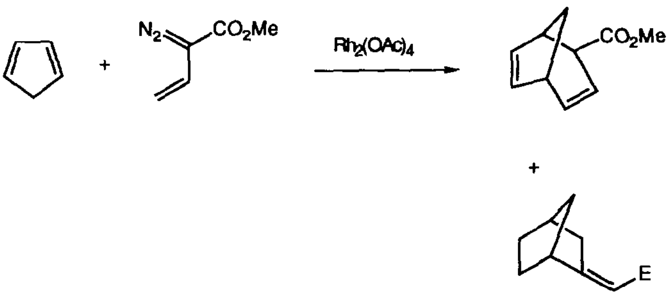
(Equation 169)



(Equation 170)

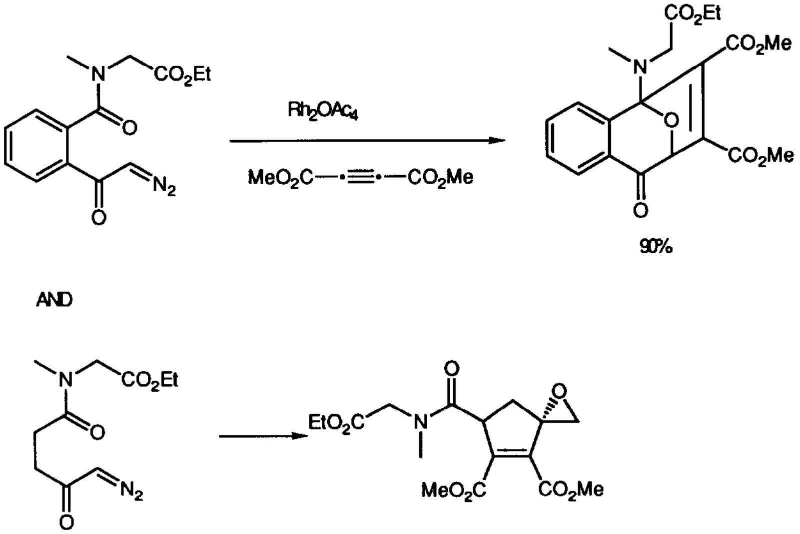


(Equation 171)

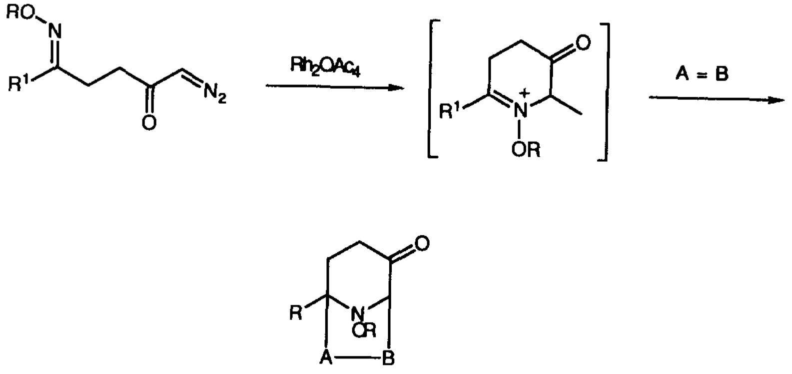


Ylids can be formed by trapping the carbene produced by catalyzed diazo decomposition with adjacent heteroatoms, leading to a variety of products (equation 172) [211], (equation 173) [212], (equation 174) [213]. Alkynes can trap the carbene to give cyclopropenes which cleave to give allyl carbenes and ultimately produce polycyclic compounds (equation 175) [214], (equation 176) [215]. ϖ-Hydroxy-α-diazoesters rearrange to β-dicarbonyl compounds (equation 177) [216]

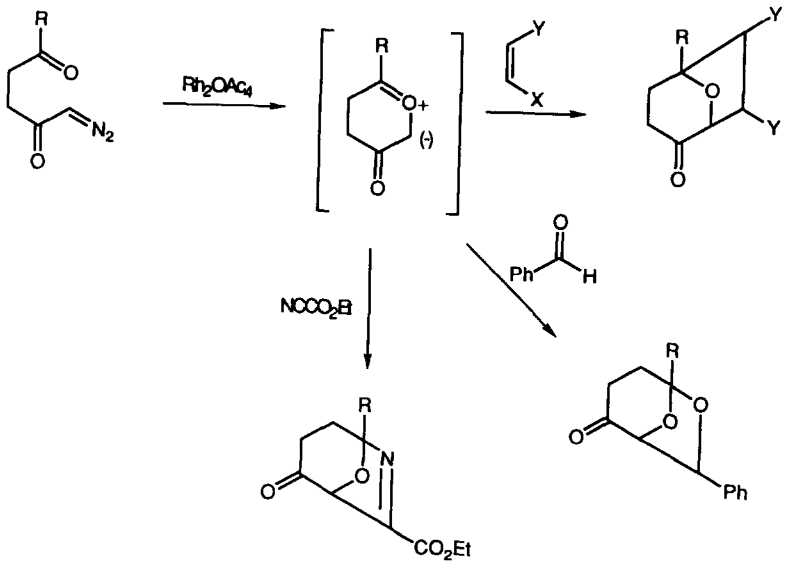
(Equation 172)



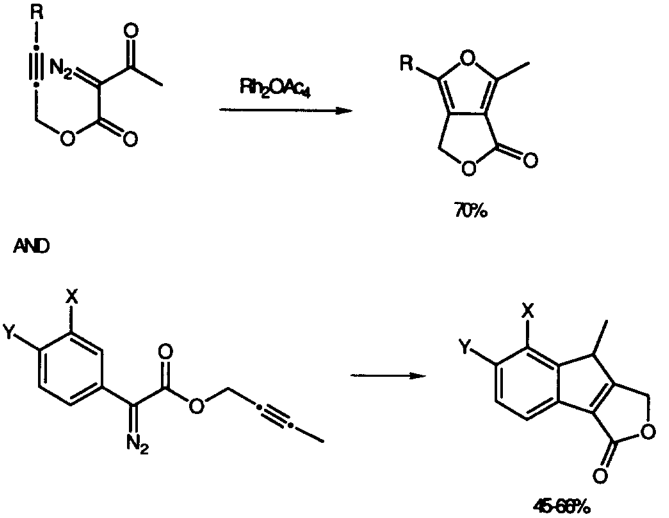
(Equation 173)



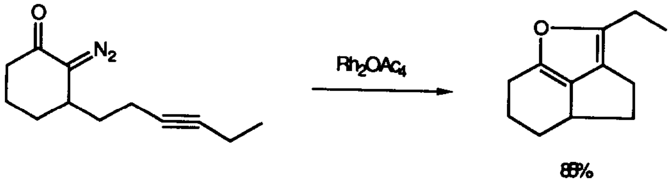
(Equation 174)



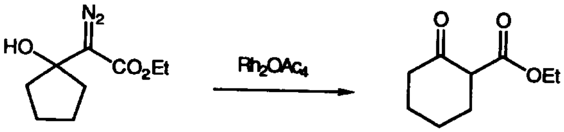
(Equation 175)



(Equation 176)

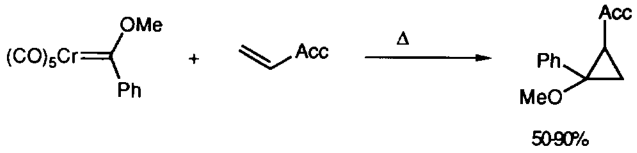


(Equation 177)



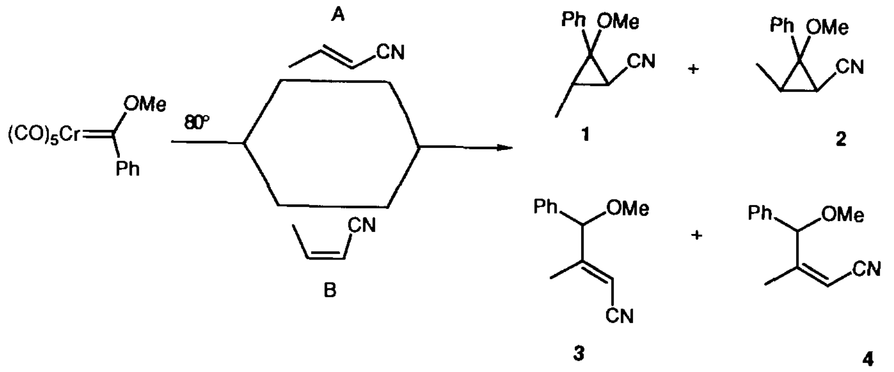
Cyclopropanes are also made by the reaction of olefins with Group(VI) carbene complexes The carbene route to donor-acceptor substituted cyclopropanes has been reviewed [217], and the full details published (equation 178) [218] Depending on the geometry of the olefin and on the solvent, C-H insertion is sometimes observed (equation 179) [219]. O-Acyl carbene complexes cyclopropanated olefins under very mild conditions (equation 180) [220] as did molybdenum carbenes (equation 181) [221] Intramolecular versions were also efficient (equation 182) [222]. Tungsten benzylidenes cyclopropanated allenes (equation 183) [223] Thermolysis of iminocarbene complexes led to intramolecular cyclopropanations (equation 184) [224].

(Equation 178)



Acc = CO_2Me, $CONMe_2$, CN, $P(O)(OMe)_2$, SO_2Ph

(Equation 179)



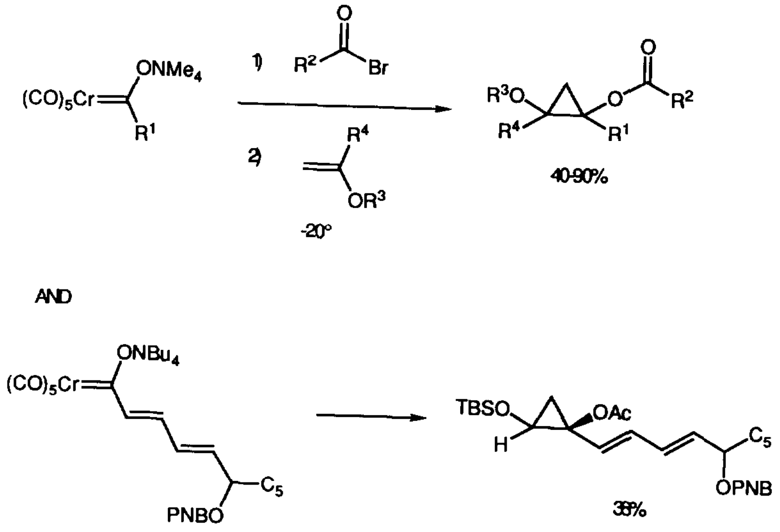
$$A \xrightarrow{C_6H_{12}} 92\%\ \mathbf{3}$$

$$A \xrightarrow{CH_3CN} 77\%\ \mathbf{1}$$

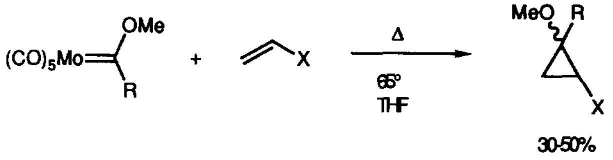
$$B \xrightarrow{C_6H_{12}} 60\%\ \mathbf{4}$$

$$B \xrightarrow{CH_3CN} 65\%\ \mathbf{2}$$

(Equation 180)

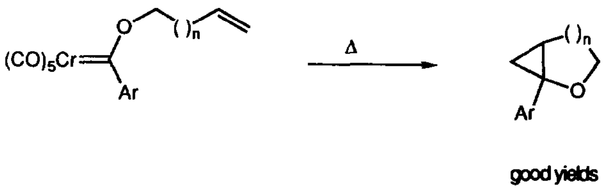


(Equation 181)

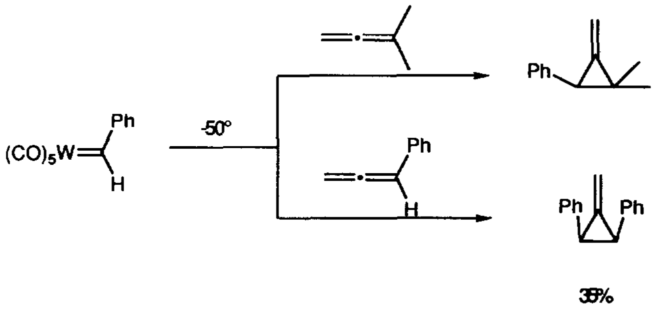


X = CN, CO_2Me, CO_2Et, $P(O)(OMe)_2$

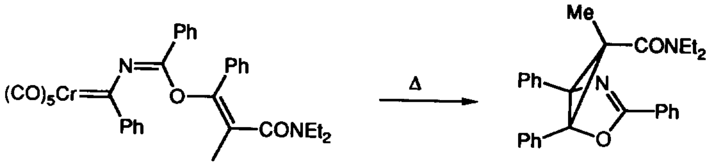
(Equation 182)



(Equation 183)



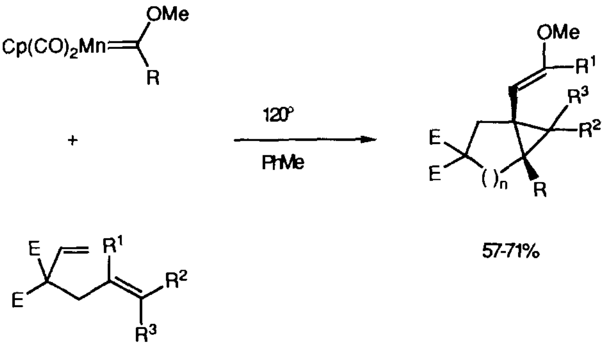
(Equation 184)



Carbene complexes having remote unsaturation underwent combined metathesis/cyclopropanation (equation 185) [225], (equation 186) [226]. Similar chemistry was observed in metal catalyzed decomposition of appropriate diazo compounds (equation 187) [227].

(Equation 185)

(Equation 186)

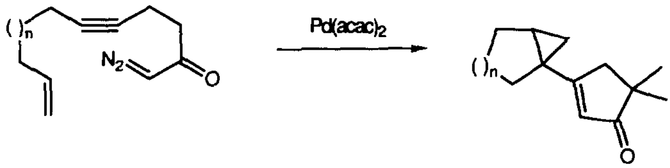


R = Me, Ph

R^1 = H, R^2 = H, Me, R^3 = H, Me, R^4 = Me, Ph

n = 1, 2

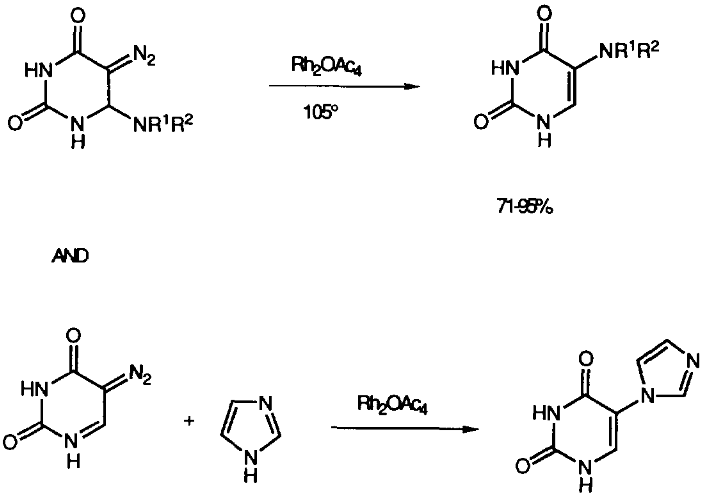
(Equation 187)



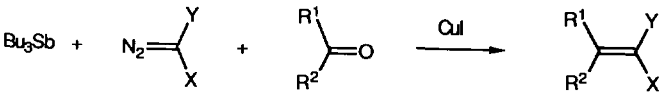
n = 1, 2

Rhodium(II) acetate catalyzed an amine rearrangement (equation 188) [228] Ketones were olefinated by diazoesters in the presence of copper catalysts and tributyl stibane (equation 189) [229]

(Equation 188)



(Equation 189)



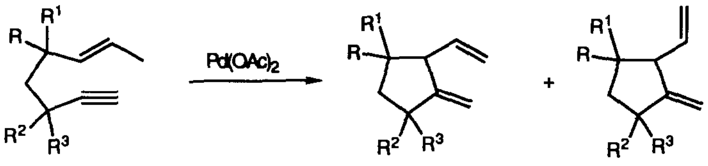
Y = CO_2Me, H, COMe

X = CO_2Me, CO_2Et, COMe

5 Cycloaddition Reactions

Palladium-catalyzed cyclization of enynes has been reviewed (69 references) [230]. This process is very useful for the synthesis of cyclic compounds from simple starting materials (equation 190) [231], (equation 191) [232], (equation 192) [233] Enynes were cyclized with incorporation of carbon monoxide using titanium chemistry (equation 193) [234]

(Equation 190)

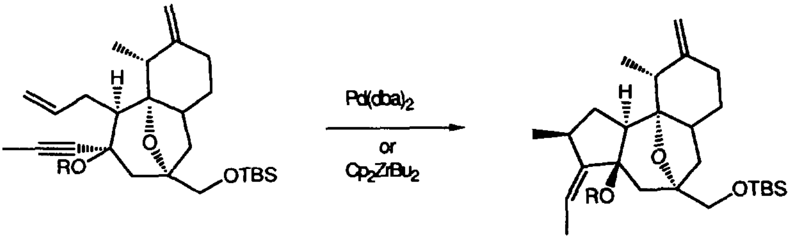


40-80%

R = PMBO, H, Me; R^1 = H, TBDMSO; R^2 = H, Me; R^3 = H, PMBO, TBDMSO

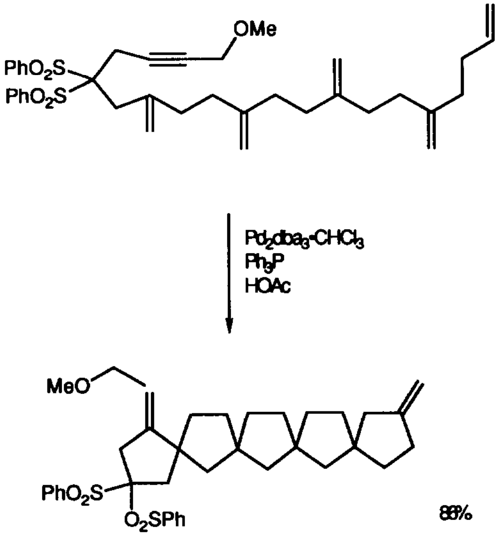
ALSO

(Equation 191)

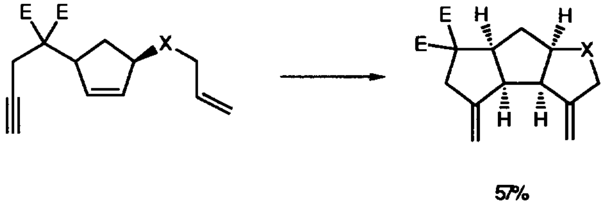


58% (Pd)
69% (ZR)

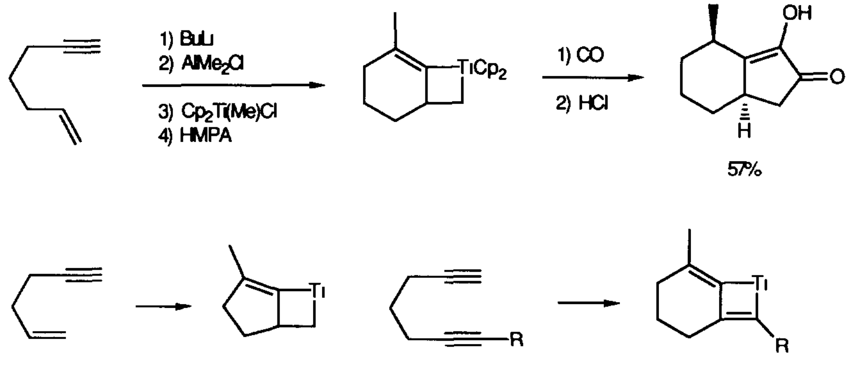
(Equation 192)



AND

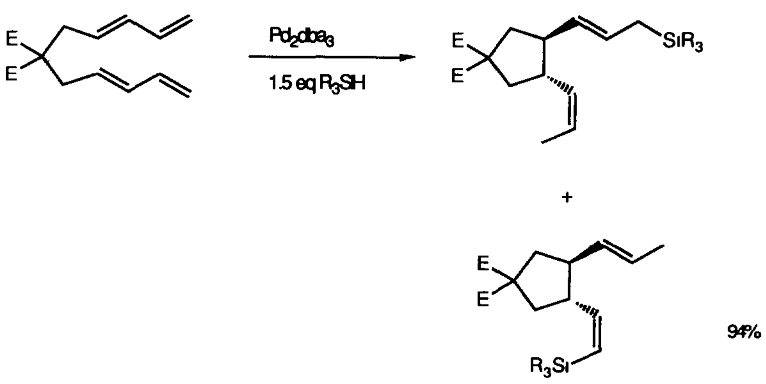


(Equation 193)

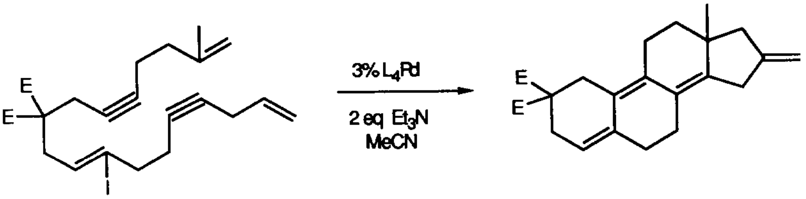


Palladium catalyzed polyene cycloadditions were also initiated by hydrosilylation (equation 194) [235] and by oxidative addition/insertion (equation 195) [236] Regio- and stereocontrolled catalytic Pd and Ni "ene-type" cyclizations have been reviewed (equation 196) [237] Cyclization was also affected by palladium catalyzed nucleophilic attack on dienes (equation 197) [238].

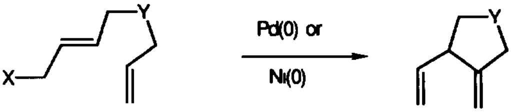
(Equation 194)



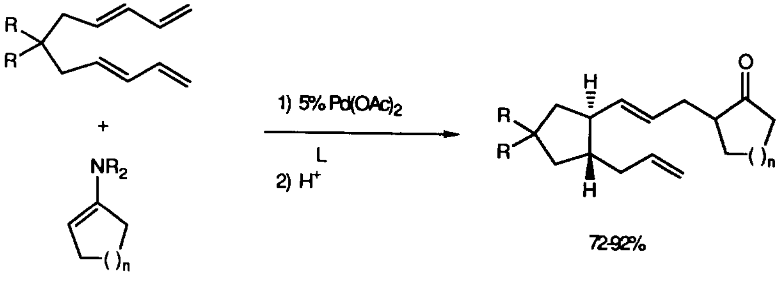
(Equation 195)



(Equation 196)



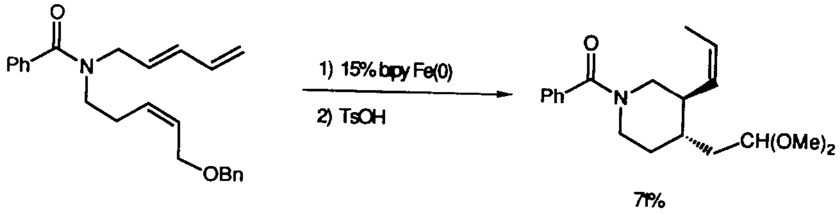
(Equation 197)



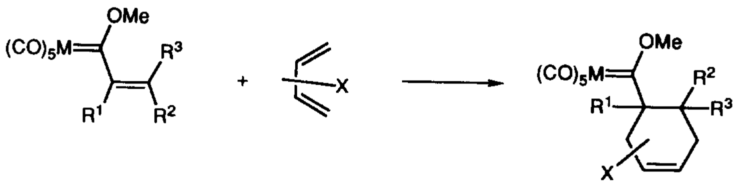
R = CO_2Et, SO_2Ph, CN

Iron(0) complexes cyclized ene-dienes (equation 198) [239] Unsaturated chromium carbene complexes underwent facile [4+2] cycloaddition reactions (equation 199) [240], (equation 200) [241].

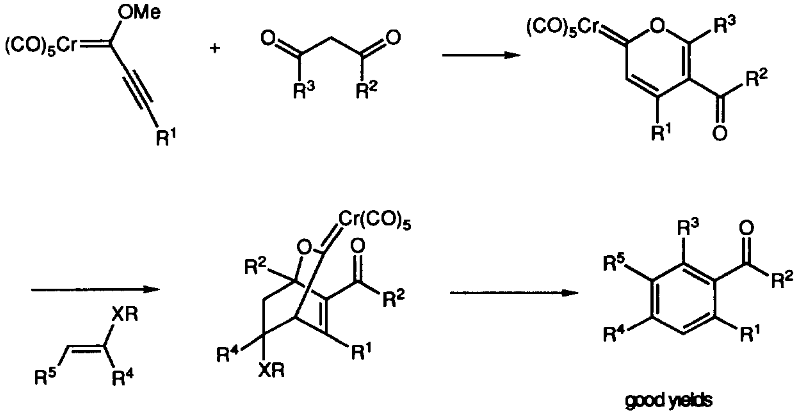
(Equation 198)



(Equation 199)

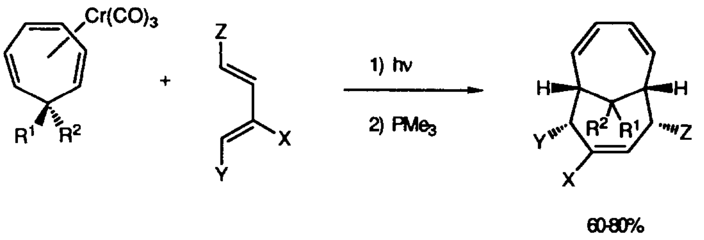


(Equation 200)



Metal cycloheptatriene complexes underwent a variety of cycloaddition reactions (equation 201) [242], (equation 202) [243], (equation 203) [244]. Cobalt complexes catalyzed the cycloaddition of alkynes to nonbornadiene (equation 204) [245]

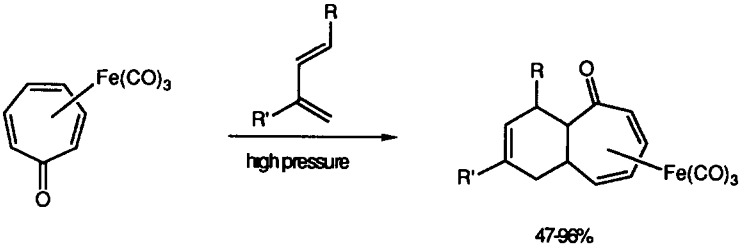
(Equation 201)



R^1 = H, OMe, Me, R^2 = H, OMe; X = H, Me, OTMS

Y = H, OAc, CO_2Me, Z = OMe, OAc, CO_2Me

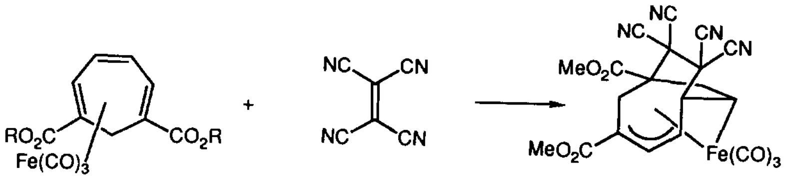
(Equation 202)



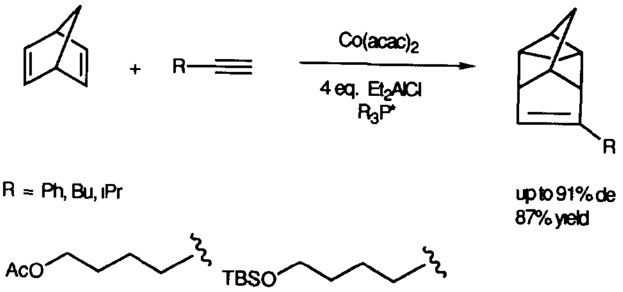
R = OTMS, OAc, H

R' = H, OTMS

(Equation 203)

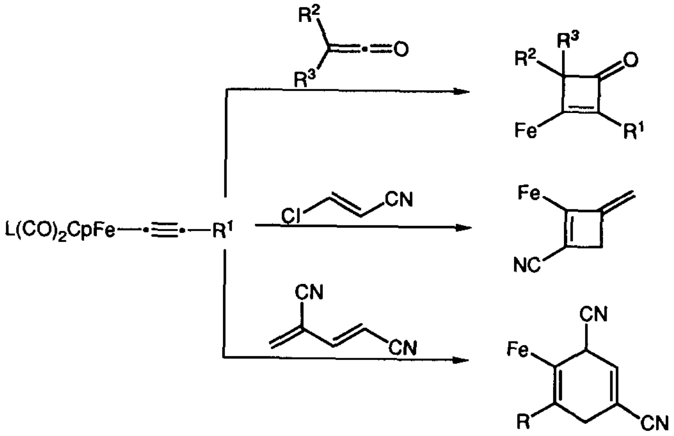


(Equation 204)

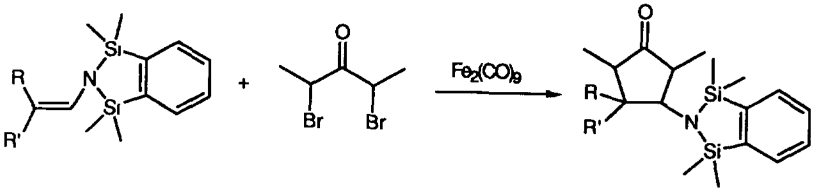


Iron acetylide complexes underwent a variety of cycloaddition reactions (equation 205) [246]. Oxallyliron complexes cycloadded to N-silyl enamines (equation 206) [247] Metal-coordinated thio and selenoketones underwent cycloaddition to cyclopentadiene (equation 207) [248]. Benzocyclobutane cobalt complexes underwent thermal cycloaddition with olefins (equation 208) [249]

(Equation 205)

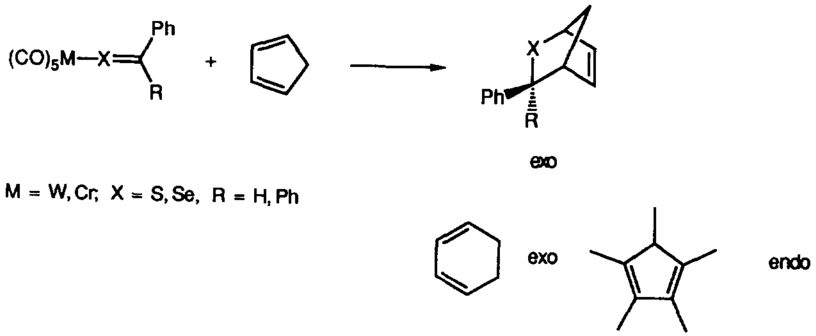


(Equation 206)

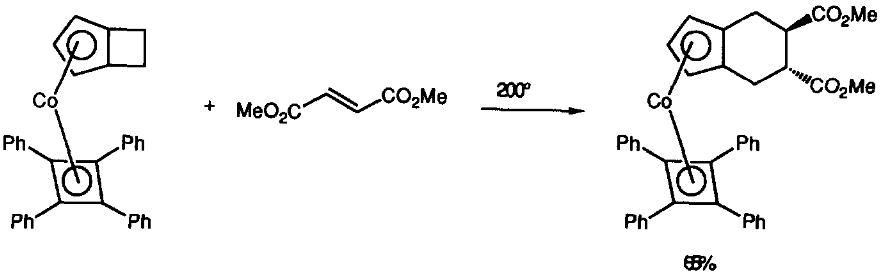


15-20%

(Equation 207)



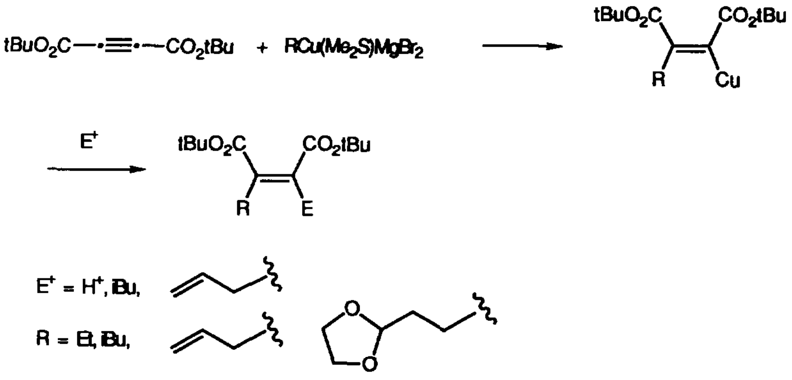
(Equation 208)



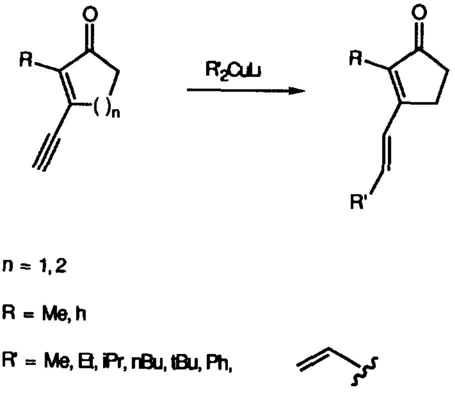
6. Alkylation of Alkynes

Cuprates added to alkynes to give vinylcopper complexes which could be further elaborated (equation 209) [250], (equation 210) [251] Trimethyl stannyl cuprates transferred the tin group to substrates (equation 211) [252].

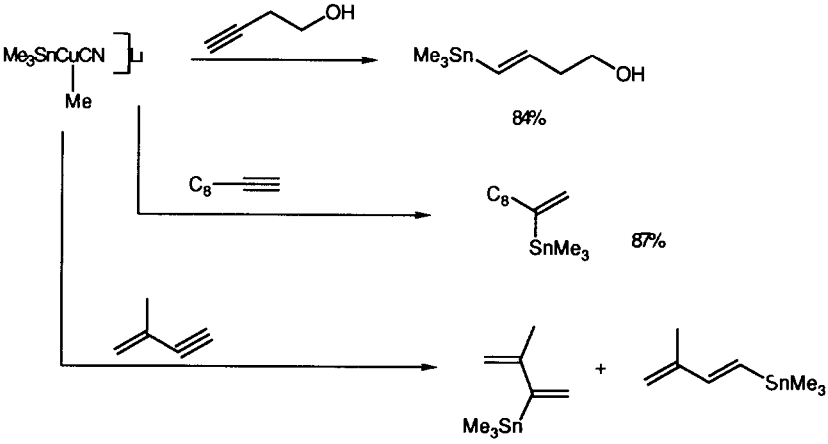
(Equation 209)



(Equation 210)

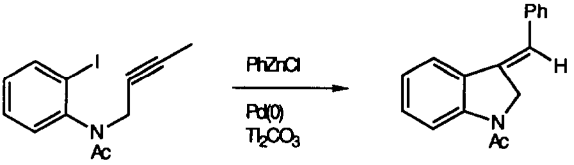


(Equation 211)

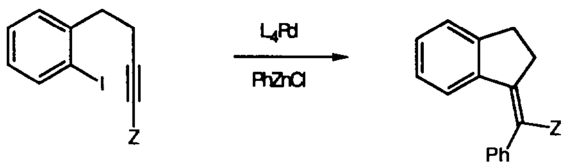


Palladium(0) complexes catalyzed the alkylative cyclization of alkynes with aryl halides (equation 212) [253], (equation 213) [254], (equation 214) [255], (equation 215) [256], (equation 216) [257].

(Equation 212)



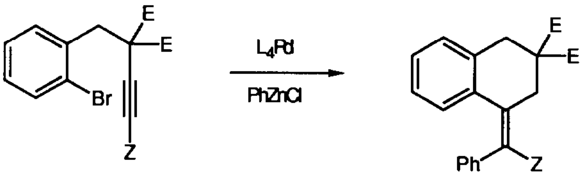
(Equation 213)



46-68%

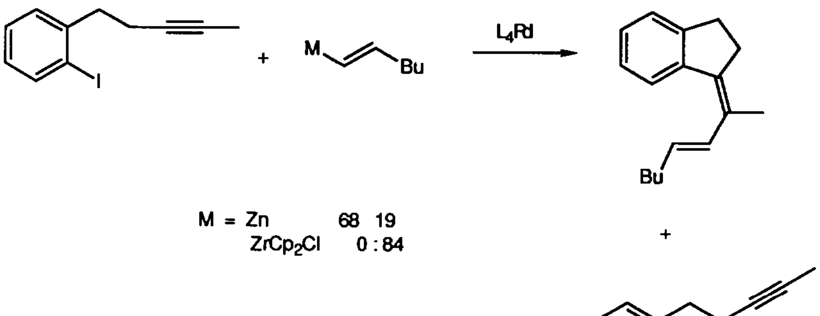
Z = H, Me, TMS

AND



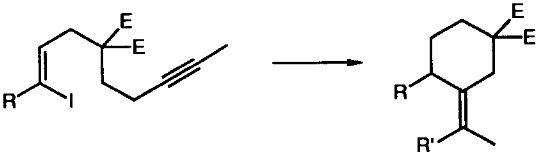
43-53%

(Equation 214)



M = Zn 68 19
ZrCp$_2$Cl 0 : 84

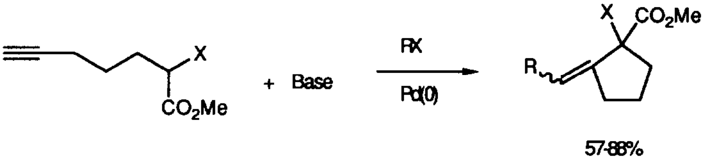
AND



85%

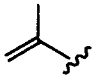
. p 643

(Equation 215)

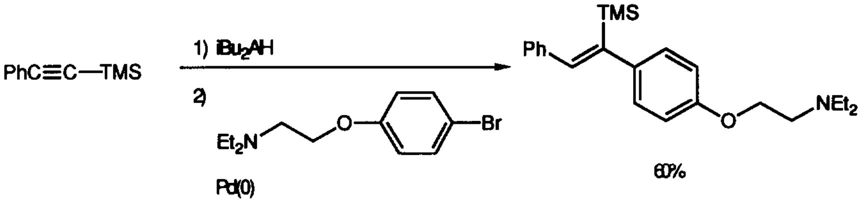


X = CO_2Me, COMe, SO_2Ph

R = Ph, MeOPh,

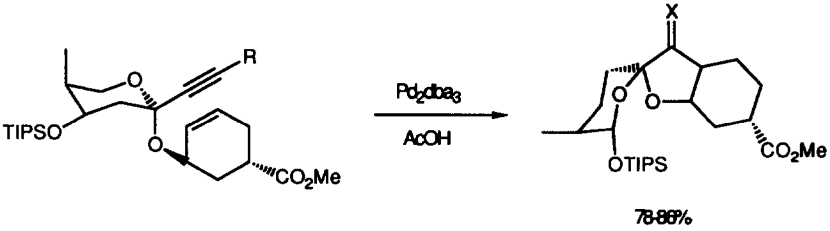


(Equation 216)

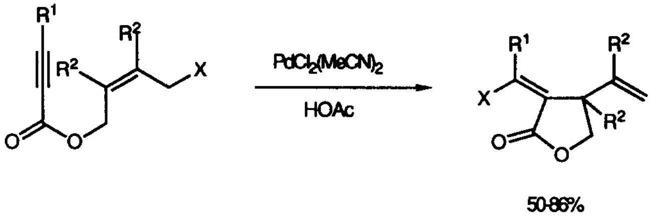


Palladium catalyzed ene-yne cyclizations (equation 217) [258], (equation 218) [259]

(Equation 217)



(Equation 218)

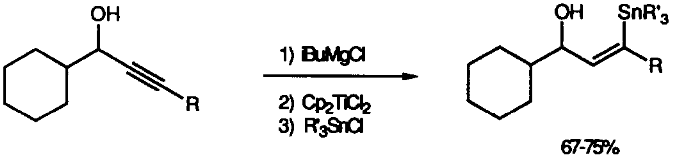


X = Br, Cl, I

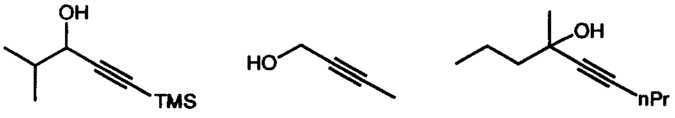
R^1 = H, C$_5$

Titanocene dichloride promoted the stannylation of alkynes (equation 219) [260]. Alkynes inserted into Zirconium-benzyne complexes to produce olefinated arenes (equation 220) [261] and unusual heterocycles (equation 221) [262]. It also catalyzed the alkylation of alkynes by trialkyl aluminum reagents (equation 222) [263].

(Equation 219)

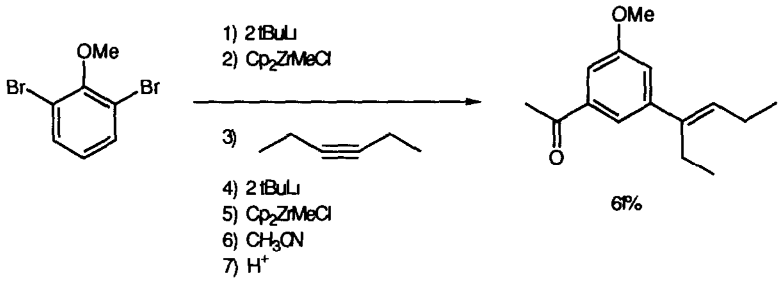


R = TMS, SiPhMe$_3$, SnBu$_3$, Bu, Ph

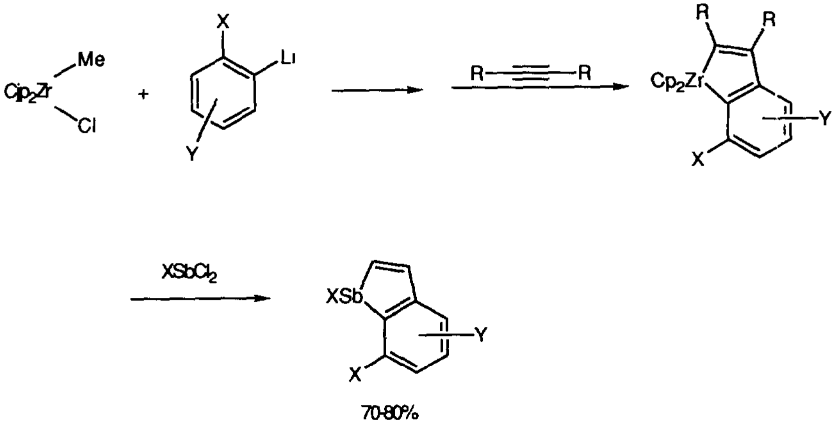


also react

(Equation 220)

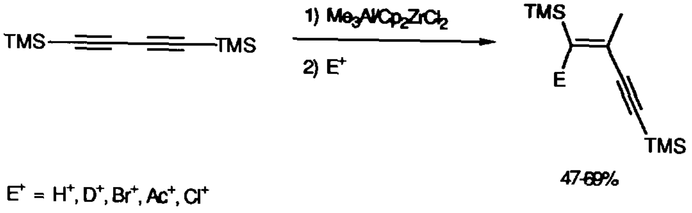


(Equation 221)



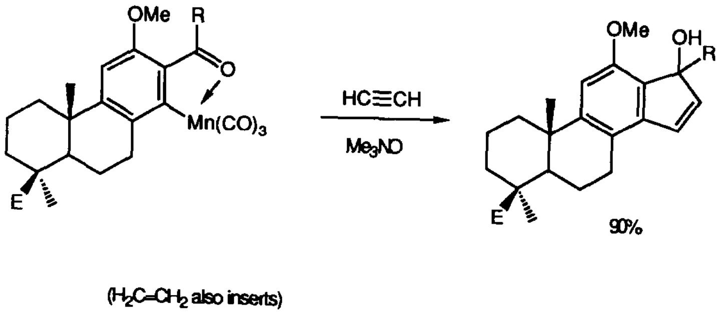
R = Me; X = H, MeO, Y = H, MeO

(Equation 222)

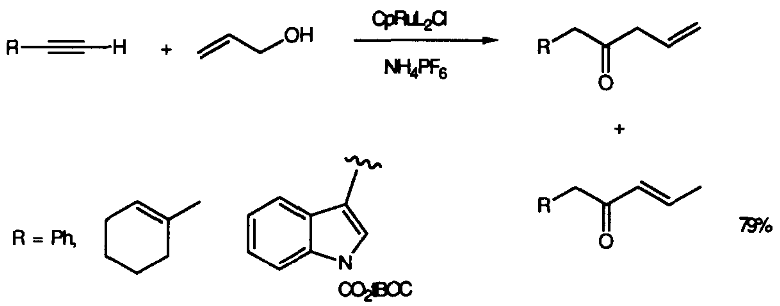


Arylmanganese compounds inserted alkynes under oxidative conditions (equation 223) [264]. Ruthenium(II) complexes catalyzed the alkylation of alkynes by allyl alcohols (equation 224) [265]. Rhodium(I) complexes catalyzed the addition of arenes to alkynes to produce styrenes (equation 225) [266].

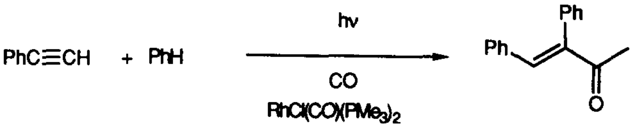
(Equation 223)



(Equation 224)



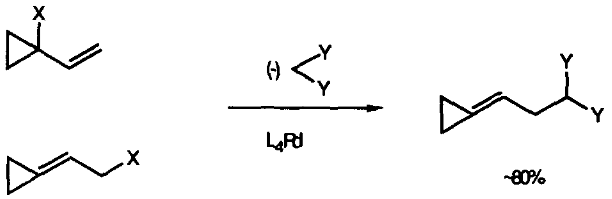
(Equation 225)



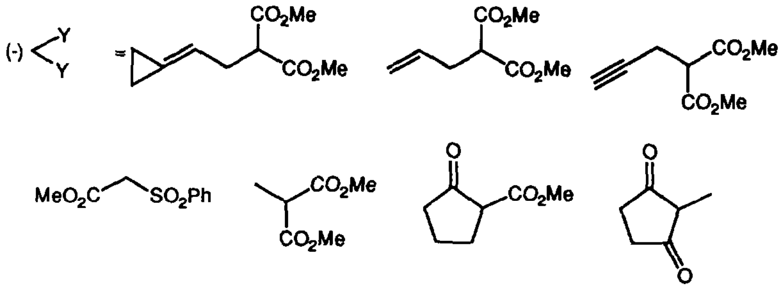
7. Alkylation of Allyl, Propargyl and Allenyl Systems

Palladium(0) catalyzed reactions of allylic substrates have remained a popular activity and is finding very wide application, although few if any conceptual advances have been made. Palladium(0) catalyzed macrocyclization reactions were the subject of a dissertation [267]. Cyclopropane-containing allyl systems were alkylated by stabilized carbanions without ring opening, in the presence of palladium(0) catalysts (equation 226) [268]. A formal nucleophilic alkylation of the α-position of ketones was achieved via palladium catalyzed alkylation of an allyl carbonate (equation 227) [269] Palladium(0) complexes catalyzed the alkylation of allyl perfluoroalkanoates by malonates and by alkyl perfluoroalkyl-ketones [270]. Allyl acetates were alkylated by tetronic acid derivatives in the presence of palladium catalysts (equation 228) [271].

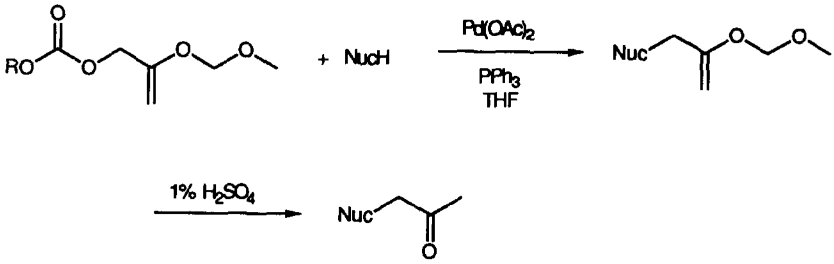
(Equation 226)

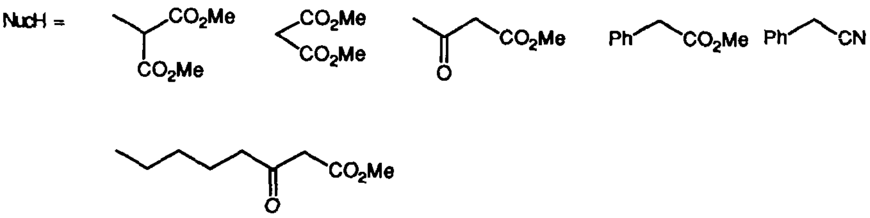


X = OAc, OCO$_2$Et, OTs

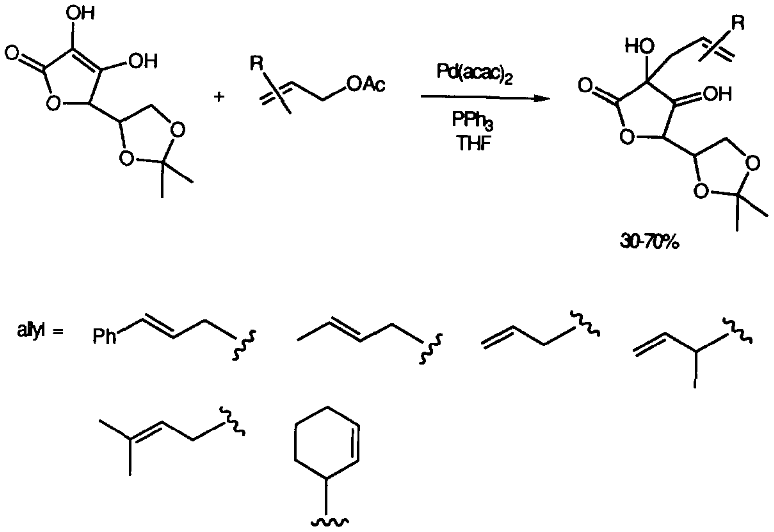


(Equation 227)



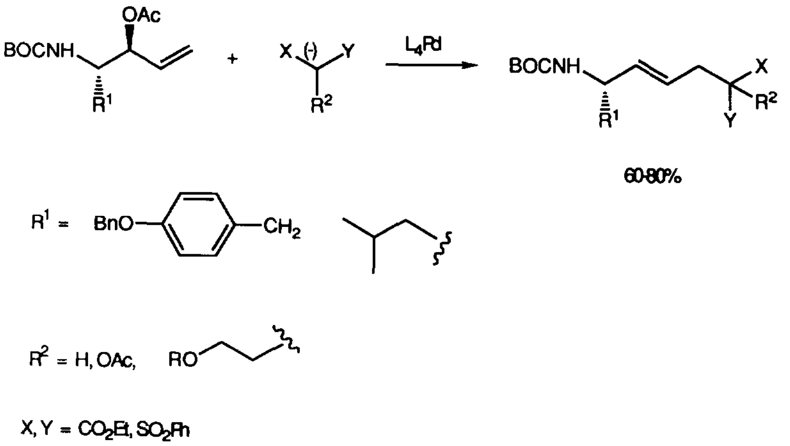


(Equation 228)

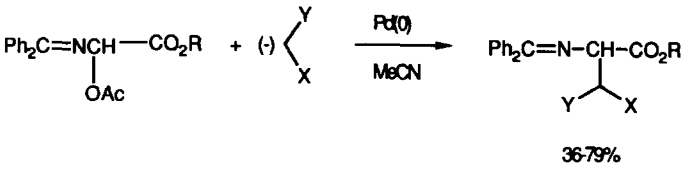


Nitrogen containing allyl acetates were alkylated by stabilized carbanions in the presence of palladium(0) catalysts (equation 229) [272], (equation 230) [273], (equation 231) [274] Silyl enol ethers alkylated allyl carbonates in the presence of palladium(0) on silica (equation 232) [275]

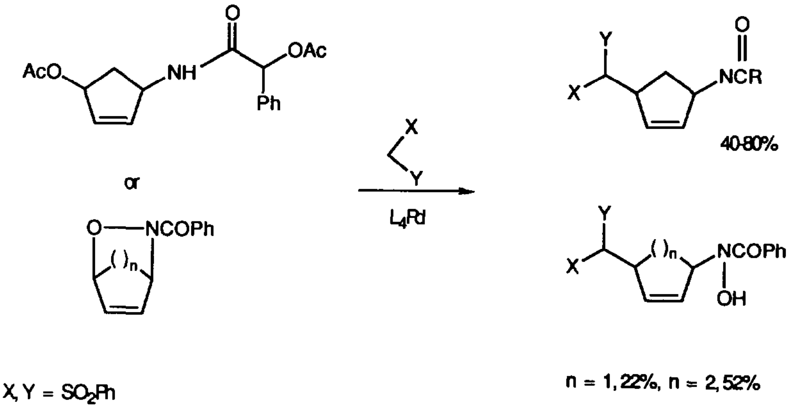
(Equation 229)



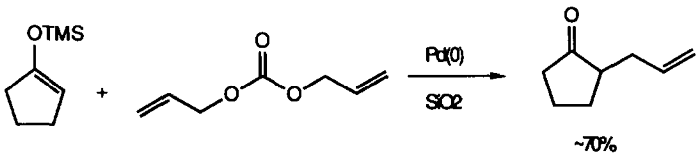
(Equation 230)



(Equation 231)

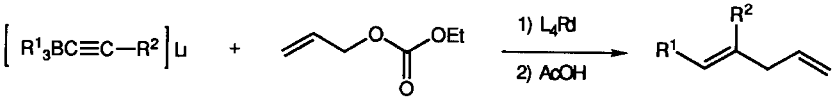


(Equation 232)



Allyl carboxylates were alkylated by organoboron compounds (equation 233) [276], (equation 234) [277], stannylated by Me_2AlSnR_3 (equation 235) [278], and alkylated by organostannanes (equation 236) [279] in the presence of palladium(0) catalysts. Thromboxane B_2 precursors were synthesized utilizing palladium(0) catalyzed allylic functionalization of allyl acetates (equation 237) [280]

(Equation 233)

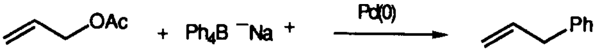


50-70%

R^1 = nBu, nC_6, nC_5, CH_2OMe

R^2 = nC_6, C_7, C_8

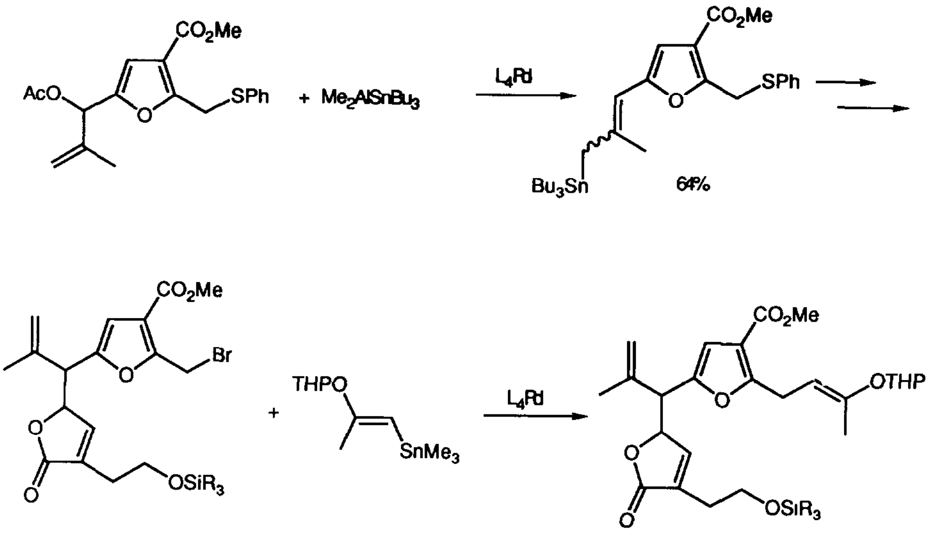
(Equation 234)



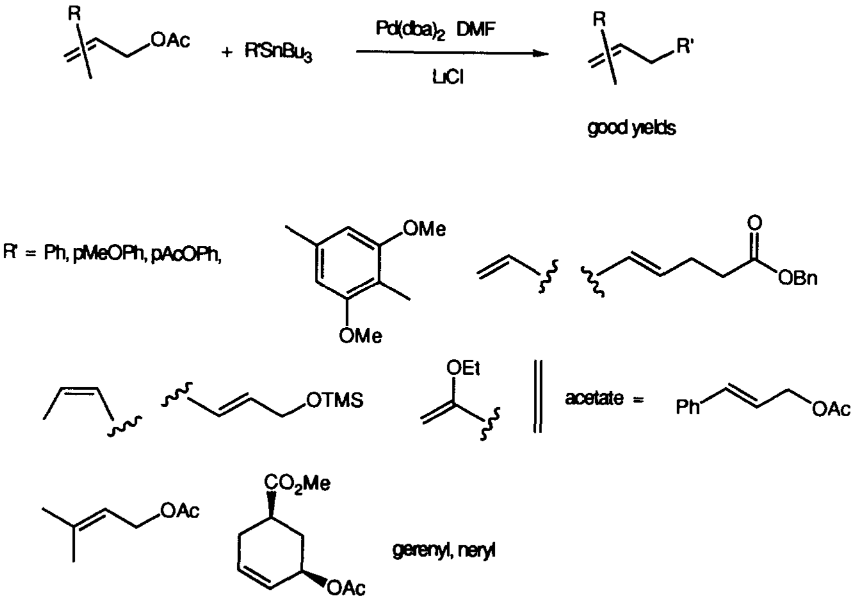
6 cases

2 - Ph's transfer

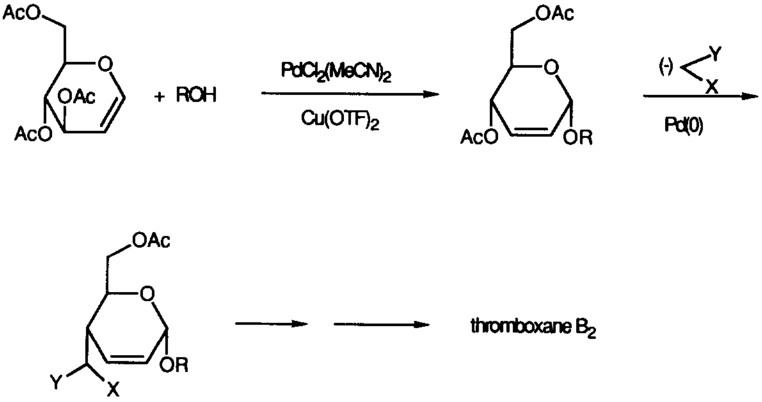
(Equation 235)



(Equation 236)

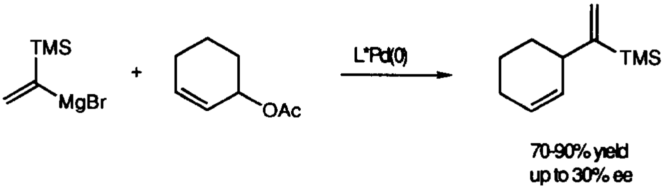


(Equation 237)

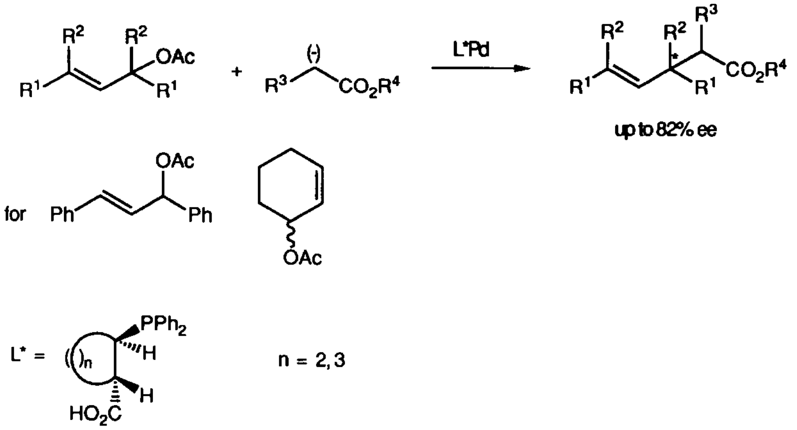


Chiral ligands were used to induce asymmetry into the palladium-catalyzed allylic alkylation process (equation 238) [281], (equation 239) [282], (equation 240) [283], (equation 241) [284], (equation 242) [285]. Functionalized allylic substrates reacted with norbornenes to produce cyclopropanes (equation 243) [286], (equation 244) [287]

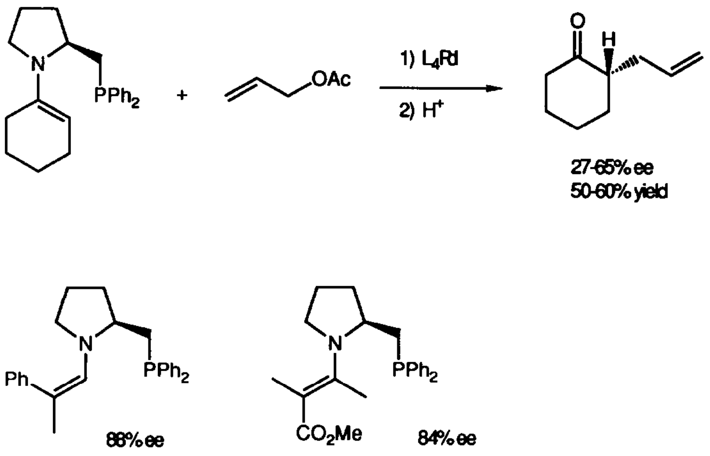
(Equation 238)



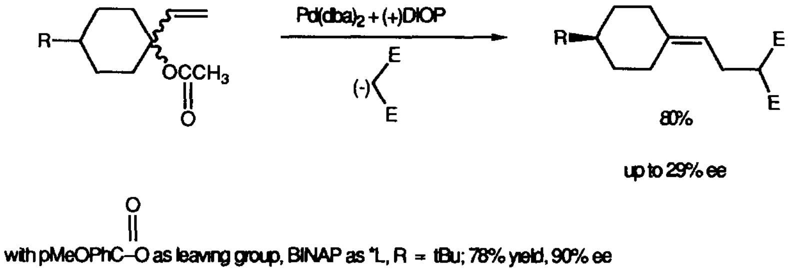
(Equation 239)



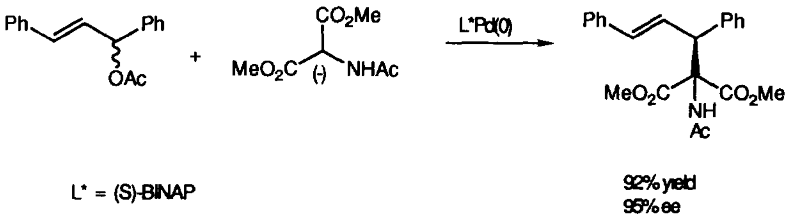
(Equation 240)



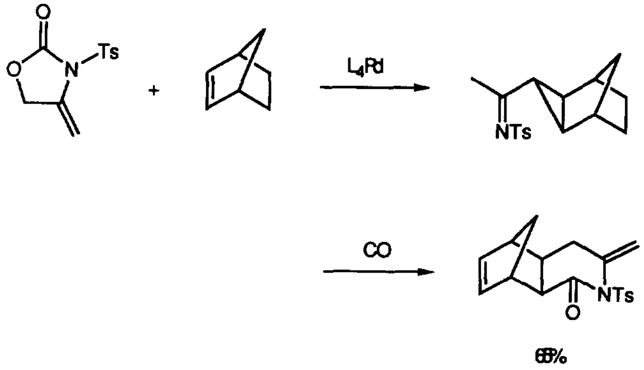
(Equation 241)



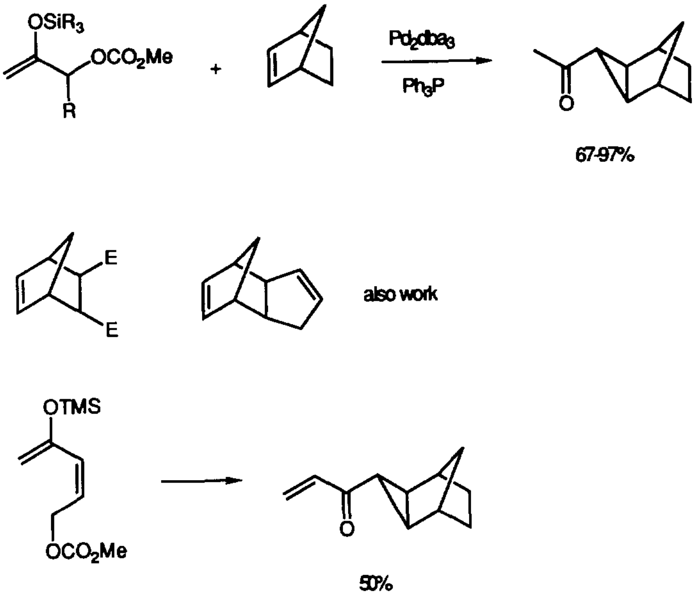
(Equation 242)



(Equation 243)

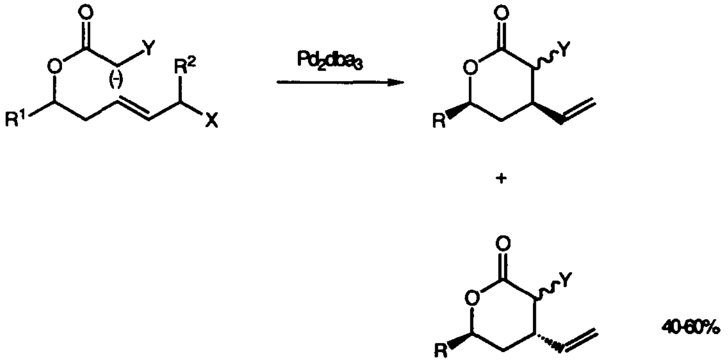


(Equation 244)

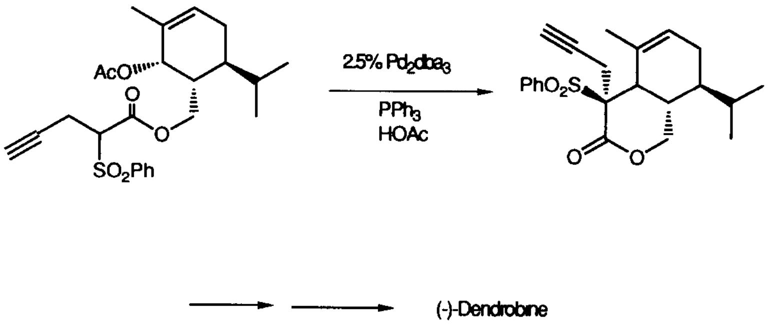


A variety of heterocycles were made by palladium(0) catalyzed allylic alkylation of acetates (equation 245) [288], (equation 246) [289], palladium catalyzed insertion of alkynes into π-allylpalladium intermediates (equation 247) [290] and insertion of alkenes into the same species (equation 248) [291], (equation 249) [292], (equation 250) [293].

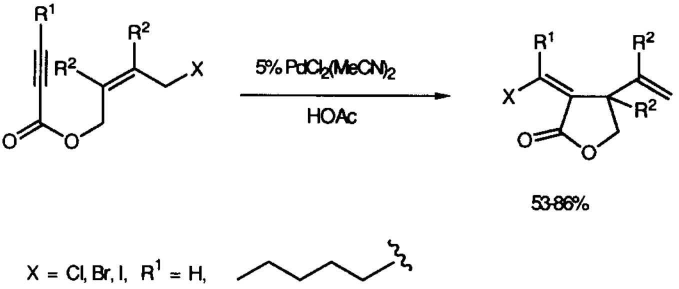
(Equation 245)



(Equation 246)



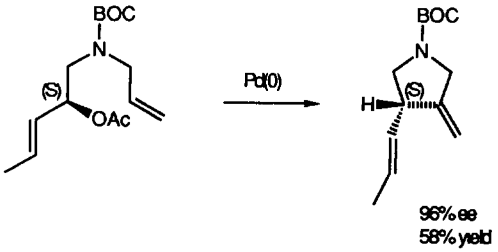
(Equation 247)



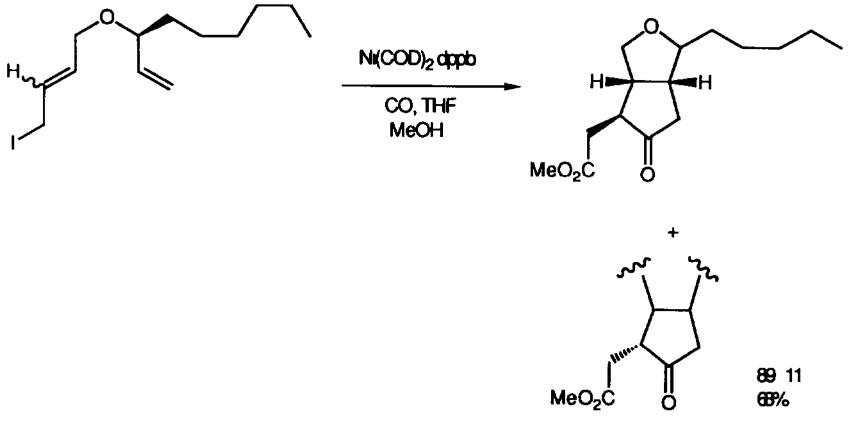
X = Cl, Br, I, R^1 = H,

R = H

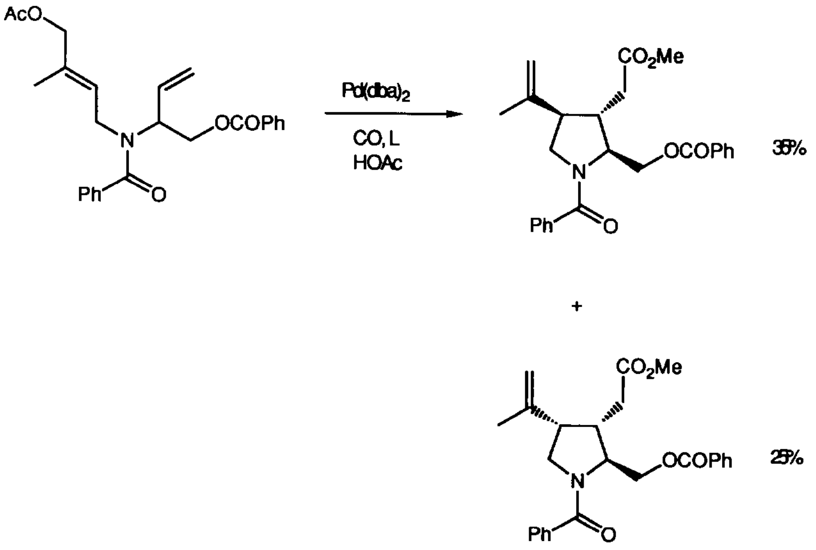
(Equation 248)



(Equation 249)

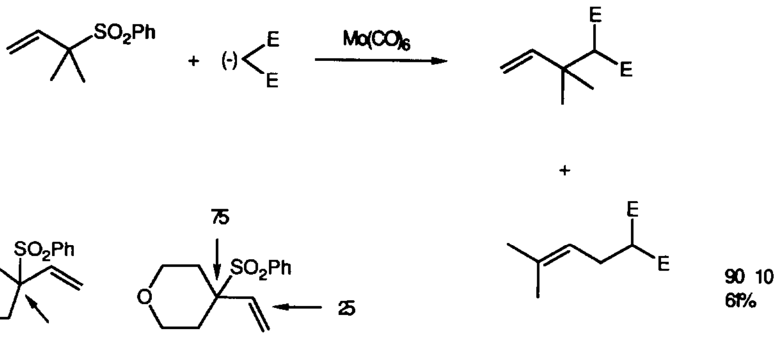


(Equation 250)



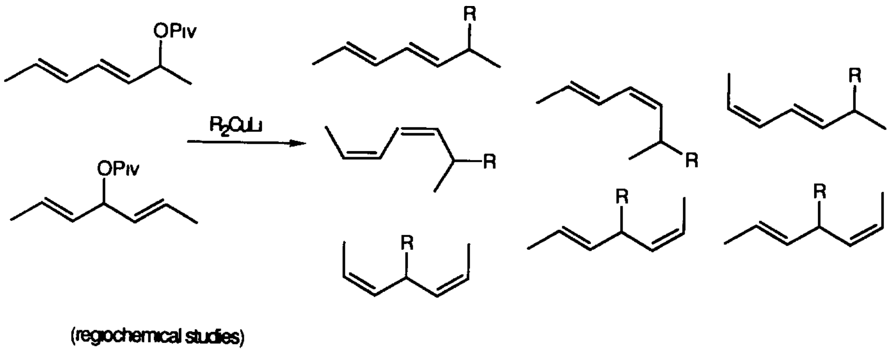
From a very broad study of catalysts, $Mo(RNC)_4(CO)_2$ proved to be the best general Mo catalyst for allyl alkylation of allyl acetates [294]. Molybdenum catalysts favored attack at the <u>most</u> substituted end of an allyl system (equation 251) [295]

(Equation 251)

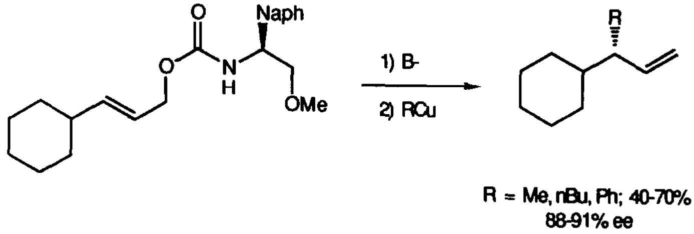


In the copper catalyzed reactions of Grignard reagents with allylic acetates and sulfones, the regiochemistry was controlled by the reaction conditions [296], (equation 252) [297] Copper complexes alkylated chiral allyl carbamates with high ee (equation 253) [298]

(Equation 252)

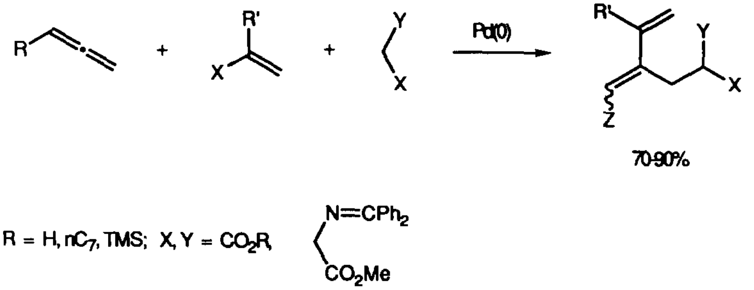


(Equation 253)

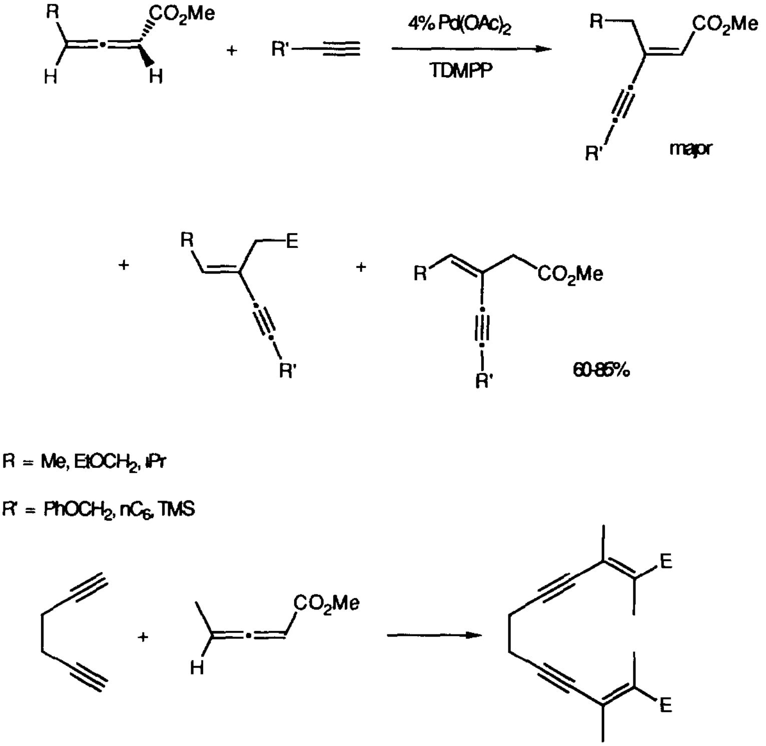


Palladium(0) complexes catalyzed the coreaction of allenes, vinyl and aryl halides and triflates, and malonates (equation 254) [299], and the alkylation of allenic esters by alkynes (equation 255) [300]. Stannyl cuprates added to allenes and the resulting vinyl cuprates were further functionalized (equation 256) [301] Allyl metal complexes alkylated allenic ethers (equation 257) [302].

(Equation 254)



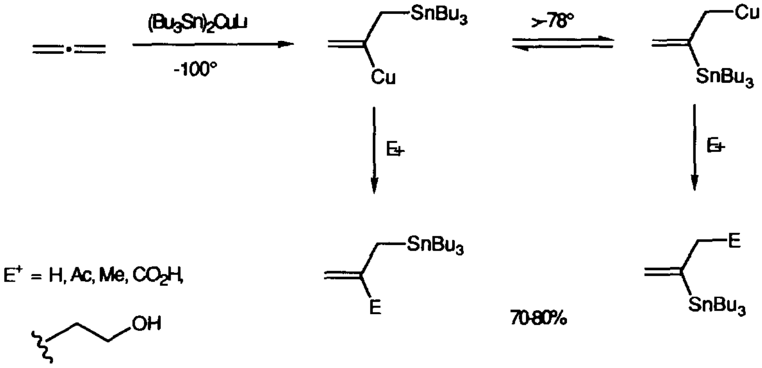
(Equation 255)



R = Me, EtOCH$_2$, iPr

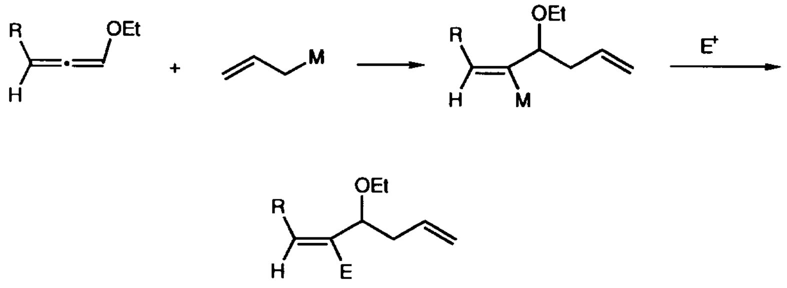
R' = PhOCH$_2$, nC$_6$, TMS

(Equation 256)



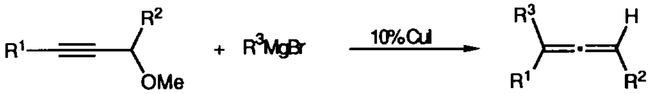
E$^+$ = H, Ac, Me, CO$_2$H,

(Equation 257)



Propargyl ethers were alkylated to allenes by Grignard reagents in the presence of copper catalysts (equation 258) [303] Perfluoroalkyl copper reagents alkylated propargyl systems to give allenes (equation 259) [304], (equation 260) [305] Palladium(0)/copper(I) catalysts alkylated propargyl carbonates with alkynes (equation 261) [306].

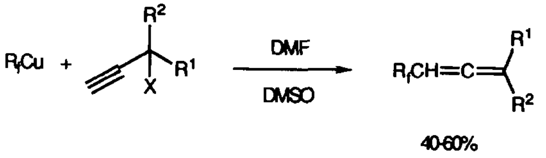
(Equation 258)



via *syn* addition, β-elimination. RMgI → anti elimination
RMgCl → syn elimination

chiral ether → chiral allene 96% ee

(Equation 259)



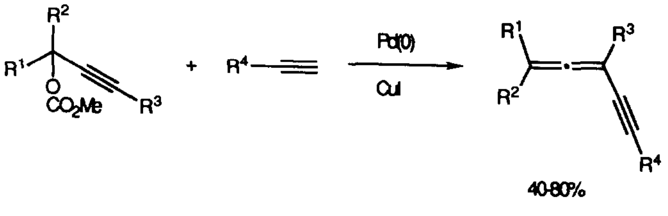
$R_f = F(CF_2)_n$; n = 1,3,6,8

R^1 = H, Me; R^2 = H, Me, $(CH_2)_5$; X = Cl, OT

(Equation 260)

$R_f = C_5F_{13}, C_8F_{17}$

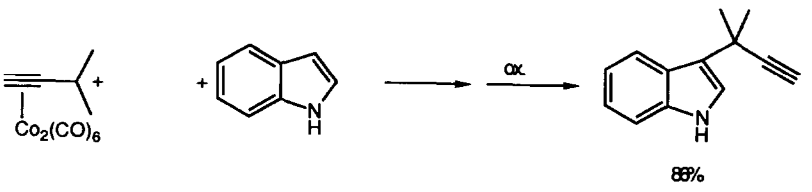
(Equation 261)

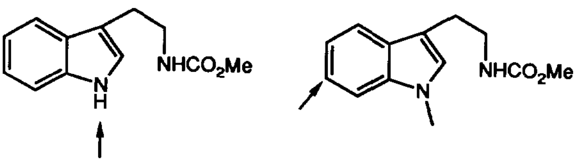


A dissertation dealing with the dynamics and stereochemistry of reactions of cobalt carbonyl stabilized propargyl cations has appeared [307] These stabilized cations were alkylated by indoles (equation 262) [308], trimethylsilylenol ethers (equation 263) [309], and phenols (equation 264)

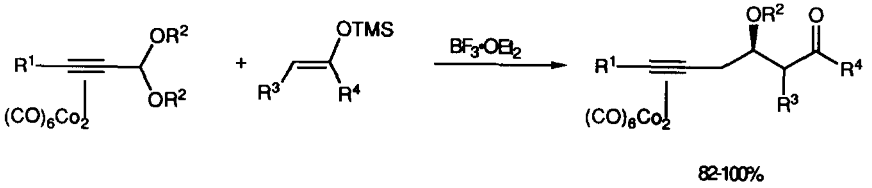
[310] Allylic halides cross coupled to allyl organomanganese(II) compounds [311].

(Equation 262)

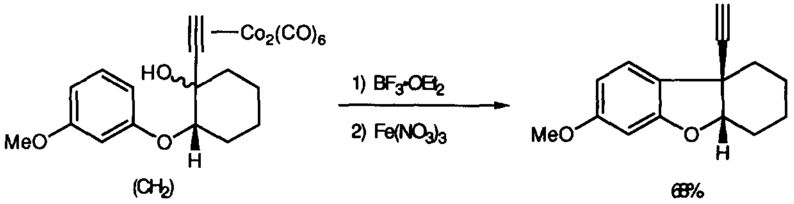




(Equation 263)



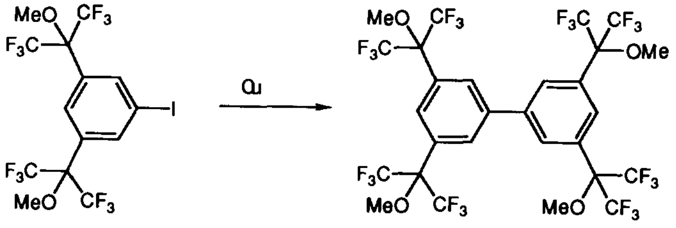
(Equation 264)



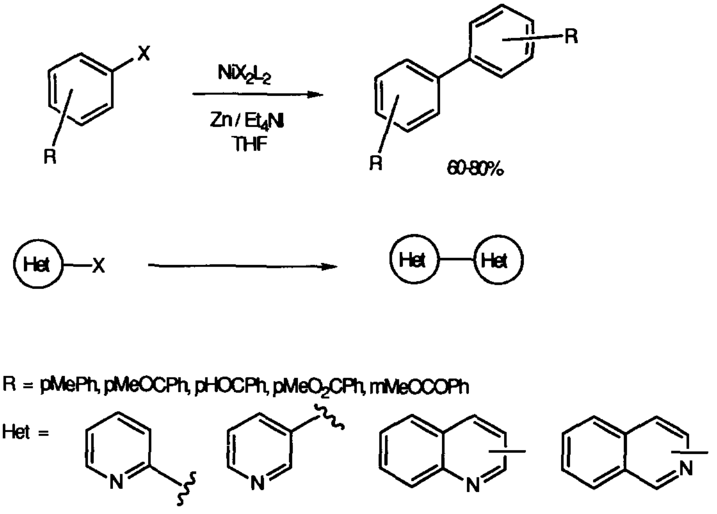
8 Coupling Reactions

Reductive homocoupling of halides has been further developed The classical Ullmann coupling of bromobenzene was compared with that effected by copper metal vapor [312] Fluoroalkyl iodobenzenes were coupled under Ullmann conditions (equation 265) [313]. Activated copper homo coupled allylic, benzylic and aliphatic bromides efficiently [314]. Nickel(II) chloride/phosphine/zinc in pyridine coupled π-chloromethyl benzoate to the biphenyl in good yield [315] A related system homocoupled a wide range of aryl and heteroaryl halides (equation 266) [316] Aryl and vinyl halides cross coupled to α-chloro esters under electrochemical reduction in the presence of nickel complexes (equation 267) [317] Chromium carbonyl amine complexes coupled a variety of halides (equation 268) [318]

(Equation 265)

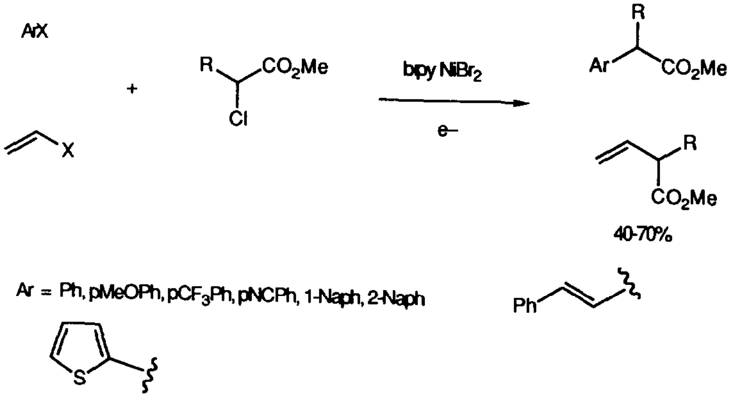


(Equation 266)



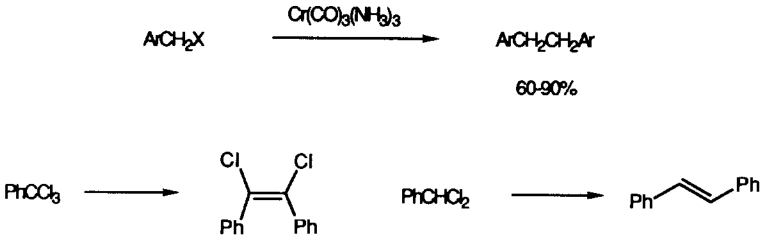
R = pMePh, pMeOCPh, pHOCPh, $pMeO_2CPh$, mMeOCOPh

(Equation 267)



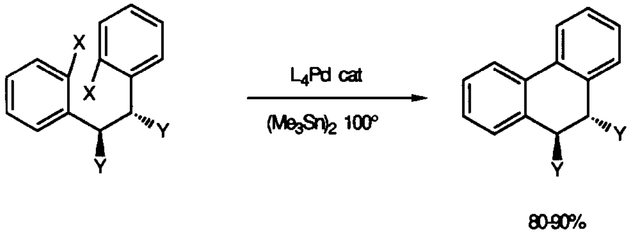
Ar = Ph, pMeOPh, pCF_3Ph, pNCPh, 1-Naph, 2-Naph

(Equation 268)



Intramolecular coupling of aryl halides was promoted by palladium(0)/hexamethyldistannane catalysts (equation 269) [319]. Arylsulfonyl chlorides coupled to biaryls in the presence of palladium(II)/titanium(IV) catalysts (equation 270) [320]. Reduced titanium species homocoupled halides (equation 271) [321]

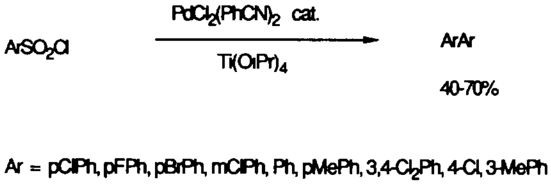
(Equation 269)



X = Br, I, OTf

Y = OH, H

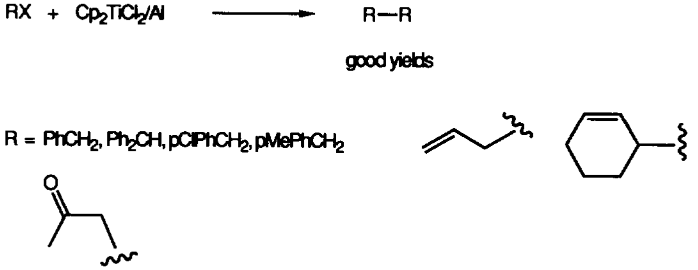
(Equation 270)



$$ArSO_2Cl \xrightarrow[Ti(OiPr)_4]{PdCl_2(PhCN)_2\ cat.} ArAr \quad 40\text{-}70\%$$

Ar = pClPh, pFPh, pBrPh, mClPh, Ph, pMePh, 3,4-Cl_2Ph, 4-Cl, 3-MePh

(Equation 271)



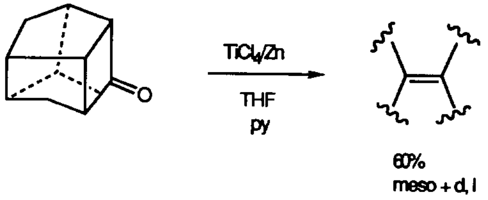
$$RX + Cp_2TiCl_2/Al \longrightarrow R{-}R$$

good yields

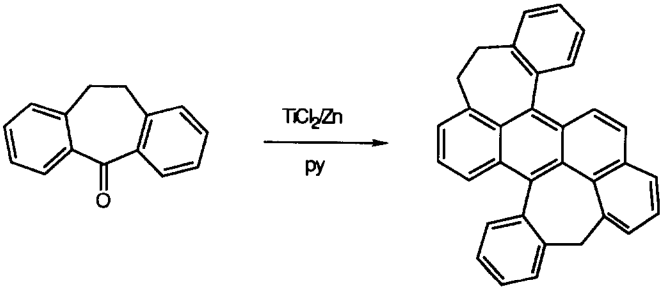
R = $PhCH_2$, Ph_2CH, $pClPhCH_2$, $pMePhCH_2$

McMurray's reagent ("Ti(0)") has been extensively utilized for the reductive coupling of ketones to olefins (equation 272) [322], (equation 273) [323], (equation 274) [324], (equation 275) [325], (equation 276) [326], (equation 277) [327]. Reduced vanadium coupled ketones to diols (equation 278) [328].

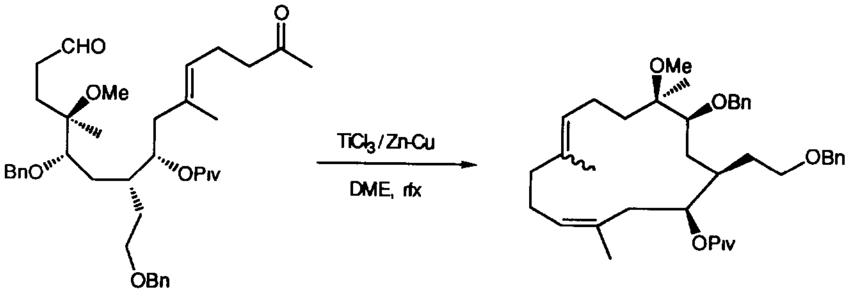
(Equation 272)



(Equation 273)

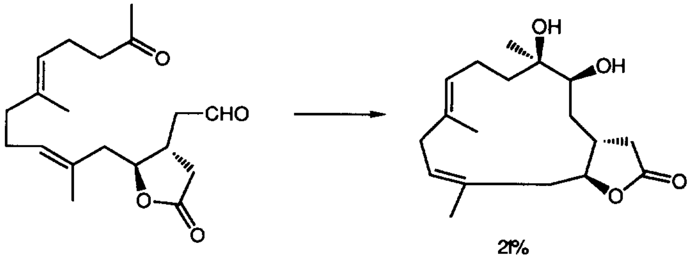


(Equation 274)



65%

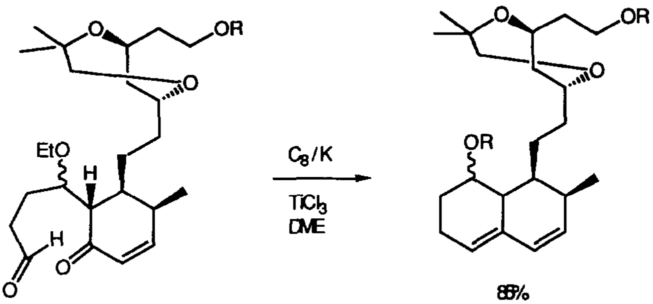
(Equation 275)



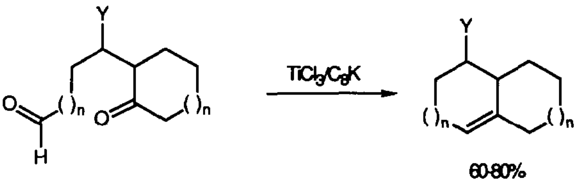
21%

(crassin, isolobophytolide)

(Equation 276)



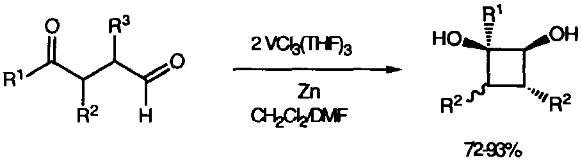
(Equation 277)



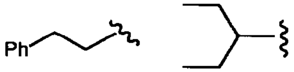
X = $OSiR_3$, H, OAc

n = 0, 1, 2

(Equation 278)



R^1 = iBu, iPr, nBu, nPr, H

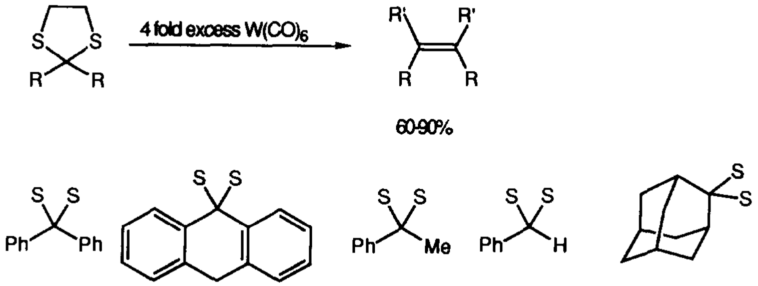


R^2 = H, NHTs, Et

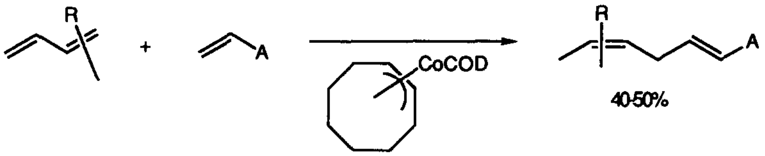
R^3 = H, OTBDMS

Tungsten hexacarbonyl coupled dithioketals to olefins (equation 279) [329]. Cobalt allyl complexes coupled dienes to electron deficient olefins (equation 280) [330] Manganese porphyrin complexes oxidatively coupled phenols to make benzylisoquinoline alkaloids [331]. Alkylcuprates and alkyllithiums were coupled (equation 281) [332].

(Equation 279)



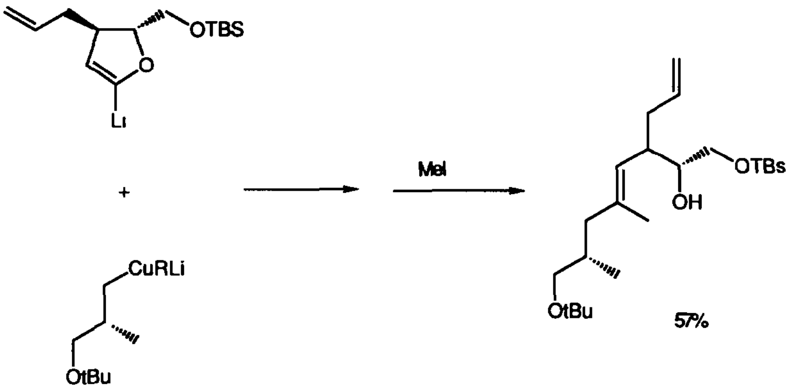
(Equation 280)



R = H, Me, 2,Me

A = CO_2Me, SO_2Ph, CO_2tBu

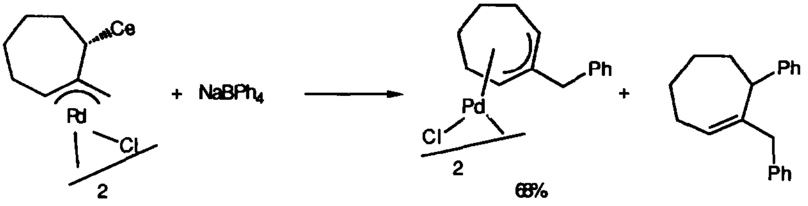
(Equation 281)



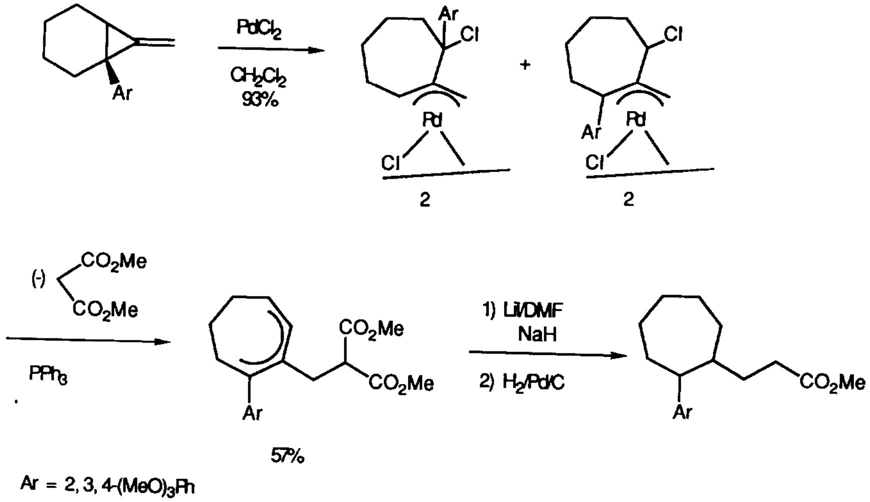
9 Alkylation of π-Allyl Complexes

Allylic halides coupled to *bis*-π-allylnickel complexes to produce biallyls [333]. π-Allylpalladium chloride complexes reacted with methyl formate to produce unsaturated carboxylic acid esters [334]. Chloromethyl allylpalladium complexes were arylated by tetraphenylborate (equation 282) [335] and stabilized enolates (equation 283) [336]. π-Allylpalladium complexes with optically active DIOP ligands allylated optically active esters with good de (equation 284) [337] Z-Stereochemistry could be forced in the alkylation of π-crotyl palladium complexes by using α-methylated phenanthroline ligands (equation 285) [338]. The stereochemistry of oxidative addition of palladium(0) complexes to allylic chlorides depended on the solvent and the ligands (equation 286) [339].

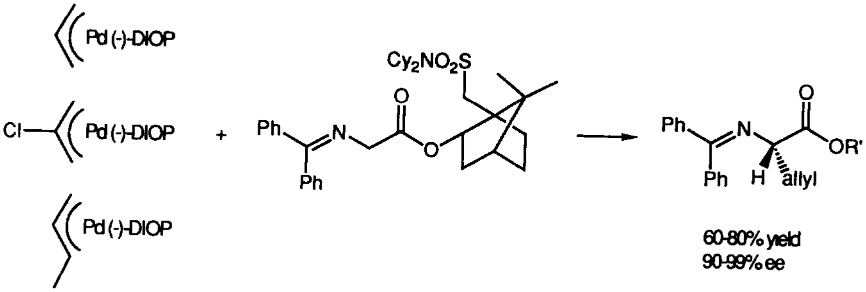
(Equation 282)



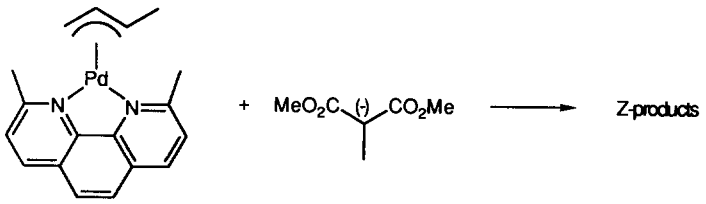
(Equation 283)



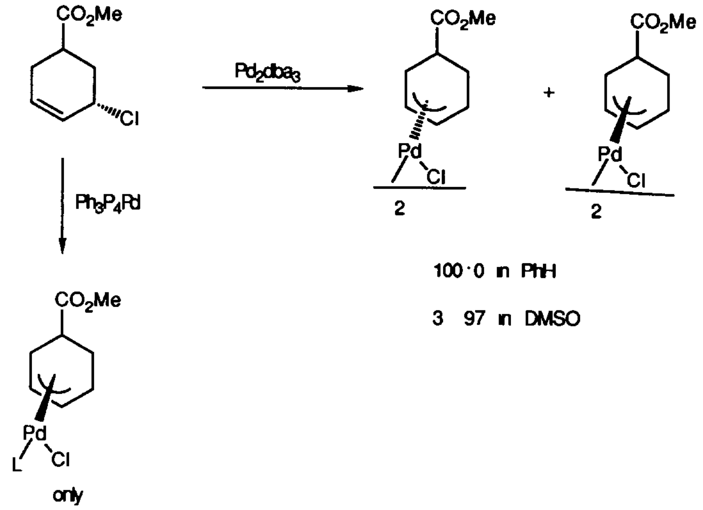
(Equation 284)



(Equation 285)

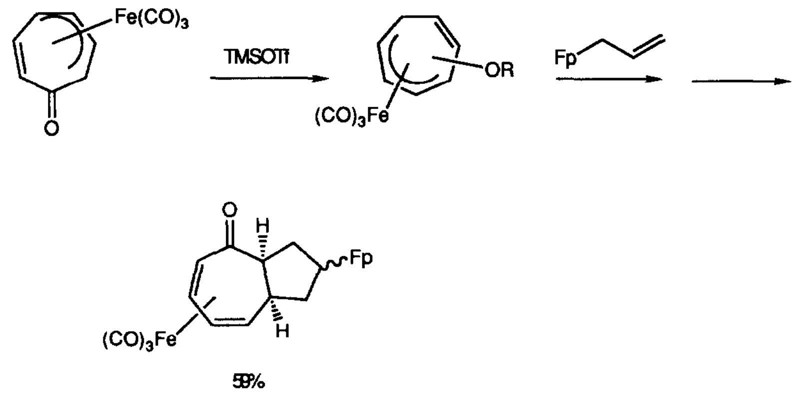


(Equation 286)

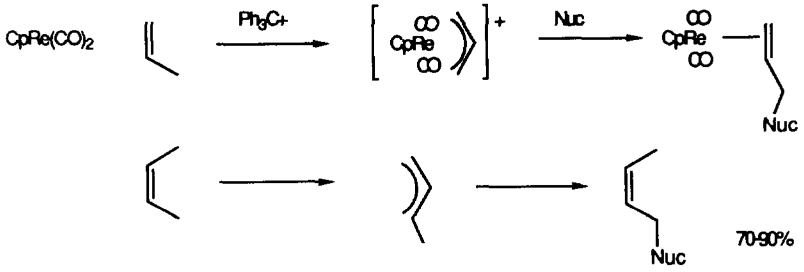


π-Allyliron complexes of tropones were alkylated by iron allyl and propargyl compounds (equation 287) [340]. Cationic rhenium π-allyl complexes were alkylated (equation 288) [341]. π-Allyl iridium and rhodium complexes underwent alkylation at the central carbon to give metallacyclobutanes (equation 289) [342]

(Equation 287)

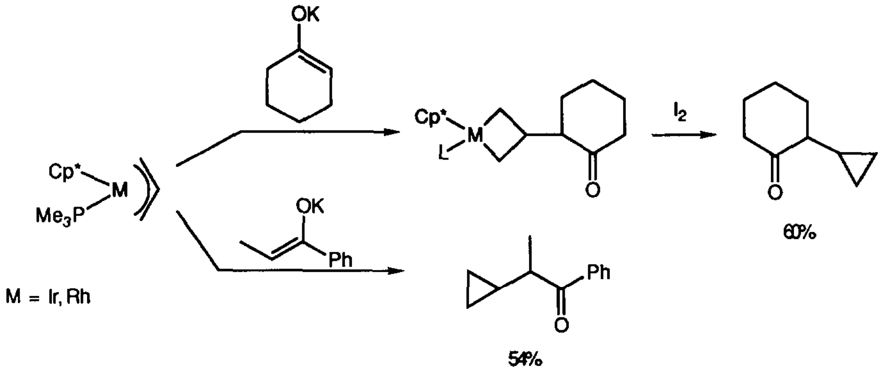


(Equation 288)



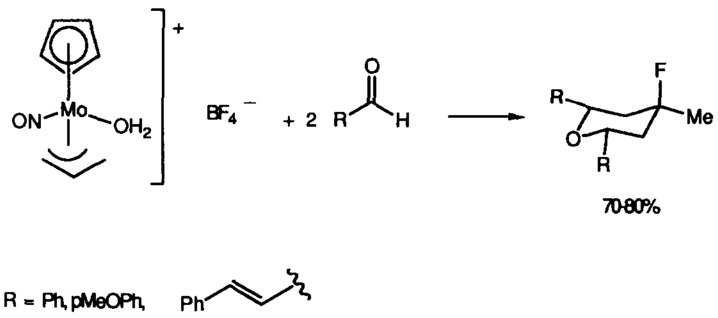
Nuc = Me$_2$CuLi, -CH(CO$_2$R)$_2$

(Equation 289)

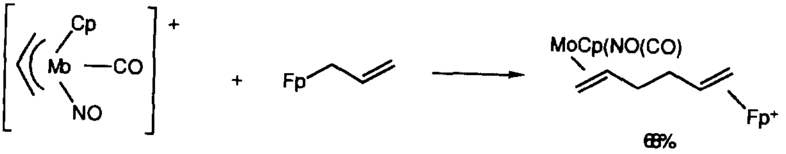


A paper dealing with controlling the regiochemistry of nucleophilic attack on unsymmetrical allyl complexes of the type CpMo(NO)(CO)(allyl)$^+$ has appeared [343]. Cationic π-allylmolybdenum complexes condensed with aldehydes (equation 290) [344], and were alkylated by allyliron complexes (equation 291) [345]. Acetyl-π-allylmolybdenum complexes were elaborated into polyhydroxy compounds with a high degree of stereoselectivity (equation 292) [346][347].

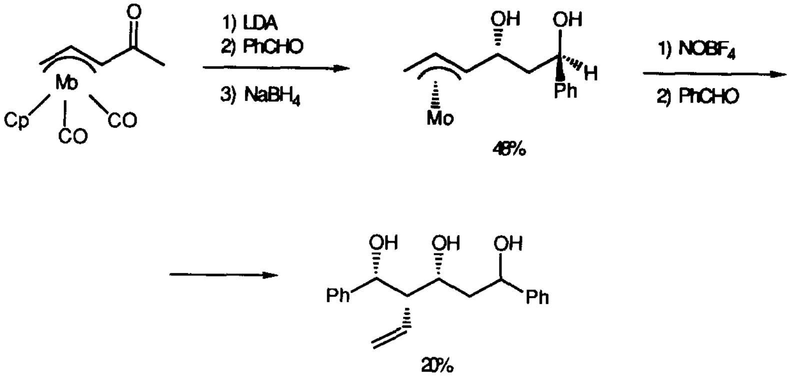
(Equation 290)



(Equation 291)



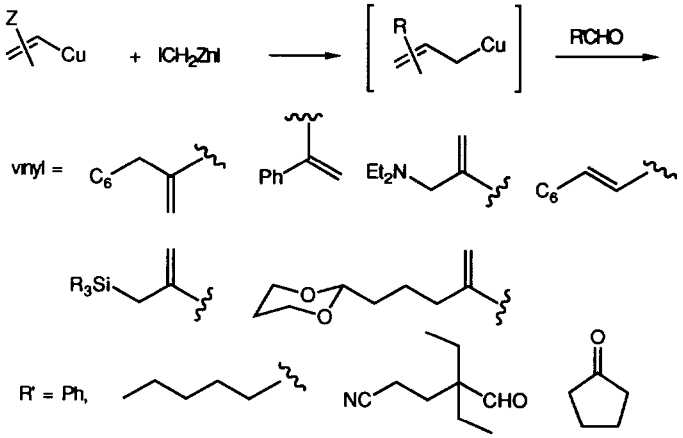
(Equation 292)



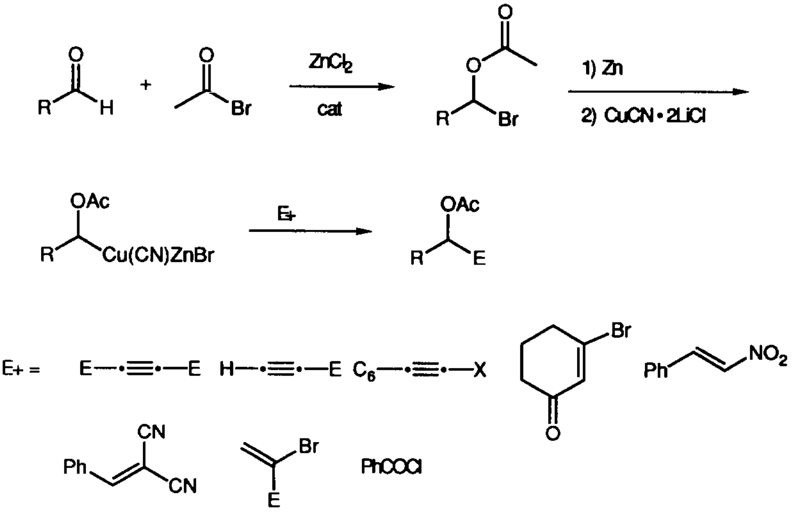
10. Alkylation of Carbonyl Compounds

Vinyl copper reagents reacted with aldehydes and CH_2I_2/Zn to give allylic alcohols (equation 293) [348]. Aldehydes and acid bromides combined in the presence of zinc chloride, and the copper complex of the resulting product alkylated a variety of electrophiles (equation 294) [349] Cuprates ring opened bicyclic allyl ethers (equation 295) [350] Manganese metal reductively allylated ketones with allylic bromides (equation 296) [351] Chromium(II) chloride carried out a similar process (equation 297) [352], (equation 298) [353].

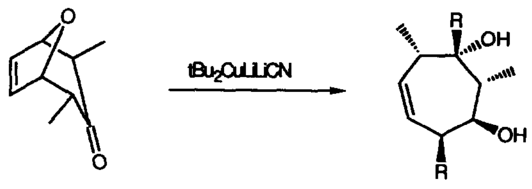
(Equation 293)



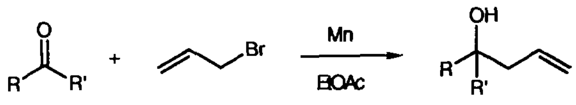
(Equation 294)



(Equation 295)

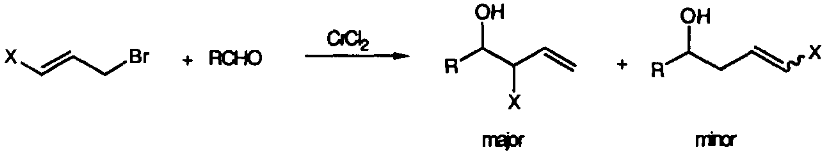


(Equation 296)

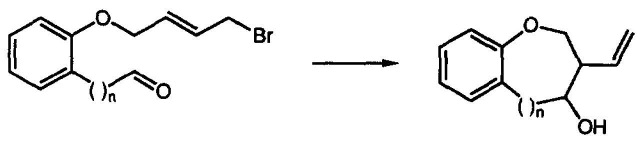


many cases
good yields

(Equation 297)

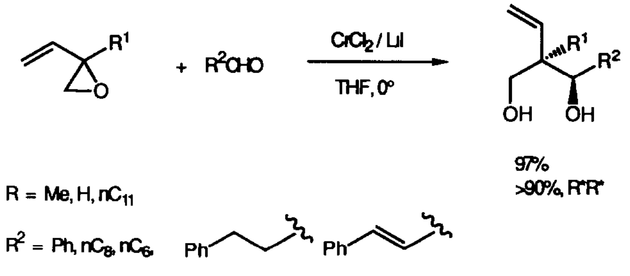


X = Cl, Br



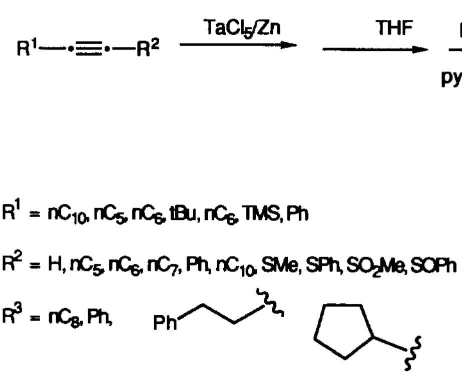
n = 0, 1, 2

(Equation 298)

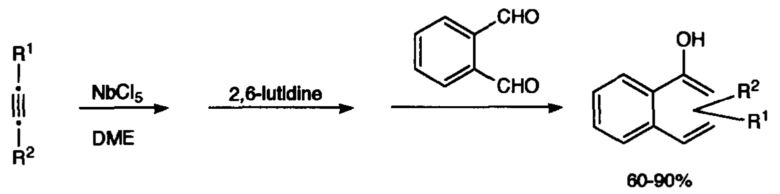


Alkynes reacted with ketones in the presence of tantalum(V) chloride and zinc to produce allylic alcohols (equation 299) [354][355]. Niobium(V) chloride condensed alkynes with dialdehydes (equation 300) [356]. Zirconium catalysts promoted the β-alkylation of naphthol by α-keto esters (equation 301) [357].

(Equation 299)



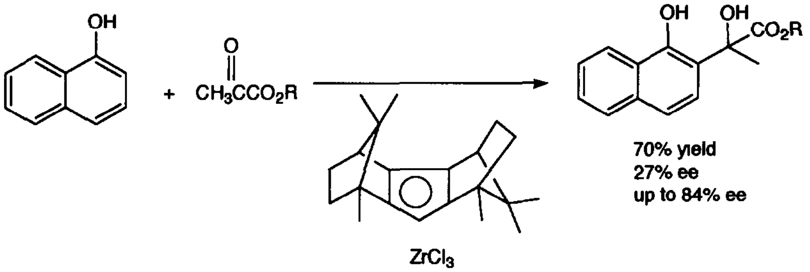
(Equation 300)



R^1 = nC$_5$, Ph, nC$_6$, nC$_7$, Me, nC$_{10}$

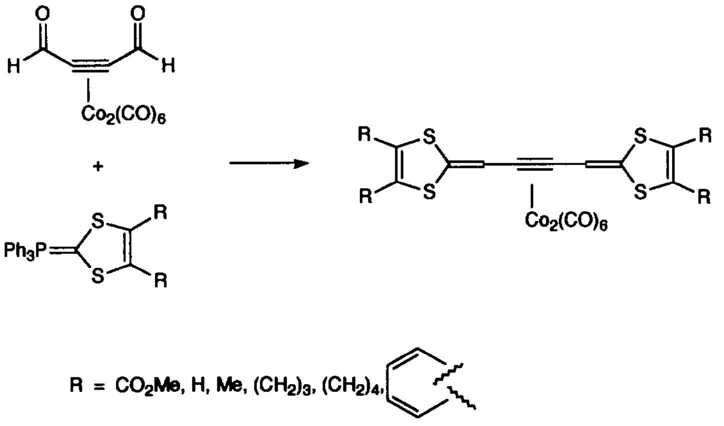
R^2 = nC$_5$, Ph, nC$_6$, H, TMS

(Equation 301)



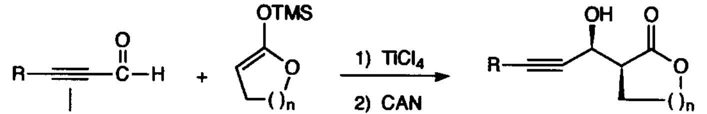
Cobalt complexed acetylenic aldehydes underwent alkylation by ylides (equation 302) [358] and trimethylsilylenol ethers (equation 303) [359] Chromium complexed benzaldehyde was allylated by chiral allyl boranes with high enantioselectivity, as were cobalt-complexed acetylenic aldehydes (equation 304) [360] Nickel chloride catalyzed the conversion of dithianes to olefins by Grignard reagents (equation 305) [361][362]. Tebbe's reagent converted a lactone into an enol ether (equation 306) [363]

(Equation 302)



R = CO_2Me, H, Me, $(CH_2)_3$, $(CH_2)_4$,

(Equation 303)



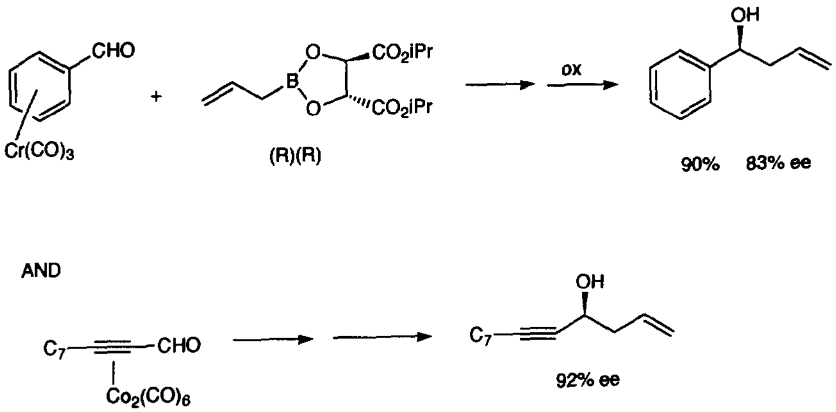
50-87%
80.20 to 95.5
erythro/threo
uncomplexed - 1·1 to 6.94

R = TMS, nBu, Ph

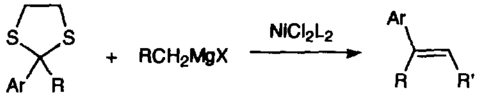
n = 1, 2, 3

› 643

(Equation 304)



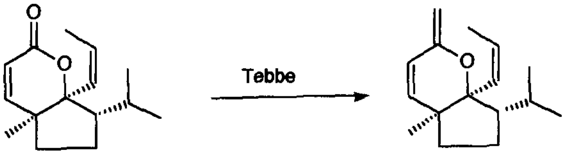
(Equation 305)



R' = TMS, H, Me_3Sn

Ar = Ph, 1-naph, oMePh, 3,5-Me_2Ph, pMeOPh

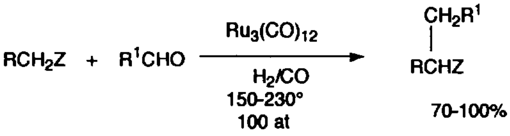
(Equation 306)



90%

Ruthenium carbonyls catalyzed the reductive alkylation of aldehydes by stabilized carbanions (equation 307) [364]. Rhodium carbonyls catalyzed the condensation/silylation reaction of enones with aldehydes (equation 308) [365].

(Equation 307)



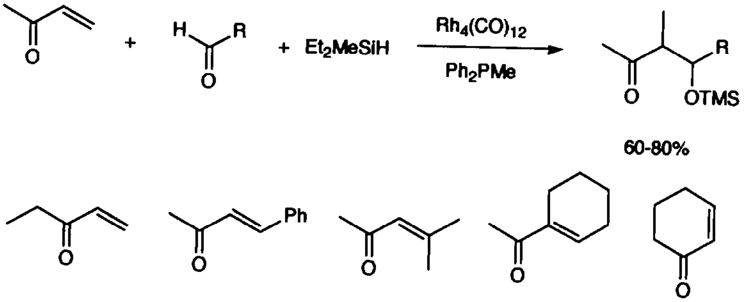
$$RCH_2Z + R^1CHO \xrightarrow[H_2/CO,\ 150\text{-}230^\circ,\ 100\ at]{Ru_3(CO)_{12}} \underset{RCHZ}{\overset{CH_2R^1}{|}}\quad 70\text{-}100\%$$

R = Ph, CN, CO_2Me, COMe

Z = CN, CO_2Me, COMe

R^1 = H, nPr

(Equation 308)

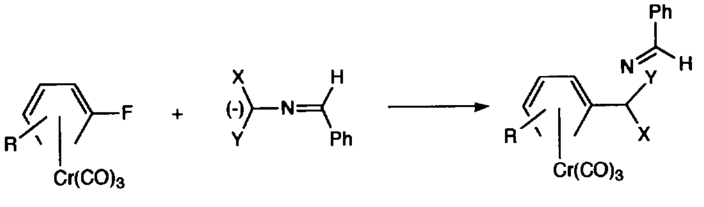


11. Alkylation of Aromatic Compounds

Applications of arene chromium tricarbonyl in asymmetric synthesis has been reviewed (21 references) [366], as has stereocontrolled synthesis using arene tricarbonyl chromium complexes as catalysts (9 references) [367], and application of η^6 arene complexes in organic synthesis (26 references) [368] The chemistry of arene-manganese complexes was the subject of a dissertation [369] "Substitution nucleophile aromatique; activation par les metaux de transition" was the subject of a review (128 references) [370]

Stabilized carbanions containing imines alkylated chromium complexed fluorobenzenes (equation 309) [371] and manganese coordinated arenes (equation 310) [372] Benzyl alcohol groups directed the site of nucleophilic attack on chromium complexes benzyl alcohols (equation 311) [373] Arenes were alkylated then acylated by a process involving nucleophilic attack on a chromium arene complex (equation 312) [374] t-Butyllithium attacked chromium complexed toluene ortho to the methyl group (equation 313) [375]

(Equation 309)



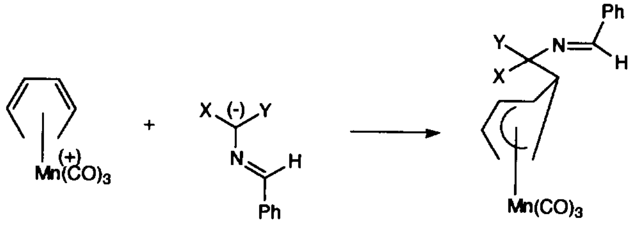
40-60$

R = OMe, mMe, pMe, pMeO

X = CO$_2$Me

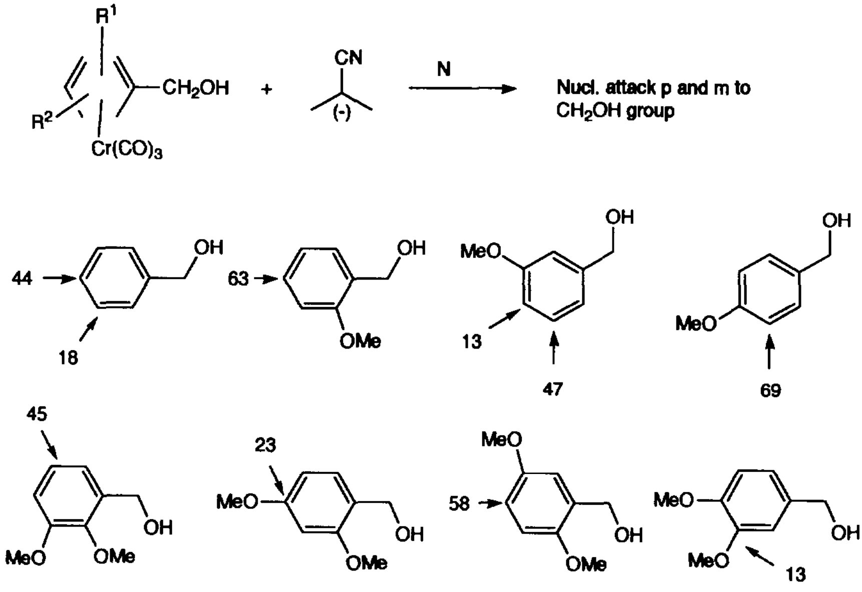
Y = CN

(Equation 310)

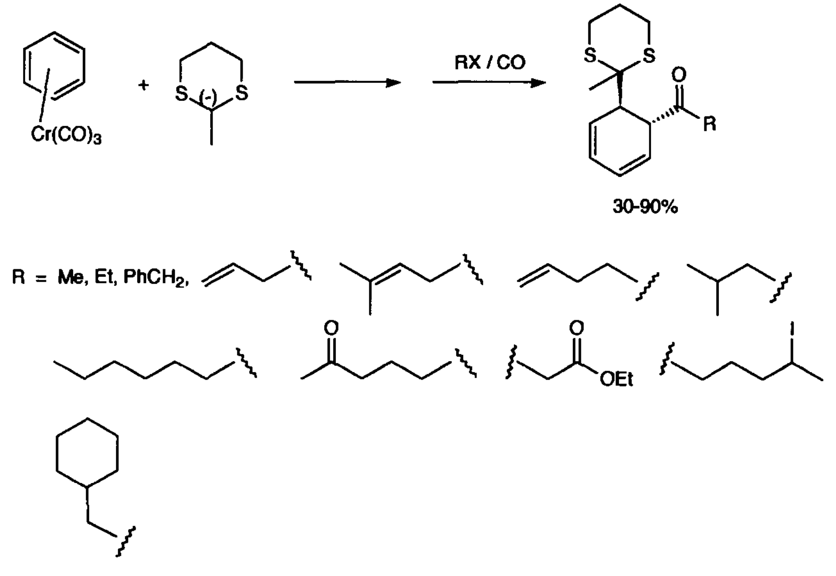


60-80%

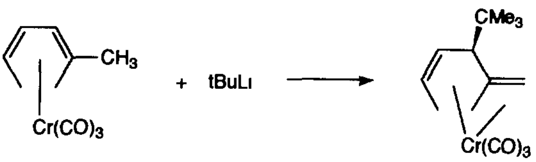
(Equation 311)



(Equation 312)

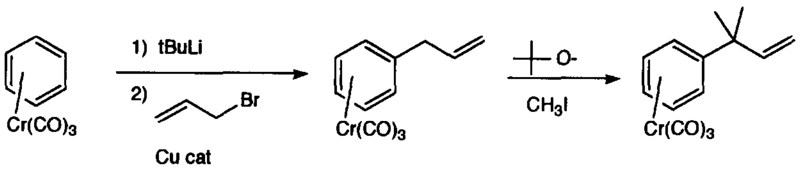


(Equation 313)

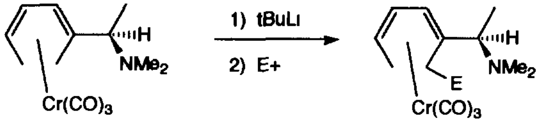


Chromium directed regio- and stereocontrol was the subject of a review [376] Arenechromium tricarbonyl complexes were lithiated then alkylated by allyl bromide (equation 314) [377] Lithiation of (η^6-1-triisopropylsiloxy-3-methoxybenzene) chromium tricarbonyl occurred at C-2 not C-4 as expected [378] Chromium complexed α-phenethyl amine underwent directed orthometallation (equation 315) [379] A high degree of stereocontrol was manifest (equation 316) [380]. Chromium complexed benzocyclobutanes were regiospecifically lithiated and alkylated (equation 317) [381] Arenechromium tricarbonyl complexes were used in the synthesis of 6,7-benzomorphans [382]

(Equation 314)



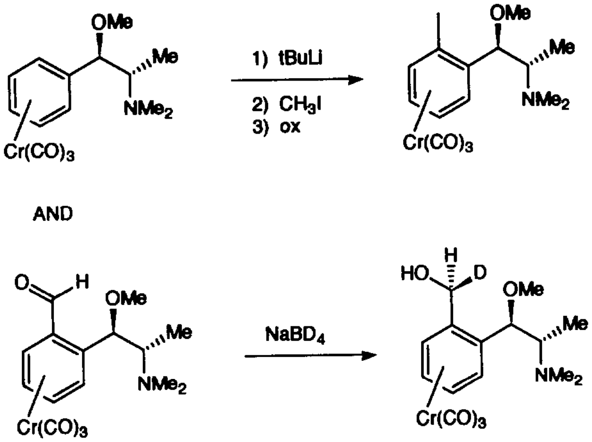
(Equation 315)



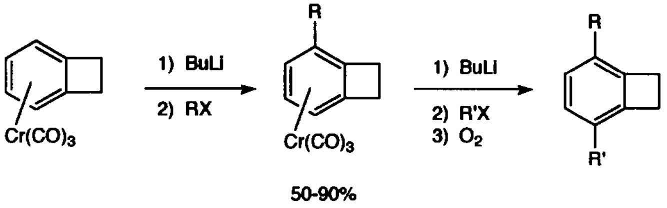
≥98% de, 40-80% yield

E+ = D, Me, Me_3Si, PPh_2, PhS

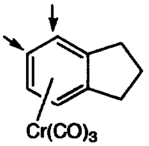
(Equation 316)



(Equation 317)



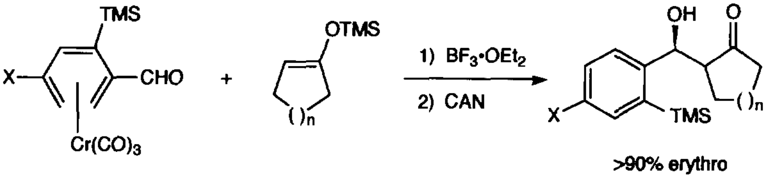
R = TMS, D, Me, I, CHO, CO_2Me



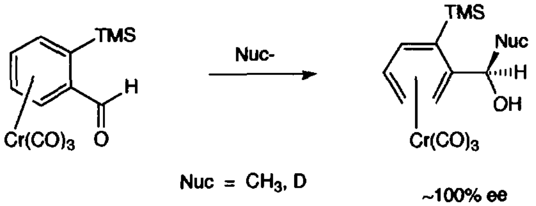
1:2 with BuLi
1:11 with TMPLi

Chromium complexed aryl aldehydes were alkylated with a high degree of stereoselectivity (equation 318) [383], (equation 319) [384], (equation 320) [385], (equation 321) [386]. Arene complexes having benzyl hydrogens alkylated formaldehyde (equation 322) [387] and bromoacetic acid (equation 323) [388] The stability of chromium complexed benzyl cations was used in a cyclization reaction (equation 324) [389]. Chromium complexed benzocyclobutanes were in equilibrium with the diene form and underwent cycloaddition reactions (equation 325) [390].

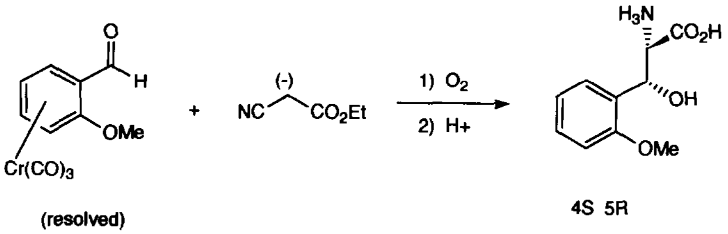
(Equation 318)



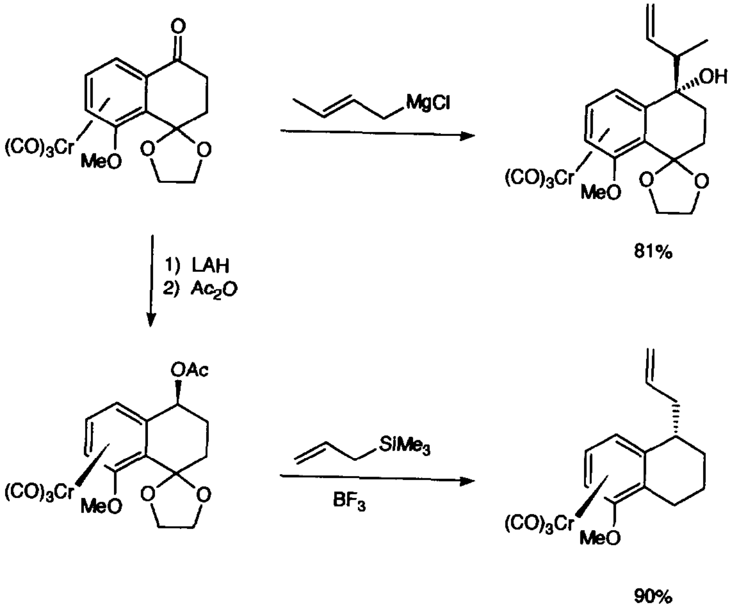
(Equation 319)



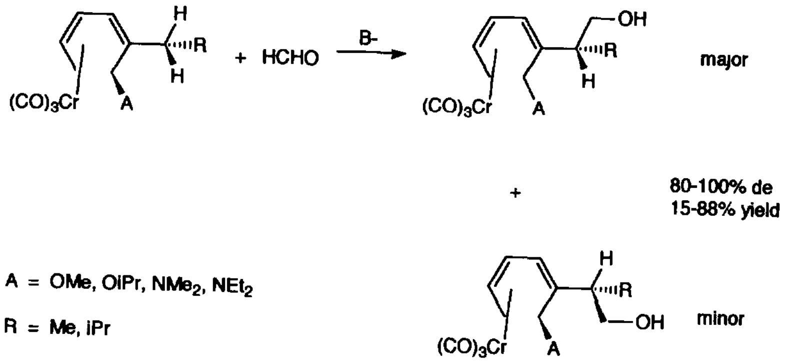
(Equation 320)



(Equation 321)



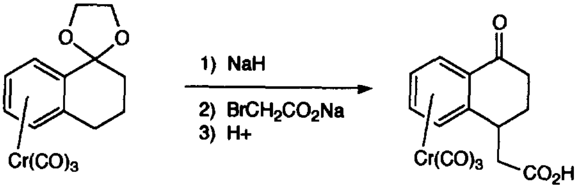
(Equation 322)



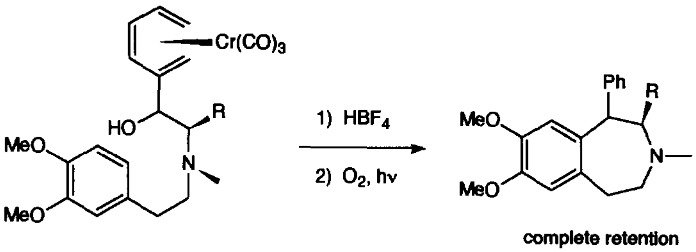
A = OMe, OiPr, NMe_2, NEt_2

R = Me, iPr

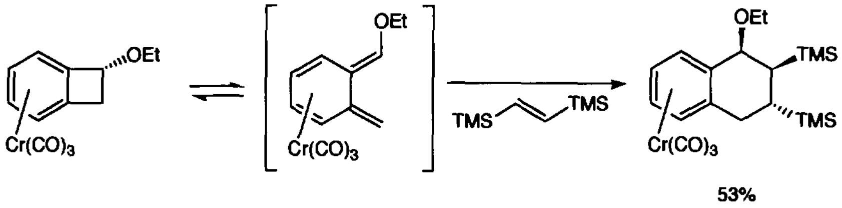
(Equation 323)



(Equation 324)

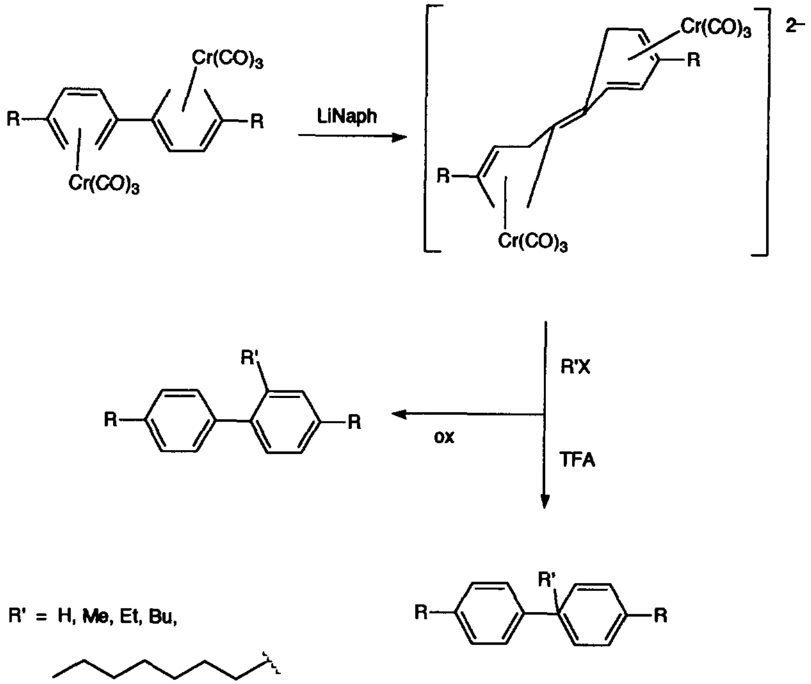


(Equation 325)

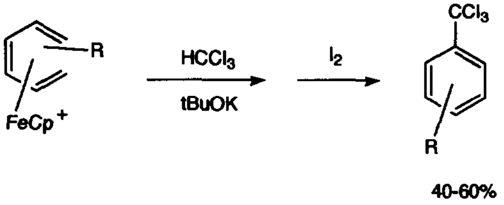


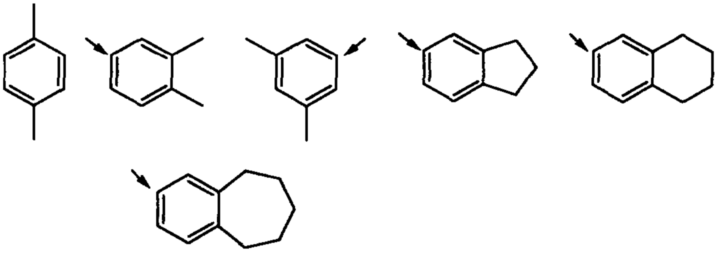
Bis complexed biphenyls were reduced then alkylated by halides (equation 326) [391]. Iron arene complexes were alkylated by chloroform (equation 327) [392] and were reduced, alkylated and acylated (equation 328) [393]

(Equation 326)

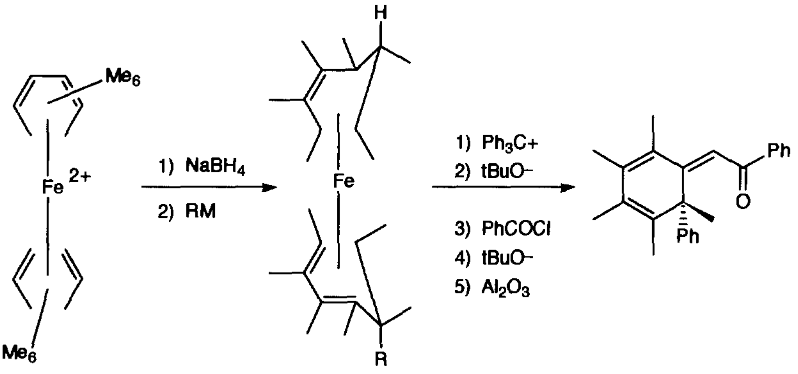


(Equation 327)





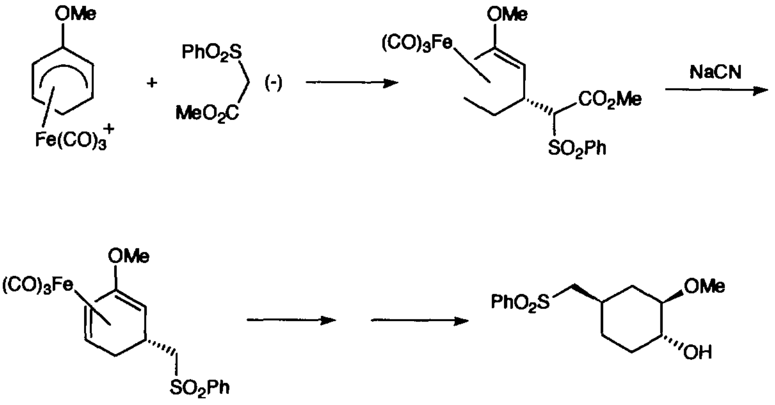
(Equation 328)



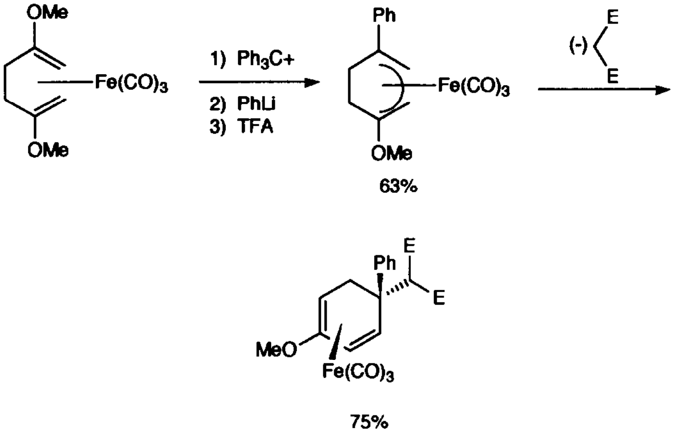
12 Alkylation of Dienyl and Diene Complexes

The alkylation of cyclohexadienyliron complexes continues to be developed for use in organic synthesis (equation 329) [394], (equation 330) [395], (equation 331) [396], (equation 332) [397] Dienyl complexes with external olefins underwent attack at either the ring or the side chain depending on conditions (equation 333) [398]. Cationic molybdenum diene complexes have also been utilized to functionalize cyclohexyl systems (equation 334), cycloheptyl systems (equation 335) [399], and pyran systems (equations 336 and 337) [400]

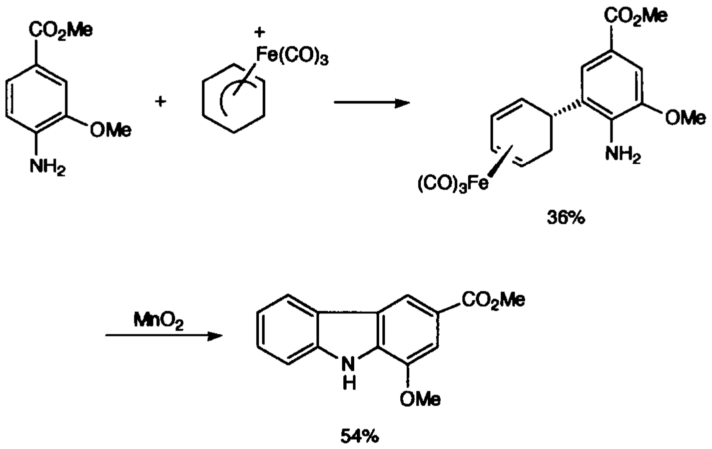
(Equation 329)



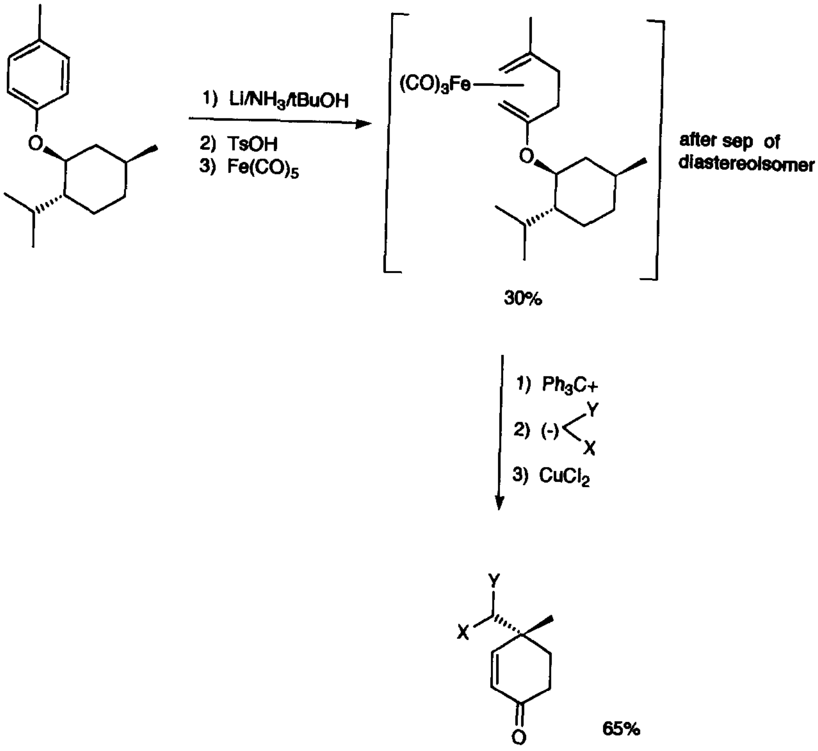
(Equation 330)



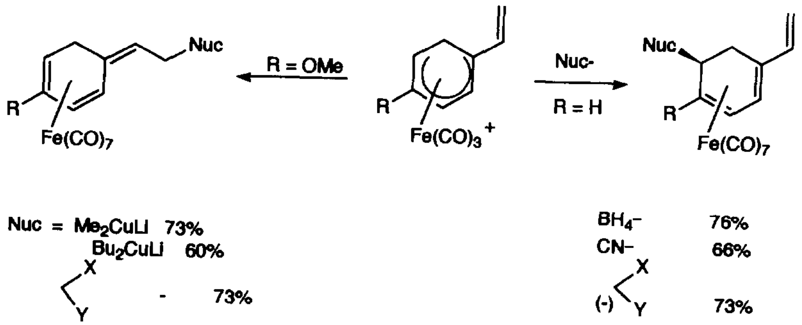
(Equation 331)



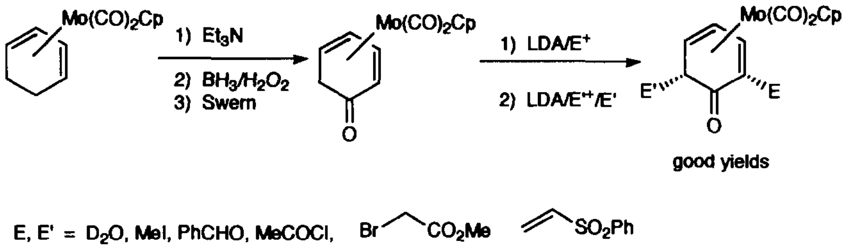
(Equation 332)



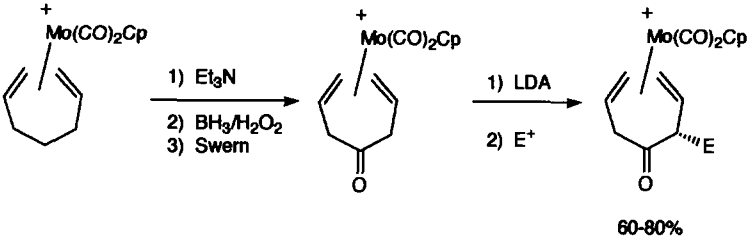
(Equation 333)



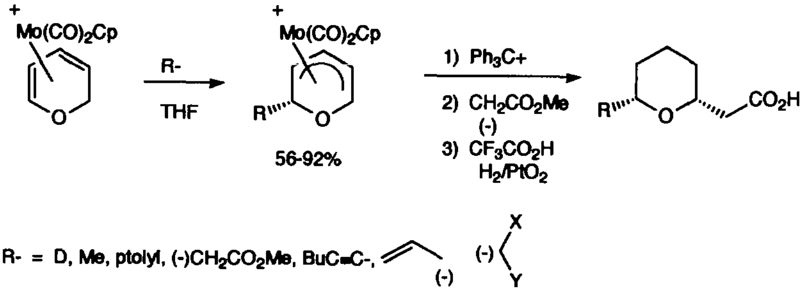
(Equation 334)



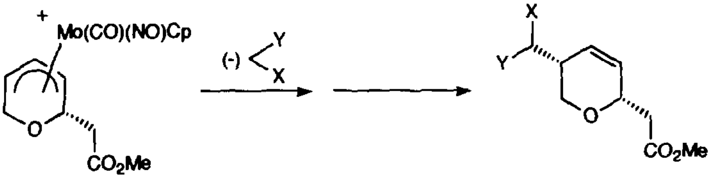
(Equation 335)



(Equation 336)

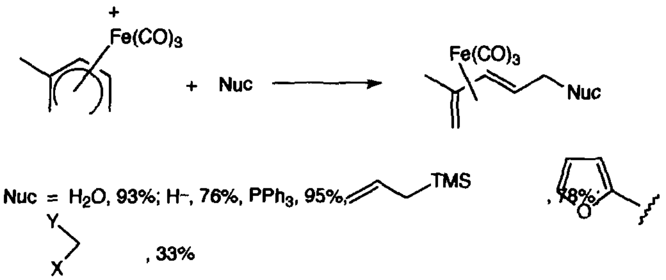


(Equation 337)

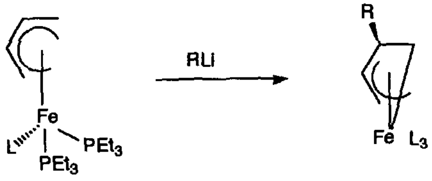


Acyclic dienyliron complexes also underwent attack by nucleophiles (equation 338) [401], (equation 339) [402], (equation 340) [403].

(Equation 338)



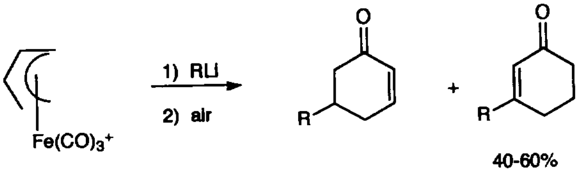
(Equation 339)



R = Me, tBu

L = CO, tBuNC

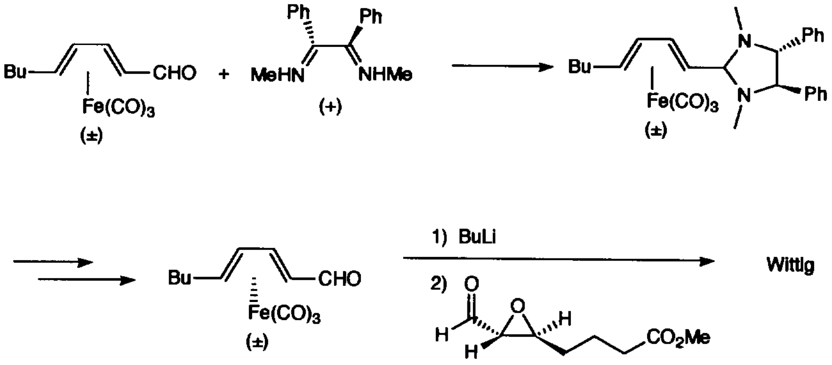
(Equation 340)



R = Me, Bu, Ph

Iron-complexed diene aldehydes were extensively elaborated (equation 341) [404], (equation 342) [405], (equation 343) [406], (equation 344) [407].

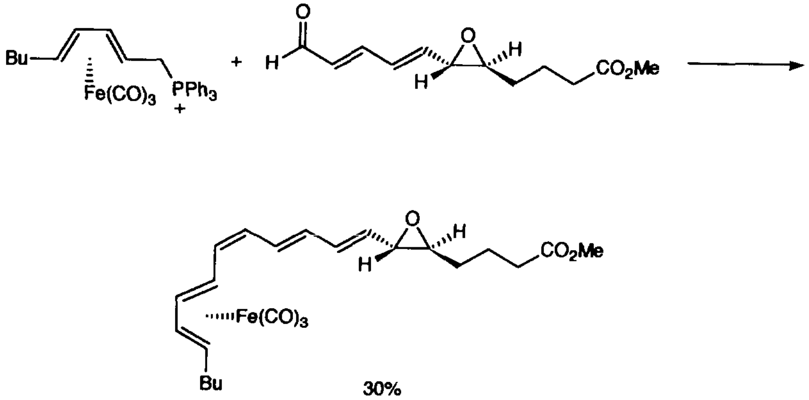
(Equation 341)



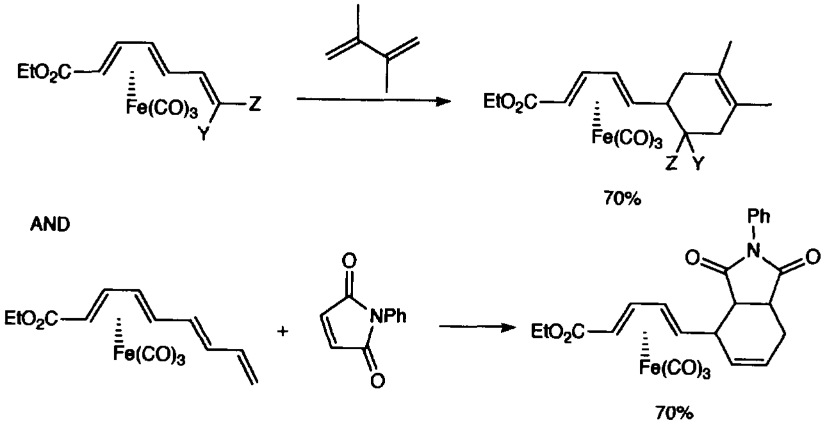
2S 5R

high selectivity for matched pair

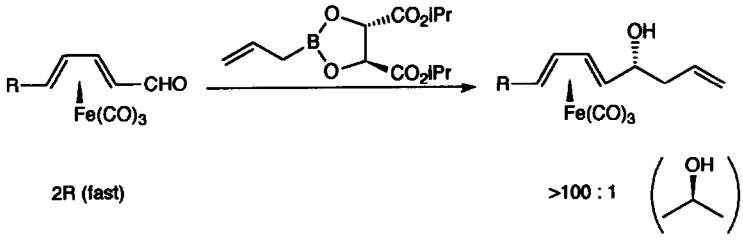
(Equation 342)



(Equation 343)

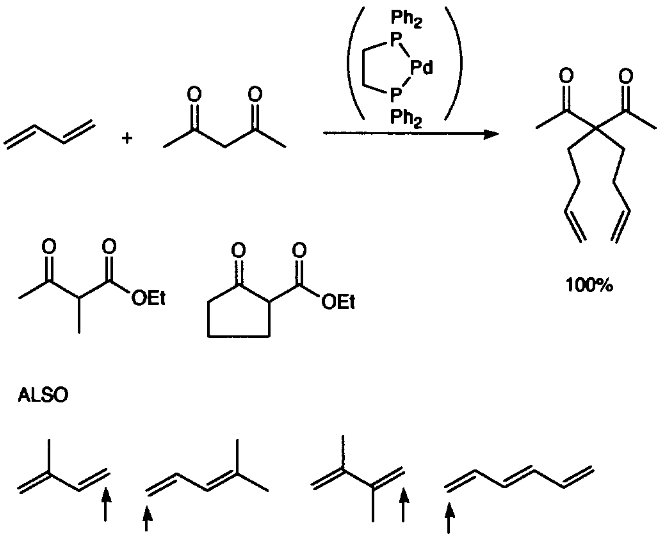


(Equation 344)

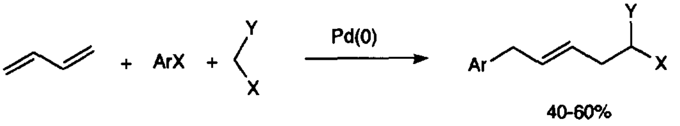


Palladium catalyzed the bis alkylation of b-dicarbonyl compounds by dienes (equation 345) [408], as well as the arylation/alkylation of dienes (equation 346) [409] Iron complexed enones underwent reaction with methyllithium to give vinyl ketene complexes which reacted with isonitriles to give vinyl ketenimine complexes (equation 347) [410], (equation 348) [411].

(Equation 345)



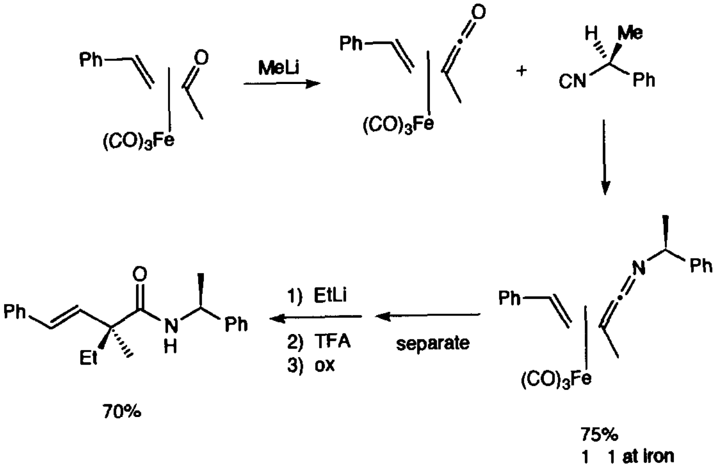
(Equation 346)



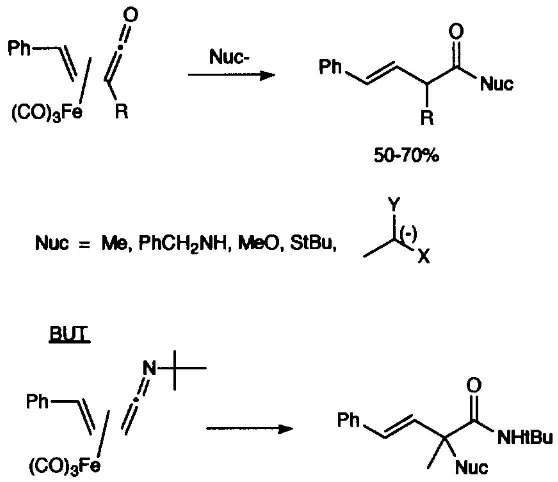
Ar = Ph, pMePh, mMePh, pClPh

X, Y = CN, CO_2Et

(Equation 347)



(Equation 348)

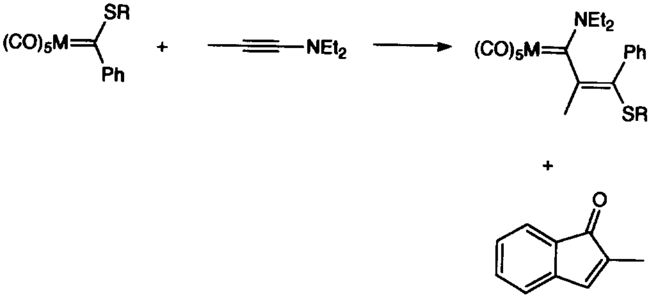


13. Metal Carbene Reactions

A large number of reviews on carbene complexes in organic synthesis have been published. These include "Carbene complexes in catalysis" (83 references) [412], "Transition metal complexes of unsaturated carbenes - synthesis structure and reactivity" (280 references) [413]; "The role of metals in carbene synthon introduction" (37 references) [414], "Carbene complexes in stereoselective cycloaddition reactions" (52 references) [415]; "Aminocarbene complexes" (23 references) [416], "Metal mediated cyclization of alkynes and carbenes - a new route to highly substituted cyclopentanoids" (8 references) [417]; "Chromium carbene complexes in the synthesis of molecules of biological interest" (18 references) [418]; "Alkene carbene complexes of tungsten and chromium Their reactions with alkynes" (32 references) [419], and "Carbene complexes derived from the activation of isocyanides and alkynes by electron-rich metal centers" (77 references) [420], and "Metal carbene complexes from alkynes" [421].

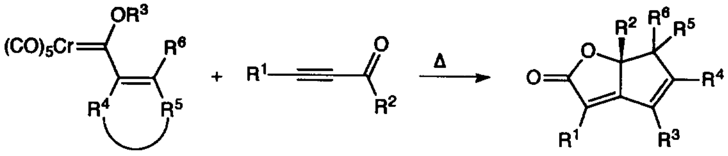
An optimized procedure for the synthesis of chromium aminocarbene complexes from amides and $K_2Cr(CO)_5$ has appeared [422]. Tungsten thiocarbenes underwent reaction with ynamines to give low yields of indenones (equation 349) [423]

(Equation 349)



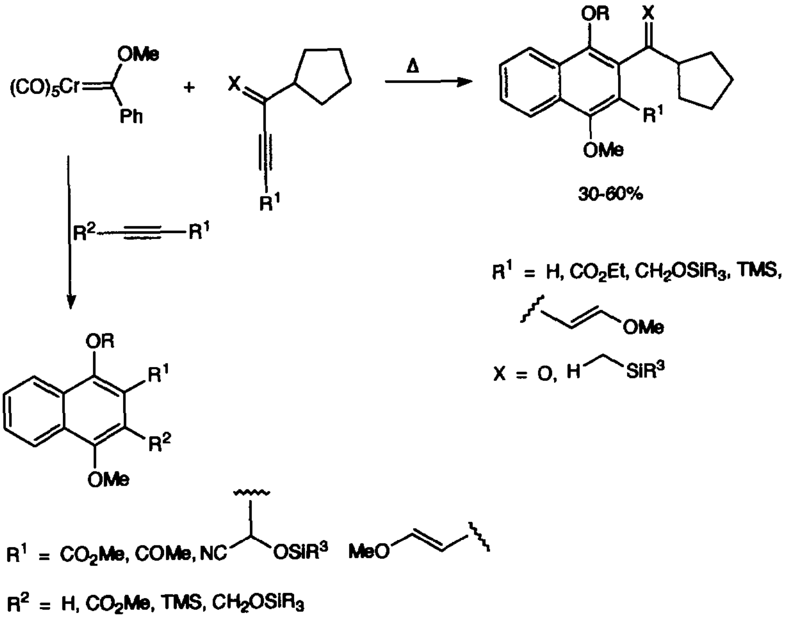
The reactions of unsaturated chromium carbene complexes with alkynes continues to produce an array of complex cyclic compounds (equation 350) [424], (equation 351) [425], (equation 352) [426], (equation 353) [427], (equation 354) [428], (equation 355) [429].

(Equation 350)

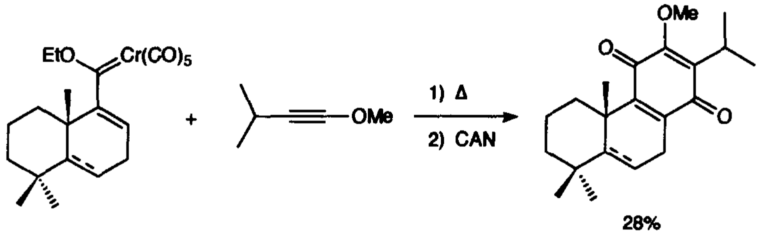


30-60%

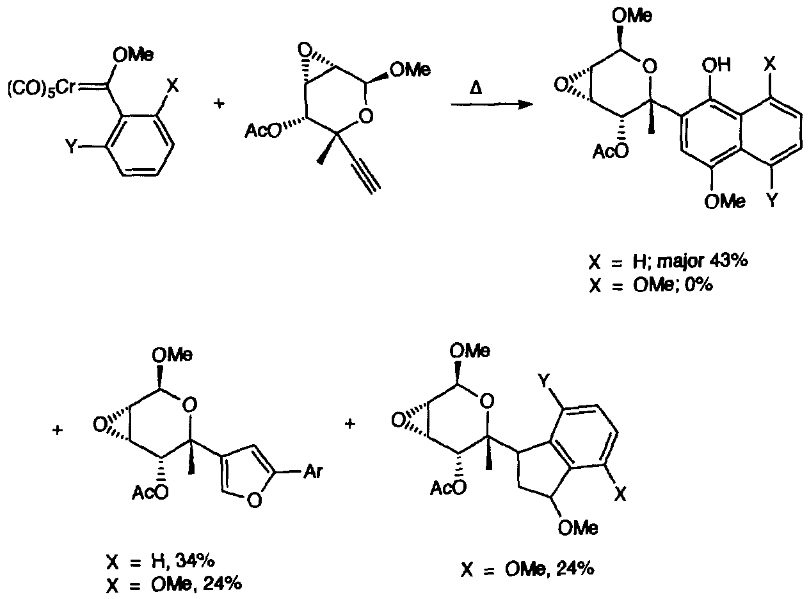
(Equation 351)



(Equation 352)



(Equation 353)

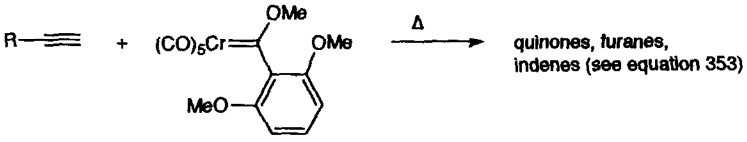


X = H; major 43%
X = OMe; 0%

X = H, 34%
X = OMe, 24%

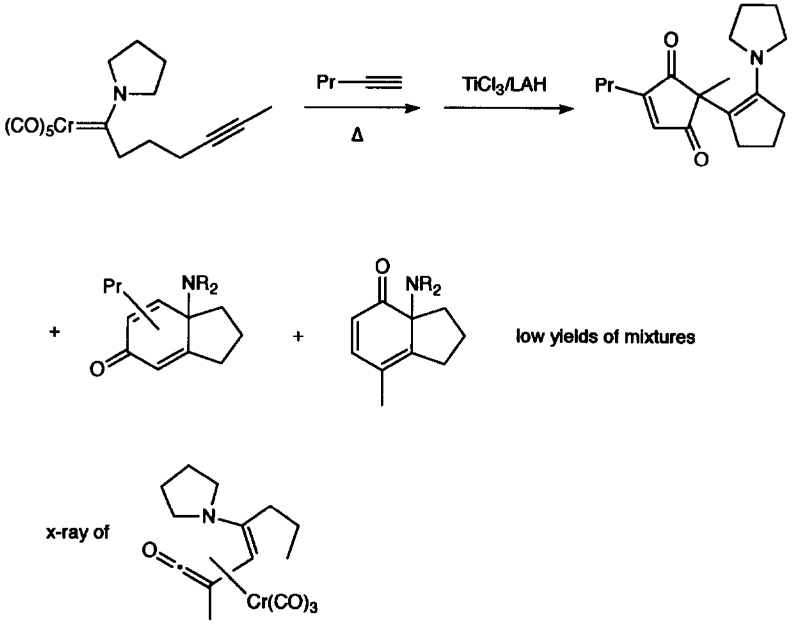
X = OMe, 24%

(Equation 354)



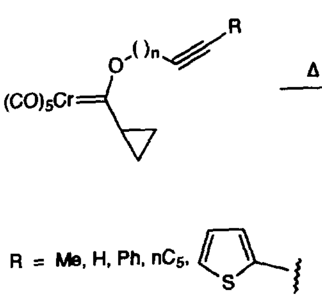
If R = O, furans, indenes predominate

(Equation 355)

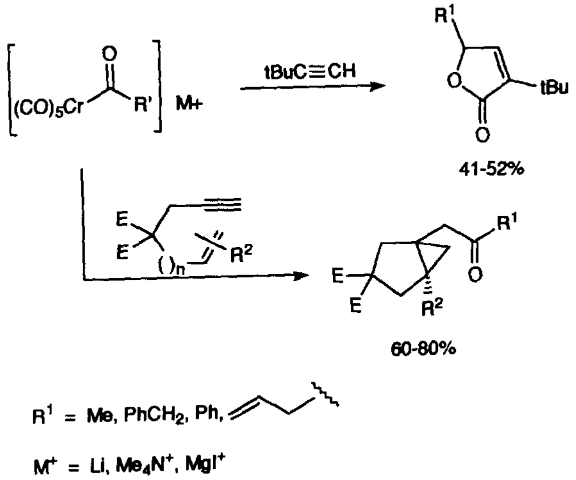


Cyclopropyl carbenes having acetylenic oxo groups formed bicyclic compounds upon thermal reaction (equation 356) [430]. Chromium acyl compounds also underwent reaction with alkynes (equation 357) [431]. Tetraalkoxy olefins underwent a 2+2 cycloaddition to the side chain of alkynyl carbenes (equation 358) [432] Photolysis of chromium alkoxy carbenes with olefins produced cyclobutanones (equation 359) [433] Photolysis of alkoxycarbenes with isoindoles gave dimeric compounds (equation 360) [434].

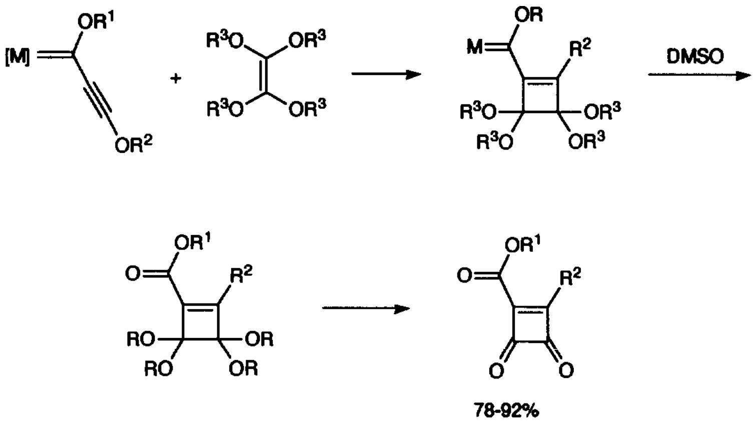
(Equation 356)



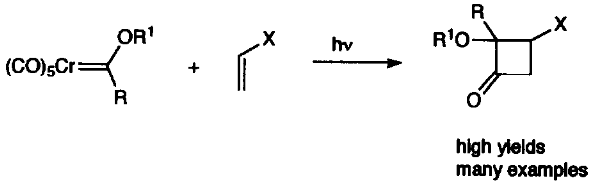
(Equation 357)



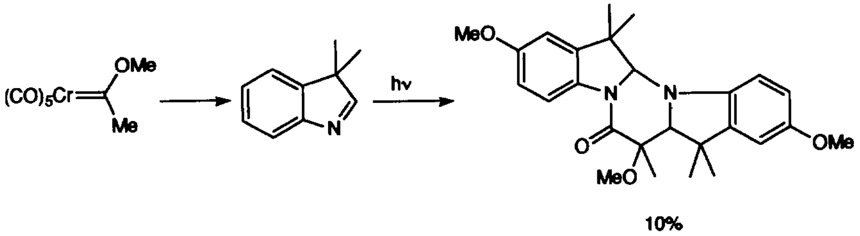
(Equation 358)



(Equation 359)

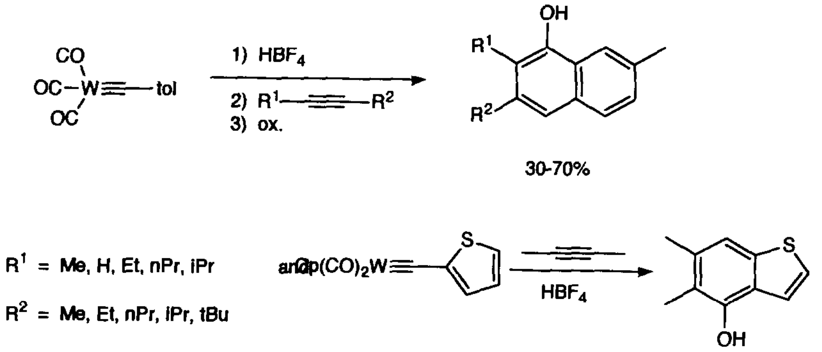


(Equation 360)

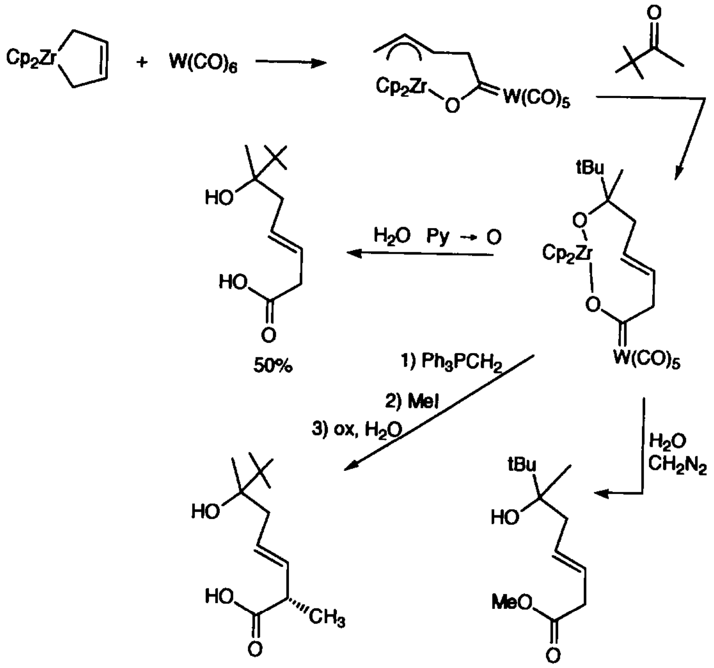


Tungsten carbyne complexes were annulated with alkynes to produce naphthols (equation 361) [435] Carbon-carbon bond formation by means of zirconocene-derived carbene complexes has been reviewed (16 references) [436] This chemistry permits the useful elaboration of the diene starting materials (equation 362) [437] [438]

(Equation 361)



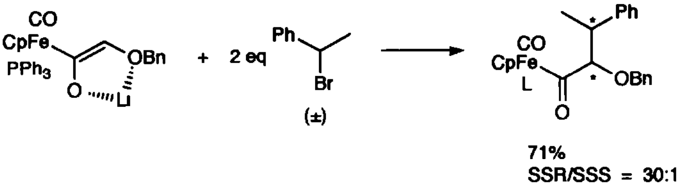
(Equation 362)



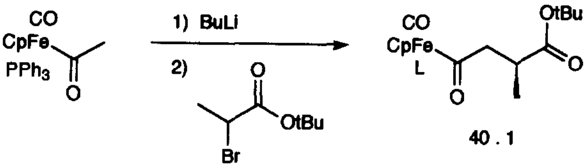
14 Alkylation of Metal Acyl Enolates

The two examples of use of this chemistry are shown in equation 363 439] and equation 364 [440]

(Equation 363)



(Equation 364)

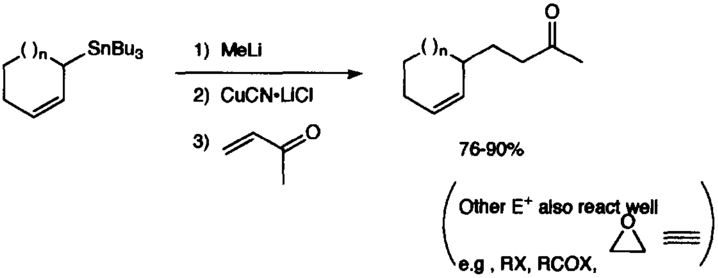


40.1

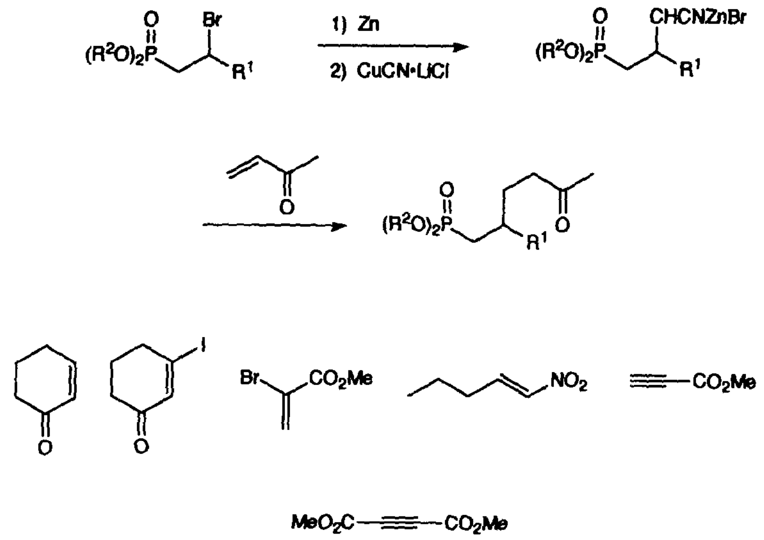
B. Conjugate Addition

Organocuprates remained the reagents of choice for conjugate addition reactions, and both new reagents and new applications continue to evolve. Cyclic allyl cuprates could not be made from tin precursors but exchange with lithium gave allyl cuprates which were efficient nucleophiles (equation 365) [441] Phosphorous-containing cuprates added 1,4- to enones (equation 366) [442]. Allyl copper/trimethyl silyl chloride was an efficient 1,4-alkylation reagent (equation 367) [443] High 1,4-selectivity was achieved in copper-catalyzed addition of Grignards to enones in the presence of trimethylsilyl chloride (equation 368) [444], (equation 369) [445] Unsaturated lactams also underwent 1,4-addition of organocopper reagents (equation 370) [446]

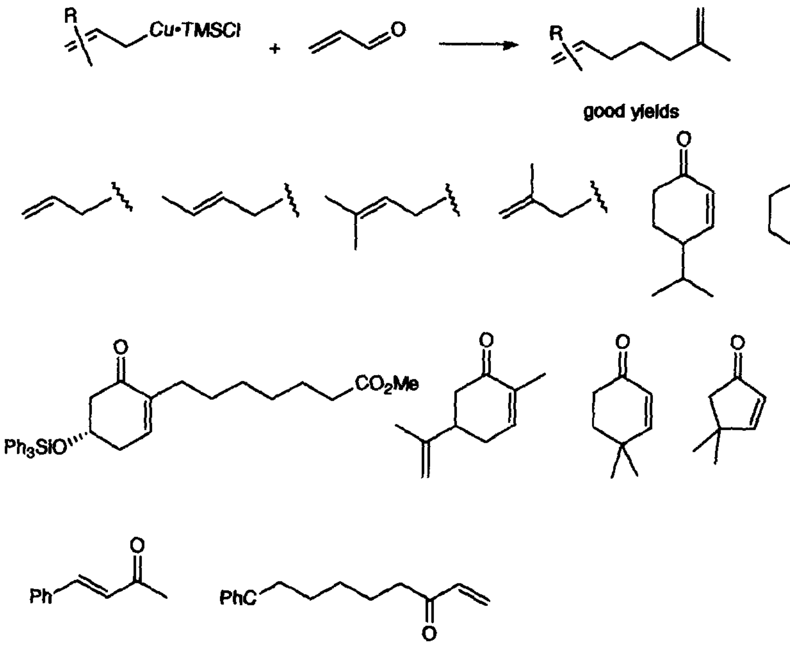
(Equation 365)



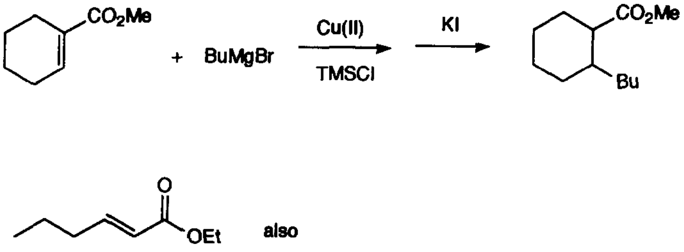
(Equation 366)



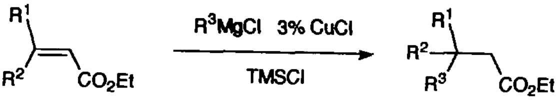
(Equation 367)



(Equation 368)



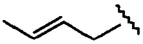
(Equation 369)



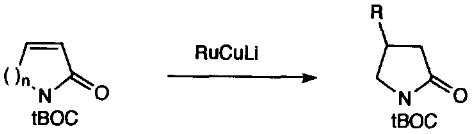
good yields

R^1 = H, Me

R^2 = Me, Pr, iPr, Ph

R^3 = Me, Bu, iPr, tBu, Ph, 

(Equation 370)



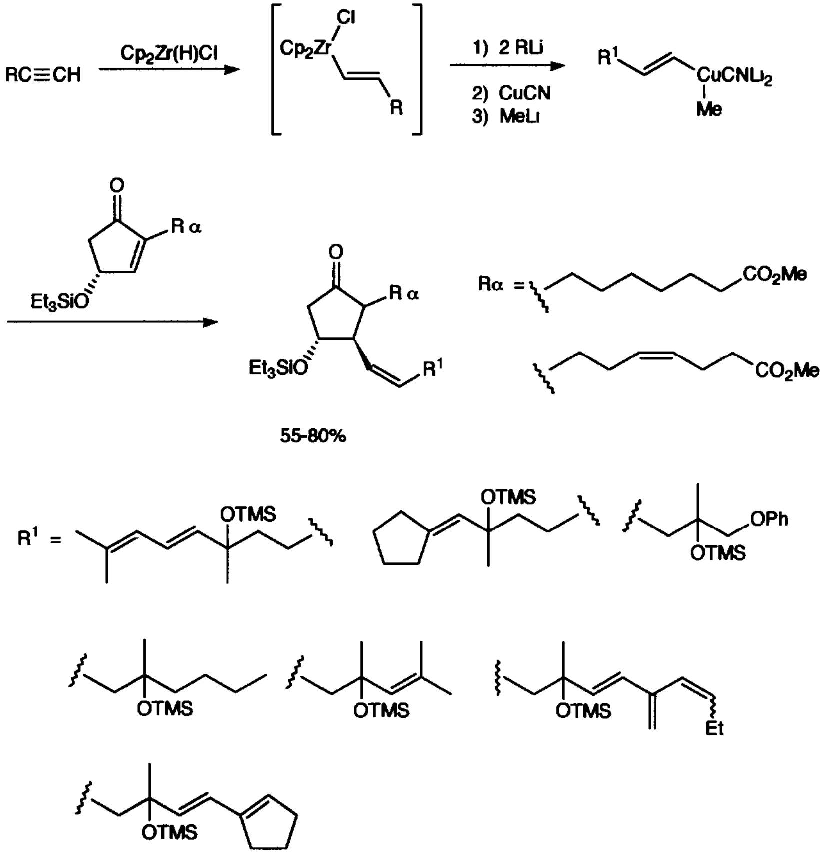
62-84%

R = Ph, Me, Bu_3Sn, $PhMe_2Si$

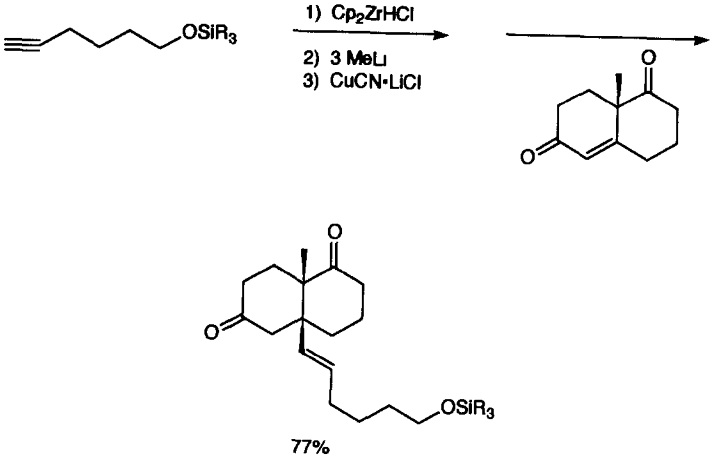
n = 1, 2, 3

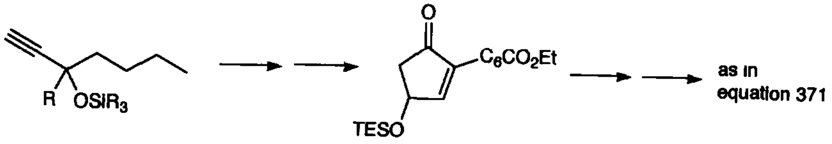
Vinylcuprates were readily derived from vinyl zirconium (equation 371) [447], (equation 372) [448], or aluminum reagents (equation 373) [449] and these reagents added cleanly in a 1,4-fashion to enones

(Equation 371)

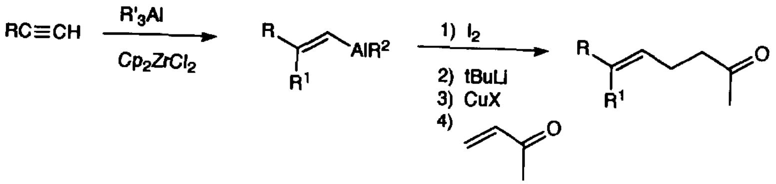


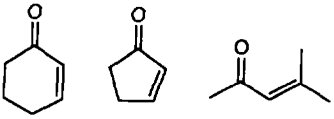
(Equation 372)





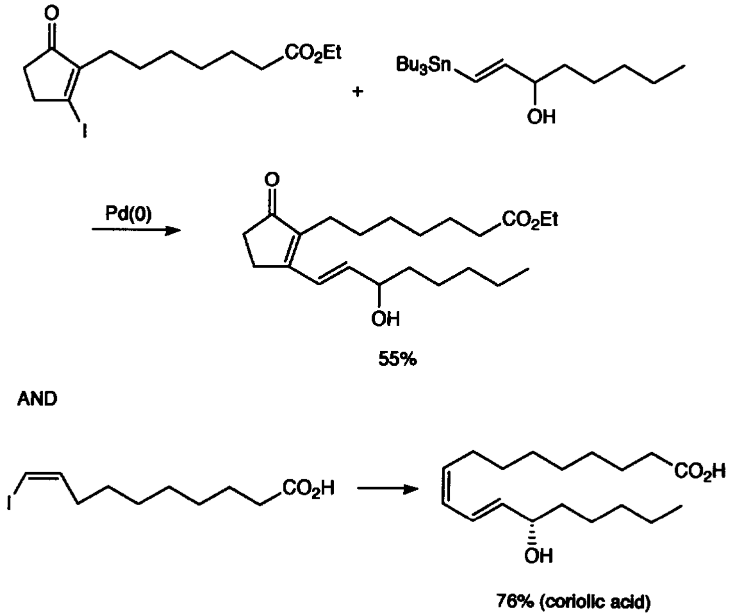
(Equation 373)



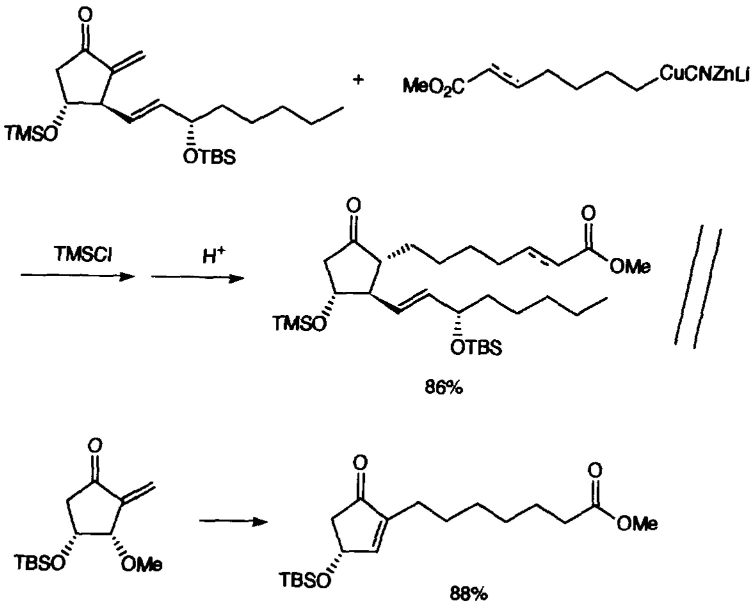


Conjugate addition reactions were central to the synthesis of prostanoids (equation 374) [450], (equation 375) [451], polycyclic compounds (equation 376) [452], (equation 377) [453], precapnellene (equation 378) [454], and steroids (equation 379) [455].

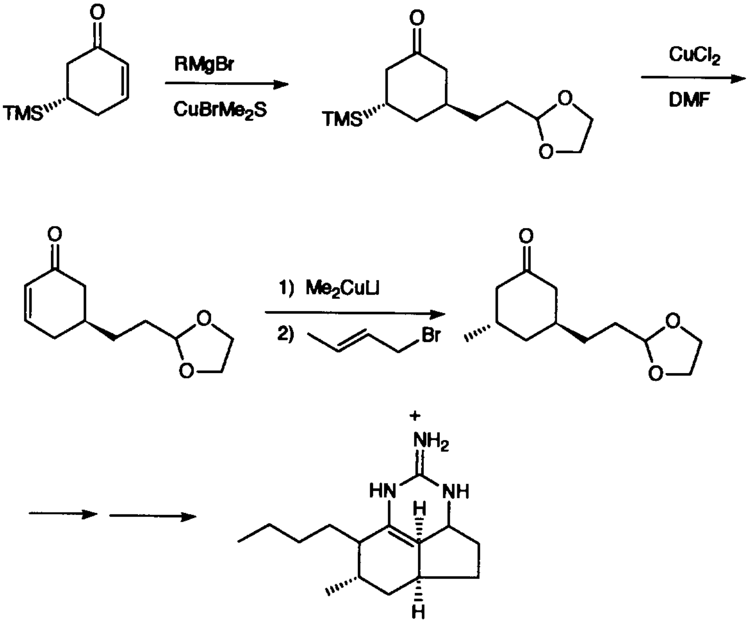
(Equation 374)



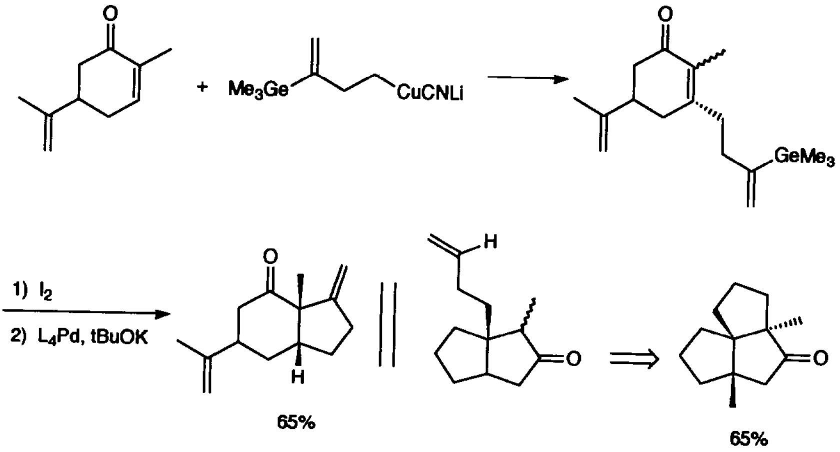
(Equation 375)



(Equation 376)

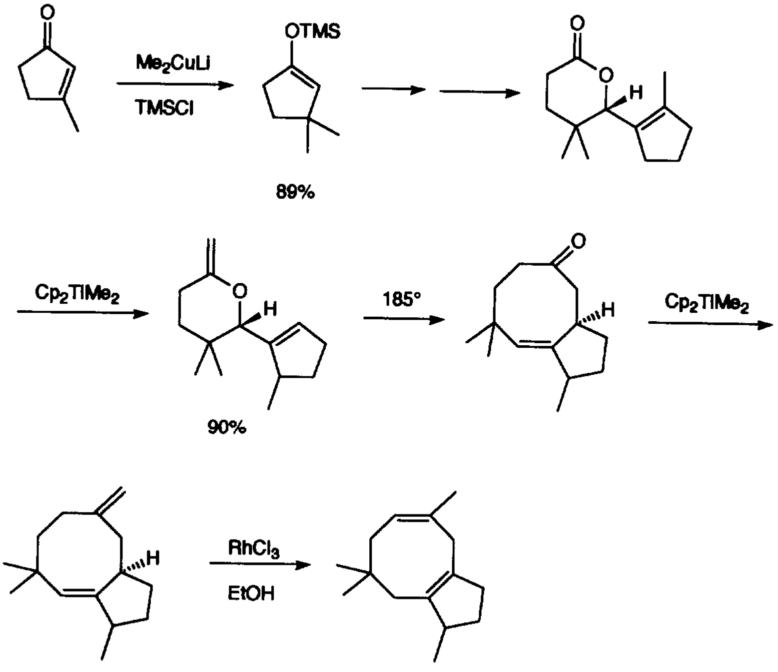


(Equation 377)



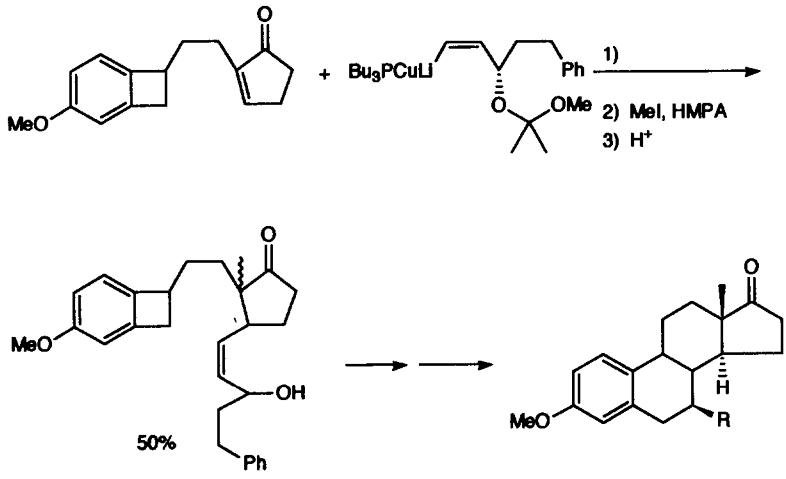
p 643

(Equation 378)



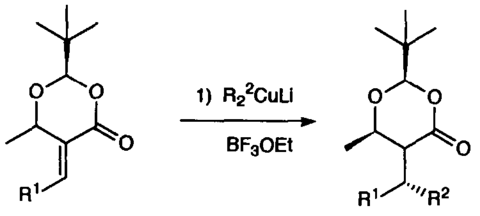
(±)-Precapnellene

(Equation 379)



Methods to control stereochemistry in the conjugate additions of cuprates warranted extensive studies. Several approaches were fruitful. Control of relative stereochemistry was achieved by constricting the enone into a rigid, hindered cyclic system (equation 380) [456] [457] [458], (equation 381) [459] or by the use of bulky ester groups (equation 382) [460].

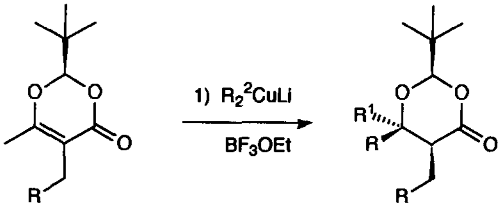
(Equation 380)



50-70%
10.1 anti/syn

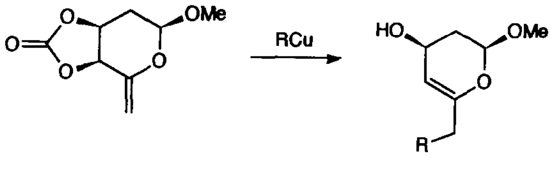
R^1 = Ph, Me, Et, Bu, H

R^2 = Me, Pr, Bu, C_6, Ph, 

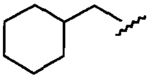


40-60%

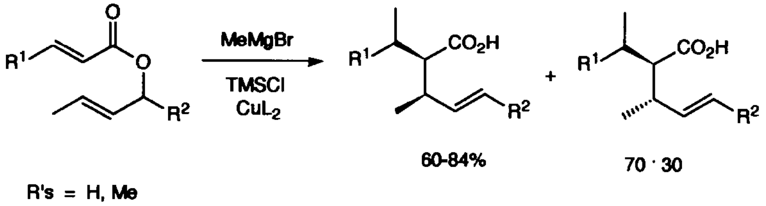
(Equation 381)



60-90%

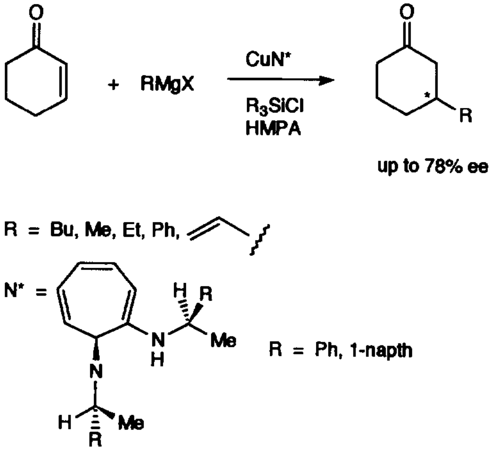
R = Me, Bu, tBu, Ph, $PhCH_2$, 1-naph, 

(Equation 382)

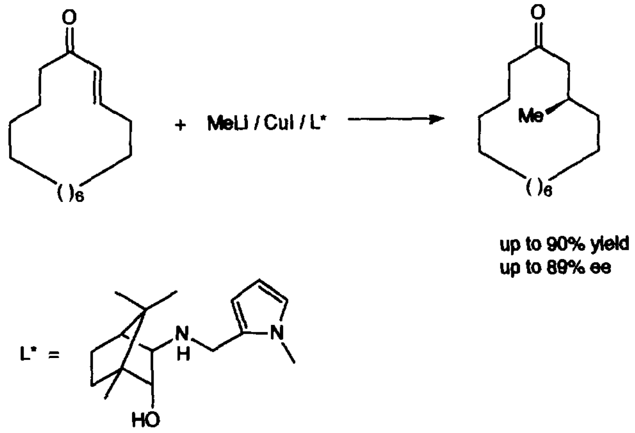


Asymmetric induction was achieved by the use of chiral ligands (equation 383) [461], (equation 384) [462], (equation 385) [463] More commonly, a chiral auxiliary somewhere in the substrate was utilized. This could be on the carbonyl group (equation 386) [464], (equation 387) [465], (equation 388) [466], α- to the carbonyl group (equation 389) [467], γ- to the carbonyl group (equation 390) [468], (equation 391) [469], (equation 392) [470], (equation 393) [471], (equation 394) [472], or δ- to the carbonyl group (equation 395) [473]. The use of chiral sulfoxides also induced asymmetry (equation 396) [474]

(Equation 383)

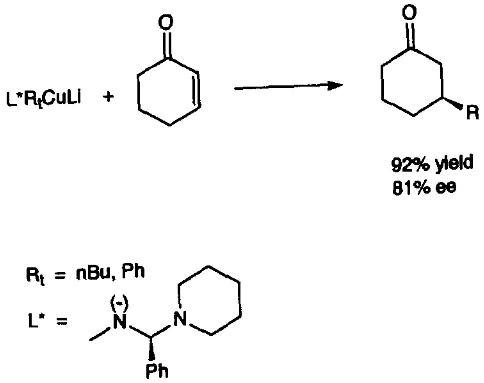


(Equation 384)



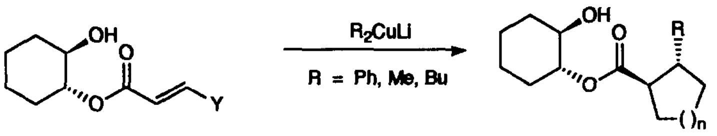
up to 90% yield
up to 89% ee

(Equation 385)



92% yield
81% ee

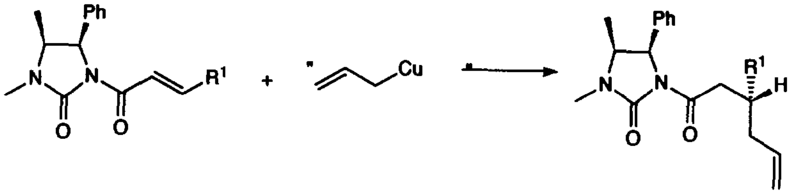
(Equation 386)



Y = $(CH_2)_3Cl$; $(CH_2)_4I$

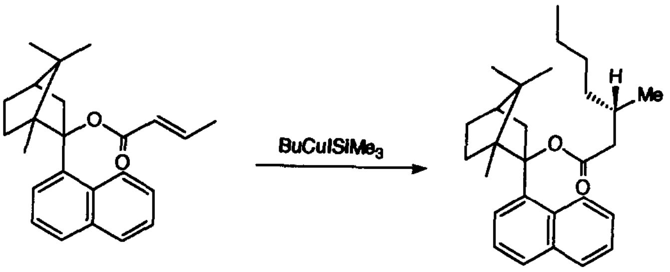
15-50%
~90 : 10

(Equation 387)



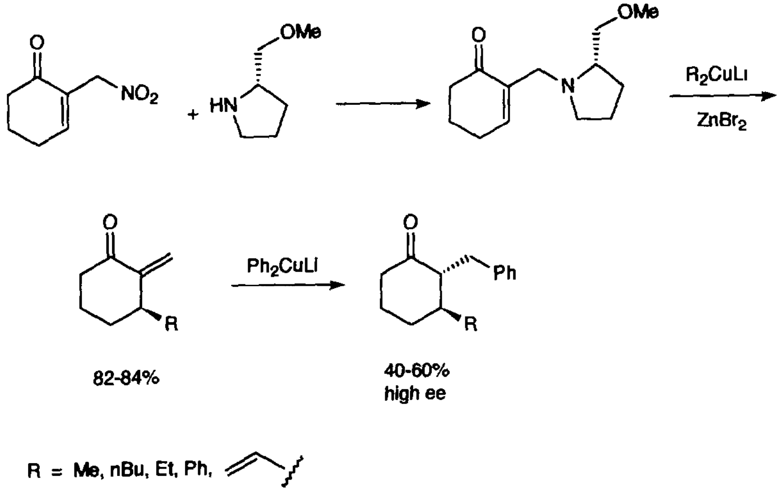
82-87% yield
97% de

(Equation 388)

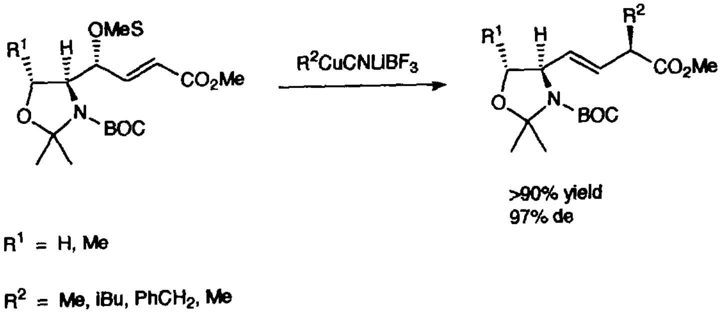


93%
98% de

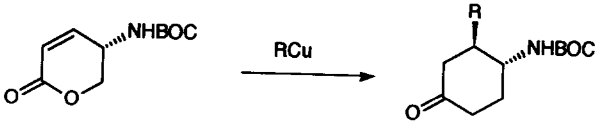
(Equation 389)



(Equation 390)



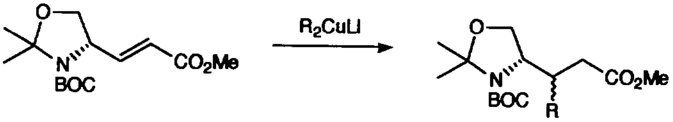
(Equation 391)



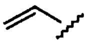
R = Pr, Me, Bu

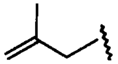
40-80% yield
>99% trans (R)(S)

(Equation 392)

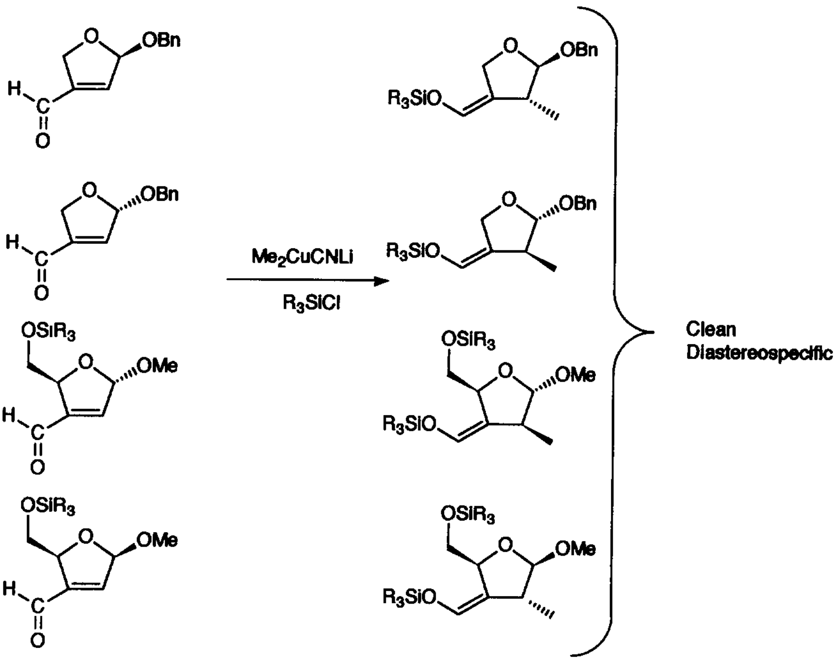


60-98%
from 70 : 30 to 97 3

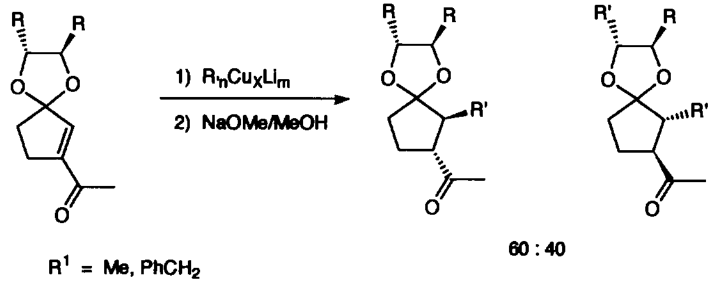
R = Me, Pr, Ph, $PhCH_2$, 



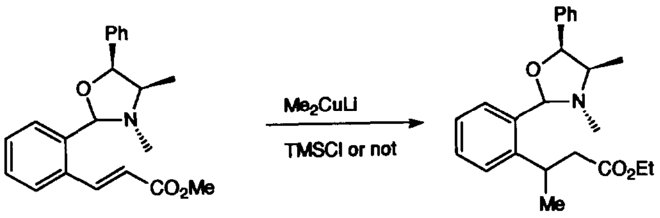
(Equation 393)



(Equation 394)

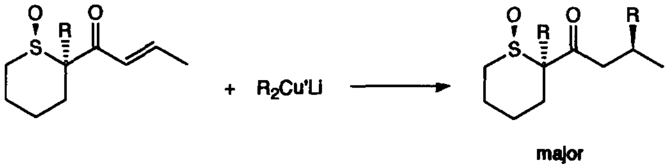


(Equation 395)



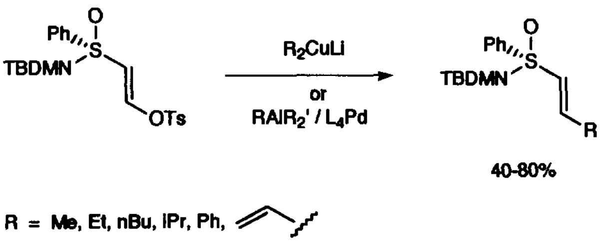
with TMSCl 61% yield, 38% ee (R)
without TMSCl 43% yield, 93% ee (S)

(Equation 396)

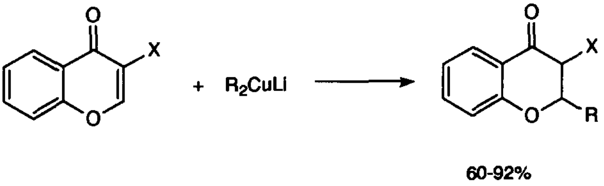


Cuprates underwent addition-elimination with β-tosyl sulfoxides (equation 397) [475], but added to other substituted enone systems (equation 398) [476], (equation 399) [477], (equation 400) [478]

(Equation 397)

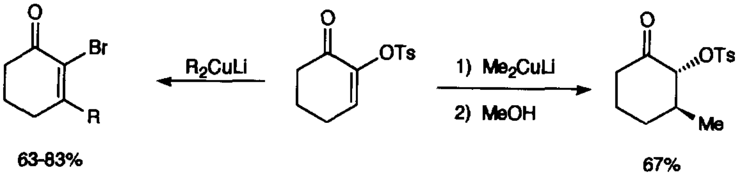


(Equation 398)



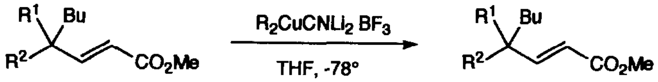
X = CHO, COMe, COPh, CO$_2$Me, CN

(Equation 399)

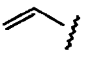


R = Me, Et, nBu, sBu, Ph

(Equation 400)



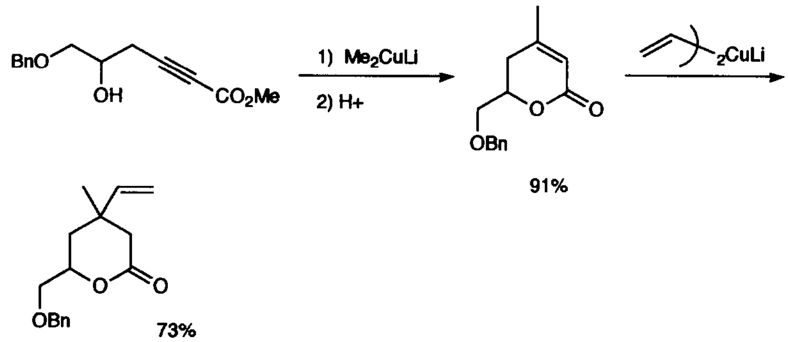
R^1, R^2 = H, Me

R = Me, nBu, Ph, Me$_2$PhSi, 

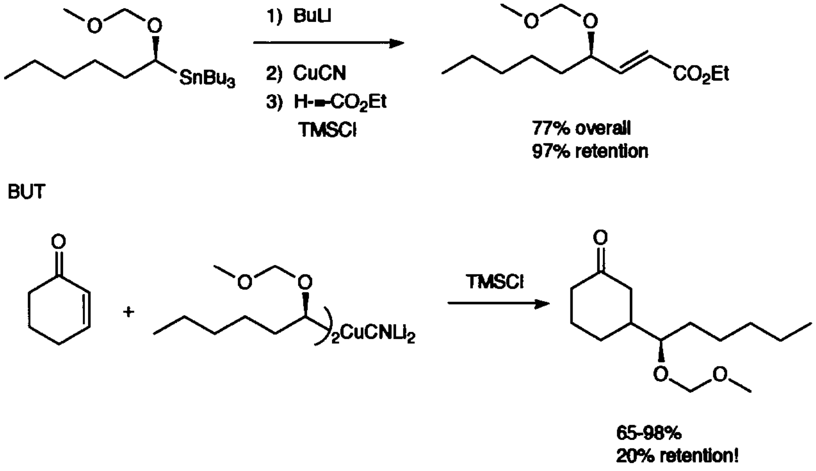
Ynones (equation 401) [479], (equation 402) [480], ynenones (equation 403) [481], (equation 404) [482], methylenecyclopropyl ketones (equation

405) [483] and epoxyenones also underwent conjugate addition with copper reagents (equation 406) [484].

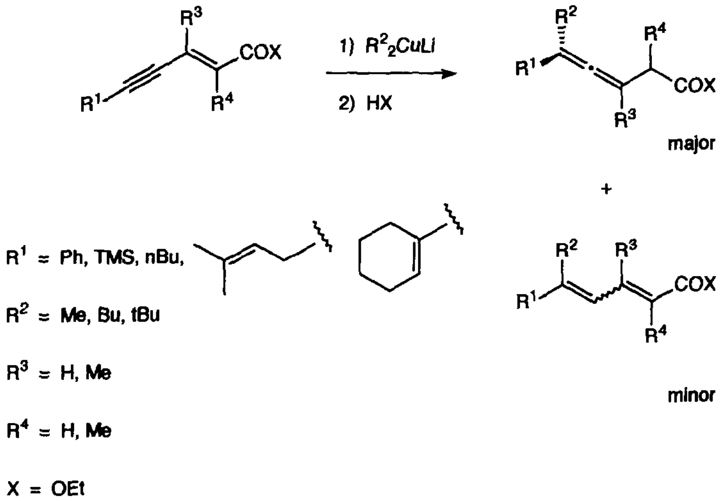
(Equation 401)



(Equation 402)



(Equation 403)



R¹ = Ph, TMS, nBu,

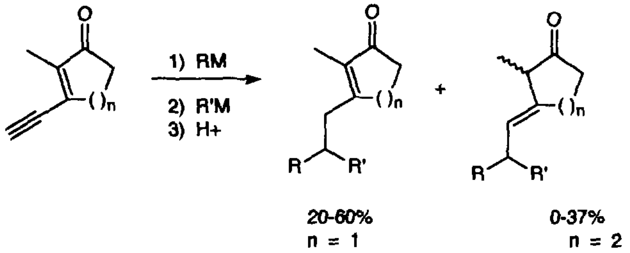
R² = Me, Bu, tBu

R³ = H, Me

R⁴ = H, Me

X = OEt

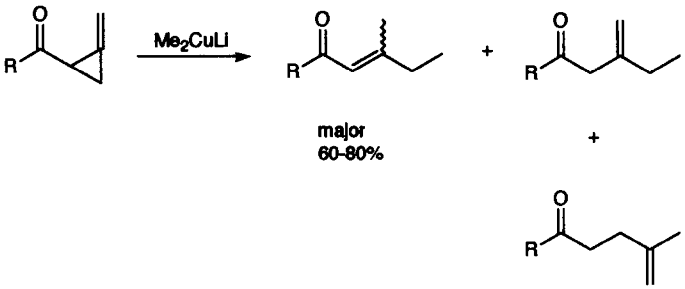
(Equation 404)



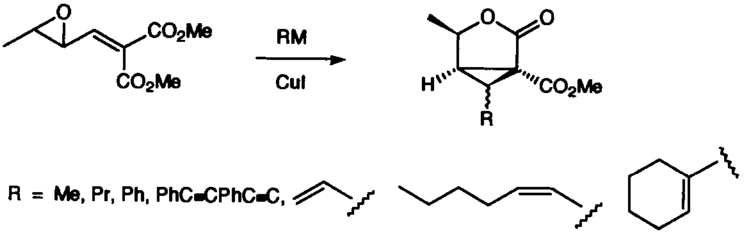
RM = $Me_2CuCNLi_2$, $Ph_2CuCNLi_2$

R'M = $Me_2CuCNLi_2$, MeLi, Ph, Me_2Si, $CuCNLi_2$

(Equation 405)

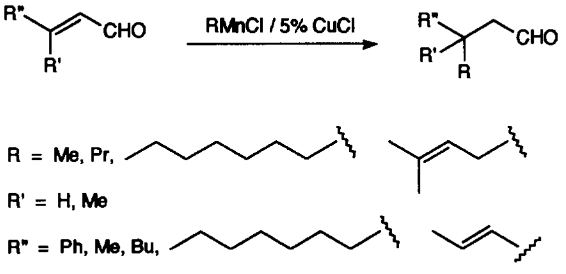


(Equation 406)

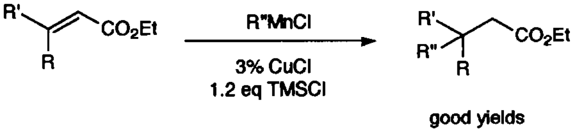


Alkylmanganese compounds added 1,4-to conjugated aldehydes and esters (equation 407) [485], (equation 408) [486] [487] in the presence of copper chloride Cyclohexenone was 1,4-alkylated by alkylmanganese compounds (equation 409) [488] but cyclopentenone, and β-methyl cyclohexenones failed

(Equation 407)



(Equation 408)

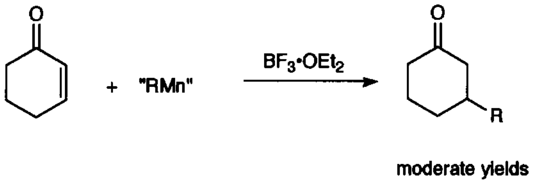


R = H, Me

R' = Me, Pr, iPr, Ph

R" = Me, Bu, iPr, tBu, Ph

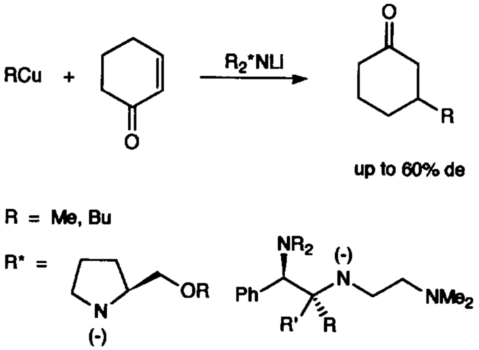
(Equation 409)



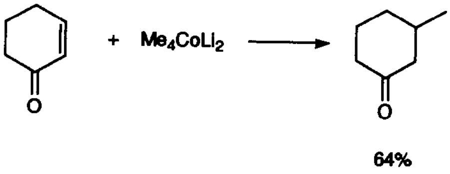
$$\text{RMn} = \begin{array}{l}\text{BuMnCl},\\ \text{Bu}_2\text{Mn},\\ \text{Bu}_3\text{MnLi}\end{array} \xrightarrow[\text{or FeCl}_3]{\text{NiCl}_2} 68\text{-}74\%$$

Cuprates added to cyclohexenone with fair diastereoselectivity in the presence of anions of chiral secondary amines (equation 410) [489]. Methyl cobalt species added to enones in fair yield (equation 411) [490] The reaction of cyclohexenone with alkyl silver, palladium, ruthenium, iron and nickel was thoroughly studied [491]. Nickel(II) catalyzed the conjugate addition of diethyl zinc to enones with fair enantioselectivity in the presence of chiral ligands (equation 412) [492] Cobalt acetylacetonate catalyzed the Michael addition enones to acrylates with low (18%) enantiomeric excess in the presence of chiral diamines [493]. Rhodium(I) complexes catalyzed the conjugate alkylation of enones by terminal alkynes (equation 413) [494]. Aluminum chloride assisted the conjugate addition of allyl tins to a,b-unsaturated iron acyl compounds (equation 414) [495]

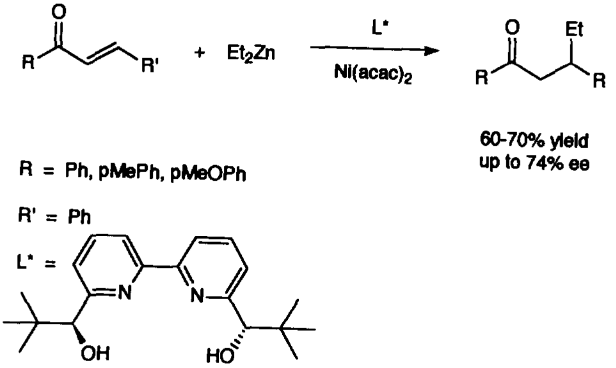
(Equation 410)



(Equation 411)



(Equation 412)

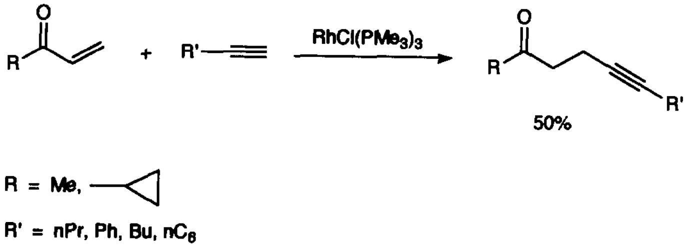


60-70% yield
up to 74% ee

R = Ph, pMePh, pMeOPh

R' = Ph

(Equation 413)

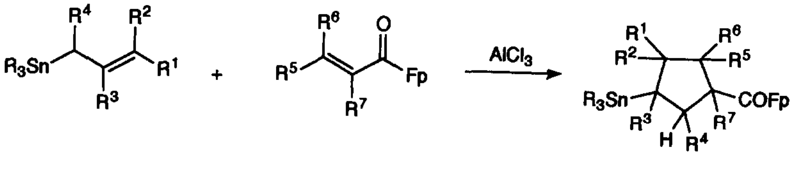


50%

R = Me,

R' = nPr, Ph, Bu, nC$_6$

(Equation 414)



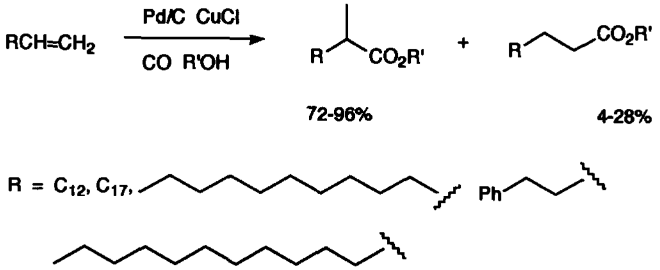
20-60%

C. Acylation Reactions (Excluding Hydroformylation)

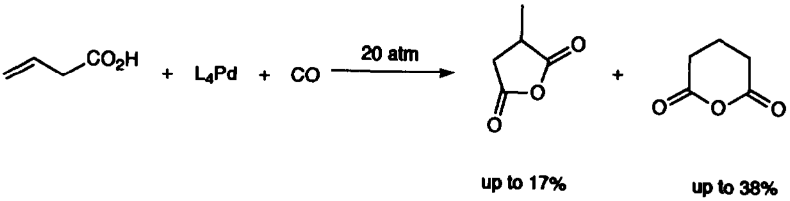
1. Carbonylation of Alkenes and Arenes

The effect of iron(III) chloride and cobalt(II) chloride on the regioselectivity of hydrocarboxylation of olefins by palladium(II) *bis*phosphine complexes was examined [496], as was the effect of temperature on rate and selectivity [497]. Palladium(II) *bis*phosphine complexes catalyzed alkoxycarbonylation of allylbenzene [498]. The role of quaternary ammonium salt cocatalysts in the palladium catalyzed carbonylation of butadiene-methanol telomers was examined [499]. Terminal olefins were alkoxycarbonylated by palladium catalysts to give primarily branched products (equation 415) [500]. Unsaturated acids underwent carbonylative cyclization (equation 416) [501]. Palladium(II) catalyzed the carbonylative cyclization of a,a'-bisallyl ketones (equation 417) [502]. Terminal olefins were alkoxycarbonylated to acrylates and diesters in the presence of palladium(II) catalysts (equation 418) [503] o-Palladated oxazolines of benzoic acids underwent carbonylative coupling (equation 419) [504].

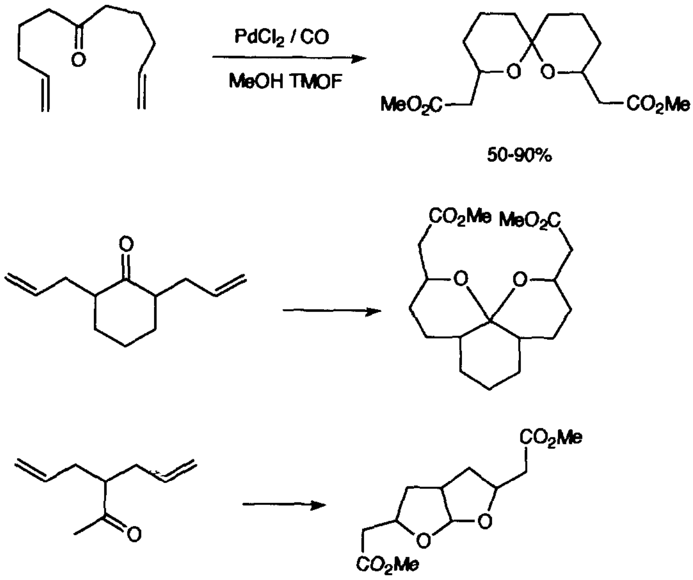
(Equation 415)



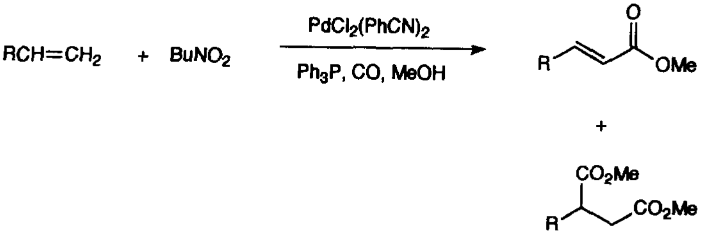
(Equation 416)



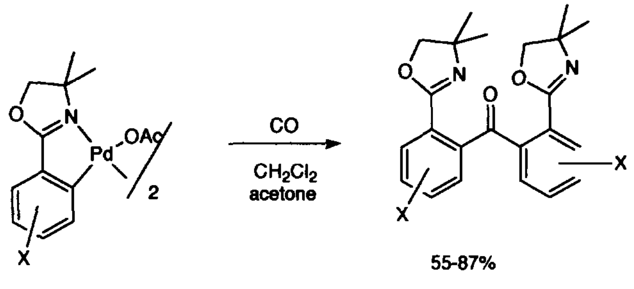
(Equation 417)



(Equation 418)



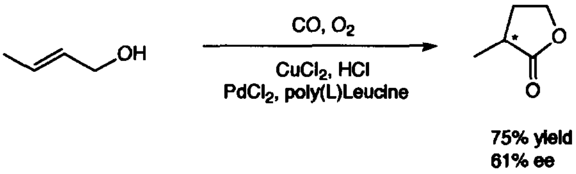
(Equation 419)



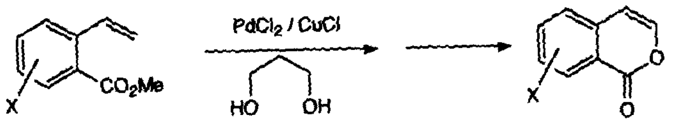
X = O, M, P, Me, OMe, pCl, pNO_2

Intramolecular alkoxycarbonylations of hydroxyalkenes promoted by palladium(II) was reviewed (11 references) [505]. Crotyl alcohol was cyclocarbonylated with reasonable ee's using palladium chloride in the presence of poly L-Leucine (equation 420) [506]. Isocoumarins were synthesized by the cycloacylation of o-vinyl esters (equation 421) [507] Olefinic amines were cyclized to lactams using rhodium(II) catalysts (equation 422) [508]. Rhodium(I) complexes catalyzed the carbonylative cyclization of 1,4-dienes (equation 423) [509], (equation 424) [510] Ligand directed rhodium(I) catalyzed hydroformylation was used effectively in a complex synthesis (equation 425) [511]

(Equation 420)

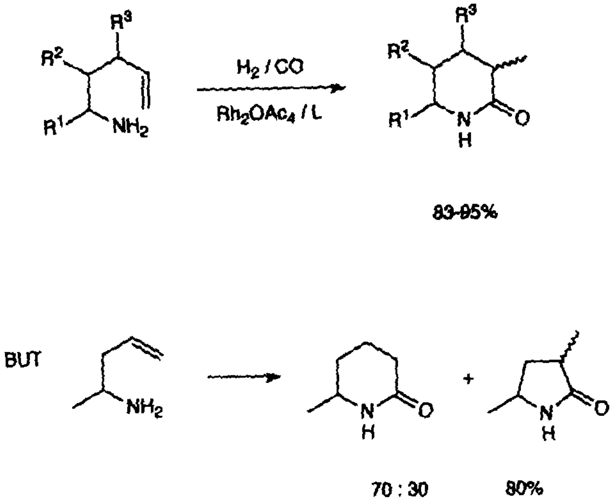


(Equation 421)



X = H, pCl, 5-Cl, 3-Me, 4-Me, 5-OMe

(Equation 422)

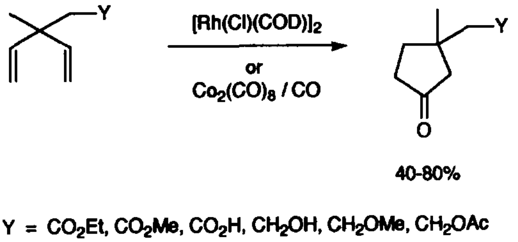


R^1 = Me, Ph, H,

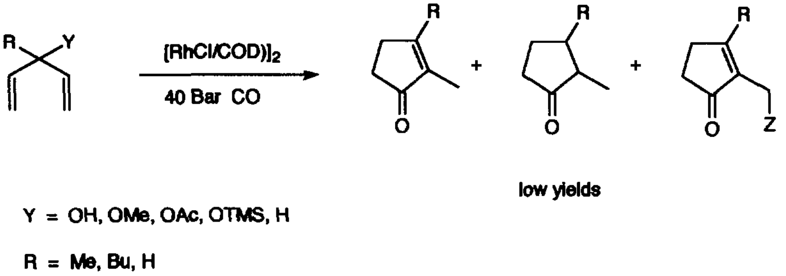
R^2 = H

R^3 = H, Me

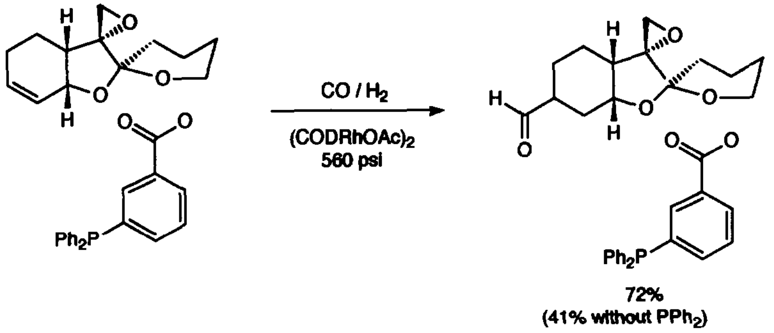
(Equation 423)



(Equation 424)

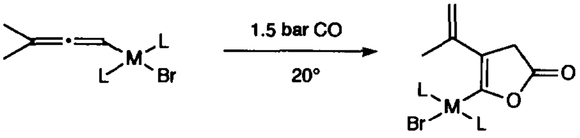


(Equation 425)



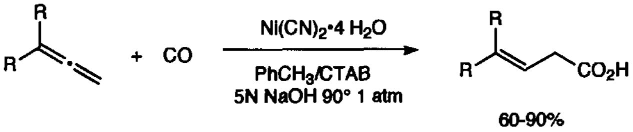
Allenic palladium complexes underwent double carbonylation (equation 426) [512]. Nickel complexes catalyzed the hydrocarboxylation of allenes (equation 427) [513] Cobalt carbonyl dimerized and carboxylated acrylonitrile (equation 428) [514] Cyclopropenes were cyclotrimerized with carbonylation by palladium catalysts (equation 429) [515] Allylcopper species incorporated carbon monoxide and 1,4-acylated enones (equation 430) [516]

(Equation 426)



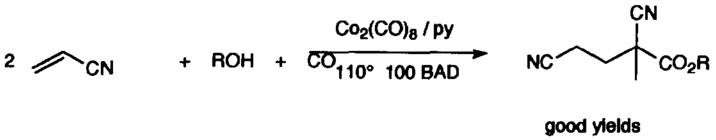
M = Pd, Pt

(Equation 427)

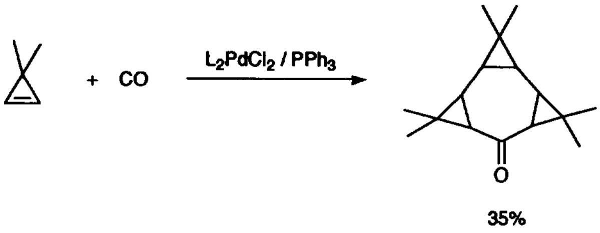


R = Me, Et, nPr, H, iBu, -(CH2)5-

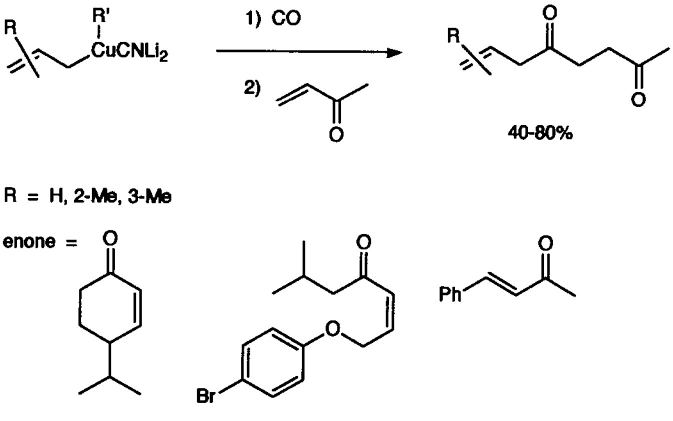
(Equation 428)



(Equation 429)

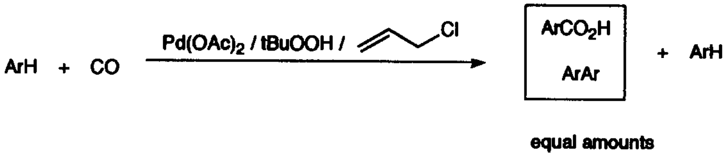


(Equation 430)

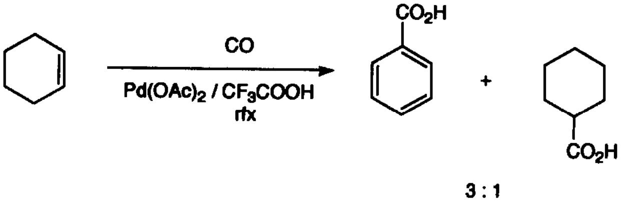


Arenes (equation 431) [517], cyclohexene (equation 432) [518], furan and thiophene (equation 433) [519], and cyclohexene (equation 434) [520] were oxidatively carboxylated by palladium or ruthenium catalysts

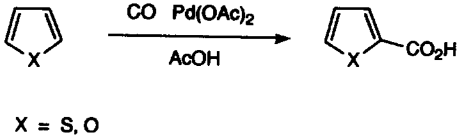
(Equation 431)



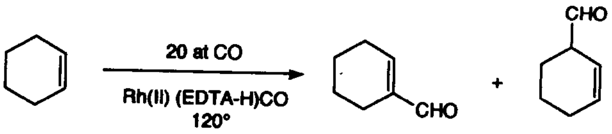
(Equation 432)



(Equation 433)

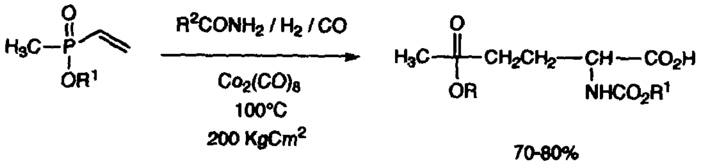


(Equation 434)

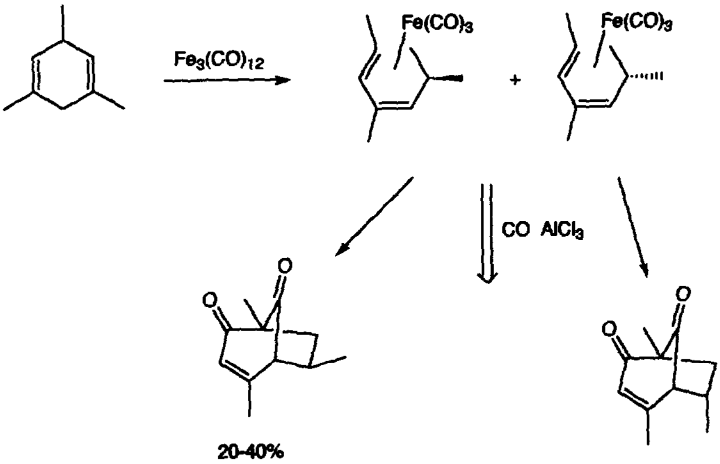


Vinyl phosphonates were aminoacylated by cobalt catalysts (equation 435) [521], while ruthenium catalysts hydroiminoformylated olefins with isocyanides in low yield [522]. Iron-diene complexes underwent multiple carbonylations when treated with aluminum chloride (equation 436) [523], (equation 437) [524].

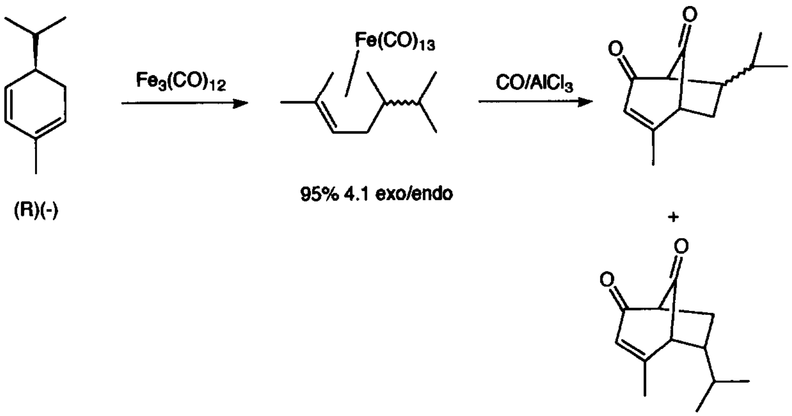
(Equation 435)



(Equation 436)



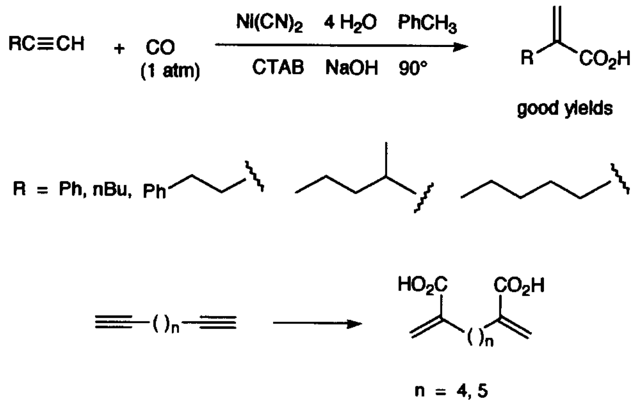
(Equation 437)



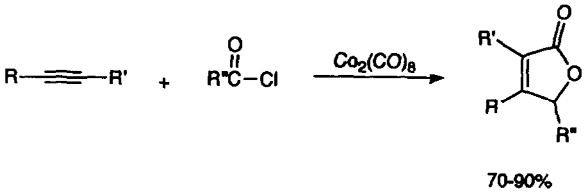
2 Carbonylation of Alkynes (Including the Pauson Khand Reaction)

Alkynes were hydrocarboxylated by nickel cyanide under phase transfer conditions (equation 438) [525] Cobalt carbonyl catalyzed the reaction of acid halides with alkynes to give butenolides (equation 439) [526], while rhodium catalyzed the addition of benzoic acid to alkynes (equation 440) [527]. Nickel(0) complexes catalyzed the addition of aldehydes to alkynes (equation 441) [528]

(Equation 438)



(Equation 439)

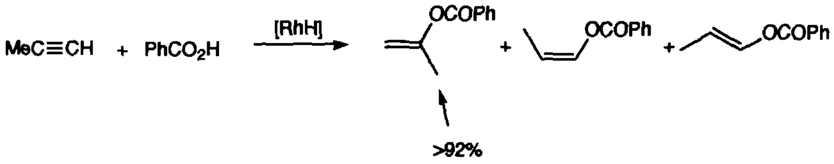


R = R' = Et

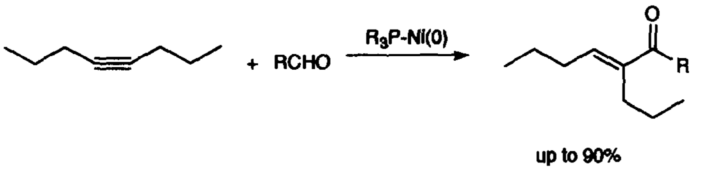
R" = Me, Et, Pr, nC$_8$, tBu, neopentyl, Ph, PhCH$_2$, ClCH$_2$CH$_2$CH$_2$

When R ≠ R', mixed regioisomers

(Equation 440)



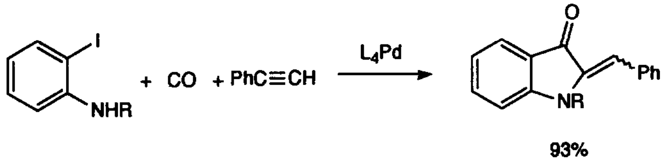
(Equation 441)



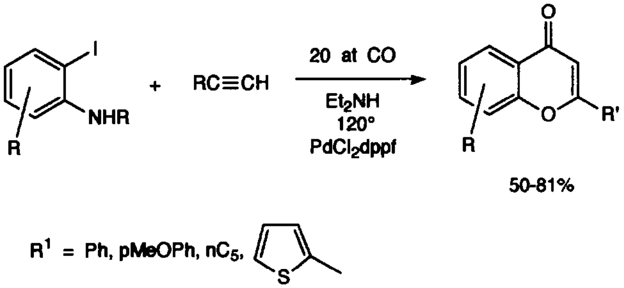
R = iPr, nPr, Ph

o-Iodoanilines (equation 442) [529] and phenols (equation 443) [430] cyclocarbonylated alkynes in the presence of palladium catalysts. Propargyl alcohols were converted to α-methylene-β-lactones by rhodium catalyzed hydrosilylation/carbonylation (equation 444) [531].

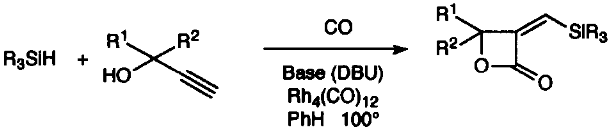
(Equation 442)



(Equation 443)



(Equation 444)

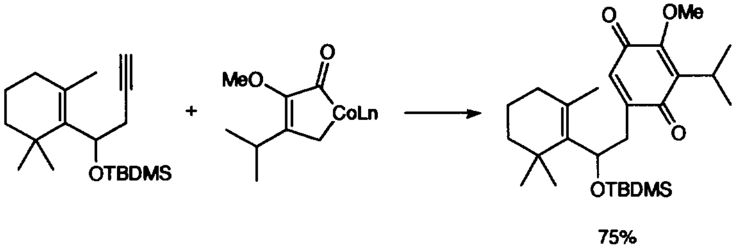


R^1 = H, Me, $(CH_2)_4$

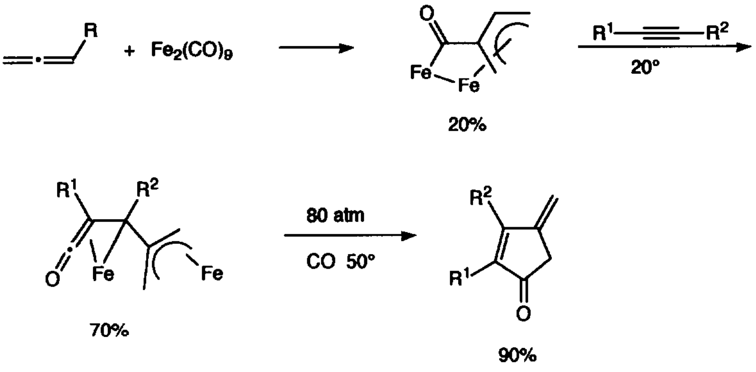
R^2 = H, Me

Alkynes were converted to quinones by reaction with cobaltacyclopentenones (equation 445) [532]. Iron carbonyl cyclocarbonylated allenes (equation 446) [533]

(Equation 445)



(Equation 446)

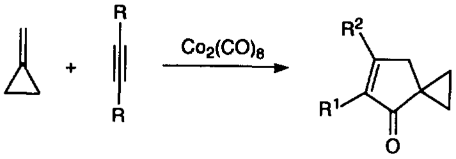


R^1 = Ph, MeOCH$_2$

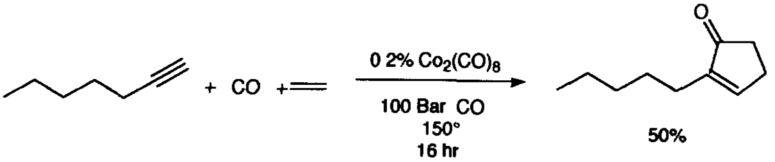
R^2 = Ph, H

Methylene cyclopropanes underwent a [2+2+1] cycloaddition to cobalt alkyne complexes (equation 447) [534]. The Pauson-Khand reaction - the combination of an alkyne, an alkene and CO to give cyclopentenones - has been extensively studied this year. These were effective in both intermolecular (equation 448) [535], (equation 449) [536], (equation 450) [537], and intramolecular (equation 451) [538], (equation 452) [539], (equation 453) [540], (equation 454) [541] versions.

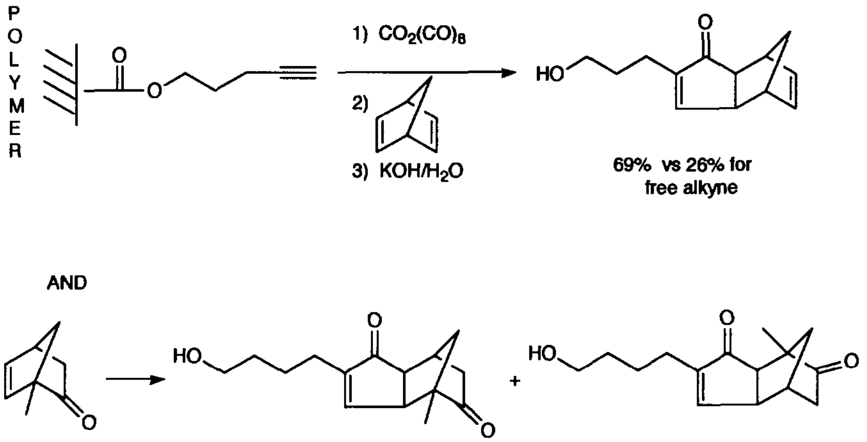
(Equation 447)



(Equation 448)



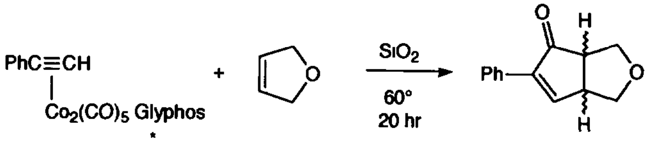
(Equation 449)



69% vs 26% for free alkyne

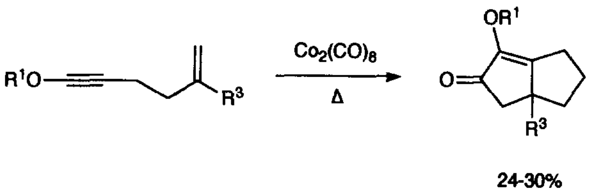
30 . 70
90% yield vs 20% for free alkyne

(Equation 450)

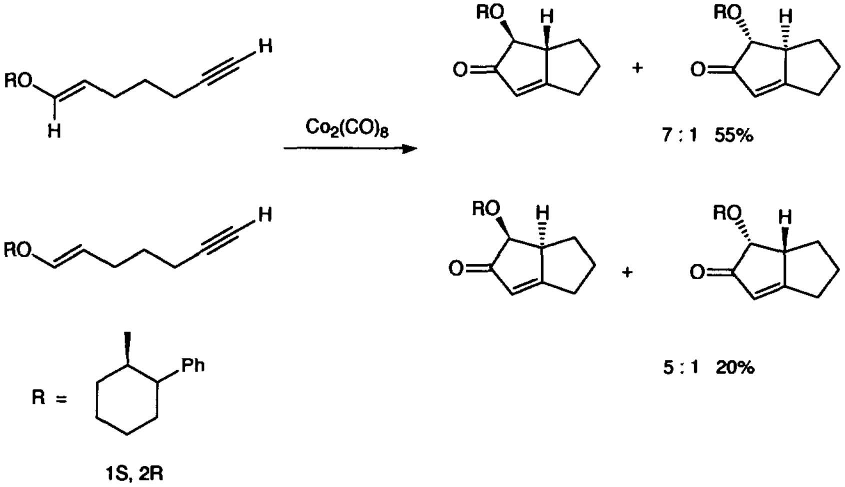


20-50%
47-59% ee

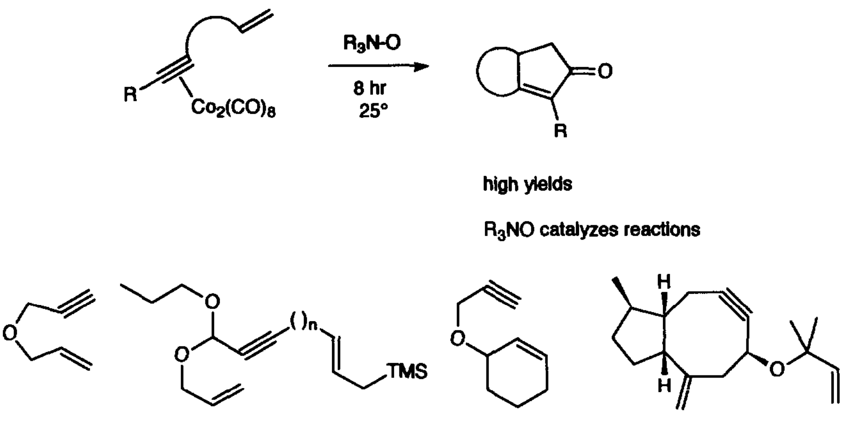
(Equation 451)



(Equation 452)



(Equation 453)

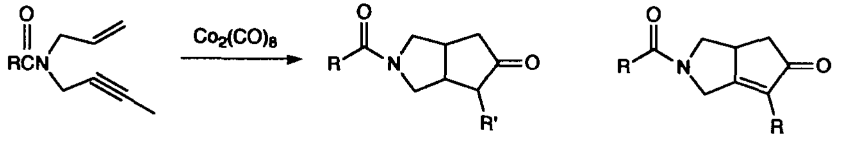


high yields

R_3NO catalyzes reactions

studied.

(Equation 454)



0-67%

0-52%

depends on conditions

R = Me; R' = H, TMS, Et

3. Carbonylation of Halides

Transition metal catalyzed carbonylation of halides is amongst the most useful of processes, and it continues to be utilized.Aryl halides were hydrocarboxylated in aqueous solution in the presence of palladium catalysts to give benzoic acids [542]. Palladium on carbon catalyzed a similar process (equation 455) [543] Iron carbonyl carbonylated dibromocyclopropanes to cyclopropane carboxylic esters (equation 456) [544]. Palladium catalyzed the carbonylation of alkyne iodonium tosylates (equation 457) [545] Benzyl halides were converted to aryl ethanols by hydrogen/carbon monoxide in the presence of dicobalt octacarbonyl [546] Alkyl iodides and benzyl halides were carbonylated by iron nitrosyl carbonyls (equation 458) [547] Carbonylation of 2-halobenzoic acids with dicobalt octacarbonyl in methanol gave half esters of phthalic acid [548] Benzyl chloride was carbonylated to phenyl acetic acids by palladium phosphine complexes under biphasic conditions [549]

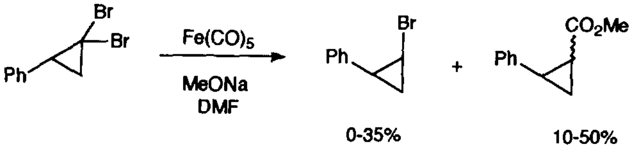
(Equation 455)

$$\mathrm{ArCl} \xrightarrow[\substack{\text{AcONa / Pd / C} \\ \text{PhCH}_3\ \ 200°}]{\text{CO / MeOH}} \mathrm{ArCO_2Me}$$

5-260 turnovers

Ar = Ph, pCF$_3$Ph, pHOPh, pMeOPh, pClPh, mClPh, 1-naphth (K$_2$Cr$_2$O$_7$ enhances)

(Equation 456)



(Equation 457)

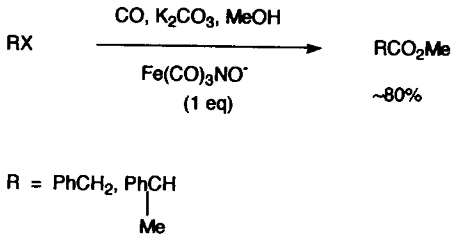
$$\mathrm{R^1C{\equiv}CI^+Ph\ OTs^-} \xrightarrow[\text{base R}^2\text{OH / CO}]{\text{Pd(OAc)}_2} \mathrm{R^1C{\equiv}C{-}CO_2R^2}$$

60-80%

R^1 = Ph, Am, Bu

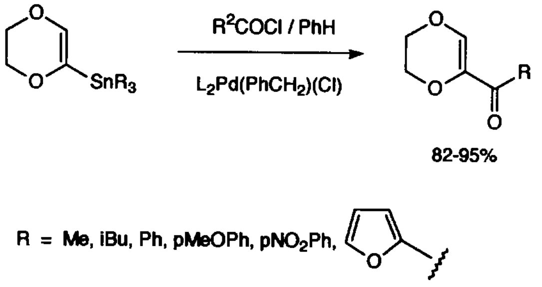
R^2 = Et, Me

(Equation 458)

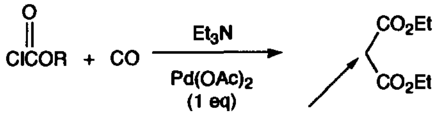


Palladium complexes catalyzed the acylation of dioxene tin reagents by acid chlorides (equation 459) [550], and the conversion of ethyl chloroformate into diethyl malonate (equation 460) [551]. Palladium also catalyzed the carbonylative coupling of aryl iodides and alkylmercuric halides to give substituted aryl alkyl ketones [552], the carbonylative homo coupling of vinyl mercuric halides to give divinyl ketones [553], and the carbonylative homo coupling of vinyl halides to give divinyl ketones [554]. Mixed iron carbonyl/cobalt carbonyl catalysts carbonylatively coupled aryl iodides to give diaryl ketones (equation 461) [555].

(Equation 459)

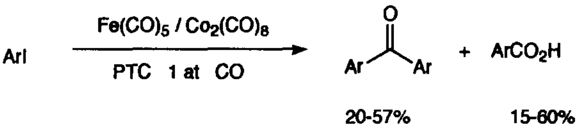


(Equation 460)



from <u>acetate</u>!

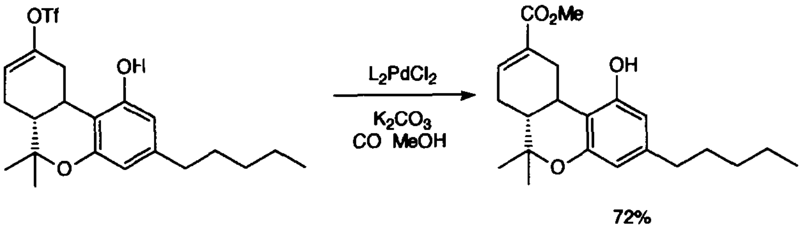
(Equation 461)



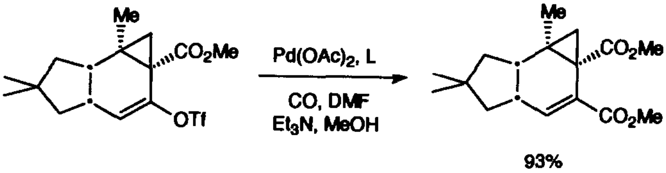
Ar = Ph, pMePh, pMeOPh, pClPh, mMePh, mClPh, oMePh

Palladium(II) complexes catalyzed the carbonylative coupling of aryl iodides with diethyl malonate to give benzoyl malonates [556]. Palladium also catalyzed the carbonylation (equation 462) [557], (equation 463) [558], and carbonylative coupling (equation 464) [559] of vinyl triflates

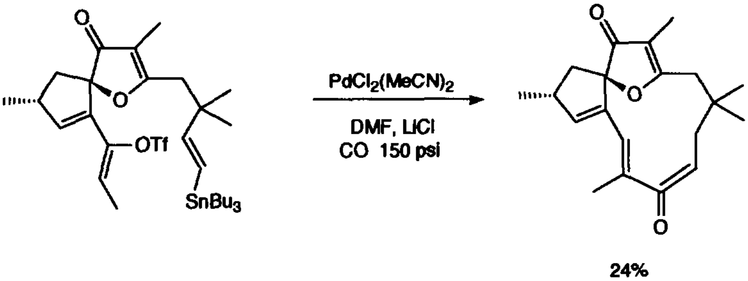
(Equation 462)



(Equation 463)

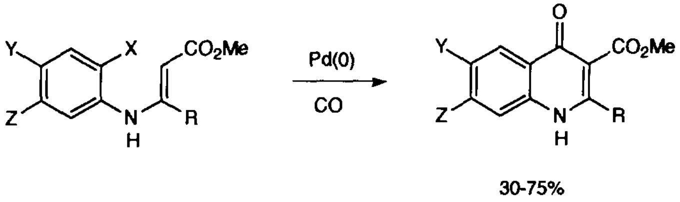


(Equation 464)



o-Haloaniline enamines were carbonylatively cyclized using palladium catalysts (equation 465) [56]. Coumarins were synthesized using similar chemistry (equation 466) [561], as were quinolones (equation 467) [562].

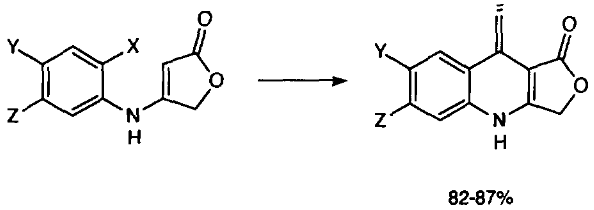
(Equation 465)



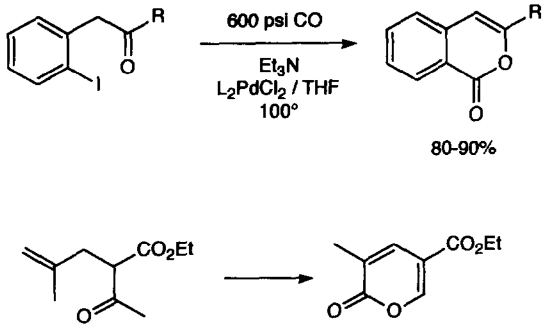
X = Br, I, Z = H, Cl, F

Y = H, F, R = Me, CH2OMe, COMe

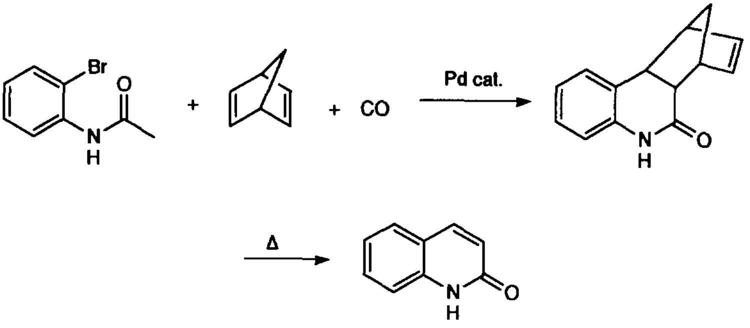
AND



(Equation 466)



(Equation 467)



4. Carbonylation of Nitrogen Compounds

The synthesis of oxamates by double carbonylation (27 references) [563] and organometallic carboxamidation (183 references) [564] have been reviewed. Palladium complexes catalyzed the synthesis of diphenyl urea from nitrobenzene, aniline, and carbon monoxide [565]. Substituted nitrobenzenes were reductively carbonylated to ureas by $Fe(CO)_5$ or $Ru_3(CO)_{12}$ [566], and by ruthenium(III) Schiffs base complexes [567] Aromatic primary amines were oxidatively carbonylated to acyclic and cyclic ureas using cobalt(II) salen complexes [568]. Palladium(0) complexes catalyzed carbonylation reactions to produce arylsulfonyl isocyanates (equation 468) [569] and imides (equation 469) [570]. Cobalt carbonyl catalyzed the N-acylation of azobenzene (equation 470) [571]

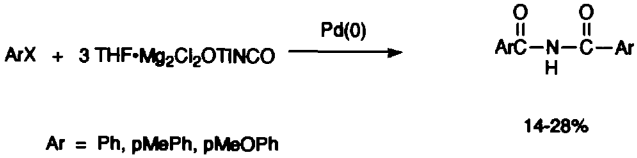
(Equation 468)

$$[ArSO_2NCl]^- \ M^+ \xrightarrow[\text{Pd (cat)}]{CO} ArSO_2NCO$$

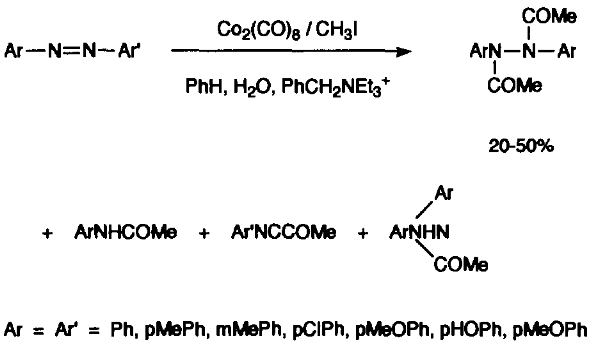
70-80%

Ar = Ph, pMePh, oClPh, oBrPh, 2-naphth

(Equation 469)



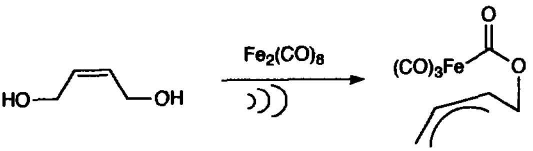
(Equation 470)



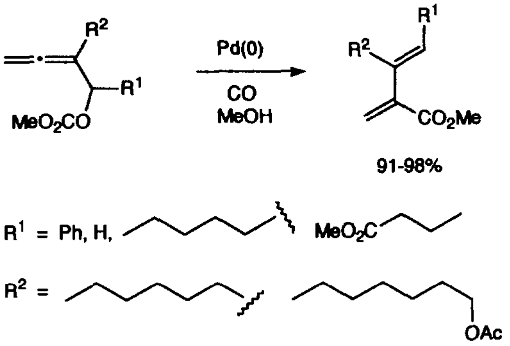
5. Carbonylation of Oxygen Compounds

Nickel cyanide catalyzed the carbonylation of allyl alcohols to carboxylic acids under phase-transfer conditions [572], while rhodium(I) complexes catalyzed the conversion of allyl alcohol to γ-butyrolactone [573] The mechanism of hydrocarbonylation of alcohols catalyzed by ruthenium-iodide complexes was studied [574]. The full details of the conversion of 2-butene-1,4-diols to ferrilactone complexes have appeared (equation 471) [575] Allenic carbonates were carbonylated to dienic esters using palladium(0) catalysts (equation 472) [576]. Lipase selectively acylated one enantiomer of a chromium benzyl alcohol complex (equation 473) [577] Cobalt carbonyl catalyzed the conversion of epoxy alcohols to butenolides (equation 474) [578]

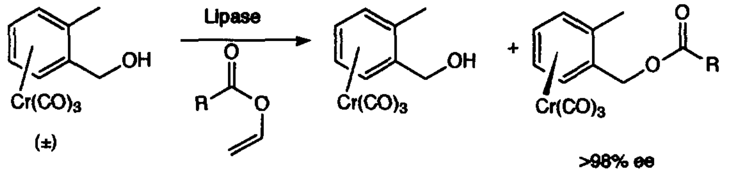
(Equation 471)



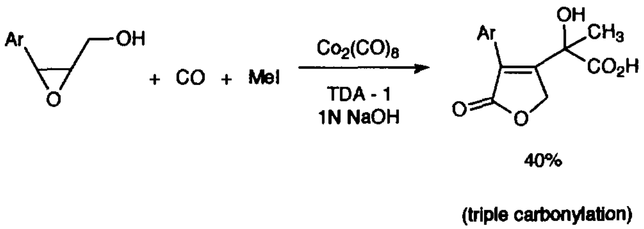
(Equation 472)



(Equation 473)



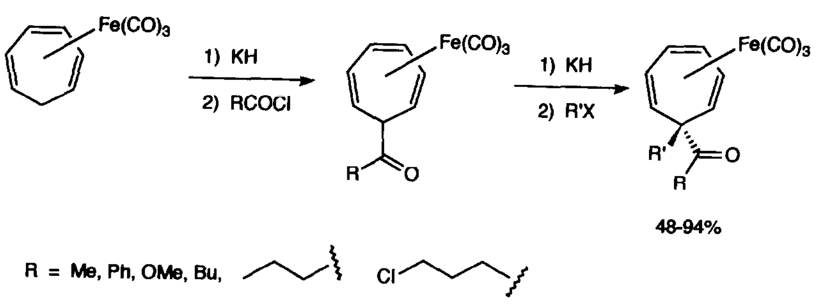
(Equation 474)



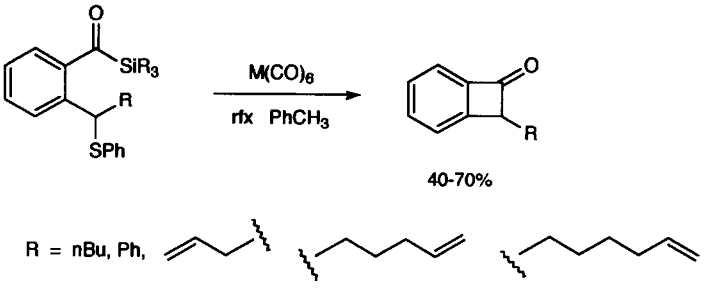
6 Miscellaneous Carbonylations

Iron-complexed cycloheptatriene was cleanly acylated by acid chlorides (equation 475) [579] Benzocyclobutanones were synthesized from aromatic acyl silanes with Group(VI) metal carbonyl catalysts (equation 476) [580] Aromatic and aliphatic hydrocarbons were carbonylated by rhodium(I) catalysts under photochemical conditions (equation 477) [581] Allyl cuprates incorporated carbon monoxide, then 1,4-acylated conjugated enones (equation 478) [582]

(Equation 475)



(Equation 476)

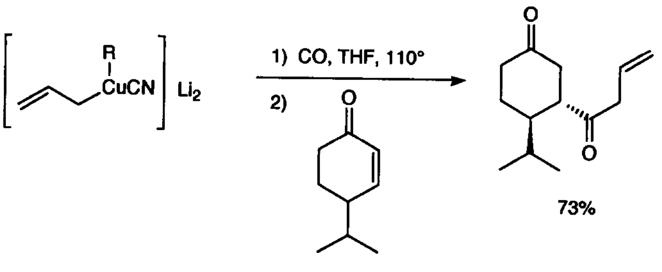


(Equation 477)

$$\mathrm{ArH} + \mathrm{CO} \xrightarrow[h\nu]{\mathrm{RhCl(CO)(PMe_3)_2}} \underset{72}{\mathrm{ArCHO}} + \underset{16}{\mathrm{ArCH_2OH}} + \underset{16}{\mathrm{ArCOAr}} + \underset{5}{\mathrm{ArAr}} + \underset{1}{\mathrm{ArCO_2H}}$$

Ar = Ph, pMePh, pMeOPh, pClPh, pNCPh

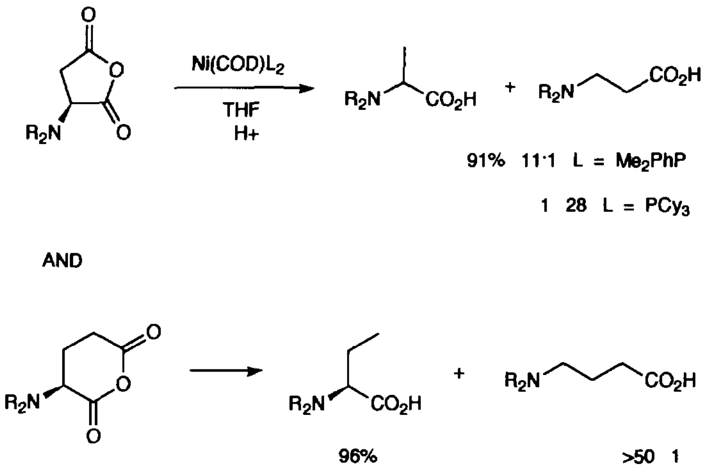
(Equation 478)



7 Decarbonylation Reactions

Nickel(0) complexes decarbonylated α-amino acid anhydrides to α-amino acids (equation 479) [583].

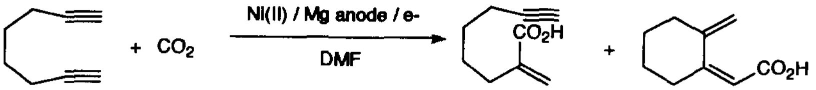
(Equation 479)



8 Reactions of Carbon Dioxide

Electrochemically generated nickel(0) complexes catalyzed the addition of carbon dioxide to diynes (equation 480) [584][585] Nickel(0) complexes catalyzed the cyclocarboxylation of diynes with carbon dioxide (equation 481) [586] (review, 23 references) [587]. Palladium(0) catalyzed the conversion of aryl halides, propargyl alcohol and carbon dioxide to cyclobutenylidene carbonates (equation 482) [588]

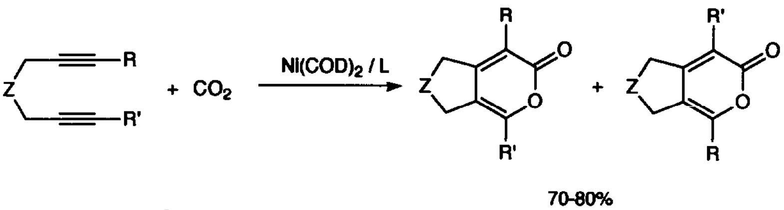
(Equation 480)



L = TMEDA, 90%

L = Bipy, 65%

(Equation 481)

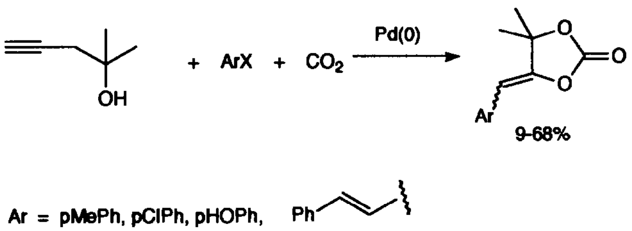


R = H, Et, TMS

R' = TMS

Z = $(CH_2)_3$

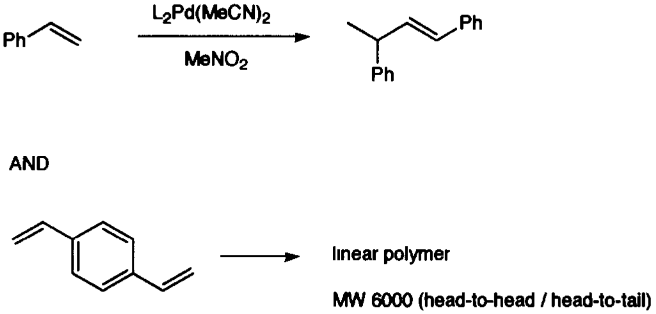
(Equation 482)



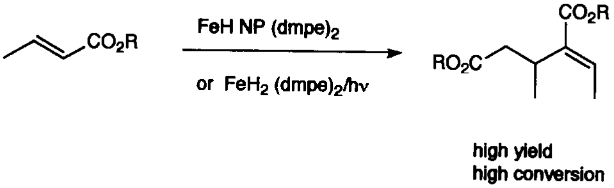
D Oligomerization (Including Cyclotrimerization of Alkynes and Metathesis Polymers).

Ethylene was dimerized to a mixture of butenes by cobalt or nickel acetylacetonates in the presence of alkylaluminum halides [589], and to 1-butene by $(h^5C_5Me_5)Ta(PMe_3)(H)(Br)(h^2CHPMe_2)$ [590] and $(CH_2{=}CH{-}CH{=}CH_2)Cp(Et)$ (diphos)Zr [591]. Chiral zirconocene dihalides catalyzed the isotactic polymerization of propene [592]. Styrene was dimerized by palladium(II) salts in nitromethane (equation 483) [595]. 3,3,3-Trifluoropropene was dimerized by stoichiometric amounts of low valent nickel catalysts [594]. Iron hydrides dimerized methacrylic esters (equation 484) [595] Nickel(0) complexes catalyzed the dimerization and trimerization of C_3-cyclic olefins [596] [597] [598] Vinyl ketones were linearly dimerized to 1,5-diketones by rhodium(I) catalysts [599] [600] [601]

(Equation 483)

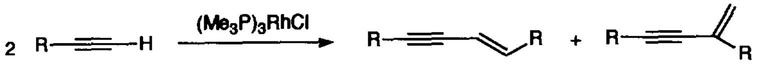


(Equation 484)



The role of dinuclear nickel complexes in alkyne oligomerization was the topic of a dissertation [602] Rhodium(I) complexes dimerized terminal alkynes to enynes (equation 485) [603] [604]. Palladium complexes catalyzed the silylation of 1,4-bis trimethylsilylbutadiyne (equation 486) [605], while molybdenum complexes oligomerized the monosilyl analog (equation 487) [606] Alkoxyacetylene was dimerized by copper(I) iodide/TMEDA (equation 488) [607]

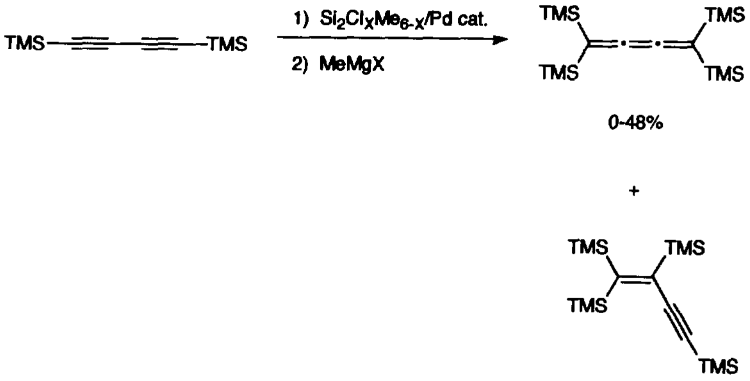
(Equation 485)



R = C_3H_7, C_4H_9, C_6H_{13}

40-80% conversion
60 · 30

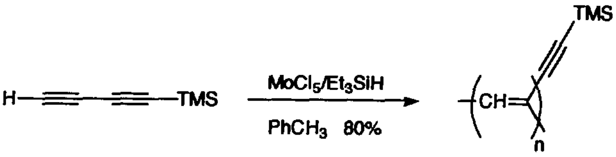
(Equation 486)



0-48%

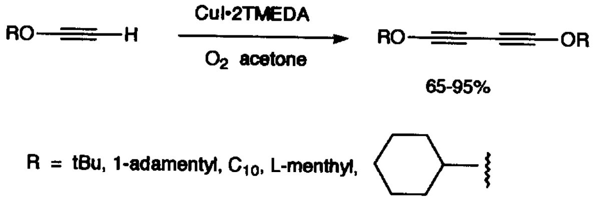
0-72%

(Equation 487)



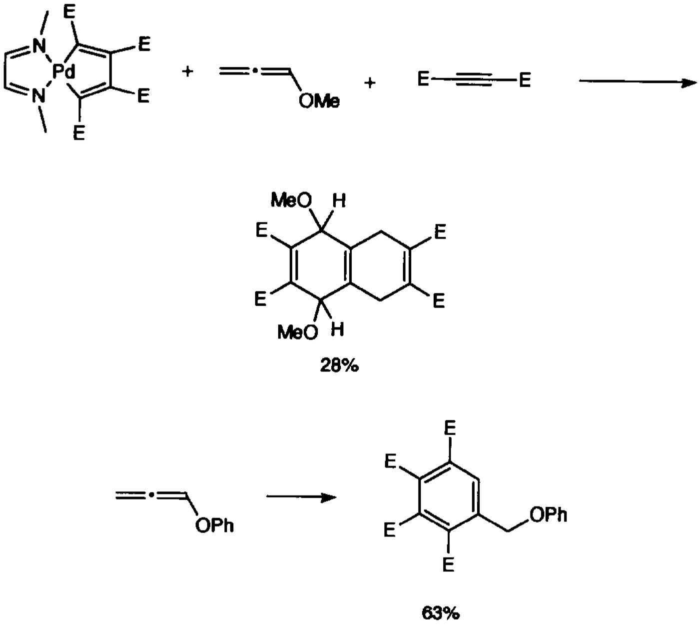
54-70%

(Equation 488)

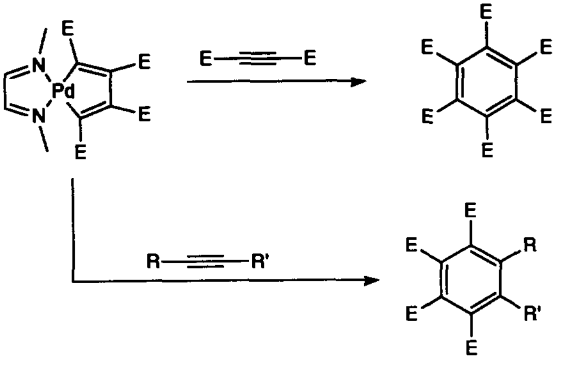


The cyclooligomerization of l^3, s^1-phosphaalkynes in the coordination sphere of a transition metal was reviewed (9 references) [608] Rhodium trichloride / aliquat 336 catalysts cyclotrimerized alkynes to benzenes [609] and cyclodimerized phenyl alkynes to 2,3-disubstituted 1-phenylnaphthalenes [610]. Palladium(0) complexes of bis imines cocyclotrimerized allenes with dimethylacetylene dicarboxylate (equation 489) [611] and cyclotrimerized alkynes to benzenes (equation 490) [612]

(Equation 489)



(Equation 490)

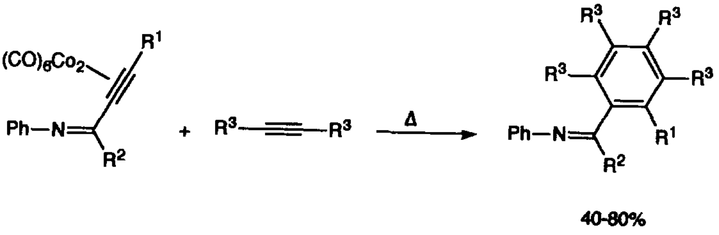


R = Et, H, TMS, Ph, $MeOCH_2$, CO_2Me

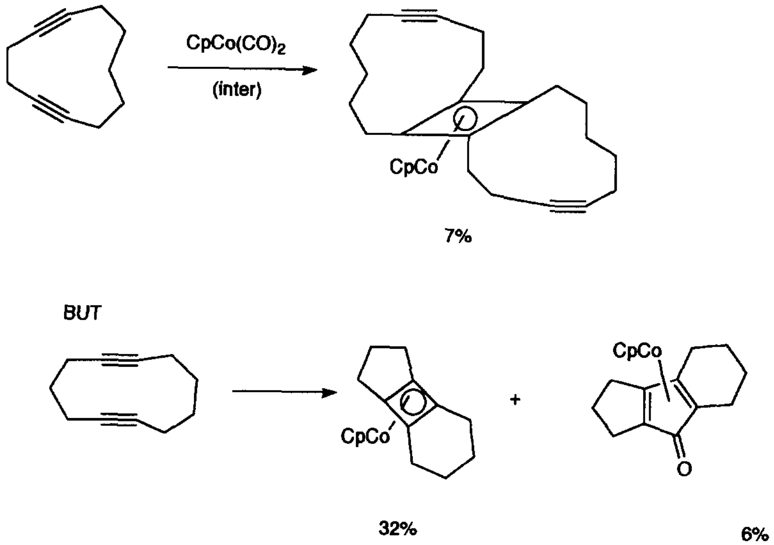
R' = Et, Bu, TMS, $PhMeOCH_2$, CO_2R, $CO_2CH_2C{\equiv}CH$

Cobalt-mediated [2+2+2] cycloadditions to the pyrrole nucleus was the topic of a dissertation [613]. Methyl propiolate was cyclotrimerized by $Ru_3(CO)_{12}$ [614]. Cobalt alkyne complexes underwent cocyclotrimerization with alkynes upon heating (equation 491) [615]. Diynes were cyclodimerized by $CpCo(CO)_2$ (equation 492) [616]. Cobalt cyclobutadiene complexes underwent reaction with dinitriles to give pyridine analogs (equation 493) [617].

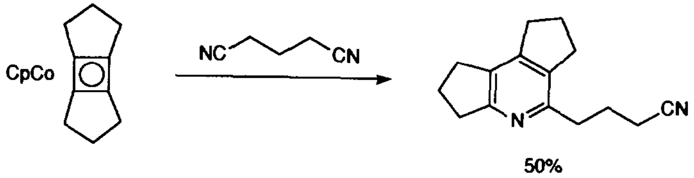
(Equation 491)



(Equation 492)

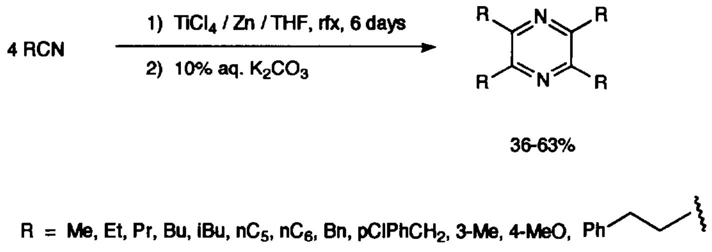


(Equation 493)



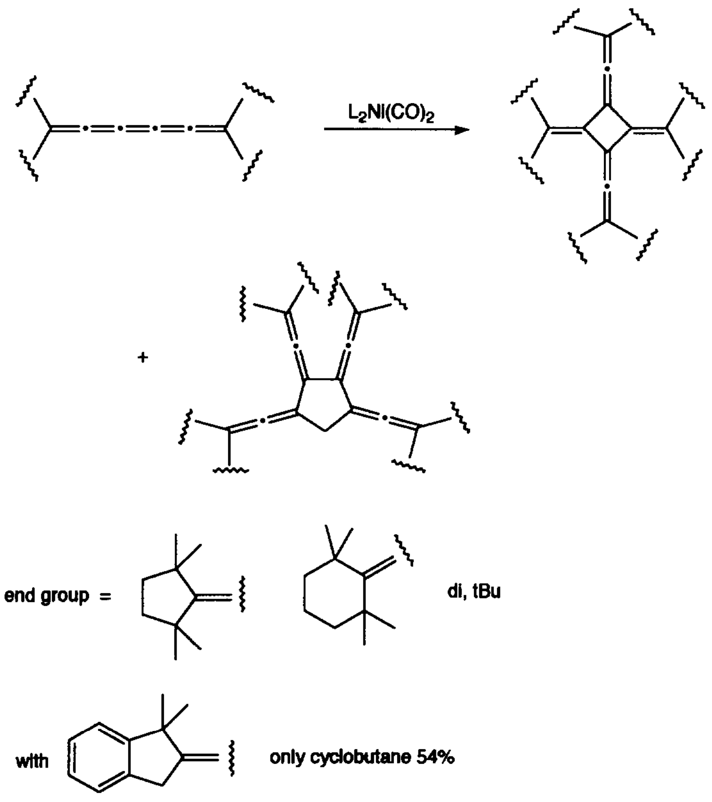
Nickel(0) mediated intramolecular cyclization has been reviewed [618]. The cobalt cluster catalyzed cocyclization of α,ω-diynes with nitriles was studied by epr [619]. Nitriles were cyclized to pyrazines by reduced titanium catalysts (equation 494) [620]

(Equation 494)



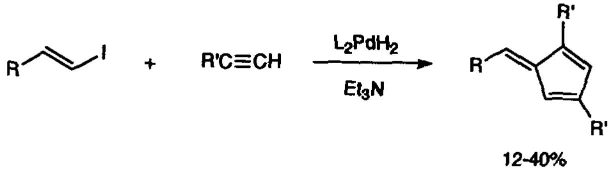
Propargyl alcohol was cyclotetramerized by nickel catalysts [621] [622]. Cumulenes were cyclooligomerized by nickel(0) catalysts (equation 495) [623] [624] Palladium(II) catalyzed reactions of 1,6-enynes: remote binding effects on cycloisomerizations, [2+2+2] cycloadditions, and skeletal rearrangements was the title of a dissertation [625].

(Equation 495)



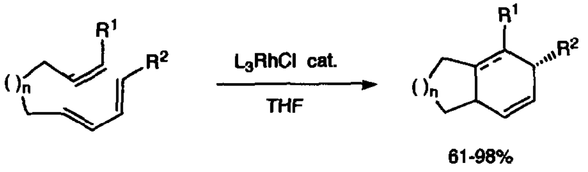
Palladium(II) complexes catalyzed the cooligomerization of vinyl halides with alkynes (equation 496) [626] Rhodium(I) complexes catalyzed the cyclization of yne dienes and trienes (equation 497) [627] Photolysis of tetraenes in the presence of copper(I) triflate gave a manifold of cyclic compounds (equation 498) [628] Nickel(0) complexes cocyclotrimerized an alkyne with carbon dioxide (equation 499) [629].

(Equation 496)



R = Ph, pMeOPh, oMePh, 2-Naph, 3-thienyl, nC_6
R' = TMS, Ph, nPr, tBu, CH_2OH

(Equation 497)



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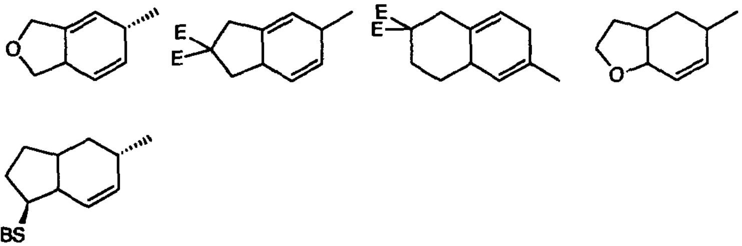
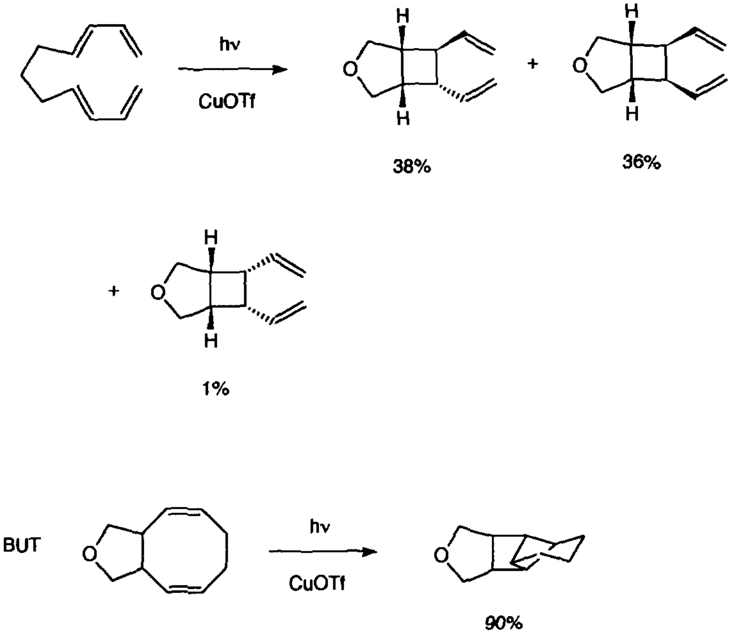
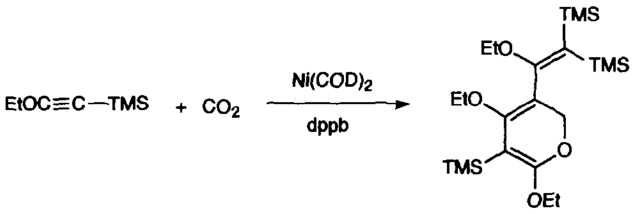


(Equation 498)

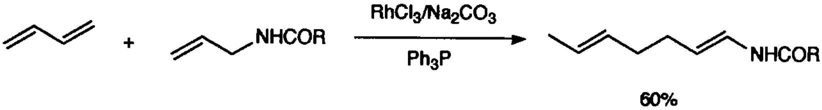


(Equation 499)

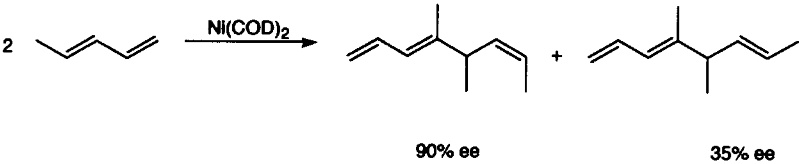


Butadiene and N-acyl allyl amine codimerized in the presence of rhodium(III) chloride (equation 500) [630]. Aromatic amine complexes of cobalt(II) catalyzed the dimerization of 1,3-dienes [631] Nickel(0) complexes catalyzed the linear dimerization of 1,3-dienes (equation 501) [632] The influence of metal halides on the cyclotrimerization of butadiene to cyclododecatriene by benzene-titanium(II) complexes was studied [633]. The mechanism of telomerization of 1,3-dienes with sulfinic acids catalyzed by palladium complexes was studied by infrared spectroscopy [634]. Water soluble ligands were developed for the biphasic palladium catalyzed telomerization of butadiene and isoprene [635] Enantioselective telomerization of butadiene with formaldehyde, nitroalkanes and enamines using palladium catalysts with chiral diphosphine ligands was developed with ee's up to 41% being obtained [636].

(Equation 500)

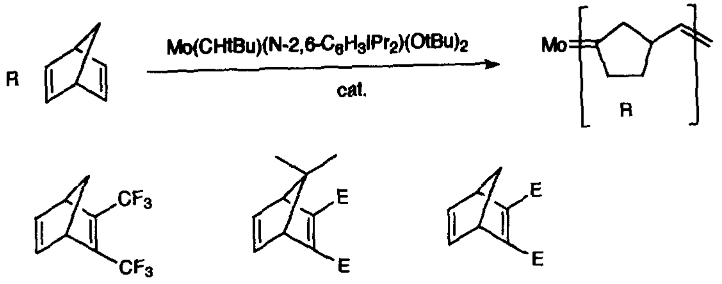


(Equation 501)

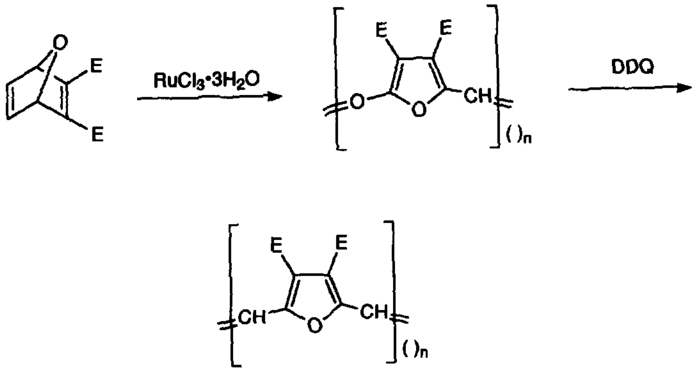


Living ring opening metathesis polymerization catalyzed by well characterized transition metal alkylidene complexes was the subject of a review (36 references) [637] The metathesis polymerization of norbornene by tungsten-carbene complexes was developed [638] Molybdenum complexes formed living polymers with norbornadienes (equation 502) [639] Ruthenium(II) chloride or osmium(III) chloride ROMPED functionalized norbornadienes (equation 503) [640]. Pyrrole was oxidatively polymerized by aluminum chloride/copper(I) chloride (equation 504) [641]

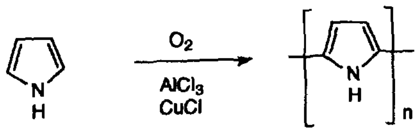
(Equation 502)



(Equation 503)



(Equation 504)

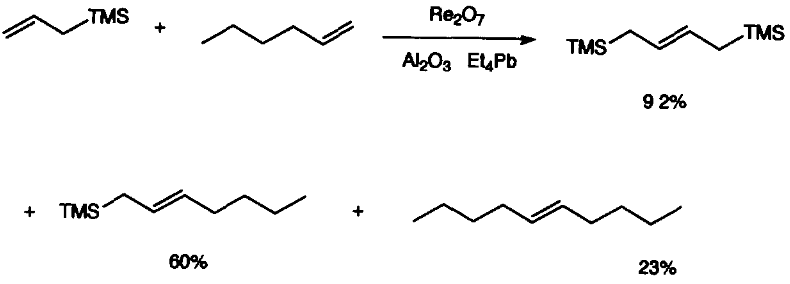


E Rearrangements

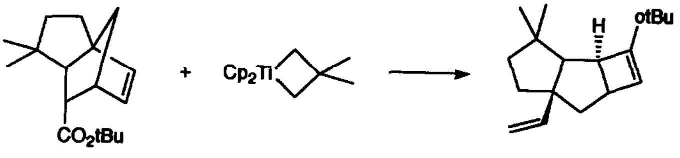
1. Metathesis

Several reviews dealing with olefin metathesis have appeared this year. These include one on reactions of acetylenes and alkenes induced by catalysts of olefin metathesis (26 references) [642]; the presence of dichlorotungsten carbenes in photocatalytic olefin metathesis reactions [643]; metathesis of imines with Fischer-type carbene tungsten complexes [644]; metathesis and isomerization of olefins with polystyrene-bonded cyclopentadienyltungsten tricarbonyl complexes has been developed [645] A number of metathesis catalyst systems including $[WBr_2(CO)_2diene]/AlCl_2Et$ [646]; MoO_3/SiO_2 [647], $Re_2O_7/Al_2O_3/CsNO_3$ [648], $Mo_3Ou(C_2O_4)_3CH_2O_3]^{2-}$ on g alumina [649] and $[Mo(NO)_4(HSO_4)_2](HSO_4)_2/EtAlCl_2$ [650] have been developed Acetylenic hydrocarbons were metathesized by $MoO_2(acac)_2$ / $AlEt_3$ / PhOH catalysts [651]. Other new metathesis catalysts include $Mo(CHTMS)(NAr)(OR)_2$ [652], $Re(C\text{-}tBu)(CHtBu)(COCMe(CF_3)_2]_2$ [653] and $[Mo(NO)(H_2NO)(X_2)L_2]^{n-}$ / $EtAlCl_2$ [654] Allylsilanes cometathesized with 1-hexene (equation 505) [655] Metathesis was used in the synthesis of capnellene (equation 506) [656]

(Equation 505)



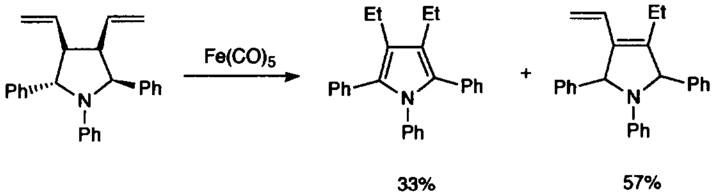
(Equation 506)



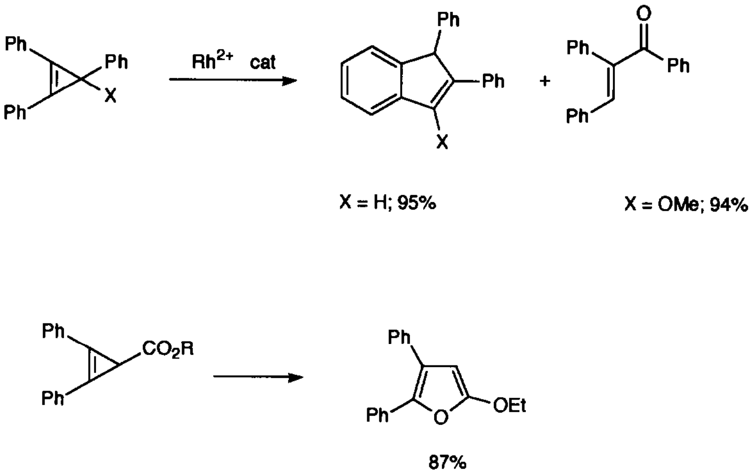
2 Olefin Isomerizations

Stereoselective isomerization of acetylenic derivatives as a new methodology in organic synthesis was the subject of a review [657], as was isomerization of olefins and diene complexes with iron tricarbonyl (132 references) [658]. Ziegler-Natta catalysts isomerized allylbenzene to trans b-methylstyrene and eugenol to isoeugenol [659]. Palladium chloride catalyzed the Z to E isomerization of styrenes [660]. Iron carbonyls isomerized divinyl tetrahydropyrroles to pyrroles (equation 507) [661]. Rhodium(II) complexes catalyzed isomerization of substituted cyclopropenes (equation 508) [662] Palladium acetate rearranged silylenol ethers to enones (equation 509) [663]

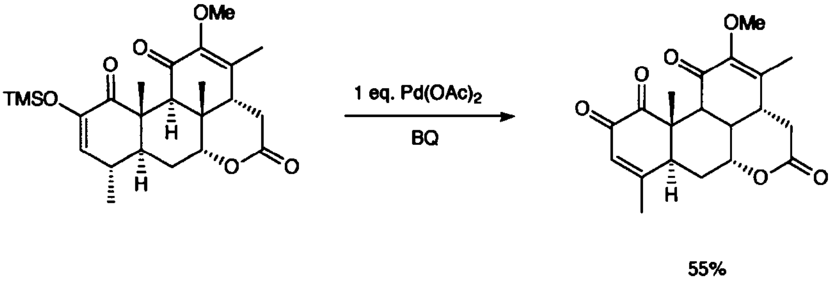
(Equation 507)



(Equation 508)



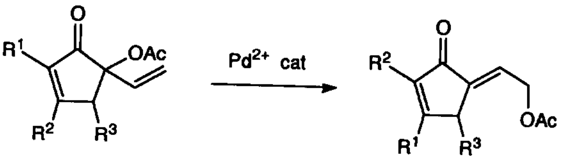
(Equation 509)



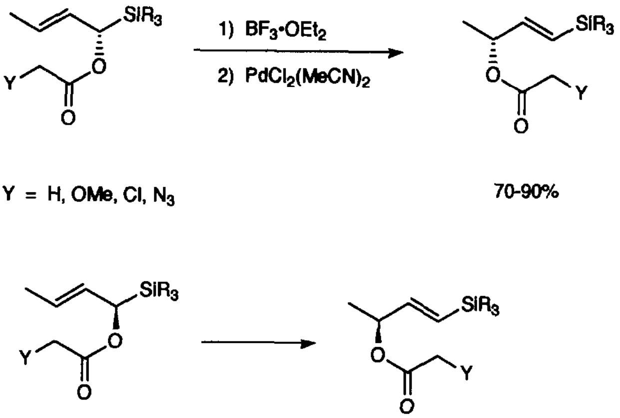
3. Rearrangement of Allylic and Propargylic Systems

Palladium(II) complexes catalyzed the allylic transposition of allyl acetates (equation 510) [664], (equation 511) [665], allyl sulfinates (equation 512) [666], (equation 513) [667], and o-allyloxycarbonyl-a-sulfenyloximes (equation 514) [668] [669]. The mechanism of the palladium(II) catalyzed Cope rearrangement of 1,5-dienes was studied and reported in detail [670].

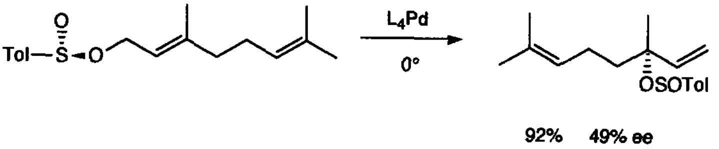
(Equation 510)



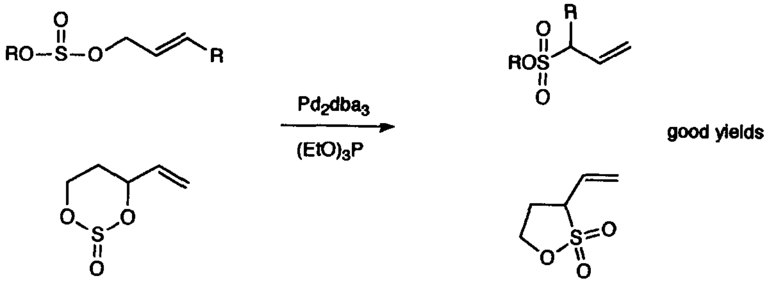
(Equation 511)



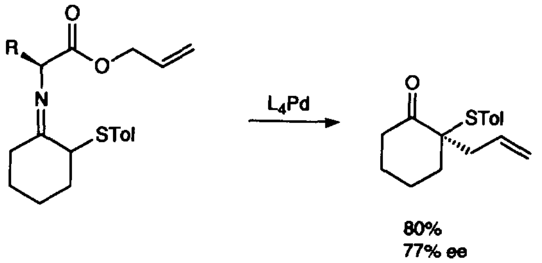
(Equation 512)



(Equation 513)

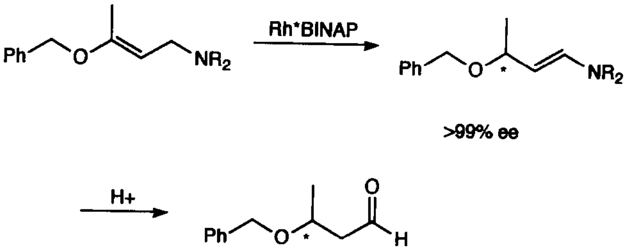


(Equation 514)

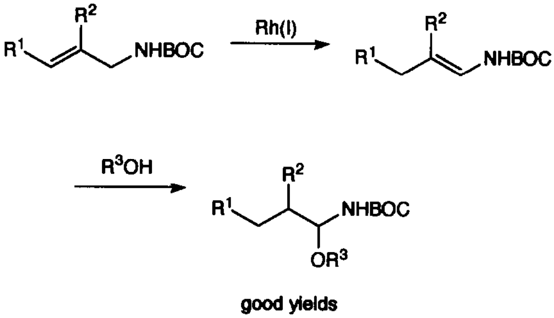


The mechanism of the rhodium(±)-Binap catalyzed rearrangement of allyl amines to enamines was fully studied and reported [671] and was used to synthesize optically active aldehydes (equation 515) [672]. Achiral catalysts were also useful for this type of rearrangement (equation 516) [673], (equation 517) [674]

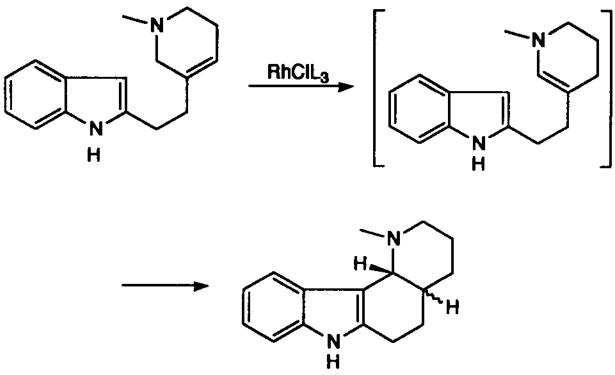
(Equation 515)



(Equation 516)

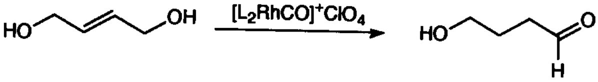


(Equation 517)

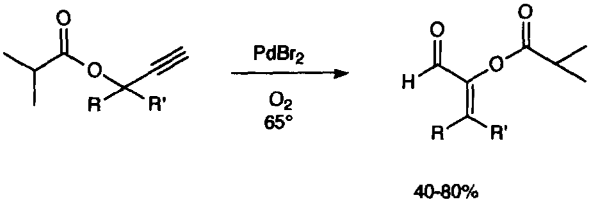


Similar rhodium(I) catalysts rearranged allyl alcohols to aldehydes (equation 518) [675] [676]. Palladium(II) salts catalyzed the rearrangement of propargyl acetates to α-acycloxyaldehydes (equation 519) [677] and palladium(0) complexes catalyzed the 1,3-diene monoepoxides to enones (equation 520) [678] and cyclopropyl allyl alcohols to dienones (equation 521) [679]. Chromium arene complexes catalyzed the rearrangement of allyl siloxanes to silyloxydienes (equation 522) [680].

(Equation 518)

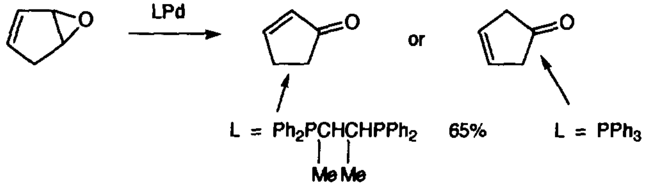


(Equation 519)

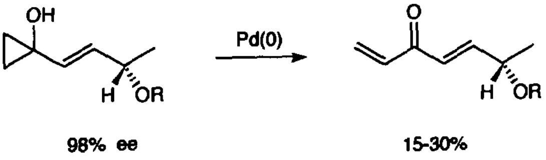


R, R' = $(CH_2)_5$, $(CH_2)_4$, $(CH_2)_{11}$
R = Et, R' = C_8
R = Ph; R' = H
R = Me; R' = C_9

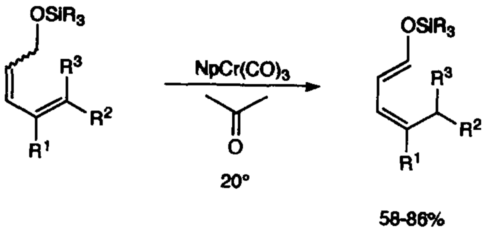
(Equation 520)



(Equation 521)



(Equation 522)



R^1 = H, nBu, Me

R^2 = H, Me

R^3 = H, Me

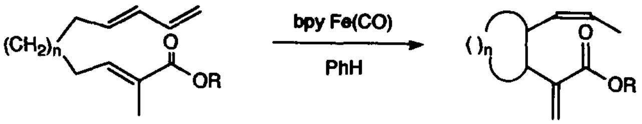
4. Skeletal Rearrangements

Transition metal complexes catalyzed isomerization of highly strained systems was the subject of a review (26 references) [681].

5. Miscellaneous Rearrangements

Palladium(0) complexes catalyzed the rearrangement of unsaturated 1,4-epiperoxides [682]. Iron(0) complexes catalyzed the cycloisomerization shown in equation 523 [683].

(Equation 523)

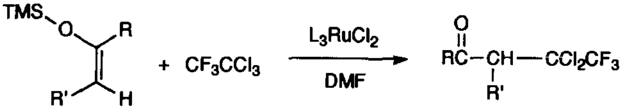


III. Functional Group Preparations

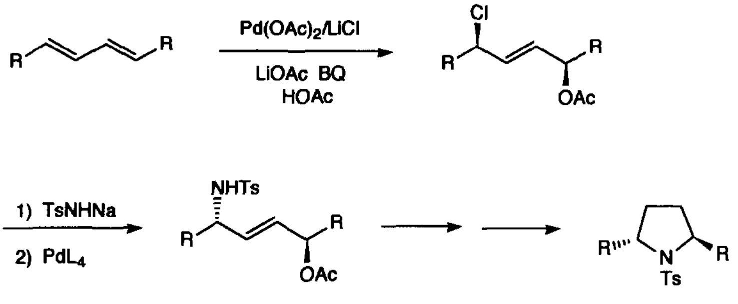
A. Halides

Bromoaromatics were converted to iodoaromatics by halide exchange over KI/CuI/alumina [684]. Ruthenium(II) complexes catalyzed the trifluorochloropropylidenation of silylenol ethers (equation 524) [685]. Improved conditions for the palladium(II) catalyzed haloacyloxylation of cyclic conjugated dienes have been developed [686] (equation 525) [687] Platinum(0) complexes catalyzed the rearrangement shown in equation 526 [688]

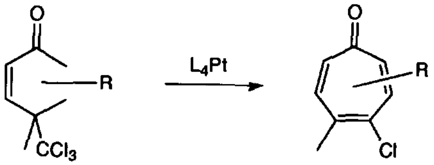
(Equation 524)



(Equation 525)



(Equation 526)

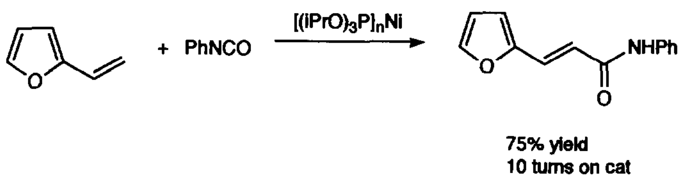


5-30%

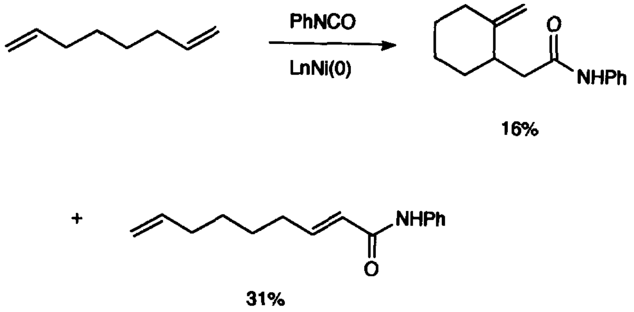
B Amides, Nitriles

Nickel(0) complexes catalyzed the addition of isocyanates to olefins to give amides (equation 527) [689], (equation 528) [690]. Cyclic amines were oxidized to amide N-oxides by hydrogen peroxide/sodium tungstate (equation 529) [691] Ruthenium(II) complexes catalyzed the condensation of formamides with aryl amines (equation 530) [692]. Aryl sulfonamides were N-acylated by isocyanates in the presence of copper(I) salts (equation 531) [693] Ruthenium chloride catalyzed the N-alkylation of amides and lactams by alcohols [694]

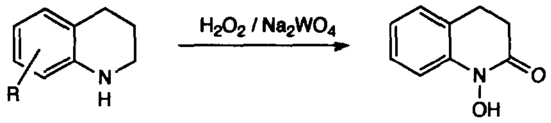
(Equation 527)



(Equation 528)



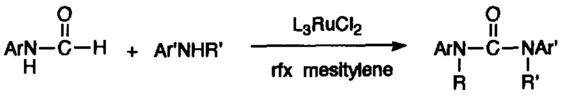
(Equation 529)



50-80%

R = H, 4-Me, 6-Me, 6-MeO, 6-MeOCNH,
6-Cl, 6-Br, 6-MeCO, 6-CN, 8-Me

(Equation 530)



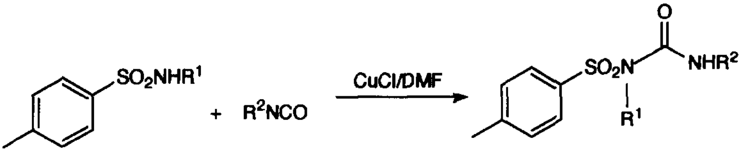
$$ArNH-C(=O)-H + Ar'NHR' \xrightarrow[\text{rfx mesitylene}]{L_3RuCl_2} ArN(R)-C(=O)-N(R')Ar'$$

76-93%

$Ar' = Ar$ = pClPh, pMeOPh, pMePh, oMePh, o,o'Me_2Ph

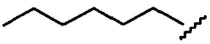
R = R' = H

(Equation 531)



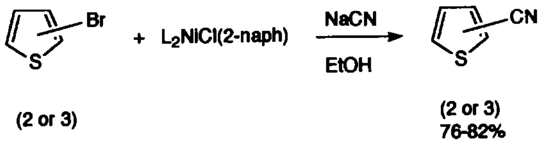
46-98%

R^1 = H, Me

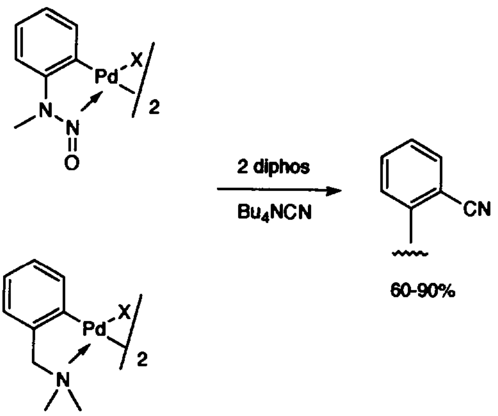
R^2 Et, Bu, tBu, Ph, 

Nickel(II) complexes catalyzed the conversion of bromothiophene to cyanothiophenes (equation 532) [695]. o-Palladated aromatics were converted to nitriles by treatment with tetrabutylammonium cyanide (equation 533) [696]. Platinum catalyzed the addition of phosphine to acrylonitrile (equation 534) [697]. Copper salts catalyzed halide exchange with iodonucleosides (equation 535) [698] Chiral titanium complexes catalyzed the asymmetric addition of TMSCN to aldehydes (equation 536) [699] Cobalt carbonyl catalyzed the ring opening of tetrahydrofuran by TMSCN (equation 537) [700], while rhodium(I) complexes catalyzed the conversion of ketals to protected cyanohydrins (equation 538) [701].

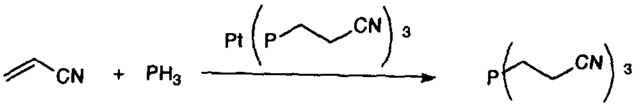
(Equation 532)



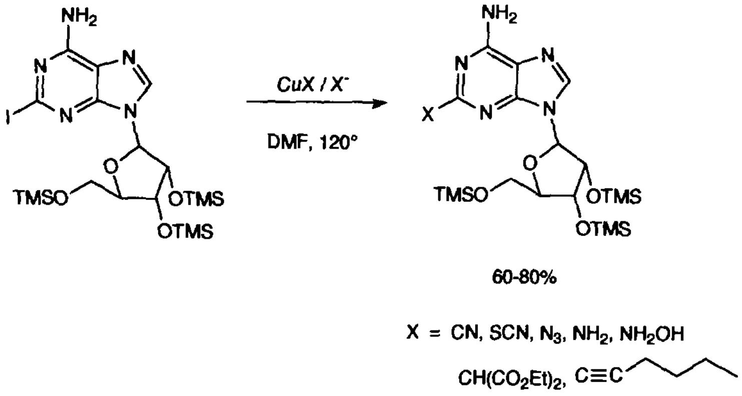
(Equation 533)



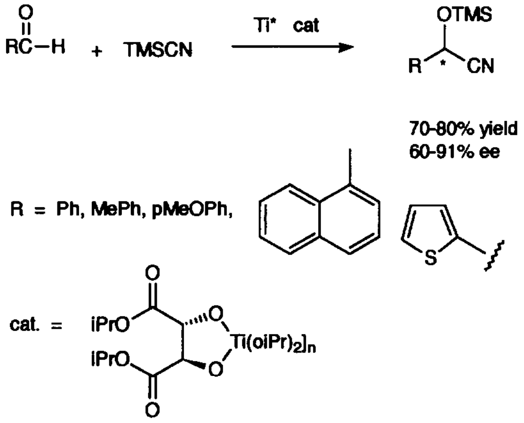
(Equation 534)



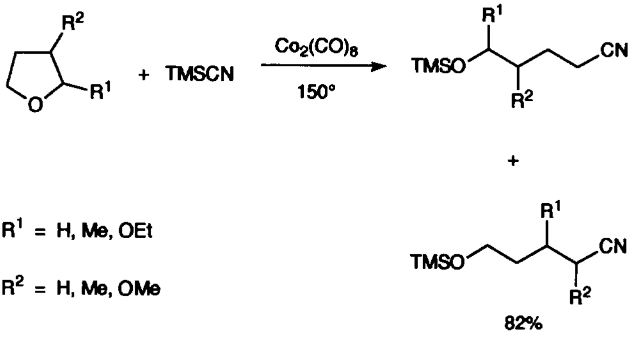
(Equation 535)



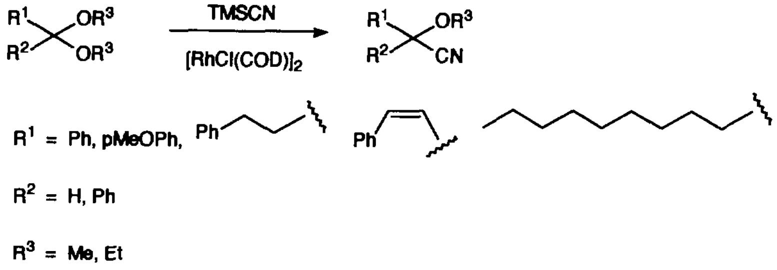
(Equation 536)



(Equation 537)



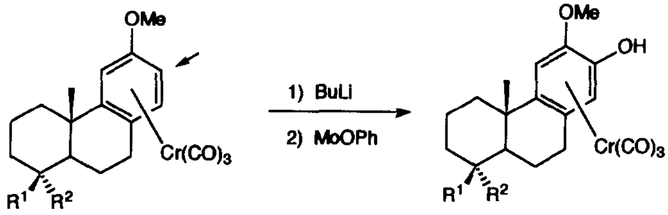
(Equation 538)



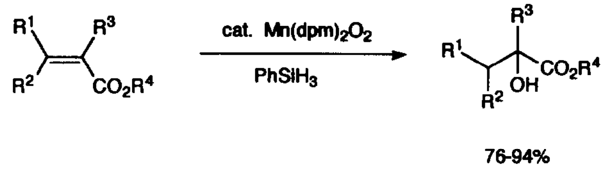
C. Amines, Alcohols

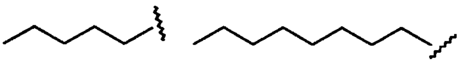
Combined palladium-copper complexes catalyzed the oxygenation of benzene to phenol or benzoquinone [702] Benzene was hydroxylated by hydrogen/oxygen in the presence of palladium(II)/iron(II) complexes [703]. The complex [MoO$_5$•Py•DMPU] was advanced as a safer alternative to MoOPh for a-hydroxylation of enolates [704]. MoOPh was used to hydroxylate lithiated arenechromium tricarbonyl complexes (equation 539) [705]. Manganese oxo complexes catalyzed the reductive a-hydroxylation of conjugated esters (equation 540) [706]

(Equation 539)



(Equation 540)



R^1 = H, Me, nPr, COMe 

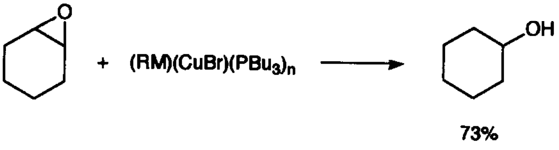
R^2 = H

R^3 = H, Me

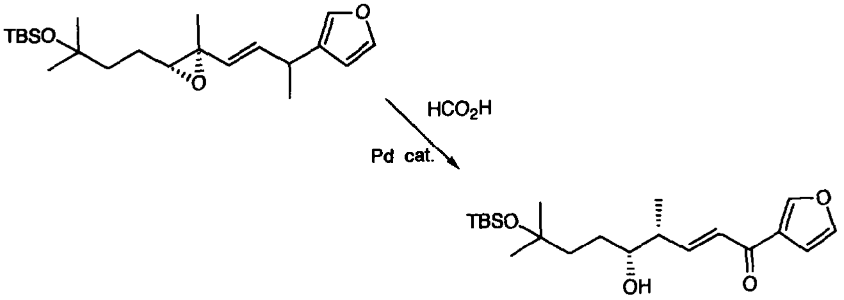
R^4 = Bn, Me, Et

Mixed cuprates reduced epoxides to alcohols (equation 541) [707], as did formic acid in the presence of palladium catalysts (equation 542) [708]. Cobalt(II) catalyzed the ring opening of epoxides by thiols (equation 543) [709] and amines (equation 544) [710]. Palladium(0) catalyzed similar processes (equation 545) [711]. Vinyl oxetanes were converted to homoallylic alcohols by reaction with organocopper complexes (equation 546) [712].

(Equation 541)

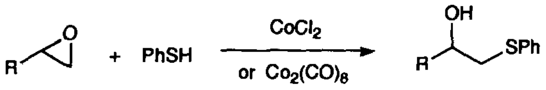


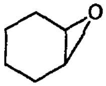
(Equation 542)



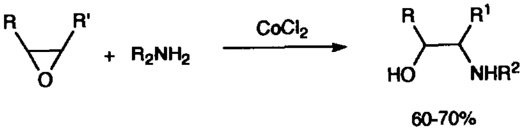
4 : 1 90%

(Equation 543)



R = Me, Et, Ph, CH_2Cl, $PhOCH_2$,  also works.

(Equation 544)

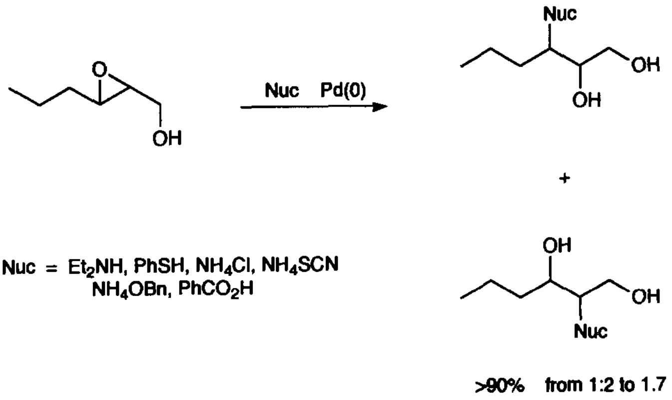


R = Et, Ph, CH_2Cl, $PhOCH_2$

R^1 = H, $(CH_2)_4$

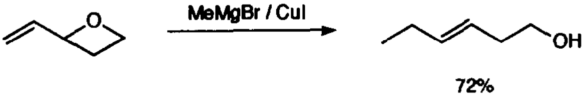
R^2 = Ar

(Equation 545)



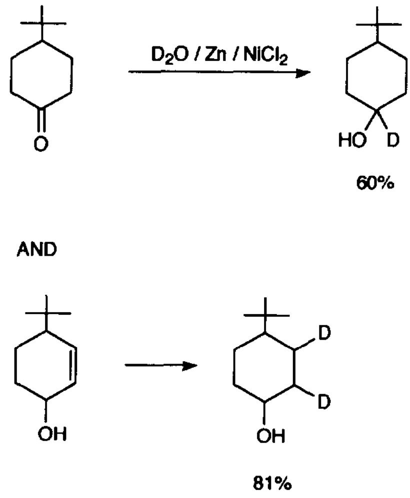
Nuc = Et_2NH, PhSH, NH_4Cl, NH_4SCN
NH_4OBn, $PhCO_2H$

(Equation 546)

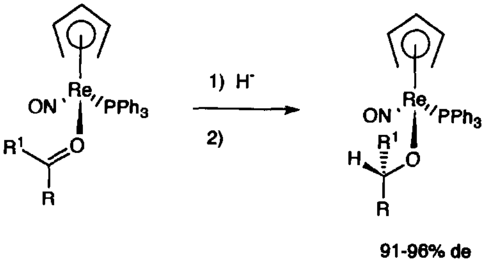


Grignard reagents reduced aliphatic, aromatic, and α,β-unsaturated ketones to secondary alcohols in the presence of titanocene dichloride [713] Ketones and aldehydes were reduced to alcohols by zinc in the presence of $NiCl_2 \bullet 6H_2O$ This reagent also reduced olefins, nitriles, and nitroaromatics [714] In D_2O as solvent, deuterium was incorporated (equation 547) [715] Aldehydes and ketones complexed to optically active rhenium complexes were reduced with high asymmetric induction (equation 548) [716] [717]

(Equation 547)

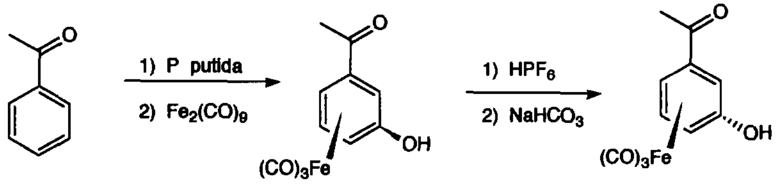


(Equation 548)

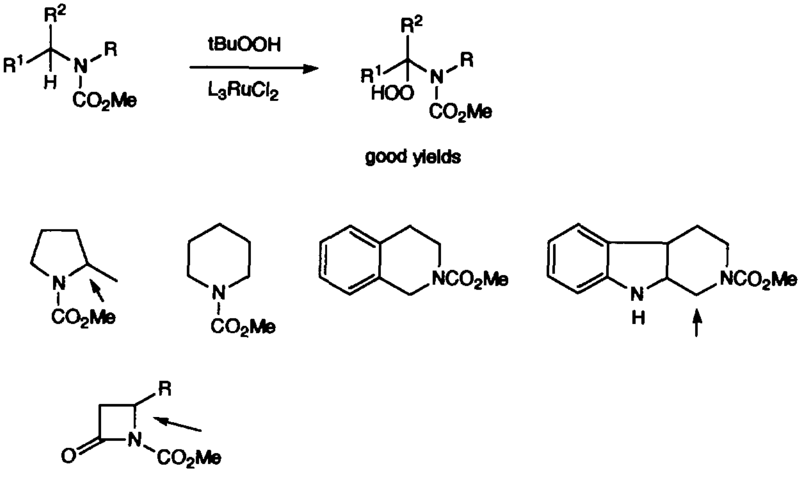


Acetophenone was microbially oxidized to the cyclohexadienol which complexed to iron (equation 549) [718]. Ruthenium complexes catalyzed the α-hydroperoxidation of amides (equation 550) [719] [720], and the varied oxidations of norborene epoxide shown in equation 551 [721]

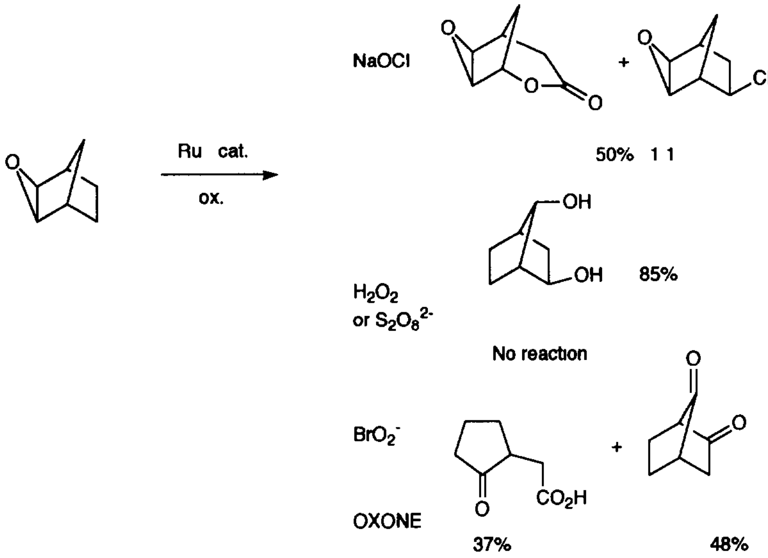
(Equation 549)



(Equation 550)

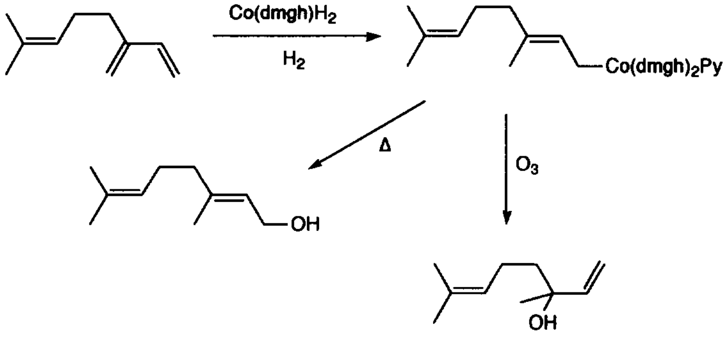


(Equation 551)

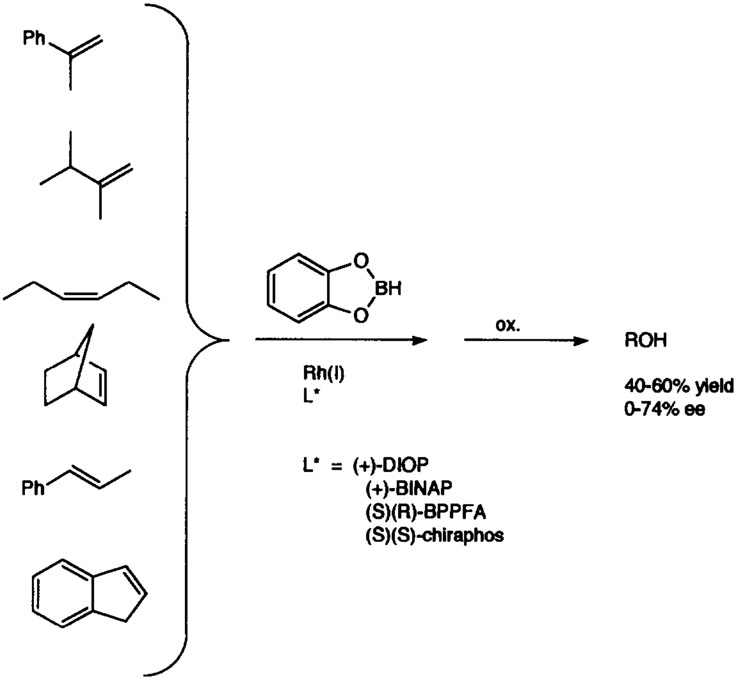


Dienes were converted into allylic alcohols by cobalt $dmgH_2$ complexes (equation 552) [722] Olefins were hydroborated and oxidized to optically active alcohols using chiral rhodium(I) catalysts (equation 553) [723]. Intramolecular (equation 554) [724], (equation 555) [725], and intermolecular (equation 556) [726] hydrosilylations/oxidations were used to produce alcohols

(Equation 552)



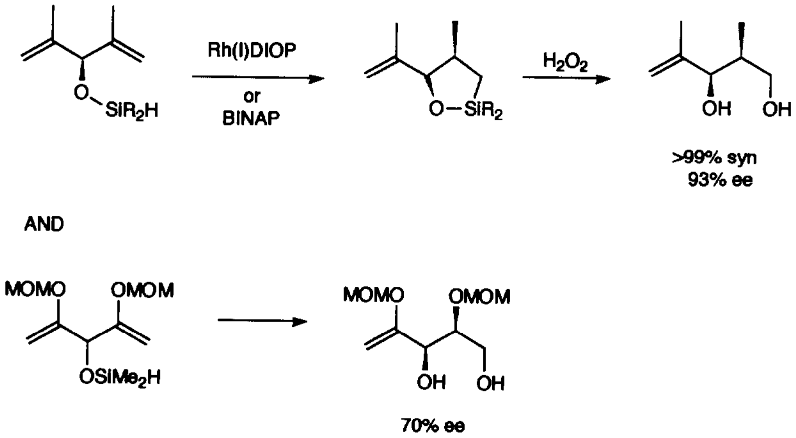
(Equation 553)



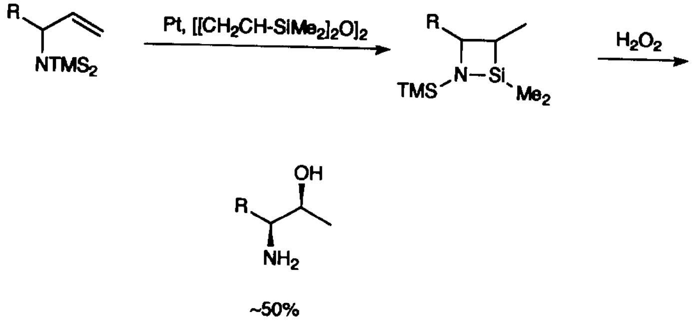
40-60% yield
0-74% ee

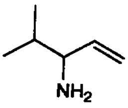
L* = (+)-DIOP
(+)-BINAP
(S)(R)-BPPFA
(S)(S)-chiraphos

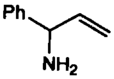
(Equation 554)

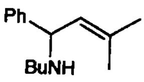


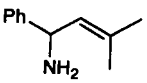
(Equation 555)

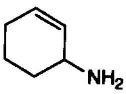




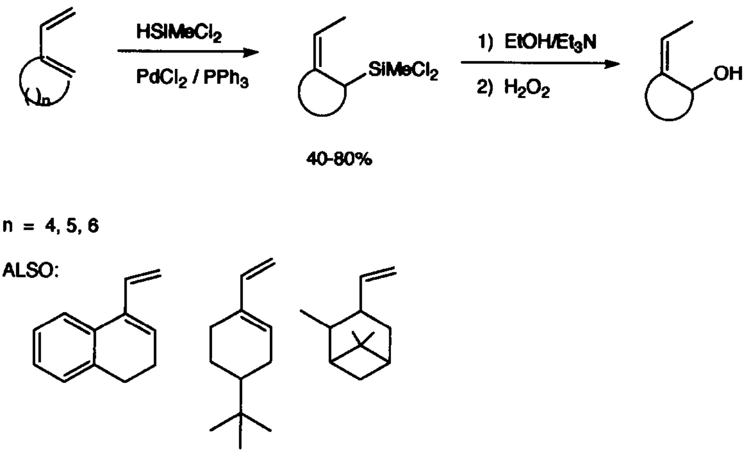






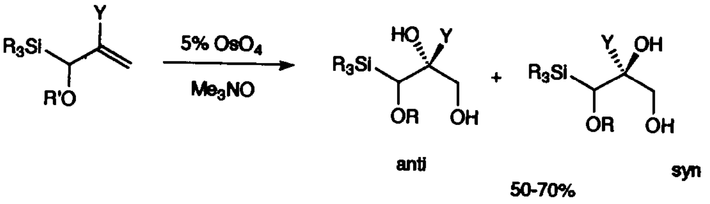


(Equation 556)



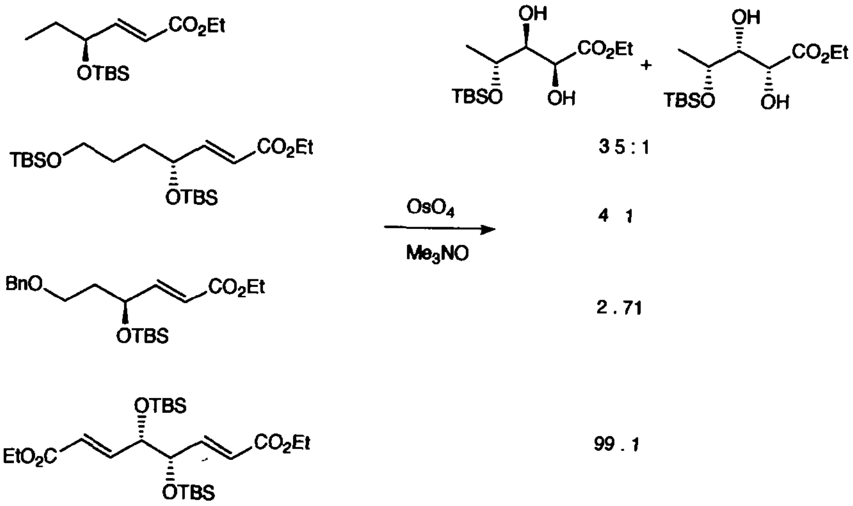
Asymmetric oxidation of olefins with osmium tetroxide has been reviewed (67 references) [727]. The highest ee's in this process have been achieved using $K_3Fe(CN)_6$ as the oxidant in place of trimethylamine N-oxide [728]. By using polymer-bound chiral alkaloid ligands and this oxidant, the hydroxylation of stilbene went in 96% yield and 87% ee, and the alkaloid could be reused [729]. Allylic ethers were *cis* hydroxylated with high stereoselectivity (equation 557) [730], (equation 558) [731] Olefins were *cis* hydroxylated by $OsO_4/Fe(CN)_6$-/DABCO (equation 559). [732].

(Equation 557)

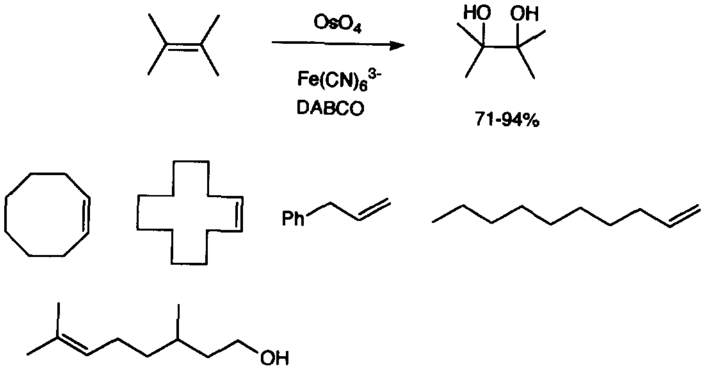


Y = H, 140:1
as size of R$_3$Si increased, % anti increased

(Equation 558)



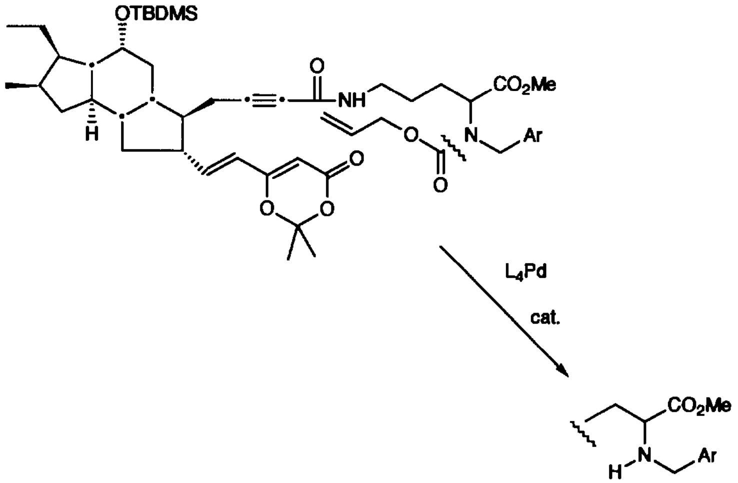
(Equation 559)



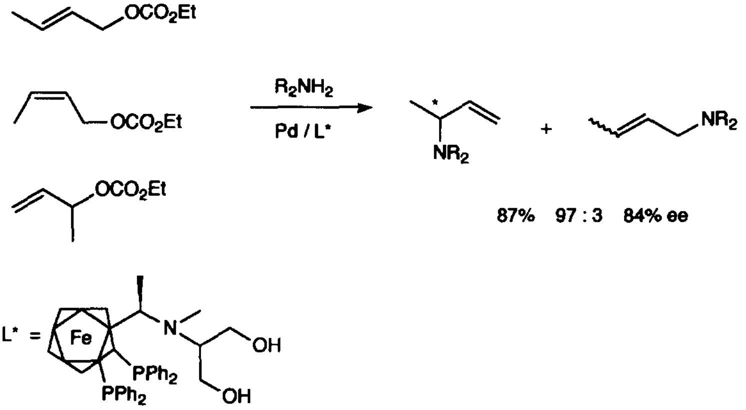
New aspects of oxypalladation of alkenes have been reviewed (54 eferences) [733]. Schiffs bases were reduced to amines by $Ru_3(CO)_{12}$ under hydrogen and carbon monoxide [734]. Platinum complexes catalyzed the reduction of halonitroaromatics to haloamines [735]. Nitrobenzene and aromatic ketones were reduced to anilines and benzyl alcohols over palladium(II)/montmorillonite silylamine catalysts [736].

Palladium(0) complexes catalyzed the removal of allyloxycarbonyl N-protecting groups in a very complex molecule (equation 560) [737]. Allylic carbonates were aminated with high ee using chiral palladium catalysts (equation 561) [738]. Optically active amines were made from aldehydes and chiral ferrocenylamines (equation 562) [739] Iron carbonyl hydrides catalyzed the reductive amination of aldehydes (equation 563) [740] [741], while ruthenium complexes catalyzed the oxidative amination of diols (equation 564) [742]

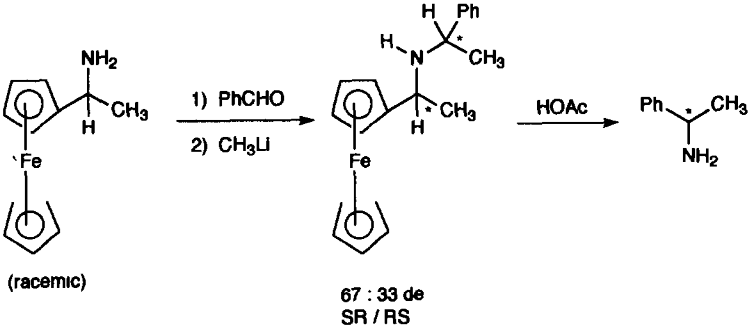
(Equation 560)



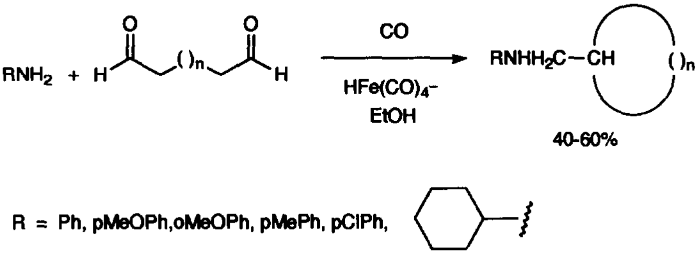
(Equation 561)



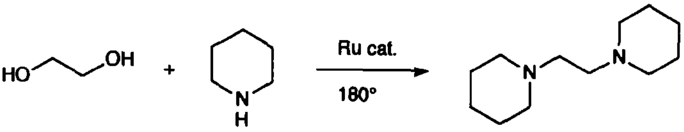
(Equation 562)



(Equation 563)

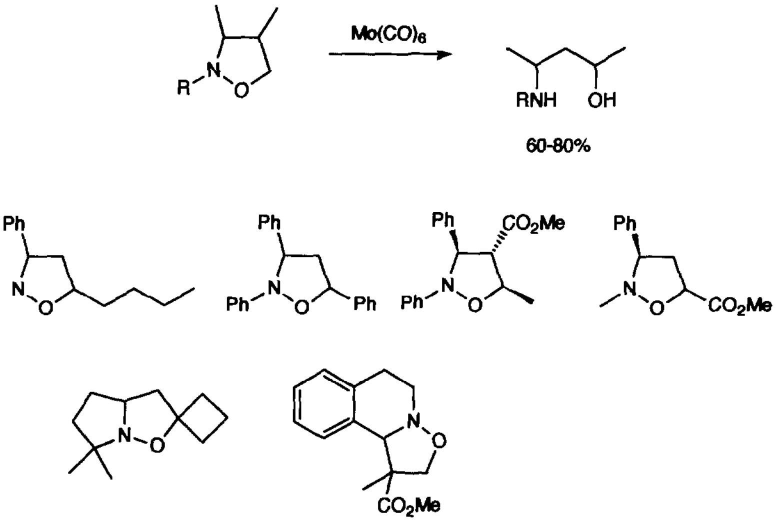


(Equation 564)

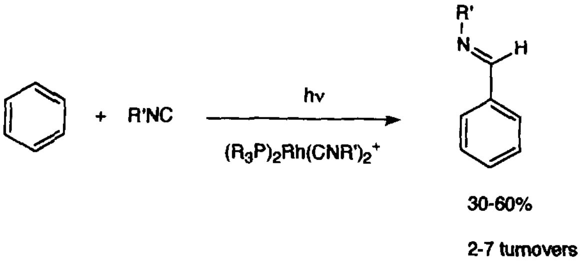


Molybdenum hexacarbonyl ring opened oxazolidines to aminoalcohols (equation 565) [743]. Cationic rhodium(I) complexes catalyzed the reaction of benzene with isonitriles to give imines (equation 566) [744] Ruthenium carbonyls catalyzed the reduction of oximes to imines (equation 567) [745] while cobalt complexes catalyzed the formation of oximes from enones (equation 568) [746] and palladium on carbon catalyzed the reduction of nitroolefins to oximes (equation 569) [747]. Palladium(0) complexes catalyzed the conversion of allyl acetates to allyl azides (equation 570) [748]

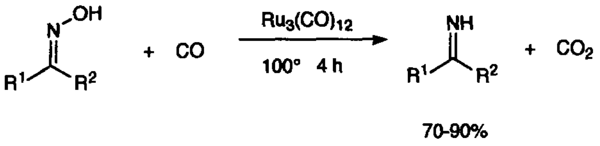
(Equation 565)



(Equation 566)



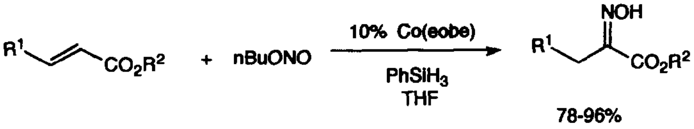
(Equation 567)



R^1 = Ph, Bu, pClPh

R^2 = Et, iPr, Ph, Me, nBu

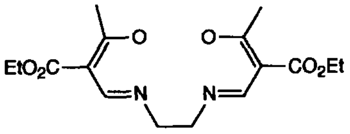
(Equation 568)



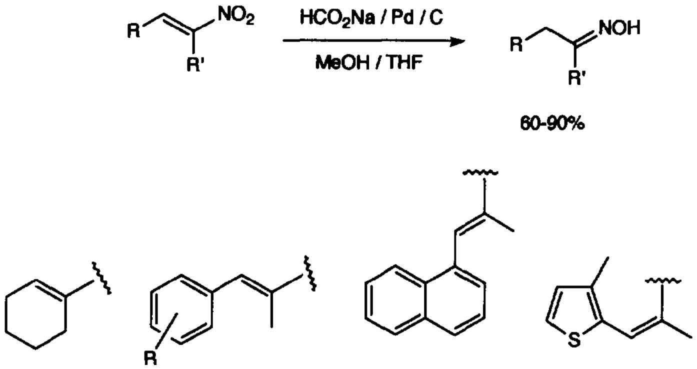
R^1 = H, Me, nPr, iPr, CO_2Et

R^2 = nBu, $PhCH_2$, tBu, ptol, Et

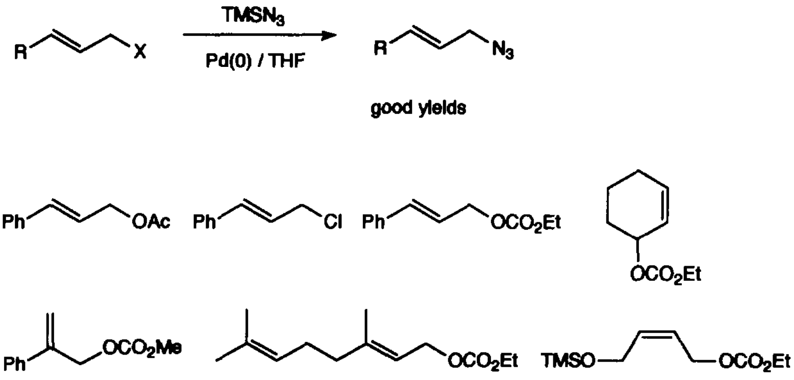
eobe =



(Equation 569)



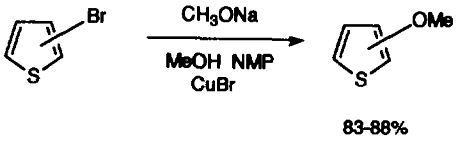
(Equation 570)



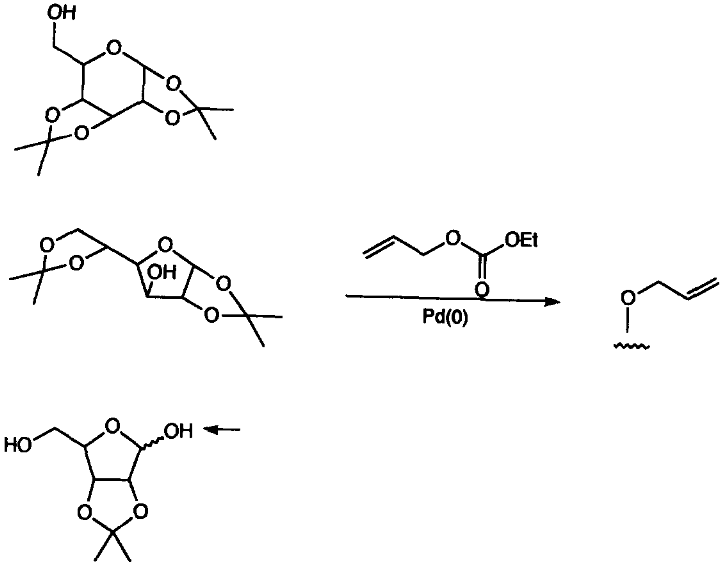
D Ethers, Esters, Acids

Copper bromide catalyzed the alkoxylation of bromothiophenes (equation 571) [749] Palladium(0) catlayzed the o-allylation of sugars (equation 572) [750] while palladium(II) catalyzed the methoxylation of norbornene (equation 573) [751]. Rhodium(II) catalyzed the rearrangement of α-diazo-β-methoxy ketones to enol ethers (equation 574) [752].

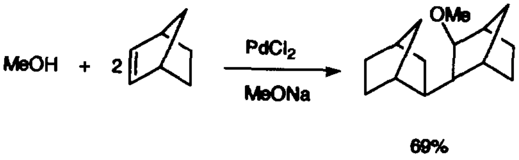
(Equation 571)



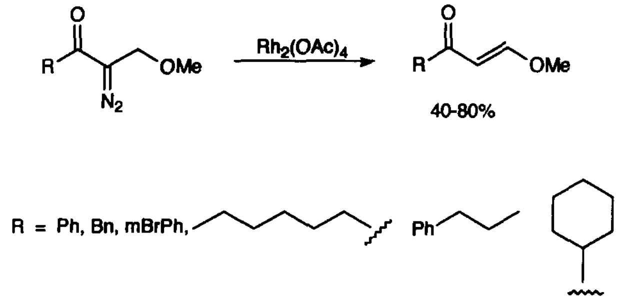
(Equation 572)



(Equation 573)

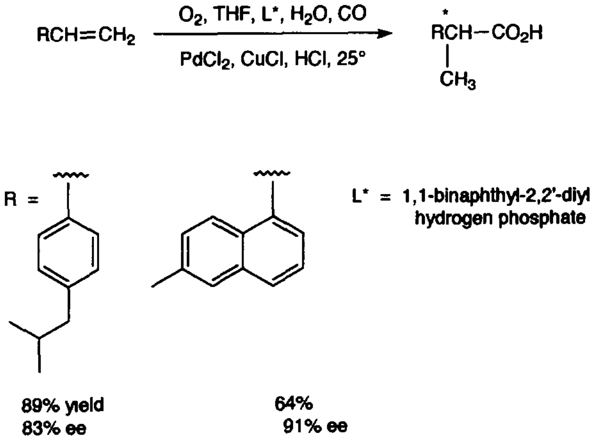


(Equation 574)

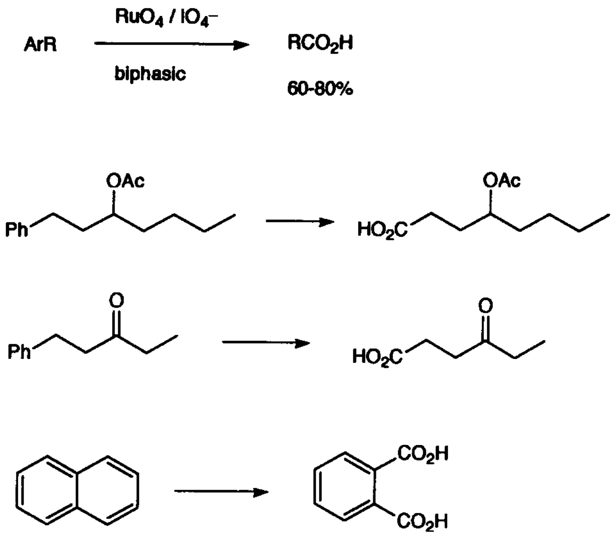


Terminal olefins were oxidized to optically active acids using Wacker type conditions with chiral catalysts (equation 575) [753]. Arenes were oxidized to acids by RuO_4/periodate (equation 576) [754], as were epoxy alcohols (equation 577) [755] Iridium oxygen complexes oxidized alcohols to carboxylic acids (equation 578) [756] Amides were converted to α-amino acids via chromium carbene complexes (equations 579 and 580) [757]

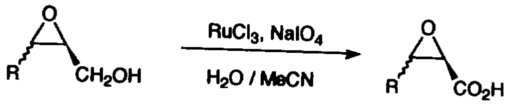
(Equation 575)



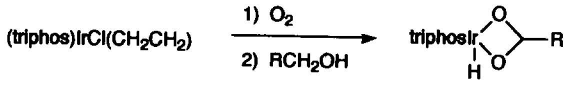
(Equation 576)



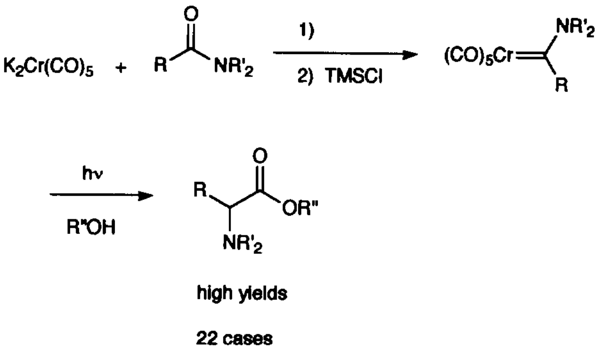
(Equation 577)



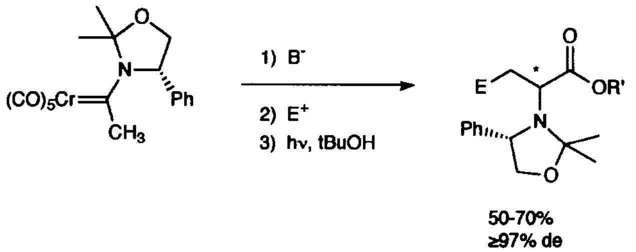
(Equation 578)



(Equation 579)

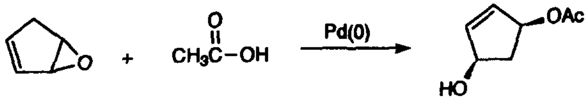


(Equation 580)

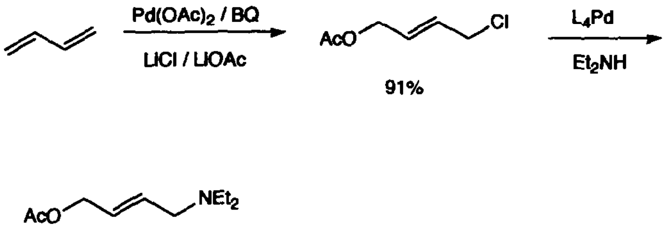


An improved procedure for the palladium(II) catalyzed chloroacetoxylation or diacetoxylation of cyclic 1,3-dienes has been developed [758]. Palladium(0) complexes catalyzed the syn 1,4-addition of acetic acid to cyclopentadiene monoepoxide (equation 581) [759]. The palladium(II) catalyzed chloroacetoxylation of dienes was used to make allyl amines, ultimately (equation 582) [760]. New procedures for the bis acetoxylation of dienes (equation 583) [761], (equation 584) [762] and the allylic acetoxylation of olefins (equation 585) [763], (equation 586) [764] were developed

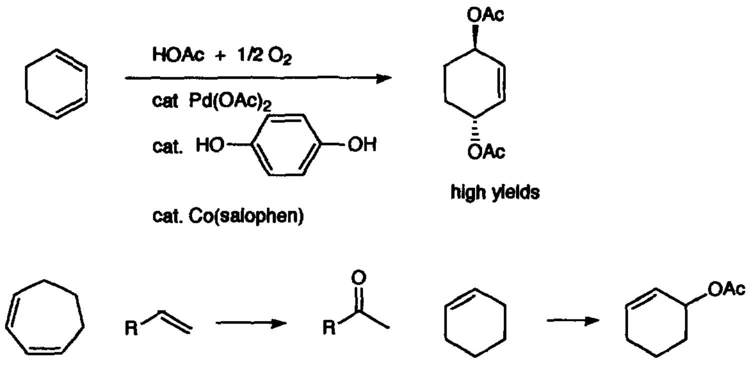
(Equation 581)



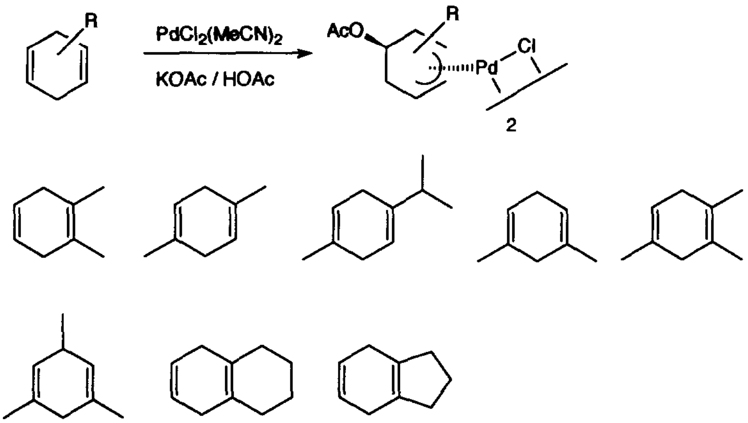
(Equation 582)



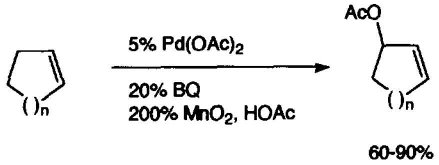
(Equation 583)



(Equation 584)

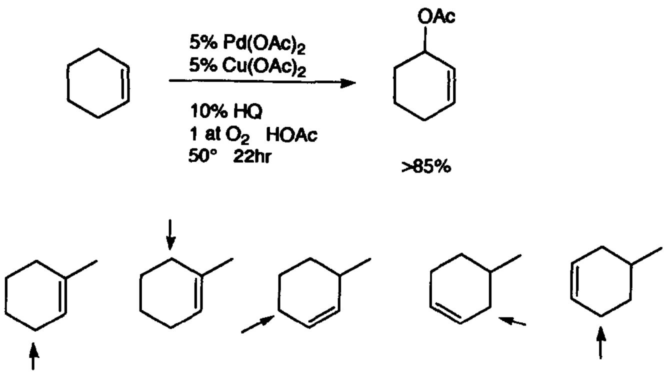


(Equation 585)



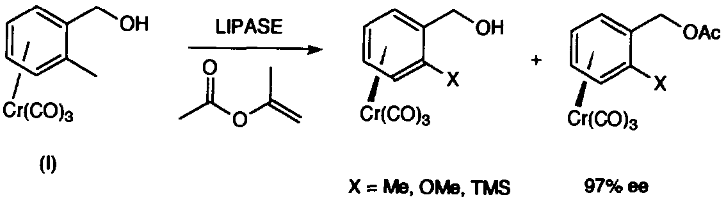
n = 1, 2, 3, 4, 6, 8

(Equation 586)

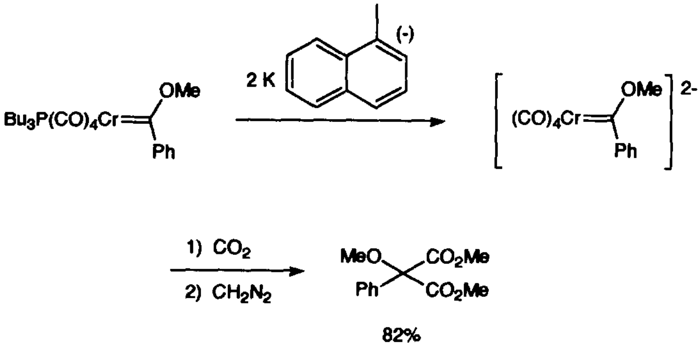


Alkanes and arenes were trifluoroacetoxylated by palladium(II) acetate in trifluoroacetic acid [765]. Lipase selectivity acylated one enantiomer of a racemic mixture of the chromium complexes of o-methylbenzyl alcohol (equation 587) [766] Chromium carbene complexes were converted to diesters by reduction followed by reaction with CO_2 (equation 588) [767] Ruthenium complexes catalyzed the addition of carboxylic acids to eneynes (equation 589) [768]. Chromium complexed phenol formed sugar acetals (equation 590) [769].

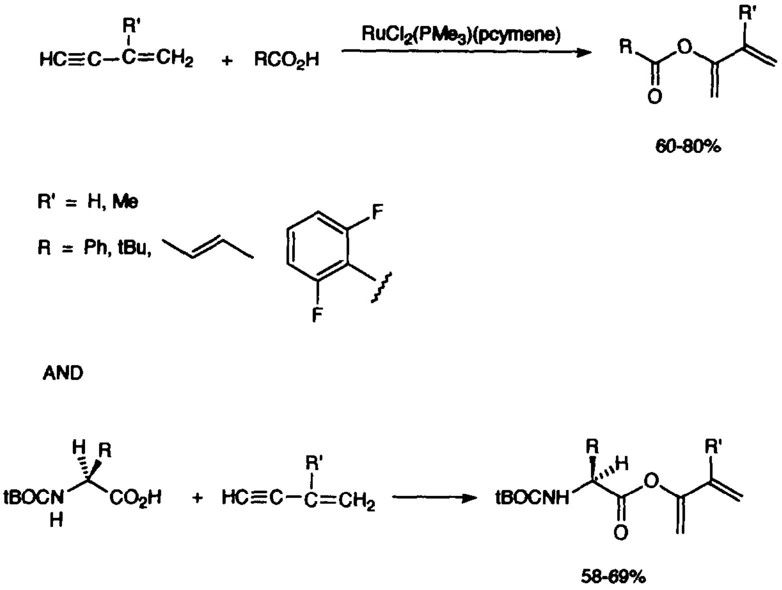
(Equation 587)



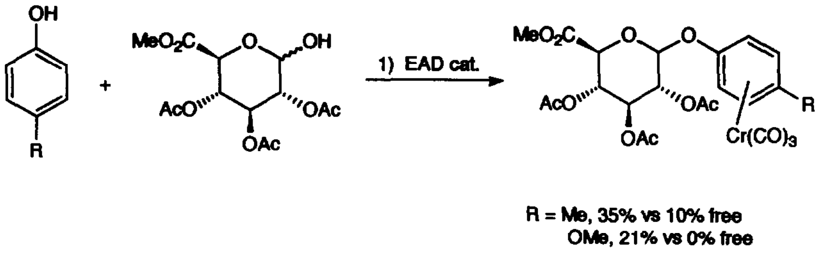
(Equation 588)



(Equation 589)



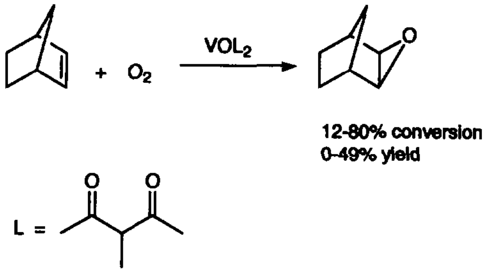
(Equation 590)



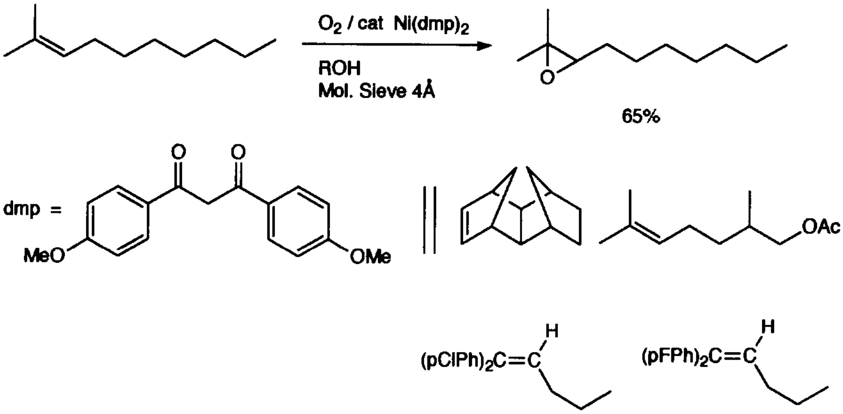
E. Heterocycles

Metal-catalyzed epoxidations continue to be very actively explored, with many references each year. Molybdenum catalyzed epoxidations of 3-chloro-1-butene [770] and cyclohexene [771] [772] with t-butylhydroperoxide have been reported. Olefins were efficiently epoxidized by hydrogen peroxide/sodium tungstate [773], by oxygen vanadate systems (equation 591) [774] and by oxygen/nickel systems (equation 592) [775].

(Equation 591)



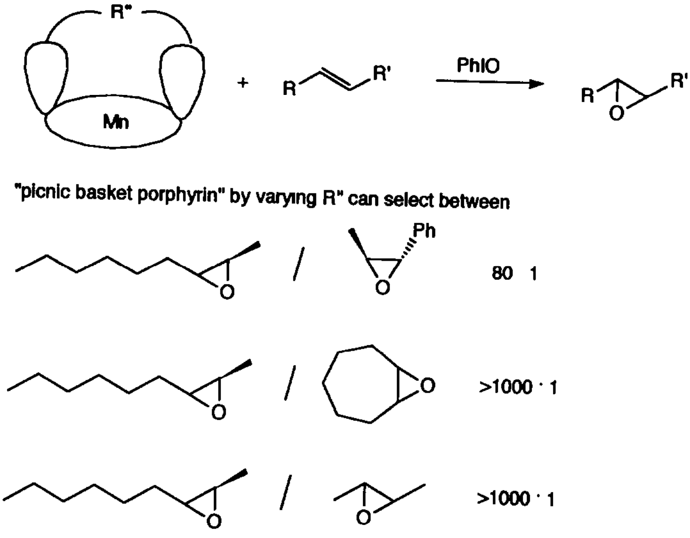
(Equation 592)



Iodosyl benzene epoxidized olefins in the presence of transition-metal phthalocyanines [776] and manganese (salen)-amino acid complexes [777] Homogeneous catalysis of epoxidation of olefins by mono- and binuclear Schiff base complexes has been reviewed (28 references) [778].

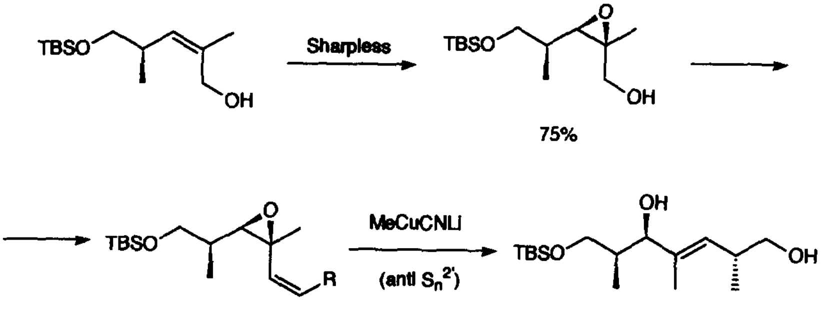
Molybdenum [779] and manganese porphyrins [780] [781] were effective olefin epoxidation catalysts Manganese "picnic basket" porphyrins catalyzed the shape-selective epoxidation of olefins (equation 593) [782]

(Equation 593)

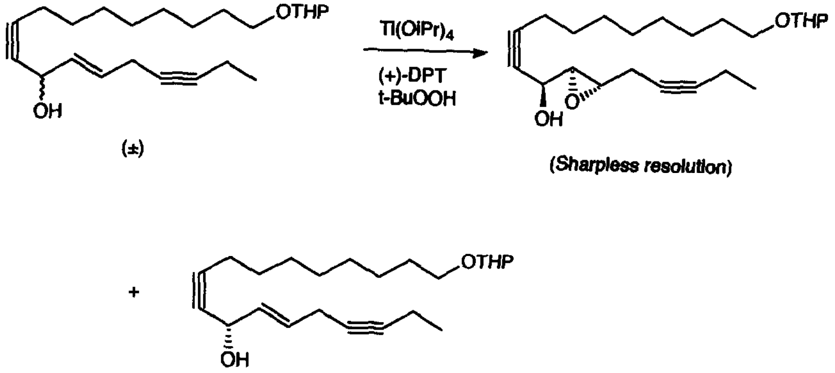


The origin of enantioselectivity in the Katsuki-Sharpless epoxidation procedure has been considered [783]. Sharpless epoxidation of 1,1-dimethoxy-3-hexen-5-ols and 1,1-dimethoxy-4-hexen-3-ols went with high enantio- and diastereoselectivity [784] This process was used extensively in total synthesis (equation 594) [785], (equation 595) [786], (equation 596) [787], (equation 597) [788], (equation 598) [789]. A relatively simple manganese compound was an efficient catalyst for the asymmetric epoxidation of olefins by iodosyl benzene (equation 599) [790].

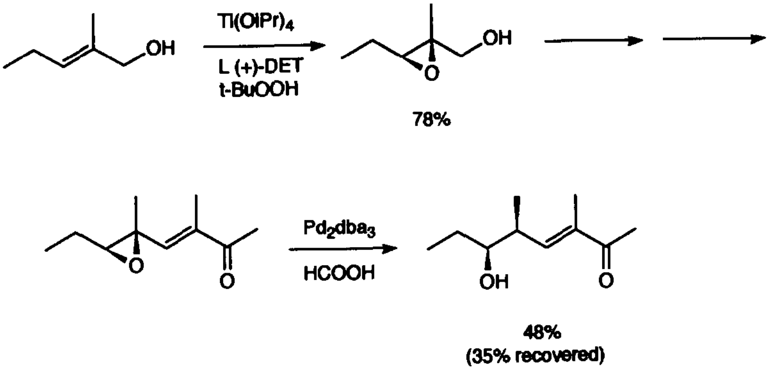
(Equation 594)



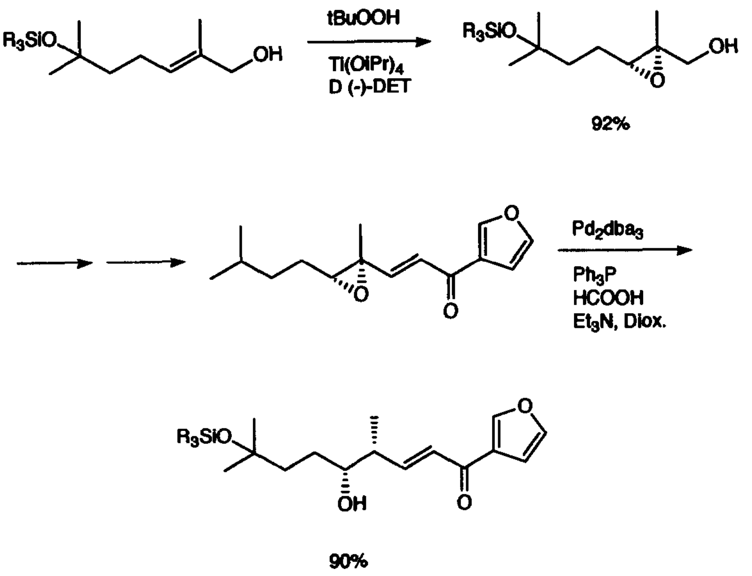
(Equation 595)



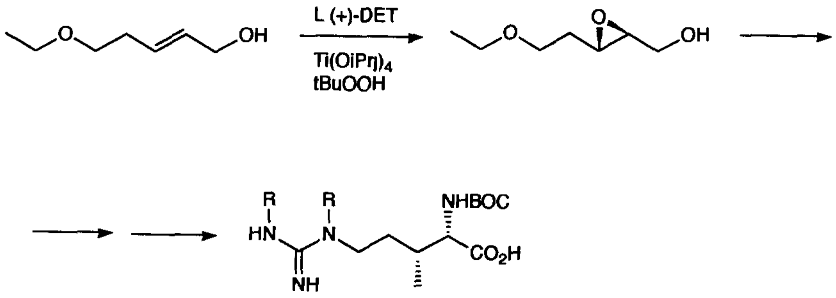
(Equation 596)



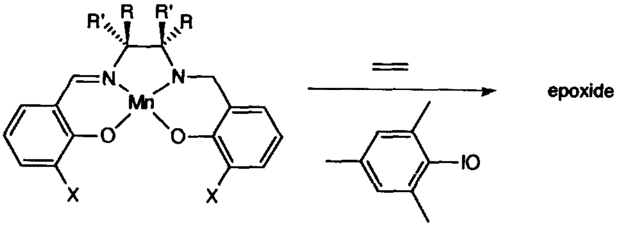
(Equation 597)



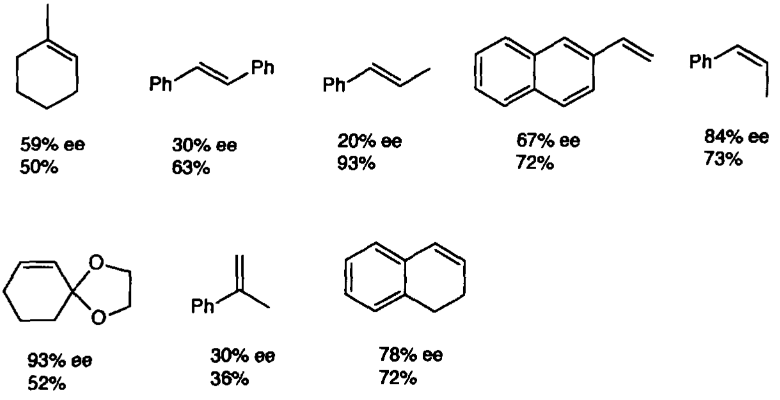
(Equation 598)



(Equation 599)

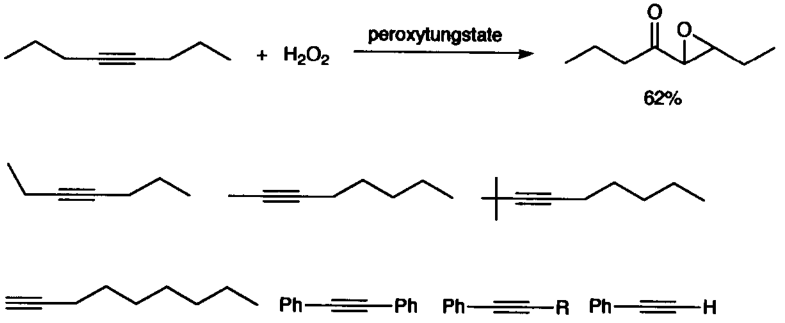


S,S-1 R = Ph, R' = H; X = H
R = H, R' = Ph; X = H
R = H, R' = Ph; X = tBu

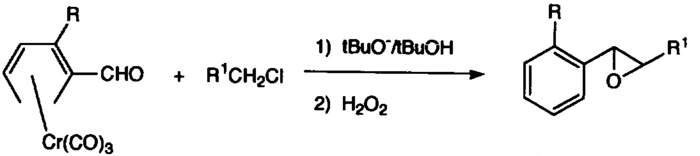


Peroxytungstate catalyzed the epoxidation of alkynes to epoxyketones (equation 600) [791] Chromium complexed benzaldehydes were converted to epoxides by treatment with alkyl chlorides/peroxide (equation 601) [792].

(Equation 600)



(Equation 601)

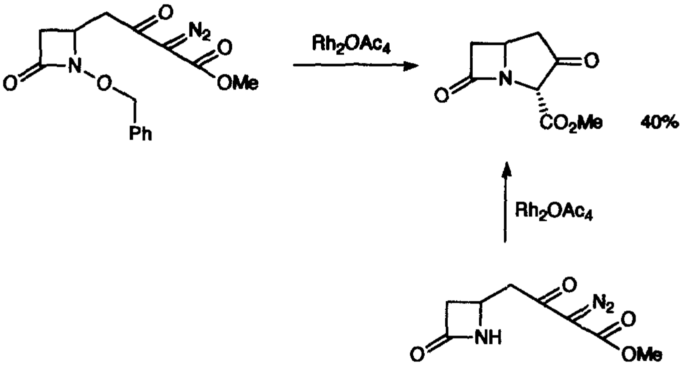


45-97% de
65-85% yield

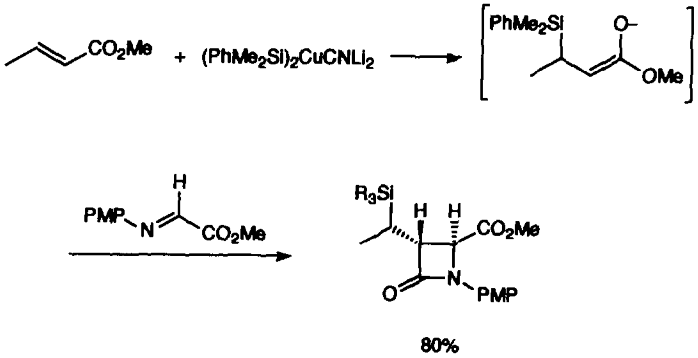
R = Me, OMe, Cl
R^1 = CN, CO_2tBu, COPh
(+)- or (-)-single enantiomer

Transition-metal mediated approaches to α-amino acids, β-lactams and mitomycin [793] and β-lactams from cycloaddition of nitrones with copper acetylides [794] were topics of dissertations. Bicyclic β-lactams were synthesized by the rhodium(II) catalyzed decomposition of diazoketones (equation 602) [795]. β-Lactams were prepared by the reaction of copper-derived ester enolates with imines (equation 603) [796]. Manganese(III) acetate produced a β-lactam from a chloroacetamide (equation 604) [797]. A wide range of optically active β-lactams were made by photolysis of optically active aminocarbene chromium complexes in the presence of imines (equation 605) [798]. A relay to (+)-thienamycin was prepared by palladium assisted alkylation of an optically active ene carbamate, itself produced from a chromium carbene complex (equation 606) [799]. Diazirines were carbonylated to diazetidinones by palladium and cobalt catalysts (equation 607) [800].

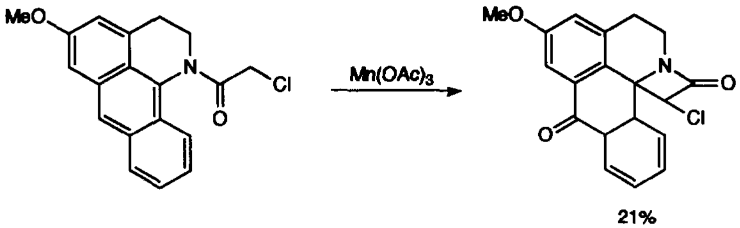
(Equation 602)



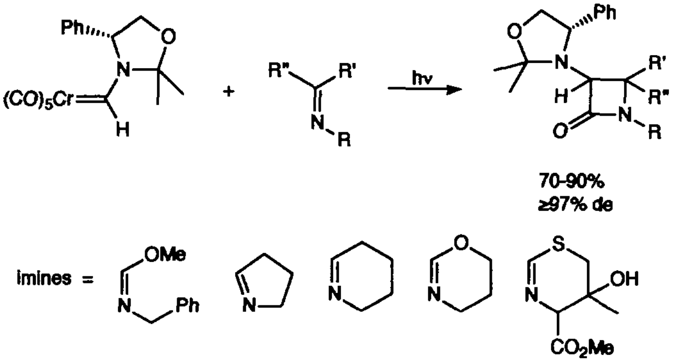
(Equation 603)



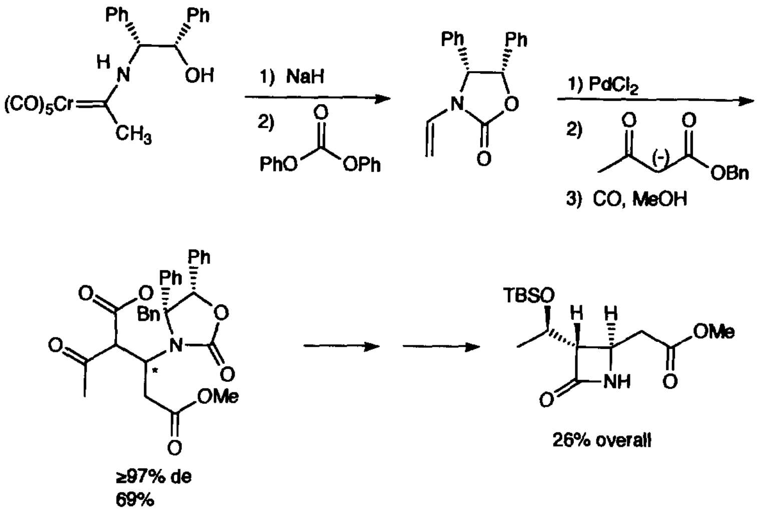
(Equation 604)



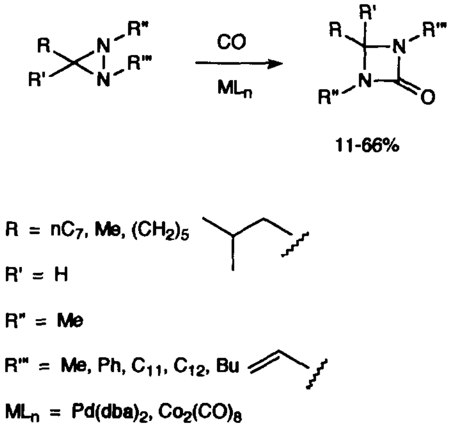
(Equation 605)



(Equation 606)

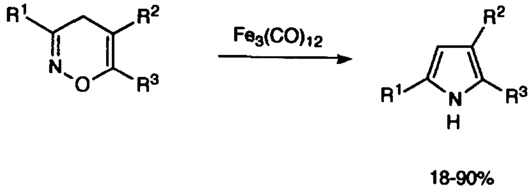


(Equation 607)



Pyrroles were made by the molybdenum (equation 608) [801] and iron (equation 609) [802] catalyzed ring contraction/extrusion reaction of six-membered heterocycles. Iron azadiene (equation 610) [803] and diazadiene complexes (equation 611) [804] were converted to pyrroles

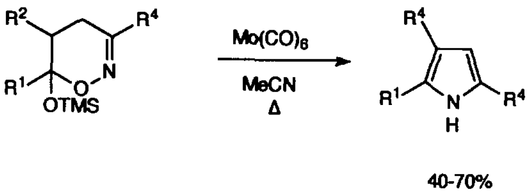
(Equation 608)



R^1 = Ph, EtO_2C

R^2, R^3 = $(CH_2)_4$, $(CH_2)_3$, $(CH_2)_5$, Et, Me, H, Ph

(Equation 609)



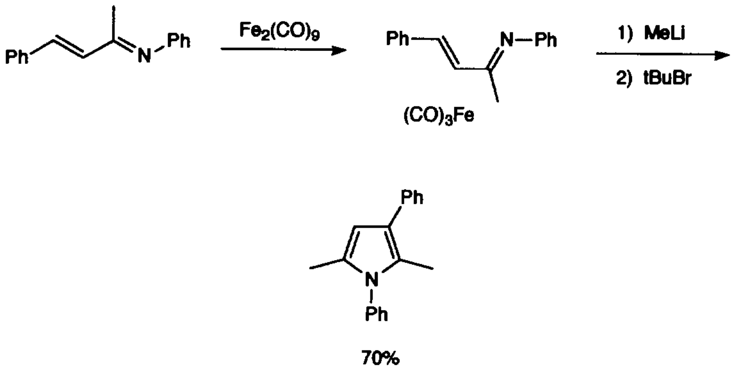
R^1 = H, Me

$(CH_2)_3$, $(CH_2)_4$

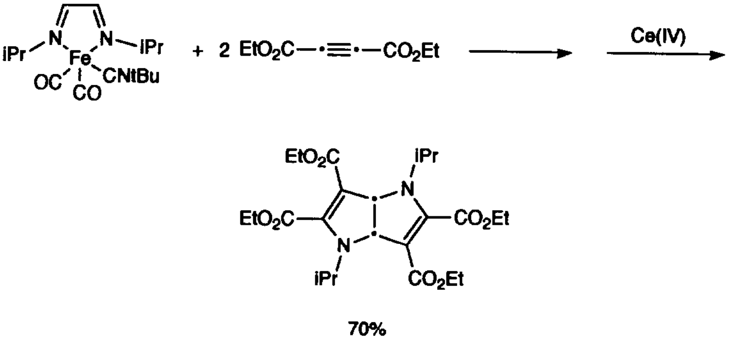
R^2 = H, Me

R^4 = Ph, CO_2Me

(Equation 610)

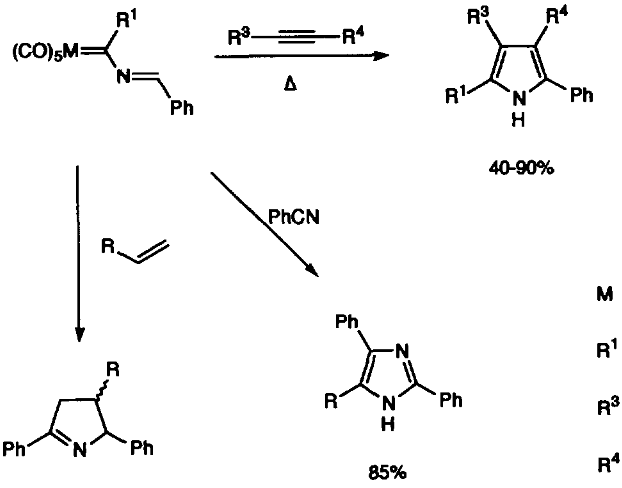


(Equation 611)



Pyrroles were synthesized from alkynes and chromium iminocarbene complexes (equation 612) [805] [806], (equation 613) [807], from ynamines and imidato carbenes (equation 614) [808], and from ω-acetylenic aminocarbene complexes (equation 615) [809]

(Equation 612)



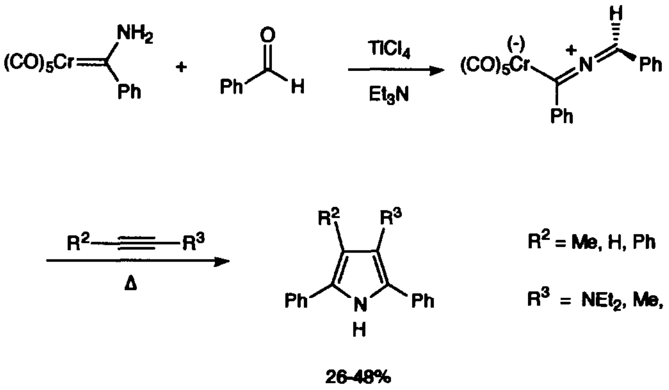
M = W, Cr

R^1 = Me, Ph, tBu

R^3 = nPr, OEt, Et

R^4 = H, nPr, COCH$_3$, Et

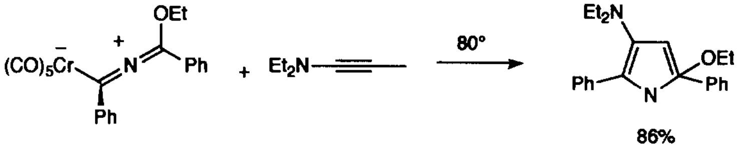
(Equation 613)



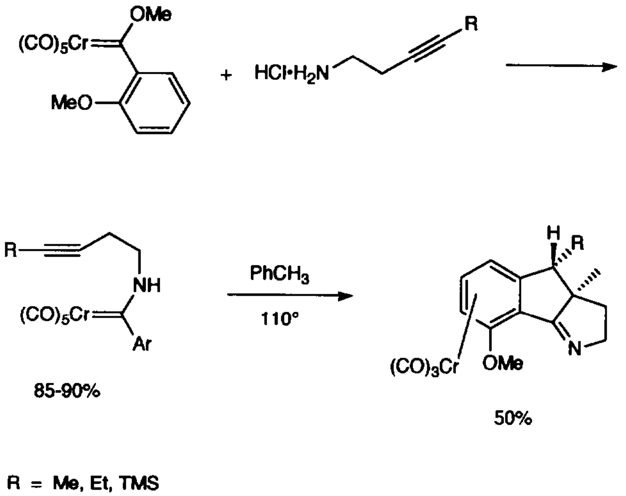
R^2 = Me, H, Ph

R^3 = NEt$_2$, Me, Ph, CH$_2$OMe

(Equation 614)

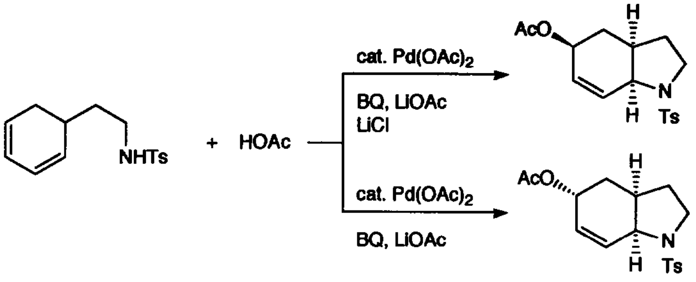


(Equation 615)

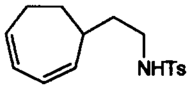


Pyrrolidines were prepared by palladium-catalyzed acetoxylation of dienes (equation 616) [810], and palladium-catalyzed insertion of dienes into α-iodo-N-tosylanilines (equation 617) [811] Indoles were prepared by palladium (equation 618) [812] and ruthenium catalyzed processes (equation 619) [813] Pyrrolidinones were made by insertion of isocyanates into o-manganated arenes (equation 620) [814] and from palladium catalyzed olefin insertion reactions (equation 621) [815].

(Equation 616)

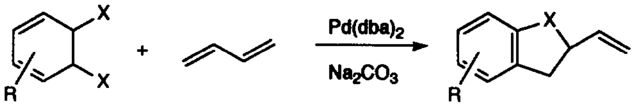


80-90%



also works.

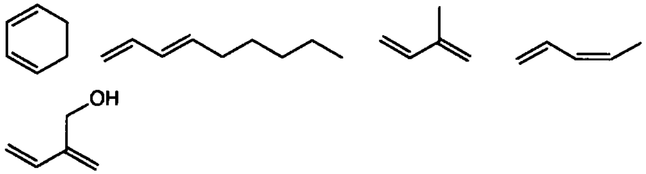
(Equation 617)



50-80%

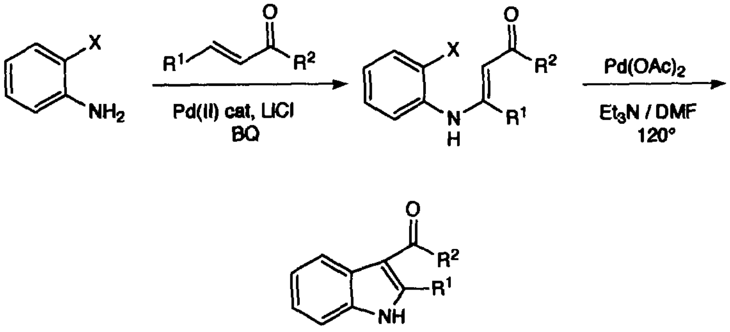
X = NHTs, OH

diene =



R = COMe, CHO

(Equation 618)



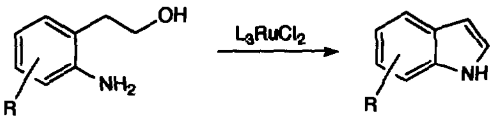
35-71%

R^1 = H, Me, CH_2CO_2Et, CO_2Me, CO_2Et, Ph

-$(CH_2)CH(Me_2)(CH_2)$-

R^2 = Me, OEt, Ph, OMe

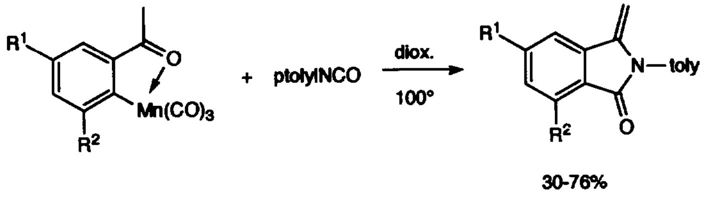
(Equation 619)



70-100%

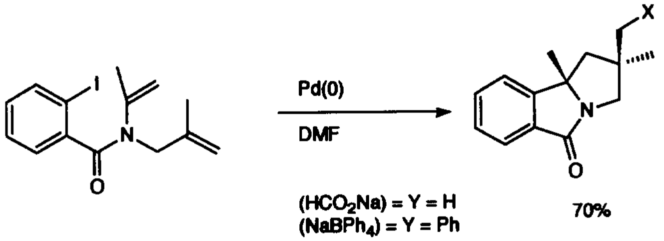
R = H, 4-Me, 5-OMe, 6-Cl, 4-Cl, 6-Br

(Equation 620)



R^1 = H, F, Cl, MeO, CF_3

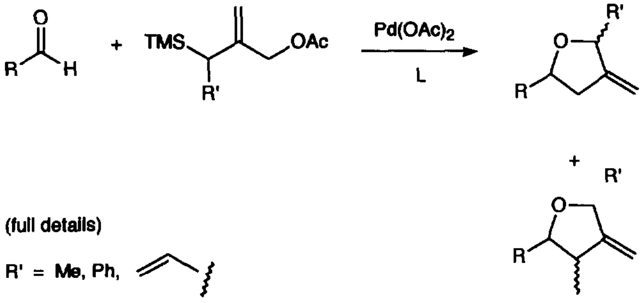
R^2 = H, F, Cl, MeO



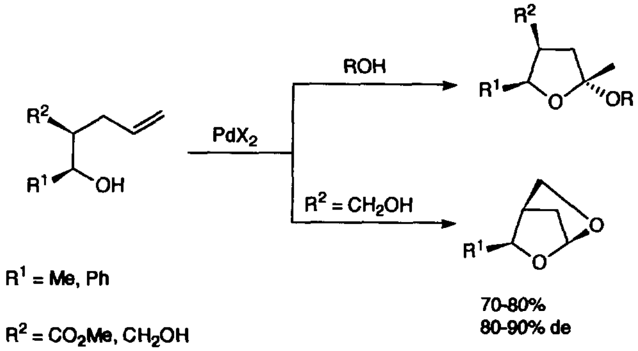
Organopalladium approaches to oxygen heterocycles [816], and a palladium catalyzed synthesis of 4-methylenetetrahydrofurans [817] were topics of dissertations. Palladium catalyzed the [3+2] cycloaddition of trimethylenemethane complexes to aldehydes (equation 622) [818] α-Olefinic alcohols cyclized to tetrahydrofurans in the presence of palladium catalysts (equation 623) [819], (equation 624) [820], (equation 625) [821]. Cobalt(II) catalyzed an oxidative cyclization of this class of compounds (equation 626) [822]. Palladium(0) complexes catalyzed the formation of cyclic acetals form allylzincs and ketones (equation 627) [823]. Furans were synthesized from allenic ketones with rhodium(I) catalysts (equation 628)

[824] and from alkynes and ketones using low-valent tantalum (equation 629) [825]

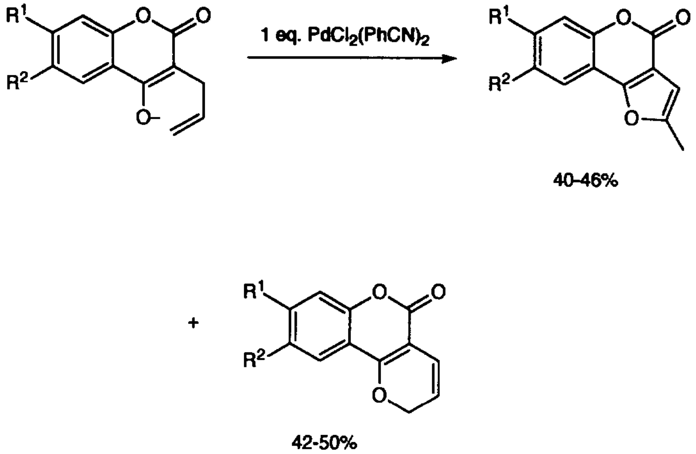
(Equation 622)



(Equation 623)



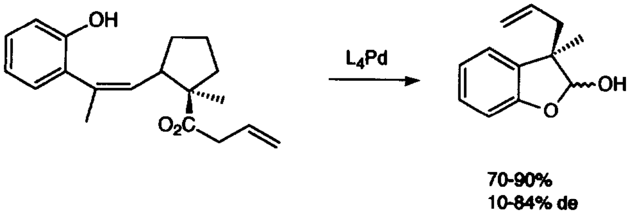
(Equation 624)



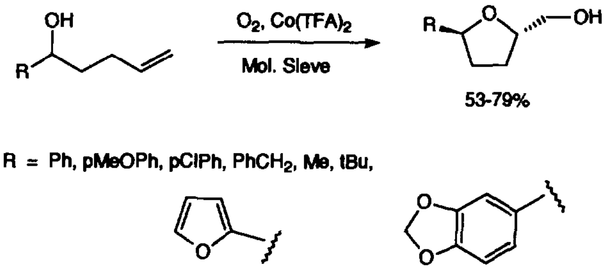
R^1 = H, Cl

R^2 = H, Me, Cl, Br

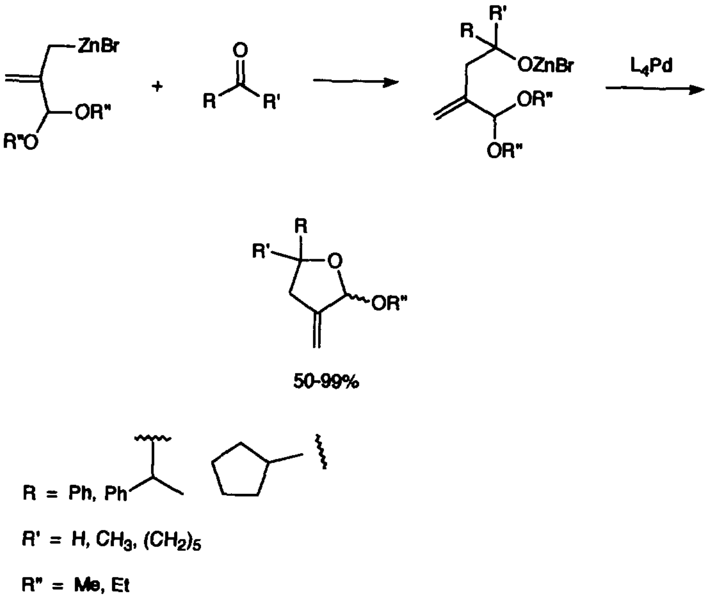
(Equation 625)



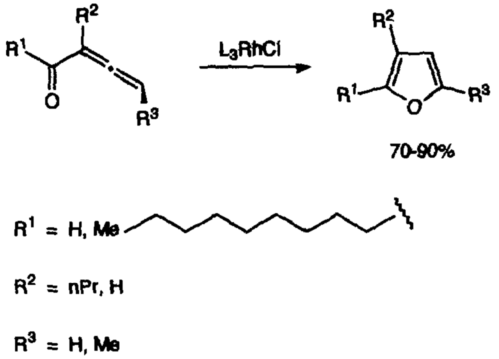
(Equation 626)



(Equation 627)



(Equation 628)

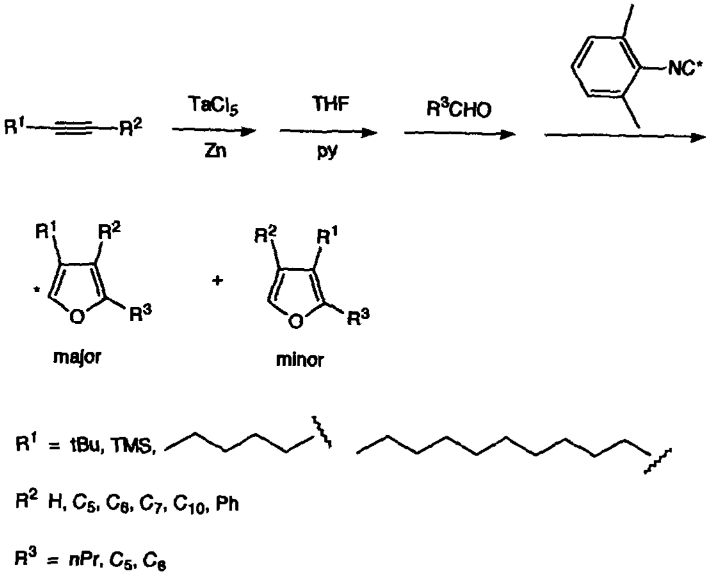


R^1 = H, Me

R^2 = nPr, H

R^3 = H, Me

(Equation 629)



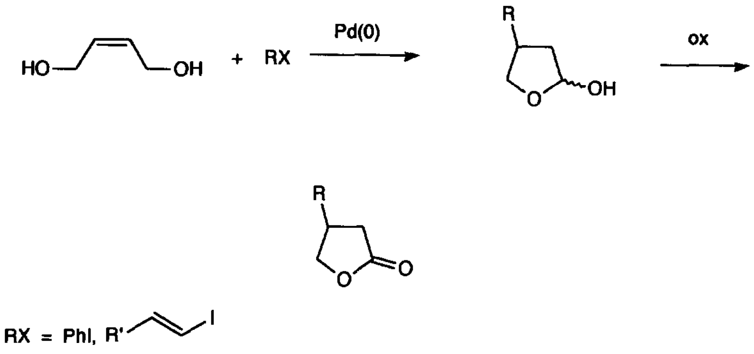
R^1 = tBu, TMS,

R^2 H, C_5, C_6, C_7, C_{10}, Ph

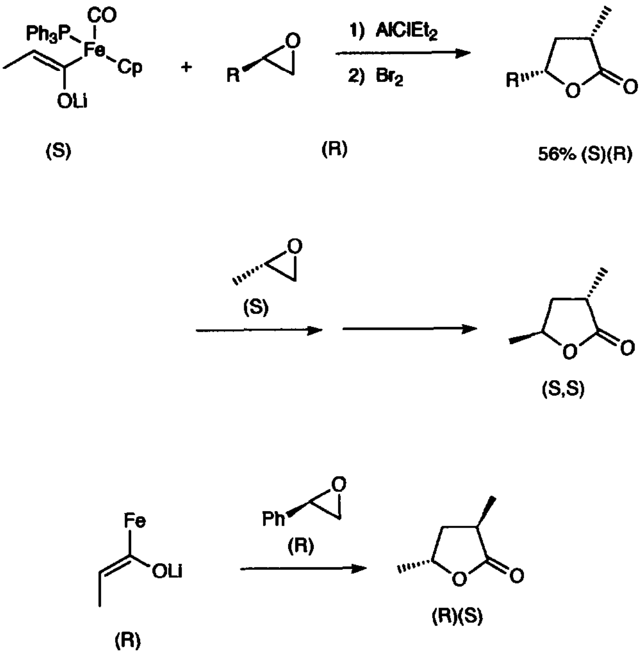
R^3 = nPr, C_5, C_6

γ-Butyrolactones were synthesized by the palladium catalyzed reaction of vinyl halides with butene diols (equation 630) [826], iron acyl enolates with epoxides (equation 631) [827], aromatic dialdehydes with rhodium(I) catalysis (equation 632) [828], reduction of 1,4-diketones with ruthenium binap catalysts (equation 633) [829], the iron(III) catalyzed reaction of alkenes with malonates (equation 634) [830], the reaction of manganese acetylides with epoxides (equation 635) [831], and transition metal catalyzed chloroacylation of olefins (equation 636) [832] Butenolides were formed in the reaction of chromium acylate complexes with alkynes (equation 637) [833] o-Iodophenol, phenylacetylene and carbon monoxide co-cyclized in the presence of palladium catalyst (equation 638) [834].

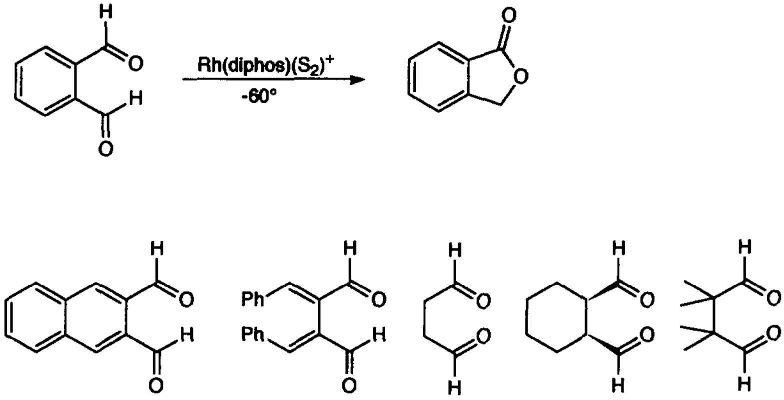
(Equation 630)



(Equation 631)

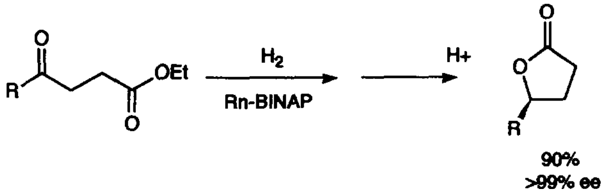


(Equation 632)

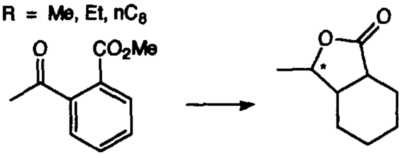


643

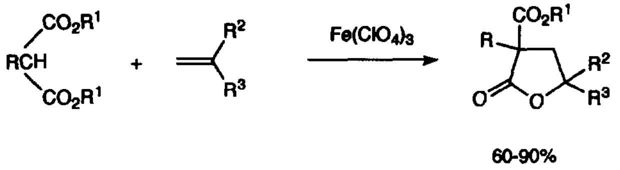
(Equation 633)



R = Me, Et, nC$_8$



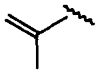
(Equation 634)



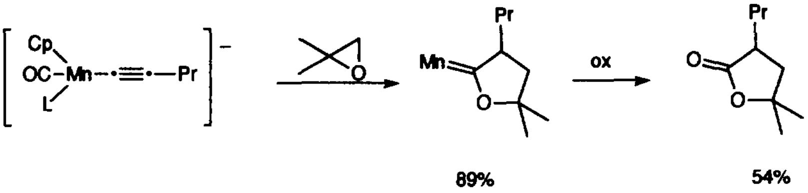
R = Et, PhCH$_2$, Me

R^1 = Me, pClPh, H, nBu, PhCH$_2$, 

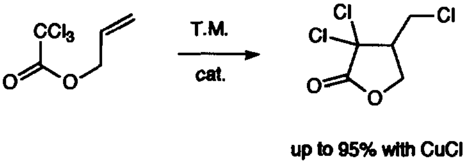
R^2 = H, Me, Ph

R^3 = Ph, pMeOPh, pClPh, tBu, nC$_6$, 

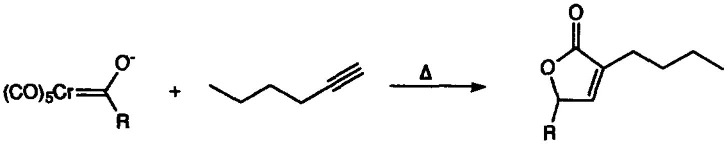
(Equation 635)



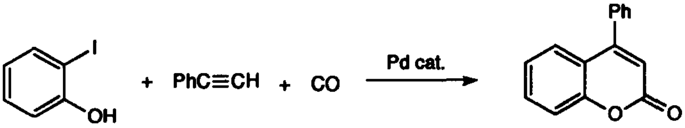
(Equation 636)



(Equation 637)

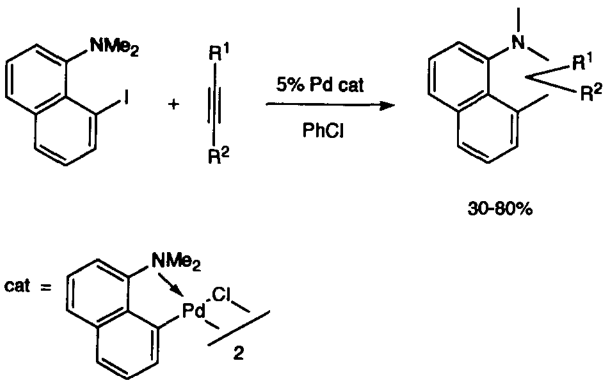


(Equation 638)

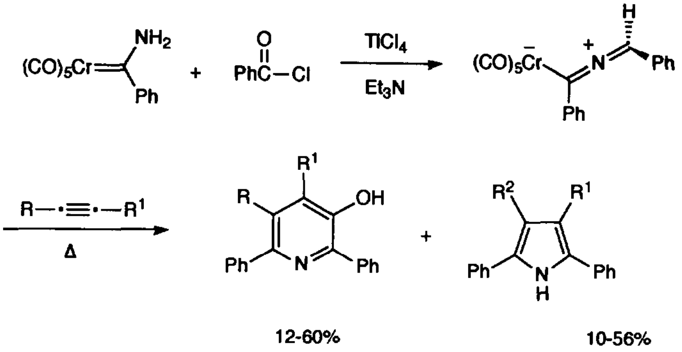


Stereocontrolled synthesis of N-methyl-1,2,3,4-tetrahydroisoquinoline derivatives via $Cr(CO)_3$ methodologies was reviewed [835]. Palladium catalyzed the formation of quinolines from alkynes and iodoaromatic amines (equation 639) [836]. Chromium aminocarbenes were used to produce a number of nitrogen heterocycles (equation 640) [837], (equation 641) [838], (equation 642) [839]

(Equation 639)



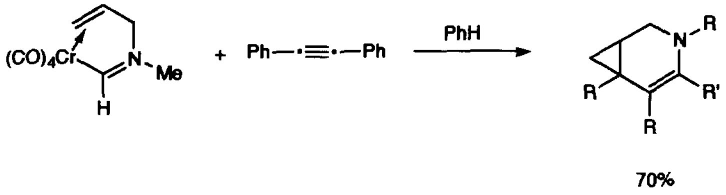
(Equation 640)



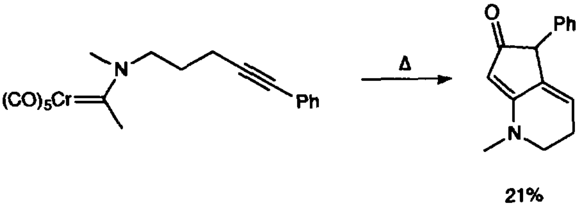
R = H, Me, Ph

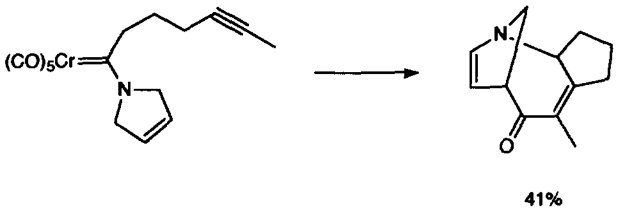
R^1 = Ph, ptol, nBu, TMS, CH_2OMe, NEt_2, CO_2Me

(Equation 641)



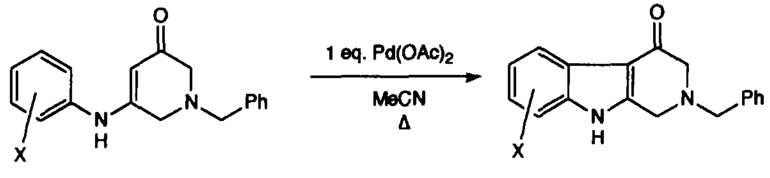
(Equation 642)





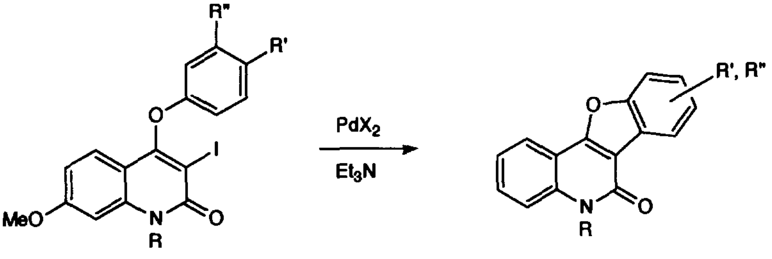
Palladium complexes catalyzed the formation of several six membered nitrogen heterocycles (equation 643) [840], (equation 644) [841], (equation 645) [842]. Nickel(0) complexes cyclooligomerized isoprene and phenylisocyanate to produce piperidinones (equation 646) [843]. Bridging carbene complexes of iron reacted with phosphinimines and alkynes to produce pyridines (equation 647) [844].

(Equation 643)



X = H, 25%; 8-MeO, 21%; 6-MeO, 23%; 5- or 7-MeO, 64%

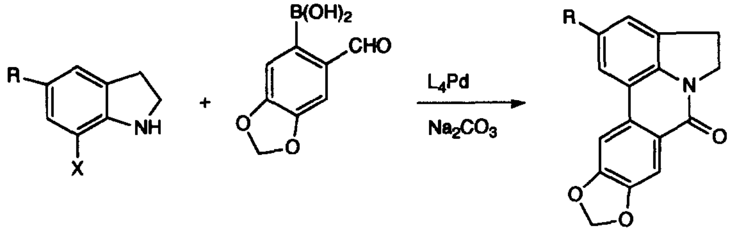
(Equation 644)



60-90%

R = Me, Et; R' = H, OMe; R" = OMe, H

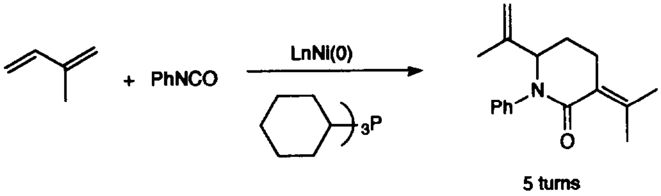
(Equation 645)



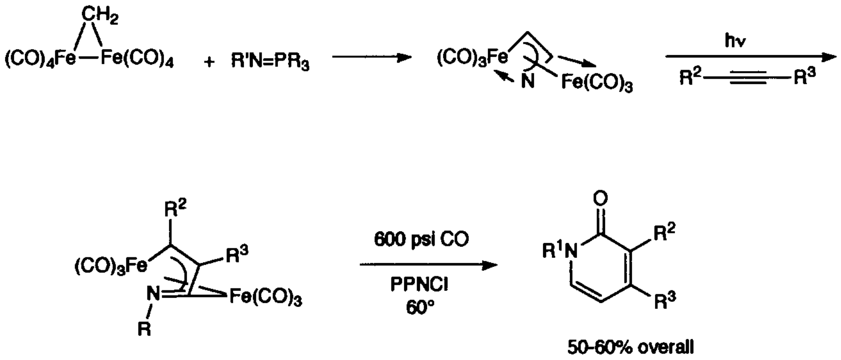
R = MeO, H

40-50%

(Equation 646)



(Equation 647)



R^1 = Ph, tBu, H

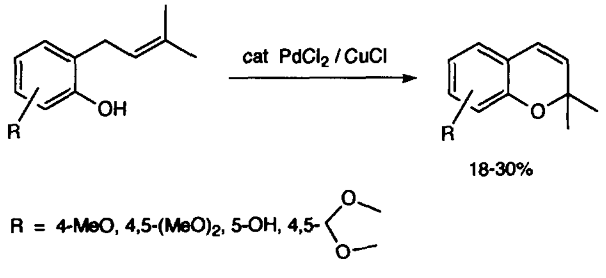
R^2 = tBu, Ph, Et, TMS

R^3 = H, D, Ph

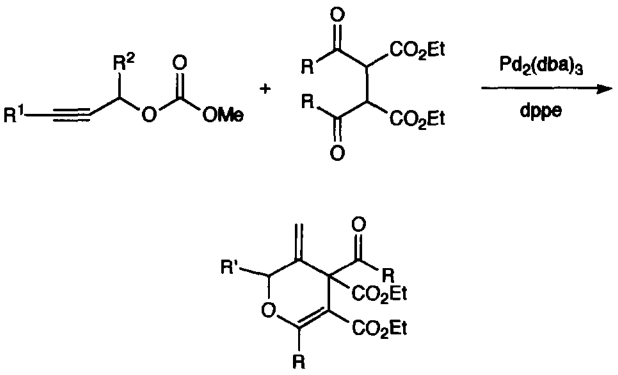
Palladium catalyzed a number of cyclizations to the pyran or coumarin ring systems (equation 648) [845], (equation 649) [846], (equation 650) [847], (equation 651) [848]. Group(VI) Cp nitrosyl carbonyl compounds effected the coupling of olefins and methyl propriolate to give oxygen heterocycles (equation 652) [849]. Vanadium complexes catalyzed the cyclization of aldehydes with alkoxy silyloxy dienes (equation 653) [850]

Palladium-bismuth systems catalyzed the formation of ketals of electron poor olefins and propane diol (equation 654) [851]

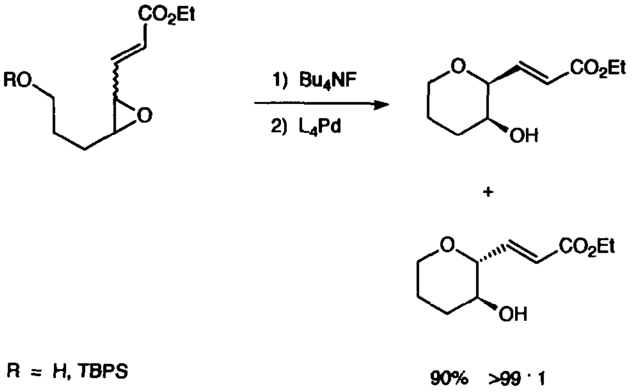
(Equation 648)



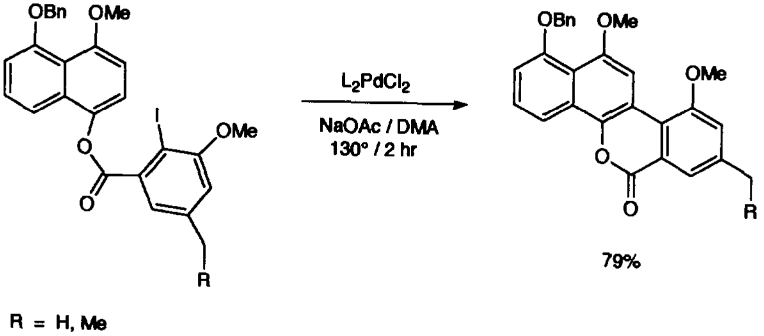
(Equation 649)



(Equation 650)

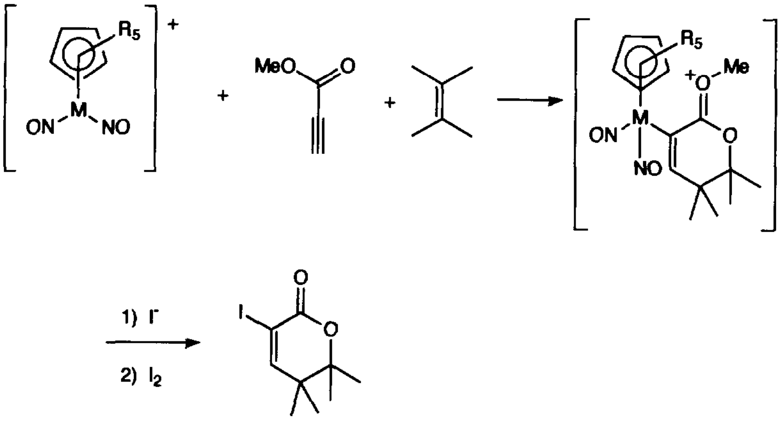


(Equation 651)

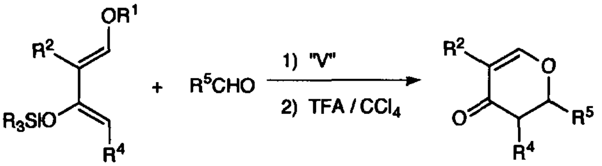


p 643

(Equation 652)



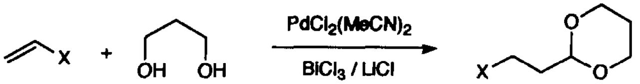
(Equation 653)



80-90%

40-85% ee

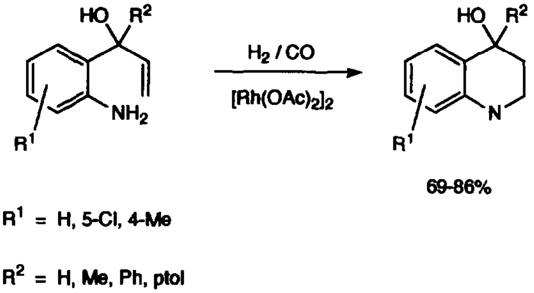
(Equation 654)



X = PhCO, MeCO, MeO_2C, Ph, CN

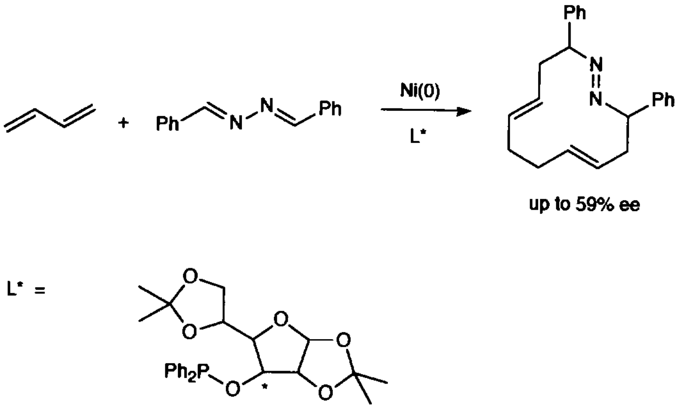
Rhodium acetate catalyzed the carbonylative cyclization of o-allyl anilines (equation 655) [852].

(Equation 655)

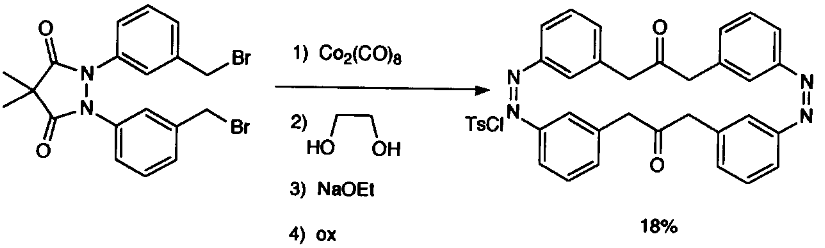


Nickel(0) complexes co-cyclotrimerized butadiene and 2,3-diazadiene (equation 656) [853]. Macrocyclic tetraazo compounds were prepared by cobalt catalyzed carbonylative coupling of bis benzyl halides (equation 657) [854]. Palladium complexes catalyzed the formation of tin-silicon heterocycles (equation 658) [855] and germanocycles (equation 659) [856] from alkynes Low valent zirconium species formed sulfur/silicon heterocycles from acetylenic silanes (equation 660) [857]. Titanio cyclobutenes were used to form arsenic and phosphorus heterocycles (equation 661) [859] while titanium oxygen heterocycles were formed from olefinic ketones (equation 662) [860].

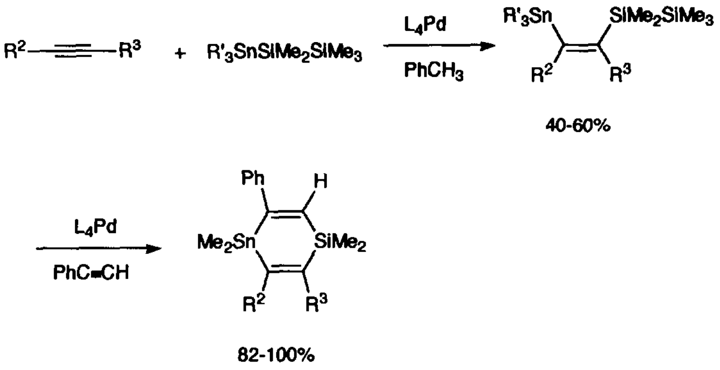
(Equation 656)



(Equation 657)



(Equation 658)

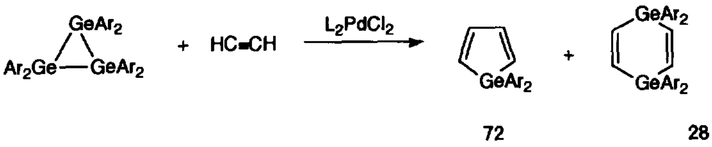


R^1 = Me, Bu

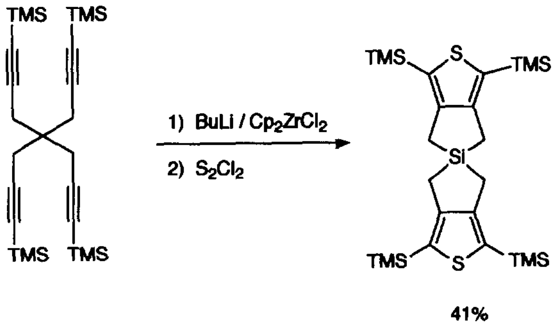
R^2 = Ph, RO_2C

R^3 = H, MeO_2C

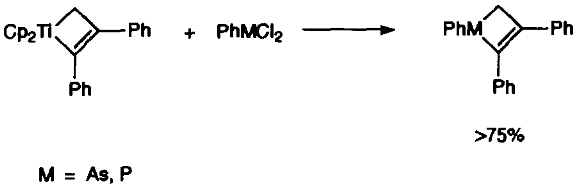
(Equation 659)



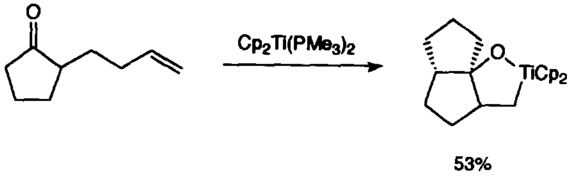
(Equation 660)

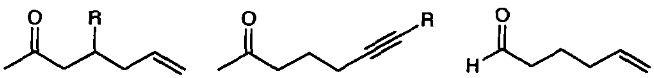


(Equation 661)



(Equation 662)

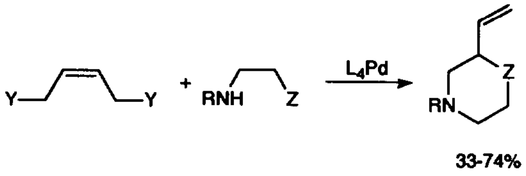




Also work

Heterocycles containing two heteroatoms were prepared by palladium catalysis (equation 663) [860], (equation 664) [861] and were alkylated by palladium(0) chemistry (equation 665) [862]. 1,3-Oxaphospholes were prepared from chromium carbene complexes (equation 666) [863], while chromium complexed benzaldehyde nitrones underwent cycloaddition to olefins (equation 667) [864]. Oxazolidinones were synthesized by intramolecular addition of carbamates to alkynes catalyzed by copper (equation 668) [865].

(Equation 663)



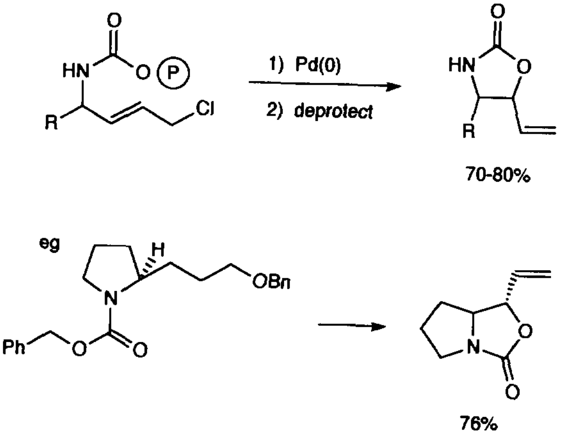
Y = OCO_2Me

R = Bn, Ts

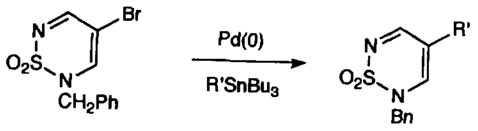
Z = NMe, NH, NBn, TsN, O

n = 3, 4

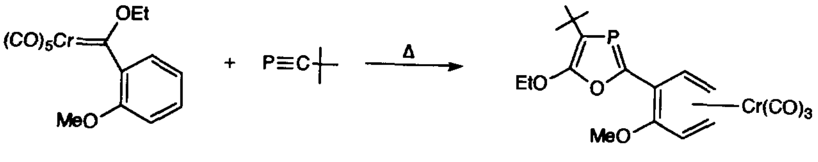
(Equation 664)



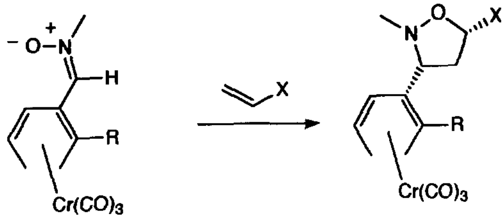
(Equation 665)



(Equation 666)



(Equation 667)

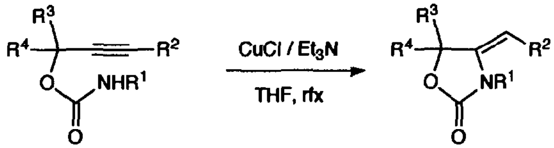


R = H, TMS

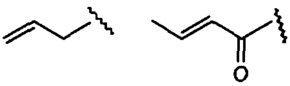
X = Ph, OEt, OAc, TMS

70-95%
>98% cis

(Equation 668)



71-93%

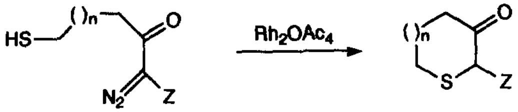
R^1 = pTs, PhCO, MeCO, Ph, pMeOPh 

R^2, R^3, R^4 = H

R^1 = Ts; R^4 = Me, Et; R^3 = Me; R^3, R^4 = $(CH_2)_5$

Rhodium(II) catalyzed decomposition of α-diazoketones was used to synthesize sulfur heterocycles (equation 669) [866], (equation 670) [867] Nickel(0) catalyzed the addition of thioacetamide to o-iodobenzamide (equation 671) [868], and nickel oxide oxidized triazene (equation 672) [869]. Optically active oxazolines were prepared by the gold-catalyzed condensation of aldehydes with isocyanoacetic ester (equation 673) [870] [871].

(Equation 669)

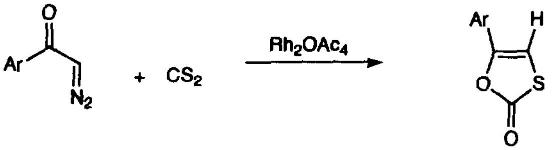


34-47%

$Z = CO_2Et, COMe, p(O)(OEt)_2$

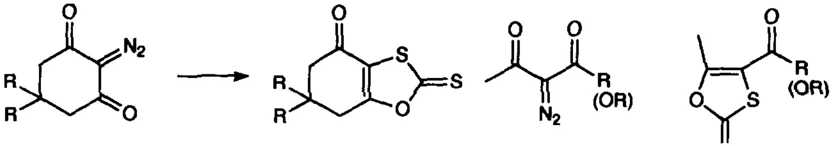
$n = 1, 2$

(Equation 670)



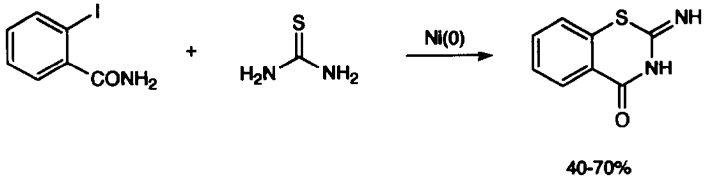
50-70%

Ar = Ph, pMeOPh, pClPh, pNO_2Ph

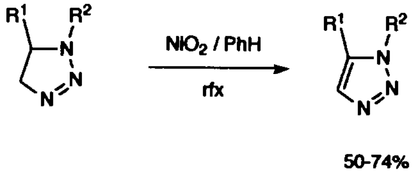


40-90%

(Equation 671)



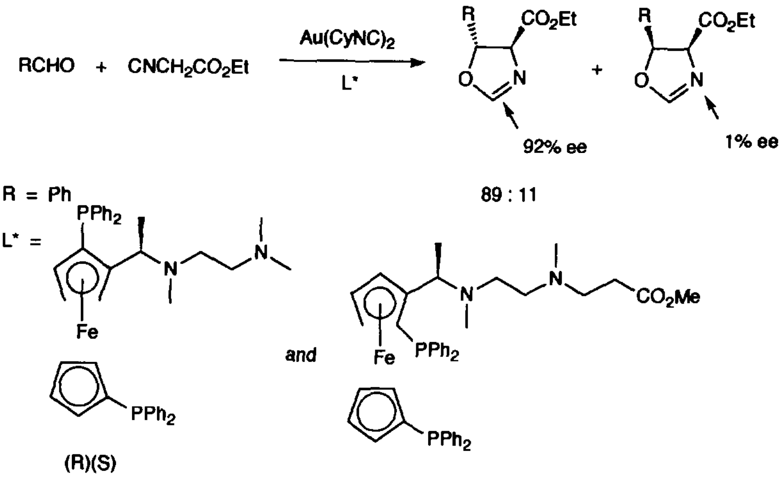
(Equation 672)



R^1 = oClPh, 2,4-Cl_2Ph, 2,6-Cl_2Ph

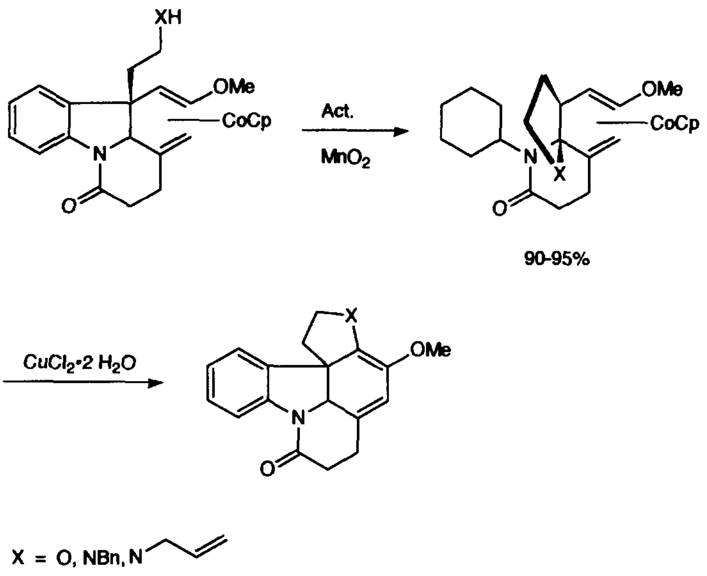
R^2 = pMeOPh, 3,4-Cl_2Ph, pClPh, Ph, mClPh, pFPh, pCF_3Ph, pBrPh

(Equation 673)

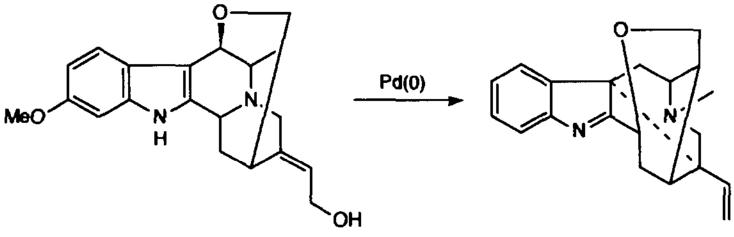


A transannular cyclization occurred when the cobalt heterocycle complex in equation 674 was oxidized by MnO_2, and a rearrangement occurred when the metal was removed with copper(I) chloride [872] Palladium(0) catalyzed the cyclization shown in equation 675 [873].

(Equation 674)



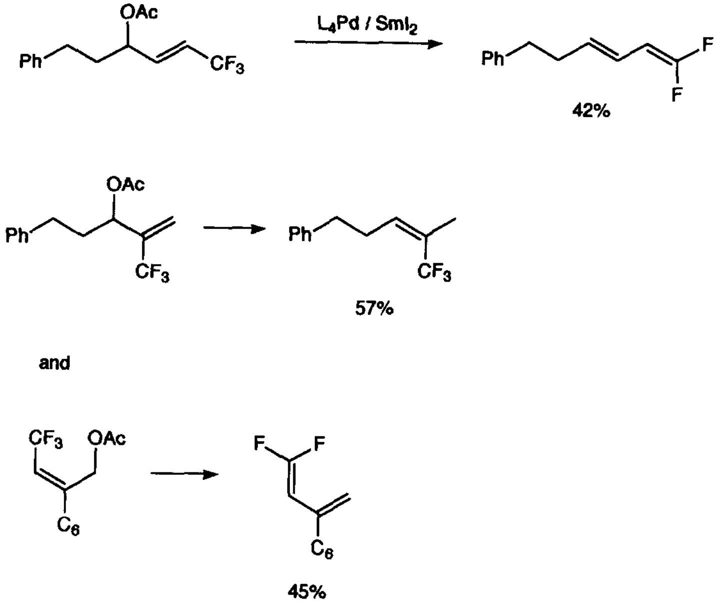
(Equation 675)



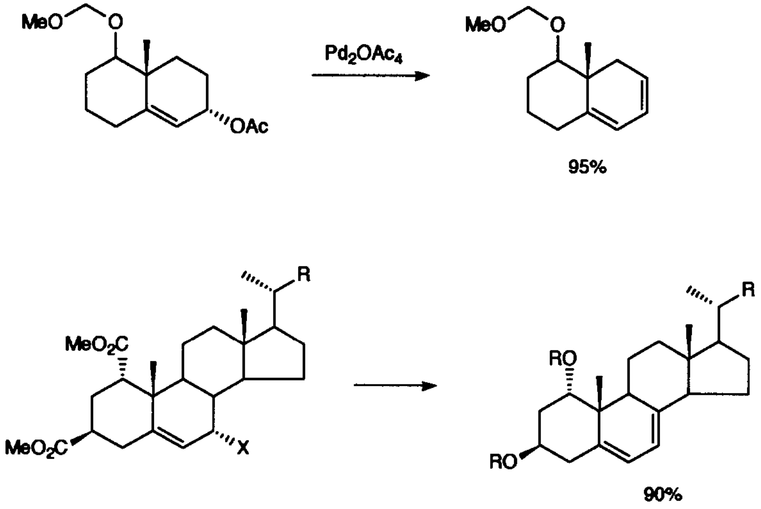
F. Alkenes, Alkanes

Palladium(0) complexes catalyzed the elimination of allyl acetates to dienes (equation 676) [874], (equation 677) [875] Palladium acetates was used to oxidize enolates to enones (equation 678) [876] Vanadium oxychlorides oxidized cyclic enones to aromatic oxygen compounds (equation 679) [877].

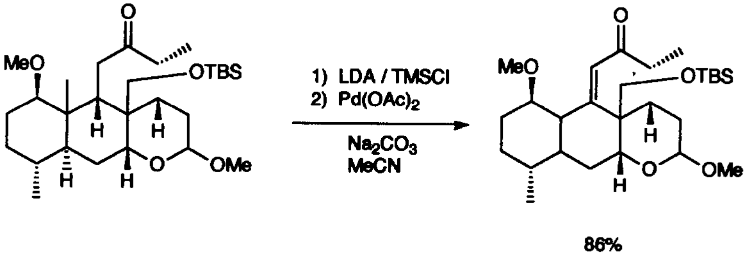
(Equation 676)



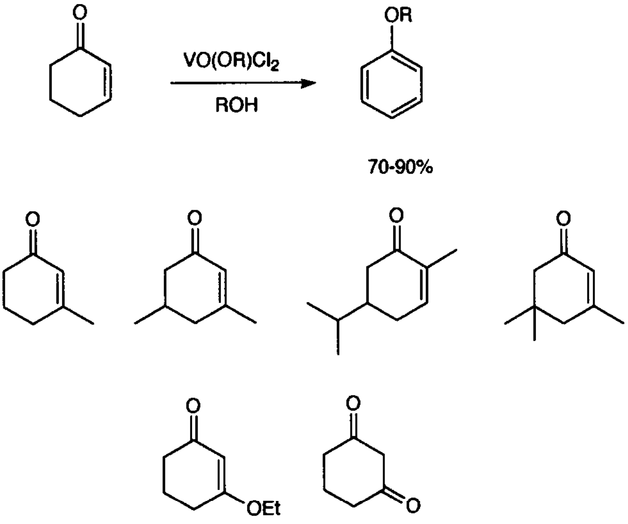
(Equation 677)



(Equation 678)

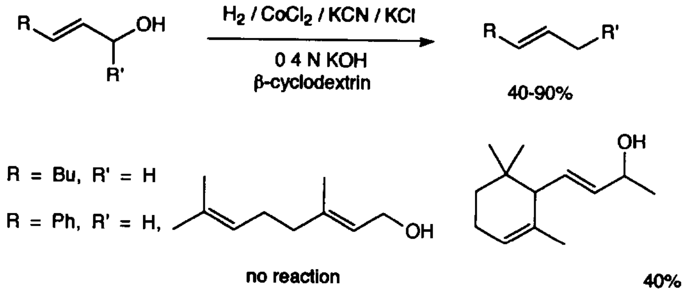


(Equation 679)

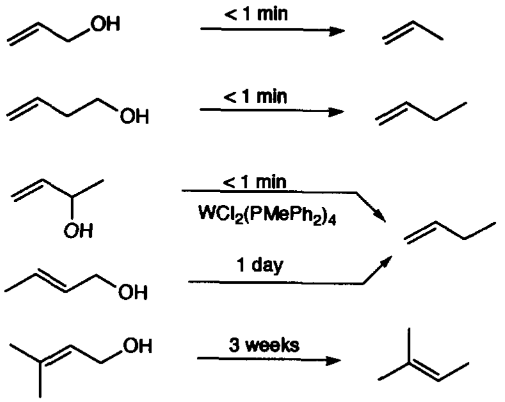


Allylic alcohols were deoxygenated to olefins by cobalt(II) chloride in the presence of cyclodextrin (equation 680) [878], and by $WCl_2(PMPh_2)_4$ (equation 681) [879]. α-Hydroxyacids were converted to olefins by $WOCl_4$ (equation 682) [880]

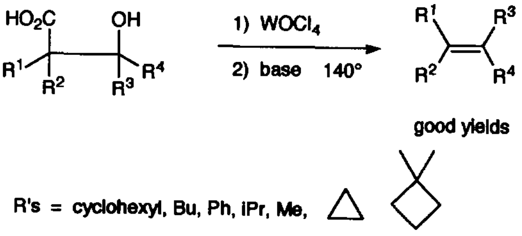
(Equation 680)



(Equation 681)

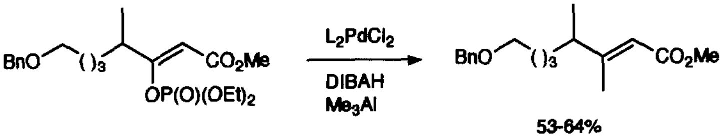


(Equation 682)



Benzyl chlorides and chlorobenzene were reduced to the corresponding hydrocarbon by Cp_2ZrH_2 and Cp_2ZrHCl [881]. Palladium catalyzed the reduction of vinylphosphonates (equation 683) [882], aryl sulfonates (equation 684) [883] and aryl triflates (equation 685) [884] to hydrocarbons. Alkoxy groups on chromium-complexed arenes could be replaced by hydrogen or by hydrides (equation 686) [885]

(Equation 683)



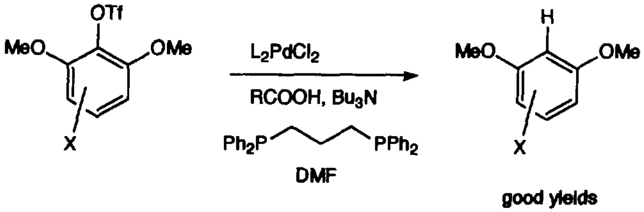
(Equation 684)

$$\mathrm{ArOSO_2R} \xrightarrow[\mathrm{Pd(OAc)_2\ cat.}\ \mathrm{DMF}]{\mathrm{Et_3NH^+HCO_2^-}} \mathrm{ArH}$$

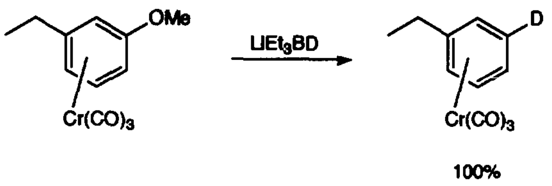
high yields

Ar = 1-naph, 4-NCPh, 4-AcPh

(Equation 685)

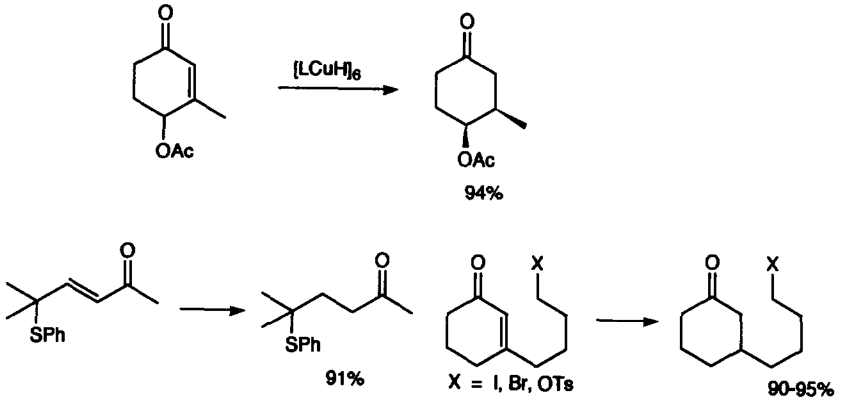


(Equation 686)

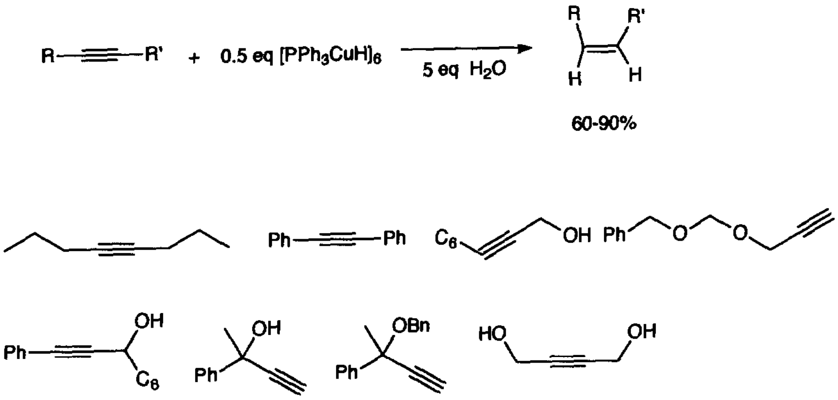


The hexamer $(LCuH)_6$ reduced conjugated enones in a 1,4-manner <u>without</u> reducing other functional groups (equation 687) [886], and reduced alkynes to cis alkenes (equation 688) [887]. Alkynes were reduced to alkenes by hydrozirconation/protonation (equation 689) [888]. The rhodium(I) catalyzed hydroboration of olefins was shown to involve both reversible complexation and olefin insertion steps [889]. Catechol borane efficiently reduced enones in a 1,4-manner in the presence of rhodium(I) catalysts (equation 690) [890].

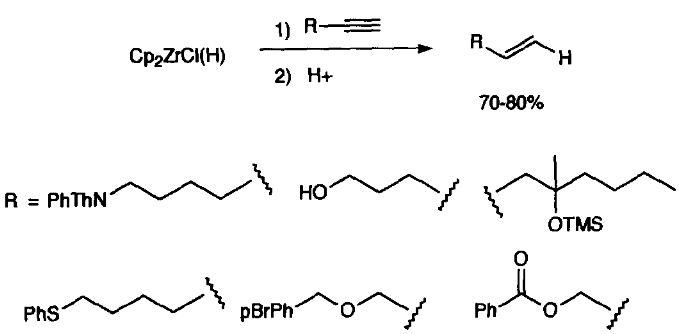
(Equation 687)



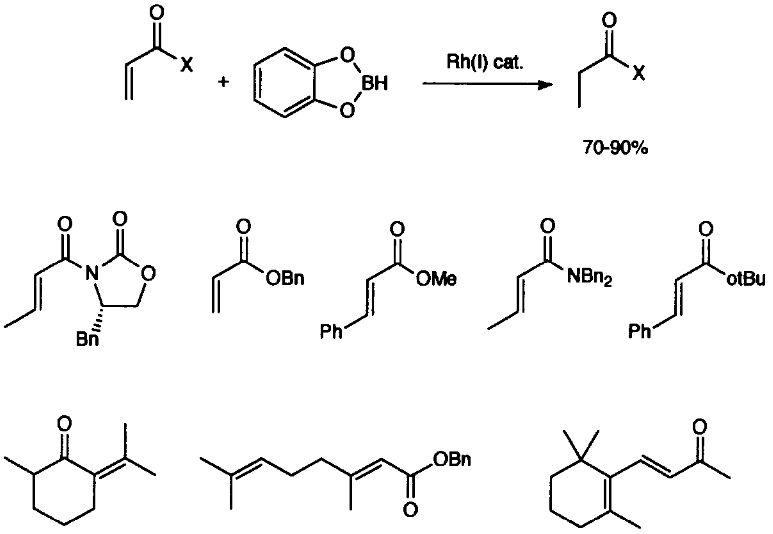
(Equation 688)



(Equation 689)



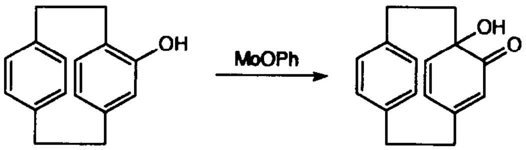
(Equation 690)



G Ketones, Aldehydes

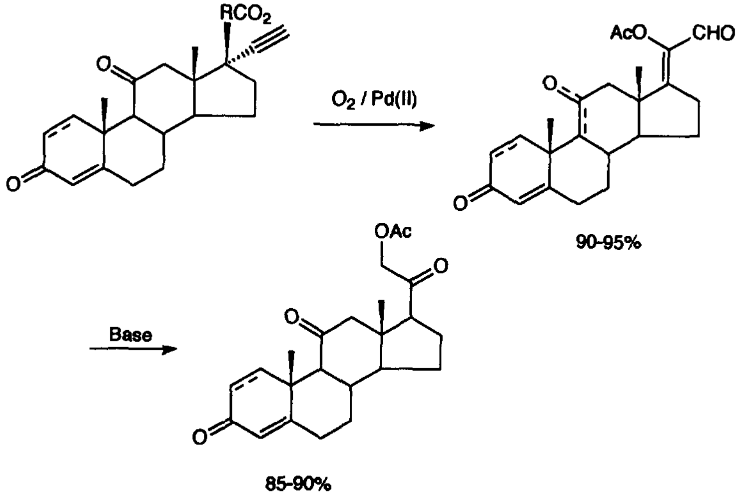
MoOPh oxidized 4-hydroxy-π-cyclophane into the α-hydroxy cyclohexadienone (equation 691) [891] Zirconium complexes catalyzed the oxidation of benzyl alcohols to aldehydes or ketones by t-butylhydroperoxide [892], as did cobalt(II) chloride in the presence of oxygen [893] Cyclopentene was oxidized to cyclopentanone using $PdCl_2/CuCl_2$ catalysts and O_2 [894] or $PdCl_2$ and t-butylhydroperoxide [895]

(Equation 691)

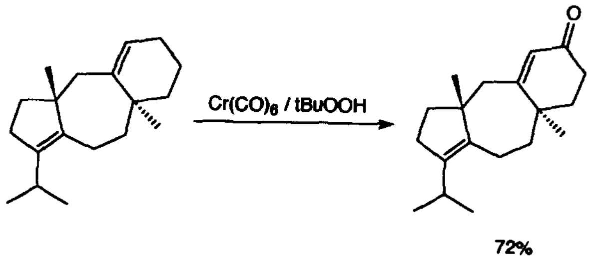


Palladium(II) catalyzed the oxidation of alkynes to α-acetoxy enones (equation 692) [896] while chromium hexacarbonyl oxidized cyclohexenes to cyclohexenones in the presence of t-butylhydroperoxide (equation 693) [897]. Ruthenium dioxide oxidatively cleaved olefins to carbonyl compounds (equation 694) [898] Palladium(0) complexes catalyzed the ring opening of endo peroxides (equation 695) [899] Platinum(II) salts catalyzed the hydrolysis of alkynes to ketones (equation 696) [900].

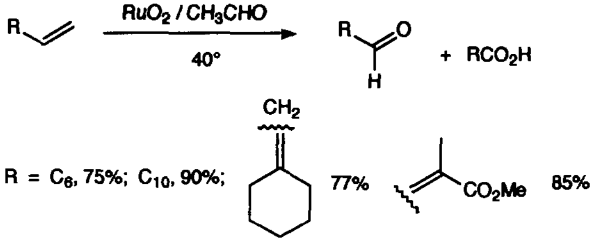
(Equation 692)

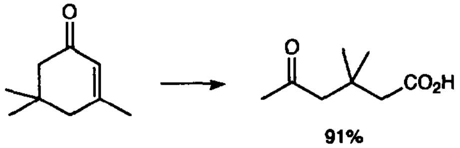


(Equation 693)

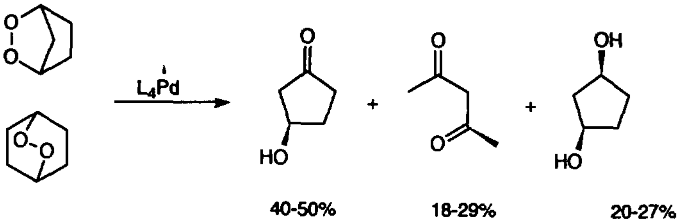


(Equation 694)

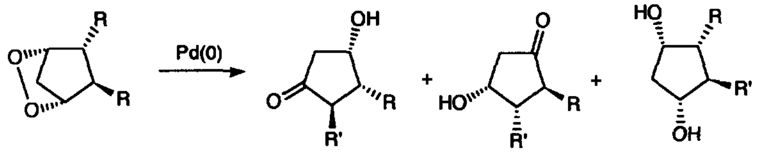




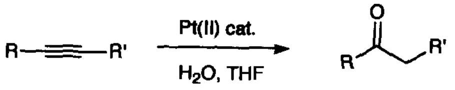
(Equation 695)



and



(Equation 696)

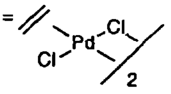


R = nBu, R' = H, R = nPr, R' = H

R = nPr, R' = Me, R, R' = nPr, R = tBu, R' = Me

R = R' = Ph; R = Ph, R' = H

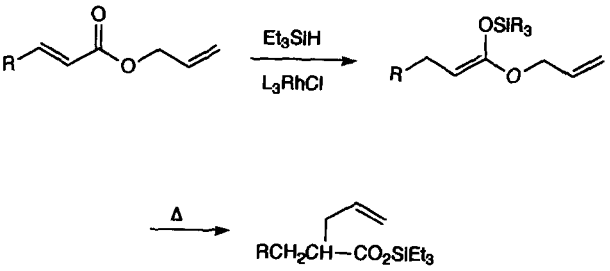
cat =



H. Organosilicon, Tin, and Germanium Compounds

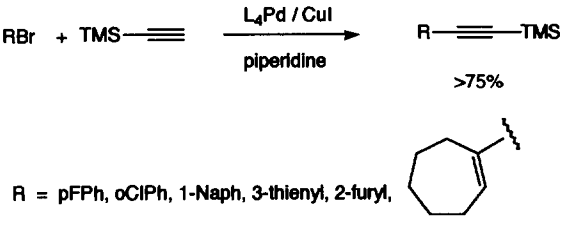
Bis isonitrile complexes of platinum, nickel and palladium catalyzed the hydrosilylation of α-methylstyrene and ketones [901]. (Bispyridine) methanol having an optically active alkoxy group α- to one nitrogen catalyzed the hydrosilylation of ketones, but with low ee [902]. Rhodium(I) complexes catalyzed the 1,4-hydrosilylation of allyl esters of acrylates (equation 697) [903].

(Equation 697)

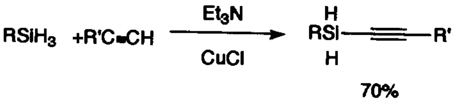


Trimethylsilyl acetylene was alkylated by aryl halides using palladium/copper catalysts (equation 698) [904] Terminal alkynes were silylated using copper chloride catalysts (equation 699) [905] Propargyl alcohols were hydrosilylated to vinyl silanes over platinum catalysts (equation 700) [906]. Disilanes added to olefins to give disilyl alkanes using platinum catalysts (equation 701) [907]. Allyl silanes were prepared by the trimethylsilylmethylation of vinyl halides (equation 702) [908]. Aryl chlorides were converted to aryl silanes using palladium(II) catalysts (equation 703) [909]

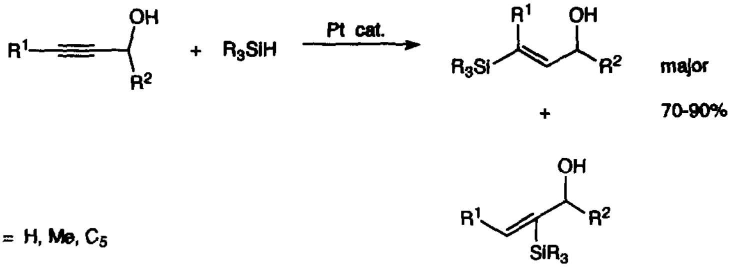
(Equation 698)



(Equation 699)



(Equation 700)



R^1 = H, Me, C_5

R^2 = H, Me, Me_2, C_5, CH_2OBn, CO_2Me, CH_2CH_2OH

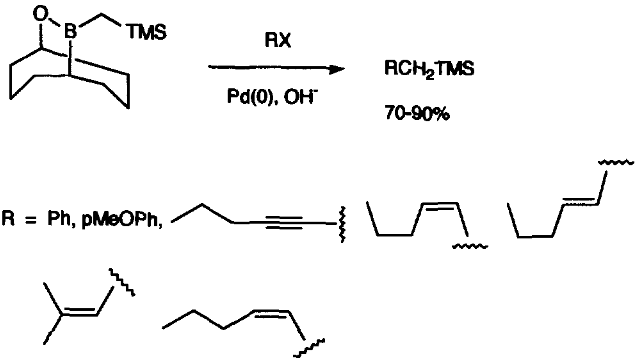
(Equation 701)

$$XMe_2SiSiMe_2X + CH_2{=}CH_2 \xrightarrow{LnPt} XMe_2SiCH_2CH_2SiMe_2X$$

X = F, 95%; MeO, 52%; Cl, 48%;, Me, 18%, pCF_3Ph, 16%; Ph, 4.3%; ptoly, 3.8%

also

(Equation 702)



(Equation 703)

$$\mathrm{ArCOCl} + \mathrm{ClMe_2S\text{-}SiMe_2Cl} \xrightarrow[\substack{\mathrm{PhNO_2} \\ 125^\circ}]{\mathrm{L_2PdCl_2}} \mathrm{ArSiMe_2Cl}$$

90%

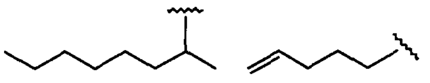
Rhodium(II) perfluorobutyrate catalyzed the silylation of alcohols with an order of reactivity of 1° > 2° » 3° (equation 704) [910]. With this catalyst the primary OH of diols cold be selectively protected. Benzyl and allyl acetates were carbonylated/reduced/silylated by cobalt carbonyl (equation 705) [911]. Nitriles were reductively silylated by the same catalysis (equation 706) [912].

(Equation 704)

$$R'OH + R_3SiH \xrightarrow[\text{cat}]{Rh_2(pfb)_4} R'OSiR_3$$

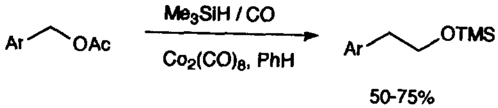
88-99%

R = $PhCH_2$, nC_8, cholesterol, glycidol, (0)-menthol, (-)-borneol, phenol, (-)-NOPOL,



1° > 2° >> 3°

(Equation 705)



Ar = pMeOPh, oMeOPh, pMePh, oMePh, Ph, pClPh, 1-naph, 2-furyl, 3-furyl, 2-thiophenyl
ferrocenyl

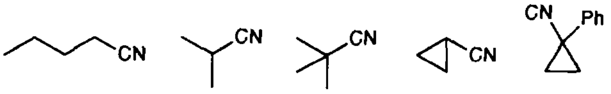
(Equation 706)

$$ArCN + Me_3SiH \xrightarrow[CO]{Co_2(CO)_8} ArCH_2NTMS_2$$

50-90%

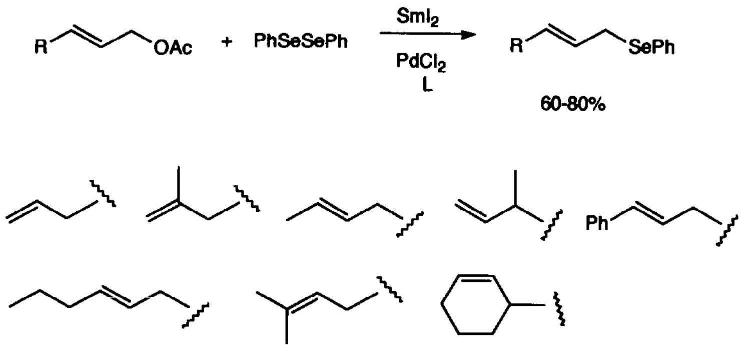
Ar = Ph, oMePh, mMePh, pMePh, pNCPh, pMeOPh, pMe_2NPh, pClPh, mClPh, $pMeO_2CPh$
2-naph

also

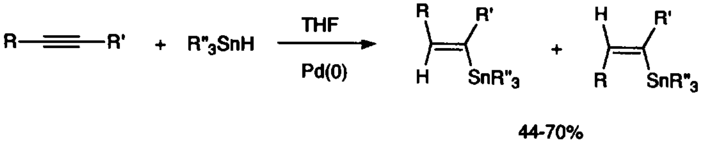


Allyl acetates were converted to allyl selenium complexes using palladium chloride/samarium(II) iodide (equation 707) [913]. Palladium(0) complexes catalyzed the hydrostannylation of alkynes (equation 708) [914] [915], the stannylation/germanylation of allenes (equation 709) [916], and the cyanogermannylation of alkynes (equation 710) [917].

(Equation 707)



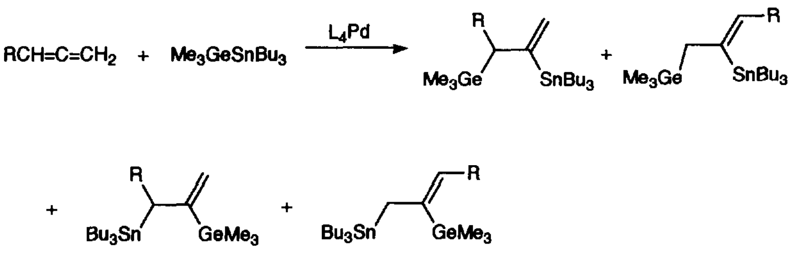
(Equation 708)



R or R' = H, Me, Et, Ph, CO_2Me, COMe

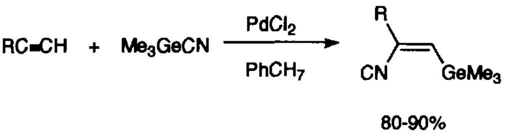
R" = Me, Ph

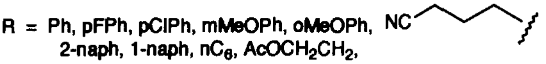
(Equation 709)



R = MeO, EtOCCH$_2$, Ph, C$_6$, tBu

(Equation 710)

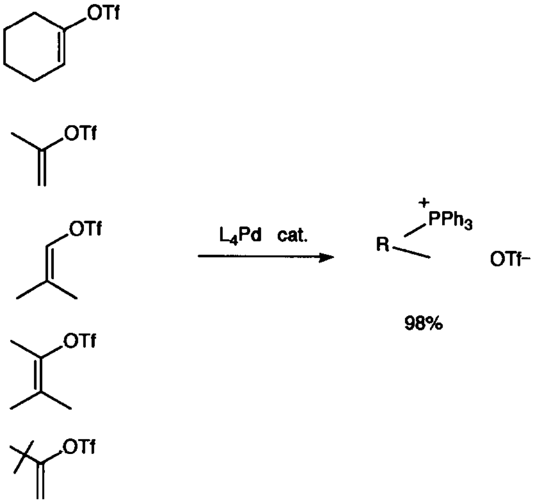




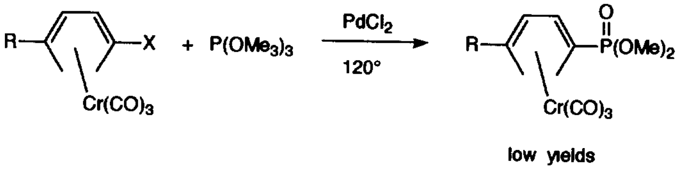
I. Miscellaneous

Palladium(0) complexes catalyzed the reaction of vinyl triflates with triphenyl phosphine to give vinylphosphonium salts (equation 711) [919]. Trimethylphosphite reacted with chromium complexed chlorobenzene in low yield (equation 712) [920] Rhodium(II) acetate coupled diazomalonates to diazadienes (equation 713) [921] Cuprates desulfurized ketene thioacetals (equation 714) [922] Platinum carbonyl complexes deoxygenated sulfoxides with CO [923] The preparation, characterization and purification of the water soluble rhodium complex $[(O\text{-}_3SPh)_3P]_3RhCl\bullet H_2O$ was reported [924] The use of allyl- and allyloxycarbonyl protecting groups which could be removed by $Pd_2(dba)_3/HCOOH$ has been developed [925]. Optically active alkaloids promoted high asymmetric induction in the osmium tetroxide conversion of olefins to diols (equation 715) [926]. Leukotrienes were labelled with metal carbonyl clusters for ir markers for in vitro analysis [927]. Cobalt and molybdenum carbonyl clusters were used in imunology [928]

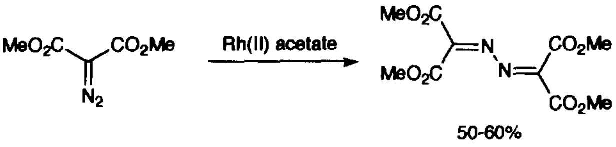
(Equation 711)



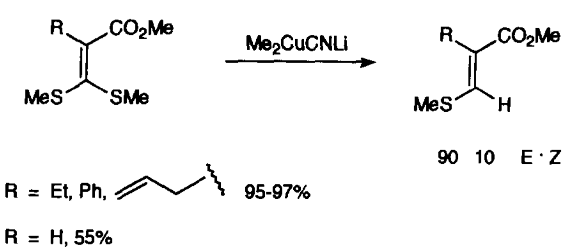
(Equation 712)



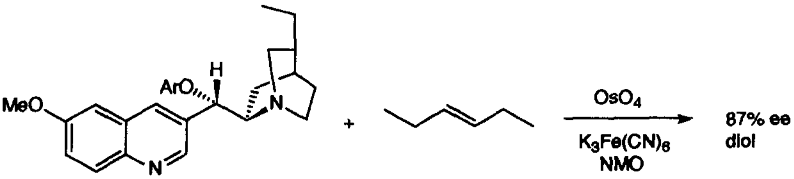
(Equation 713)



(Equation 714)



(Equation 715)



IV Reviews

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