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TRANSITION METALS IN ORGANIC SYNTHESIS
ANNUAL SURVEY COVERING THE YEAR 1989*

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*Reprints of this review are not available.
Previous review see J. Organomet. Chem., 380(1990)169–428.

I. General Comments

This annual survey covers the literature for 1989 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.

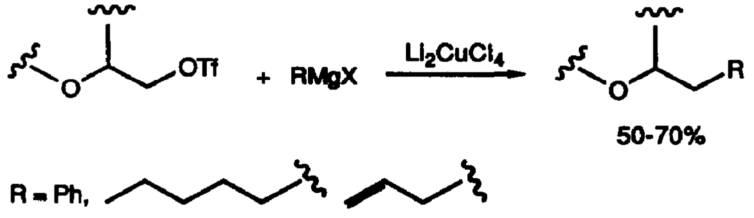
The papers in this survey are grouped primarily by reaction type rather than by organometallic reagent, since the reader is likely to be more interested in the organic transformation effected than the metal causing it. Oxidation, reduction, and hydroformylation reactions are specifically excluded, and 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

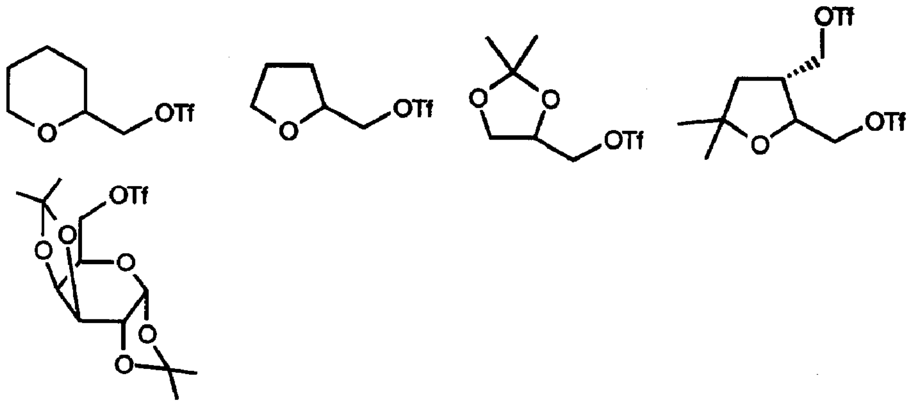
A. Alkylations

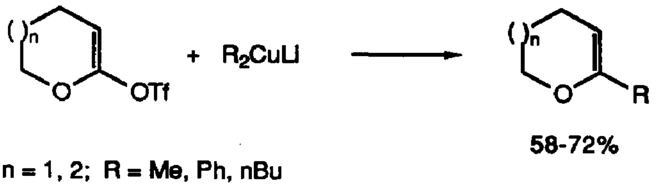
1. Alkylation of Organic Halides, Tosylates, Triflates, Acetates and Epoxides

The use of transition metal systems to alkylate organic triflates was on the rise. Copper(II) salts catalyzed the alkylation of α-alkoxy triflates by Grignard reagents (equation 1) [1]. O-trifluoromethyl ketene acetals were similarly alkylated by dialkylcuprates (equation 2) [2]. Secondary tosylates were alkylated with clean inversion by dialkylcuprates (equation 3) [3].



(1)





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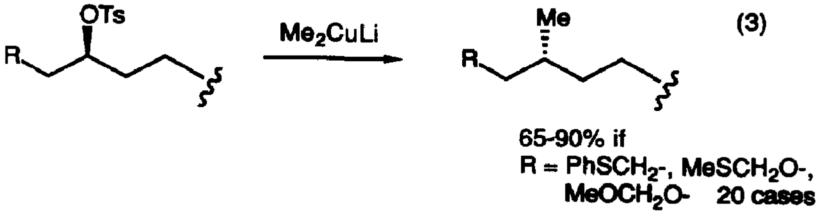
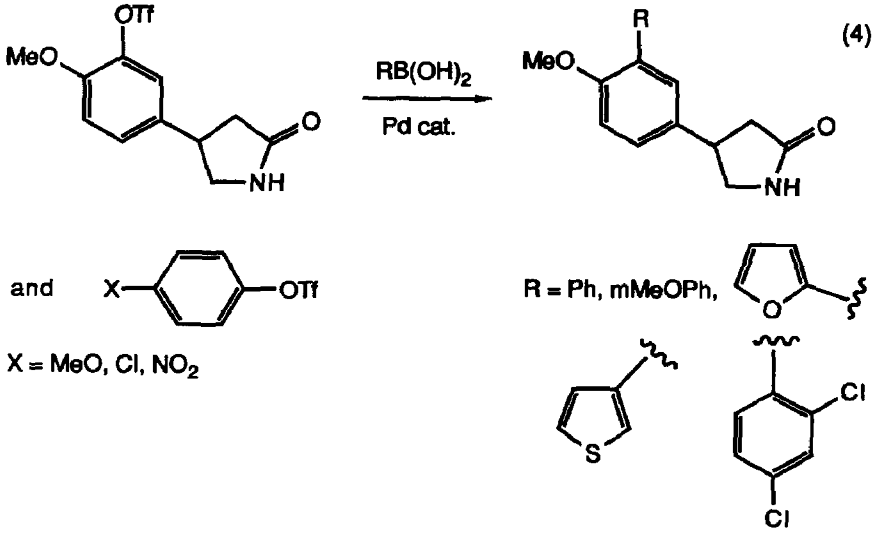


(3)

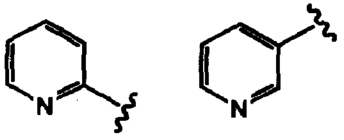
The palladium(0) catalyzed alkylation of aryl triflates (equation 4) [4], (equation 5) [5], (equation 6) [6], heteroaromatic triflates (equation 7) [7], and vinyl triflates (equation 8) [8], (equation 9) [9], (equation 10) [10], (equation 11) [11] by a variety of main group organometallics was an efficient process.

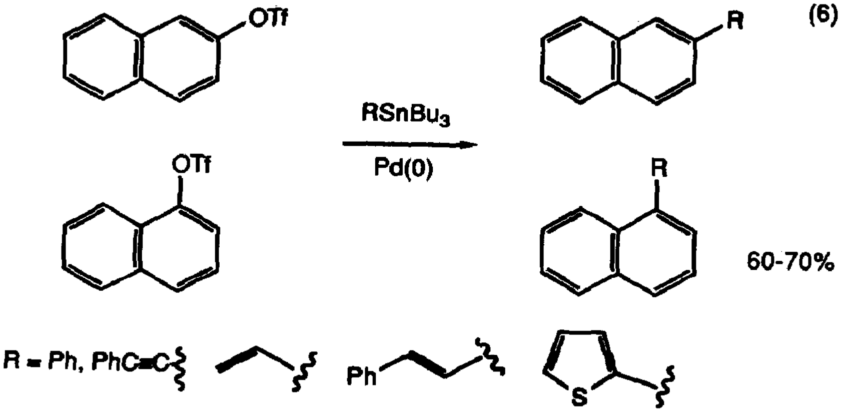


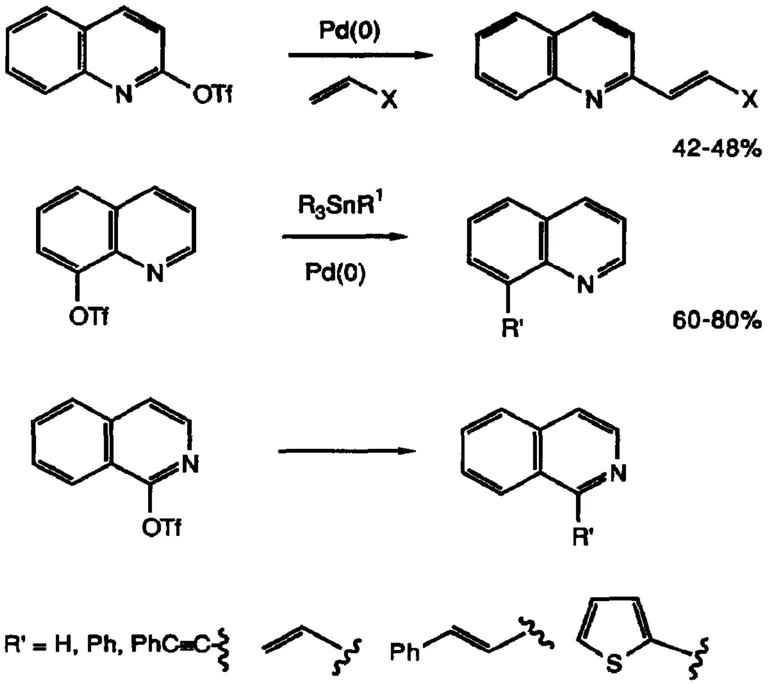
$$ArOTf + R_3Al \xrightarrow{L_4Pd} ArR \qquad (5)$$

60-90%

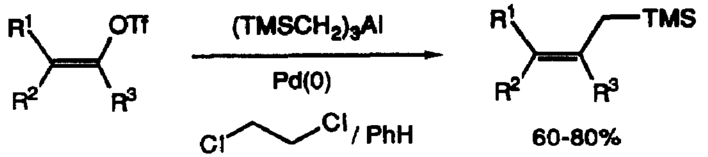
Ar = Ph, oMePh, pMePh, mMePh, pClPh, mHOPh, 1-Naphth



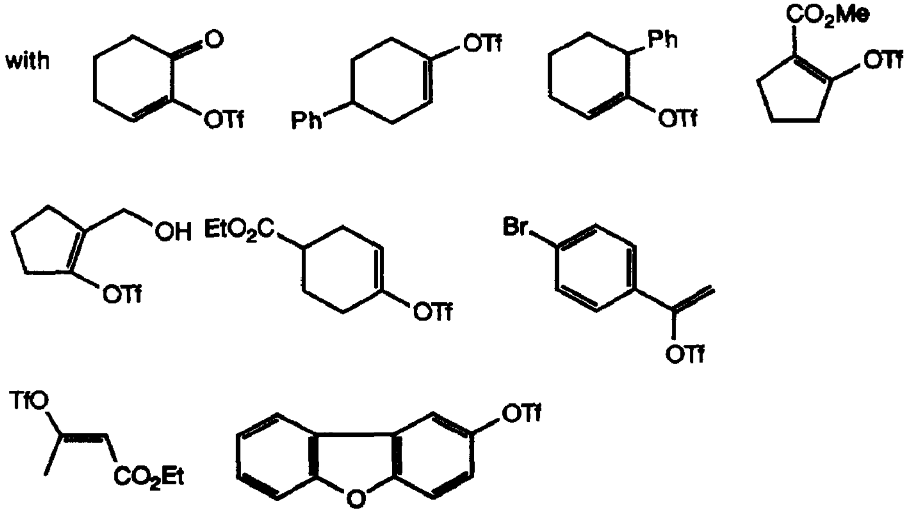



Pd(0)
X
OTf
N
42-48%
R$_3$SnR^1
Pd(0)
R'
60-80%
R' = H, Ph, PhC≡C
Ph
S

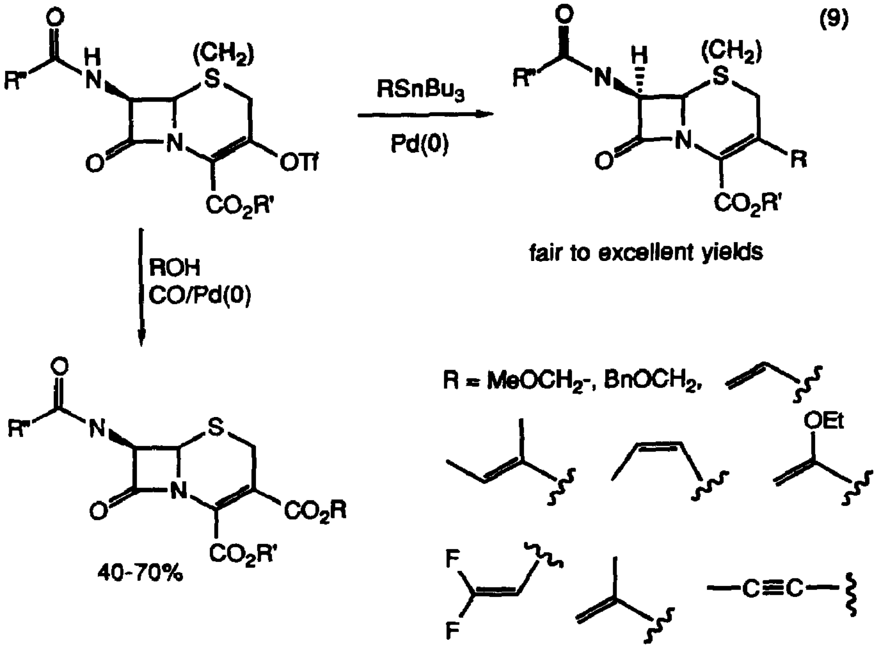
(7)


R^1
R^2
R^3
OTf
(TMSCH$_2$)$_3$Al
Pd(0)
Cl
Cl / PhH
TMS
60-80%

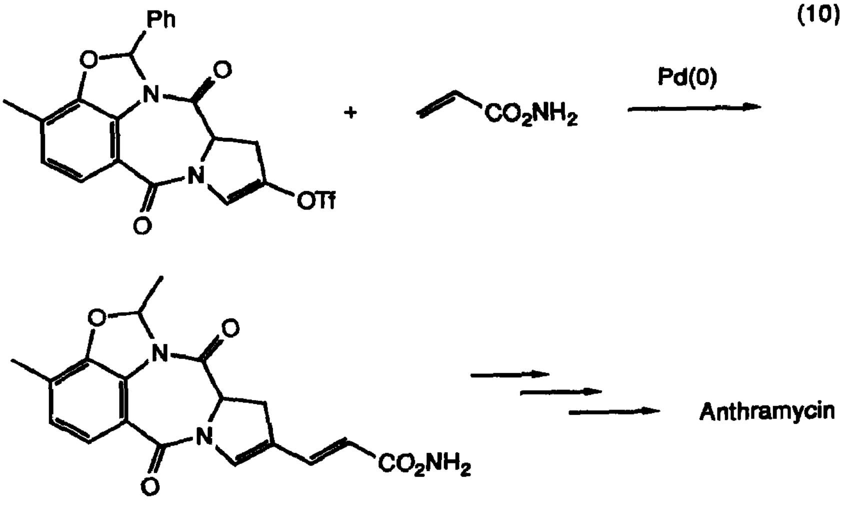
(8)


with
O
OTf
Ph
CO$_2$Me
OH
EtO$_2$C
Br
TfO
CO$_2$Et

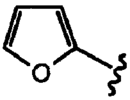
(9)


RSnBu$_3$
Pd(0)
ROH
CO/Pd(0)
OTf
CO$_2$R'
CO$_2$R
(CH$_2$)
fair to excellent yields
40-70%
R = MeOCH$_2$-, BnOCH$_2$,
OEt
F
F
—C≡C—

(10)


Ph
OTf
CO$_2$NH$_2$
Pd(0)
Anthramycin

(11)

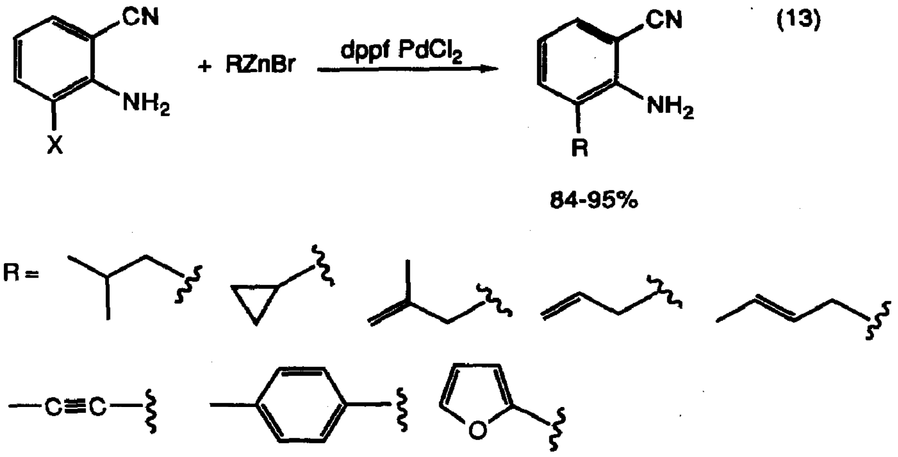
Ar = Ph, pMeOPh, pCF$_3$Ph, oMePh, 2,6-Me$_2$Ph, 2,3,4-(MeO)$_3$Ph, 

The palladium catalyzed cross-coupling of aryl and vinyl halides with vinyl or aryl metals has been reviewed (37 references) [12]. Aryl zinc, magnesium, and heteroaryl zinc halides underwent palladium(0) catalyzed coupling with aryl and heteroaryl halides to give biaryls [13]. Alkylzinc halides underwent a similar coupling process (equation 12) [14], (equation 13) [15]. Allenic zinc halides were arylated by aryl halides in the presence of optically active palladium phosphine complexes to give optically active allenes (equation 14) [16]. Isonitriles were converted to imines by dialkylation (equation 15) [17]. Palladium(0) complexes also catalyzed the alkylation of vinyl halides by organozinc species (equation 16) [18], (equation 17) [19], (equation 18) [20], (equation 19) [21]. Substituted phenylalanines were prepared by the palladium(0) catalyzed alkylation alkylation of iodoalanine by aryl halides (equation 20) [22].

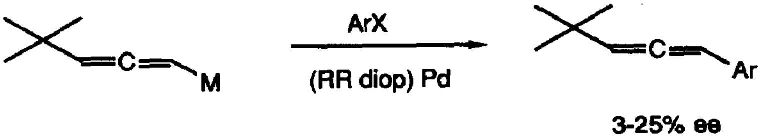
$$\mathrm{ArBr} + \mathrm{RZnX} \xrightarrow[\mathrm{THF/HMPA}]{\mathrm{PdCl_2L_2}} \mathrm{Ar{-}R} \qquad (12)$$

50-90%

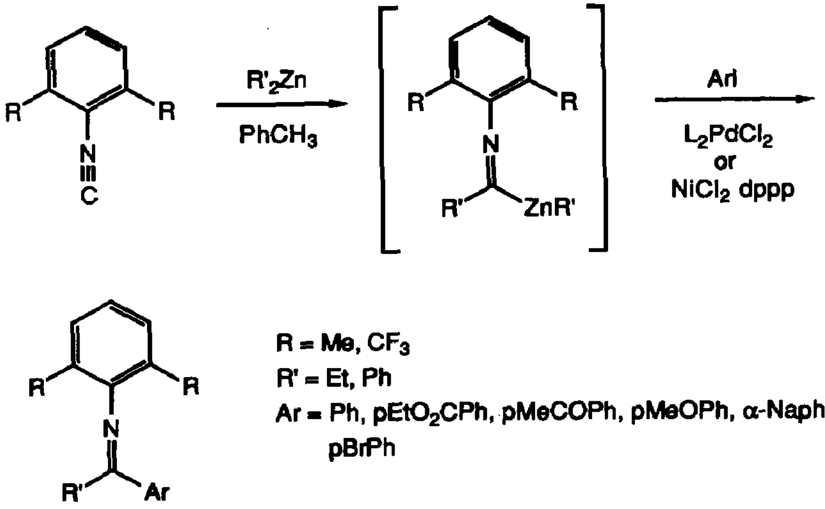
Ar = pMeCOPh, mMeCOPh, pHCOPh
R = Bu, Ph



(13)



(14)



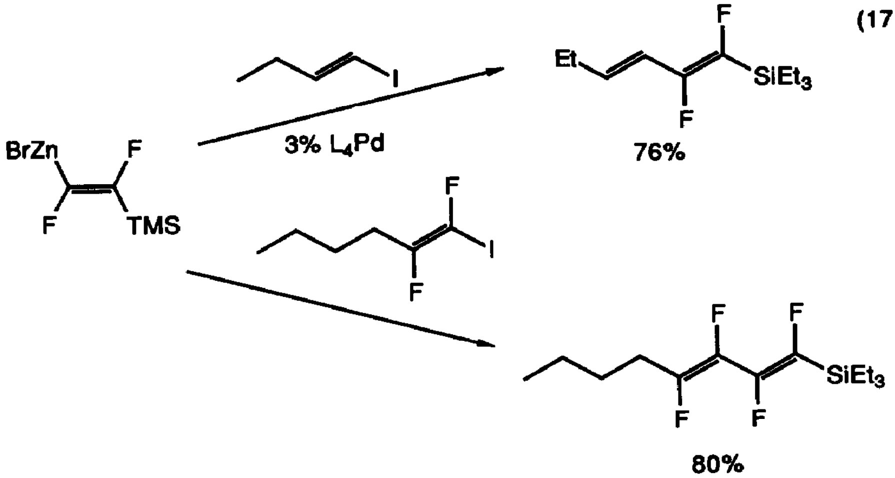
(15)

R = Me, CF$_3$
R' = Et, Ph
Ar = Ph, pEtO$_2$CPh, pMeCOPh, pMeOPh, α-Naphth
pBrPh

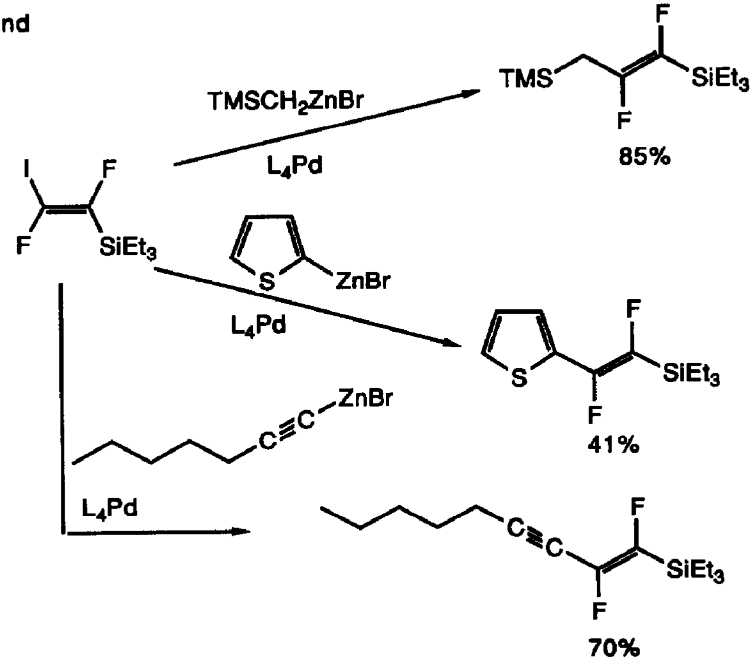

BrZn(CH2)9OtBu
+
Bu
I
L4Pd
Bu
(CH2)9OtBu

(16)

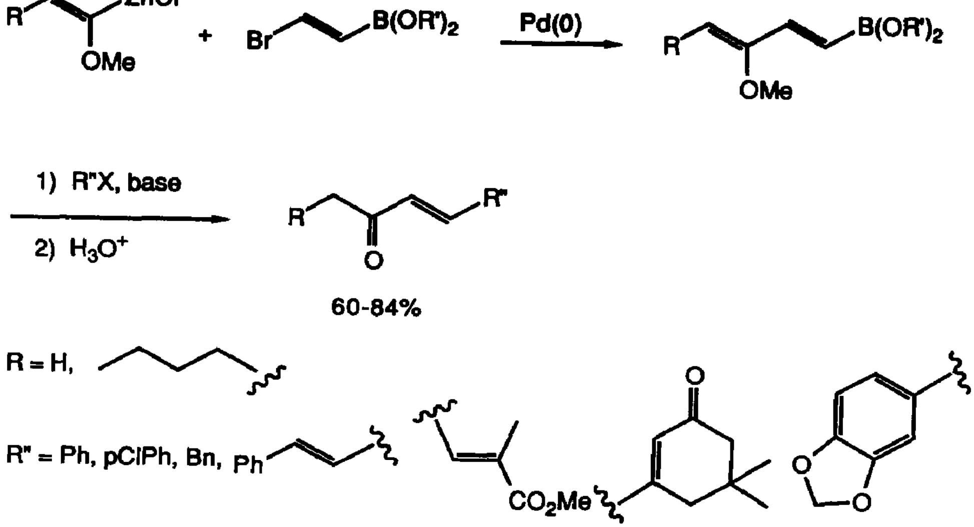
(17)


I
3% L4Pd
Et
F
SiEt3
F
76%
BrZn
F
F
TMS
F
I
F
F
F
SiEt3
F
F
80%

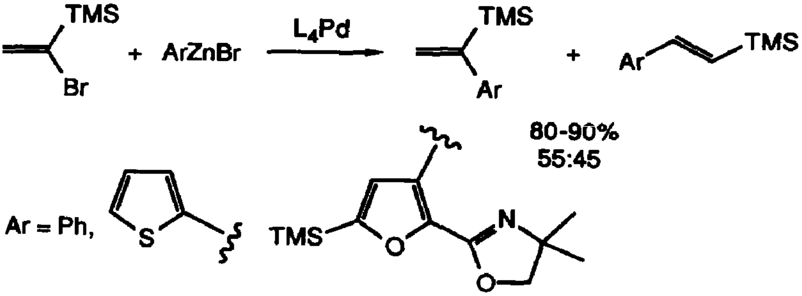
and


TMSCH2ZnBr
L4Pd
TMS
F
SiEt3
F
85%
I
F
F
SiEt3
S
ZnBr
L4Pd
S
F
SiEt3
F
41%
C≡C
ZnBr
L4Pd
C≡C
F
SiEt3
F
70%

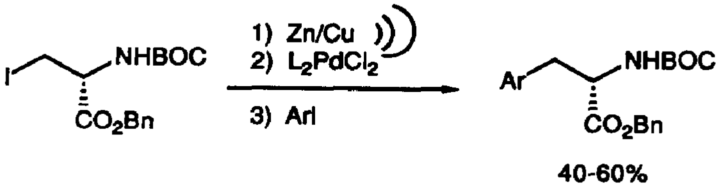
(18)



(19)

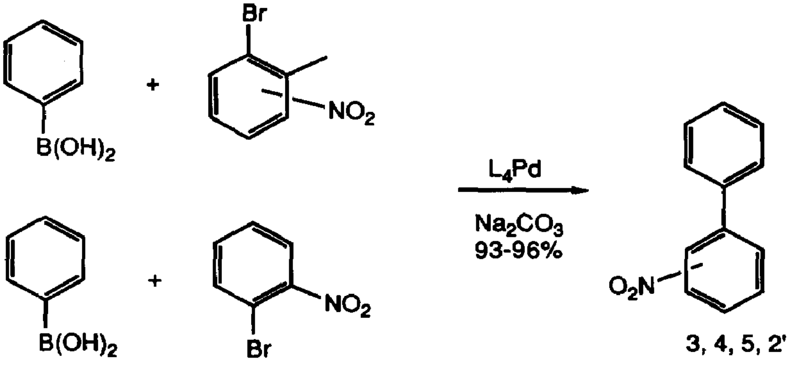


(20)

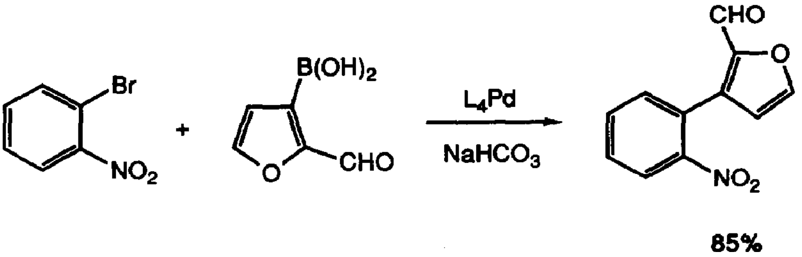


Ar = Ph, 1-Naphth, 2-AcOPh, 2-FPh, 2-MeOPh, 4-AcOPh, 4-BrPh, 4-MePh, 4-NO_2Ph

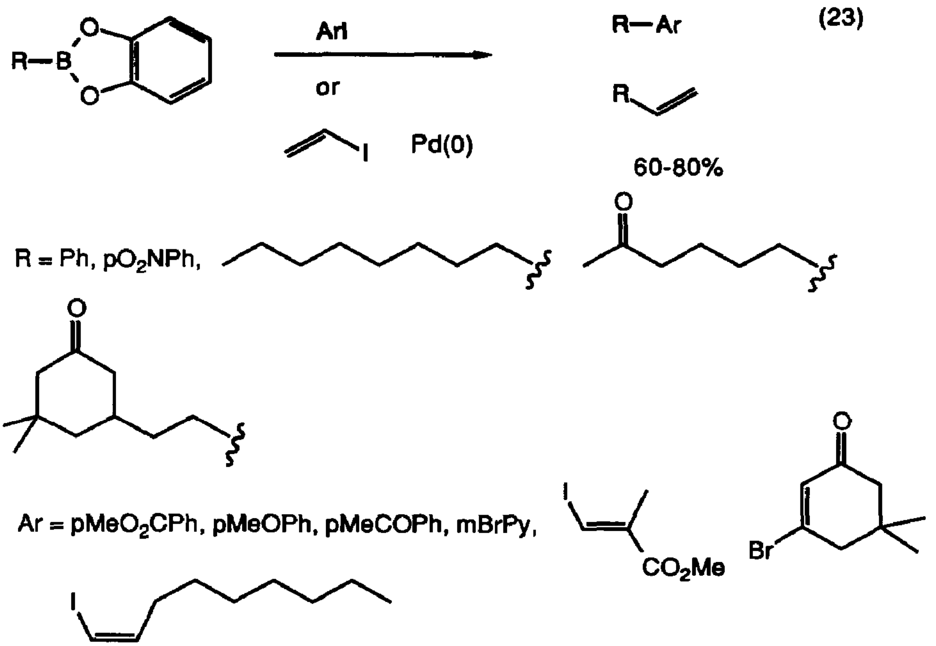
The palladium-catalyzed cross-coupling reactions of aryl and vinylic boron compounds with organic halides has been reviewed (69 references) [23], as has the palladium-catalyzed coupling reactions of organic zinc, aluminum, tin and boron reagents with organic electrophiles (33 references) [24]. Palladium catalyzed the synthesis of new tricyclic systems from heterocyclic boronic acids and halides [25]. Nitro biphenyls were prepared by the palladium catalyzed coupling of bromonitroarenes with phenyl boronic acid, (equation 21) [26], (equation 22) [27]. Alkyl catechol boranes alkylated aryl and vinyl iodides in the presence of palladium(0) catalysts (equation 23) [28]. 2-Iodobenz-4-pyrones were arylated by aryl boronic acids in the presence of palladium(0) catalysts (equation 24) [29], as were bromoisoquinolines (equation 25) [30]. The boron-to-palladium transmetallation was also useful for other alkylations **(equation 26) [31], (equation 27) [32].**



(21)

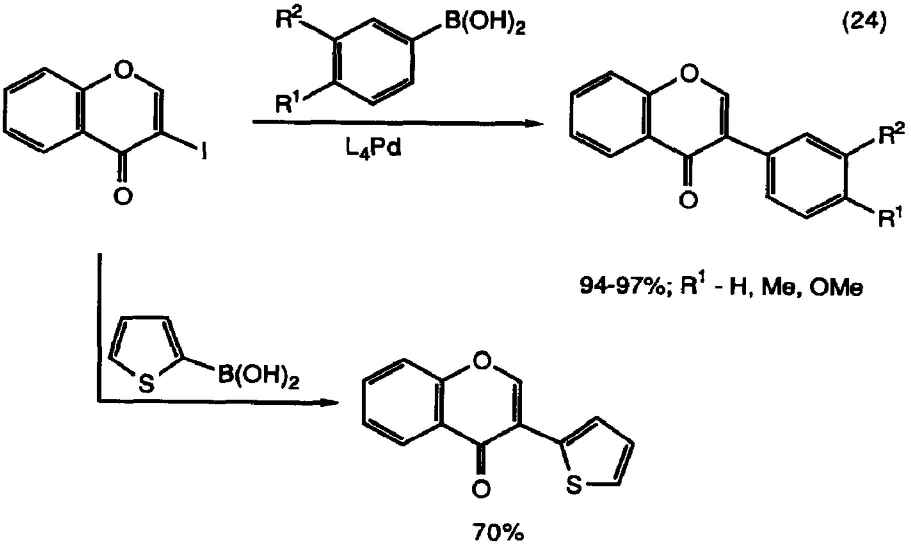


(22)



(23)

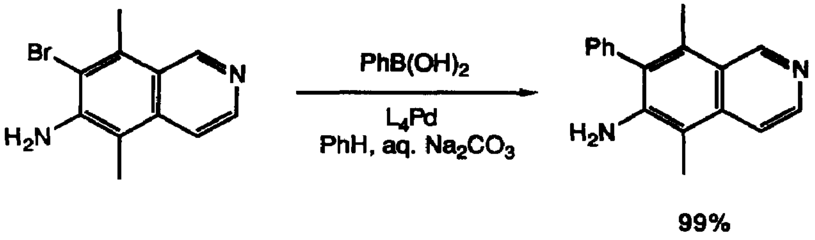
R—B
ArI
or
Pd(0)
R—Ar
R
60-80%
R = Ph, pO₂NPh,
O
Ar = pMeO₂CPh, pMeOPh, pMeCOPh, mBrPy,
I
CO₂Me
Br



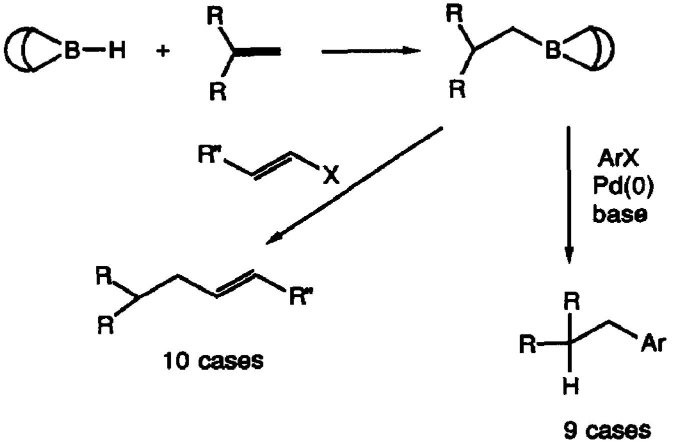
(24)

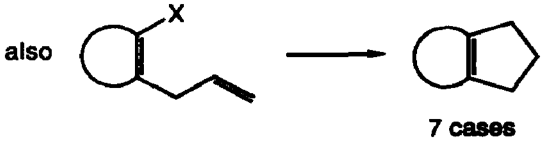
O
I
R²
B(OH)₂
R¹
L₄Pd
94-97%; R¹ - H, Me, OMe
S
B(OH)₂
70%

(25)

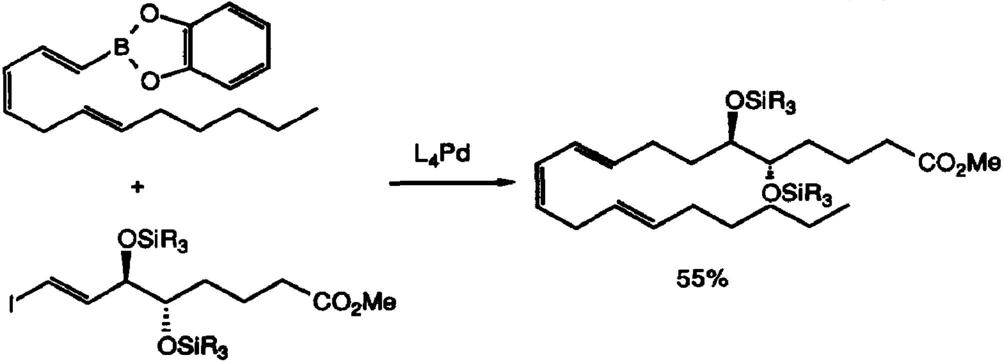

Br
H₂N
N
PhB(OH)₂
L₄Pd
PhH, aq. Na₂CO₃
Ph
H₂N
N
99%

(26)

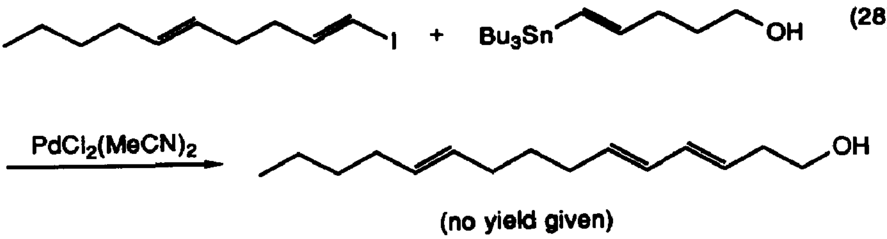

B—H
+
R
R
R
R
B
R"
X
ArX
Pd(0)
base
R
R
R"
10 cases
R
R
Ar
H
9 cases


also
X
7 cases

(27)


B
O
O
+
OSiR₃
I
CO₂Me
OSiR₃
L₄Pd
OSiR₃
CO₂Me
OSiR₃
55%

Palladium catalyzed oxidative addition/transmetallation from tin continues to be used extensively in organic synthesis. Long chain polyunsaturated alcohols (equation 28) [33] elaborated aromatics (equation 29) [34], (equation 30) [35], (equation 31) [36], (equation 32) [37], ene-ynes (equation 33) [38], diyne imines (equation 34) [39], isoflavones [40], unsaturated ketones (equation 35) [41], (equation 36) [42], and tropolones (equation 37) [43] were all made by this chemistry.



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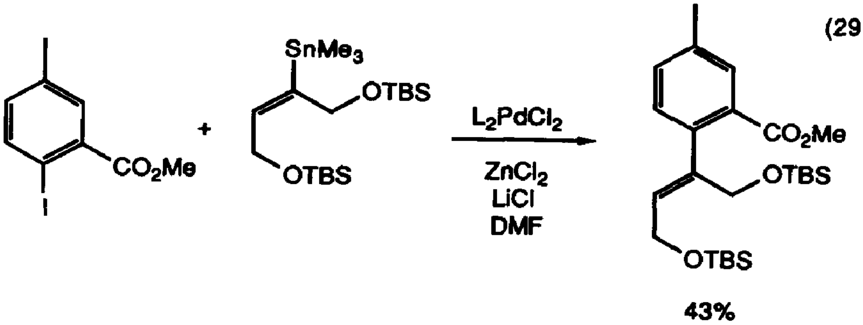
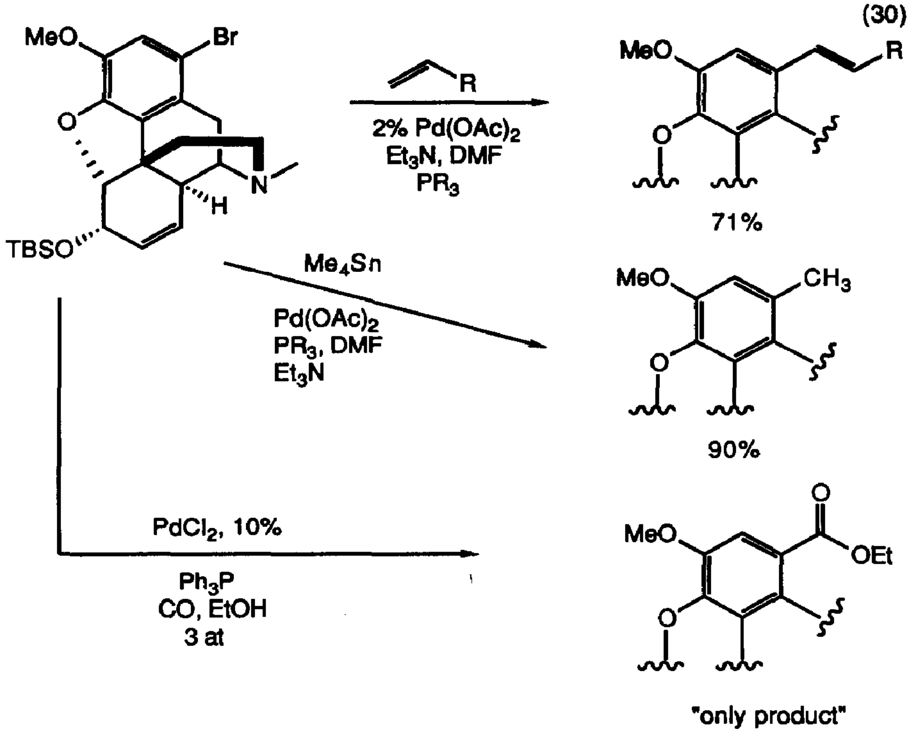


(29)

(30)



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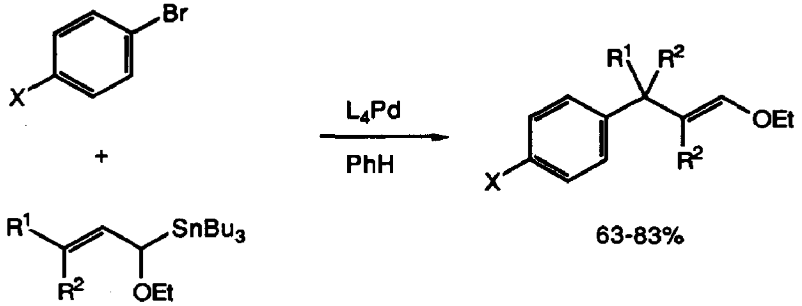
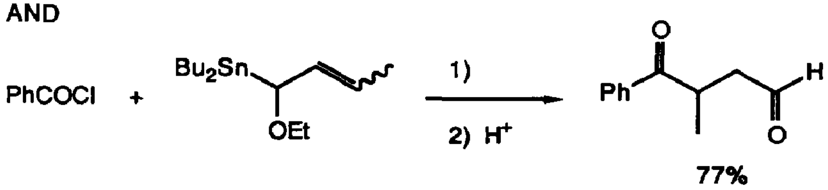
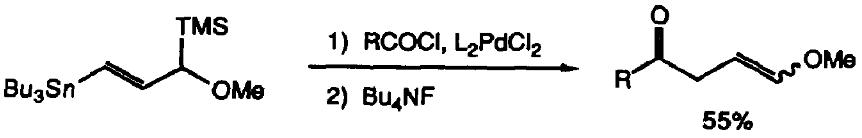


X = H, F, Cl, OMe, Me, COMe, CHO

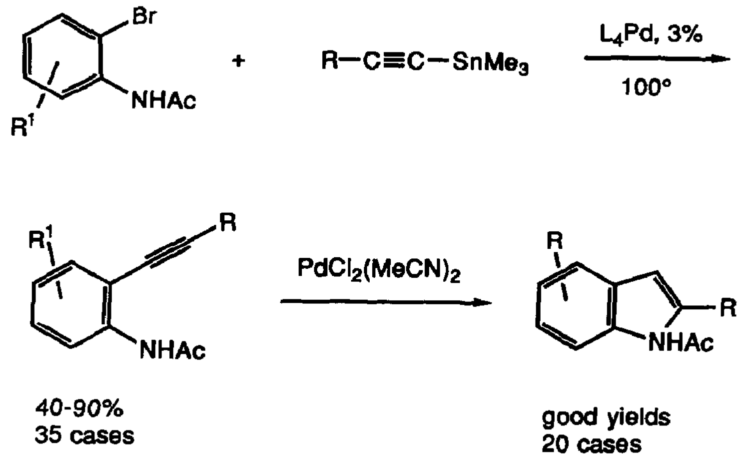
AND



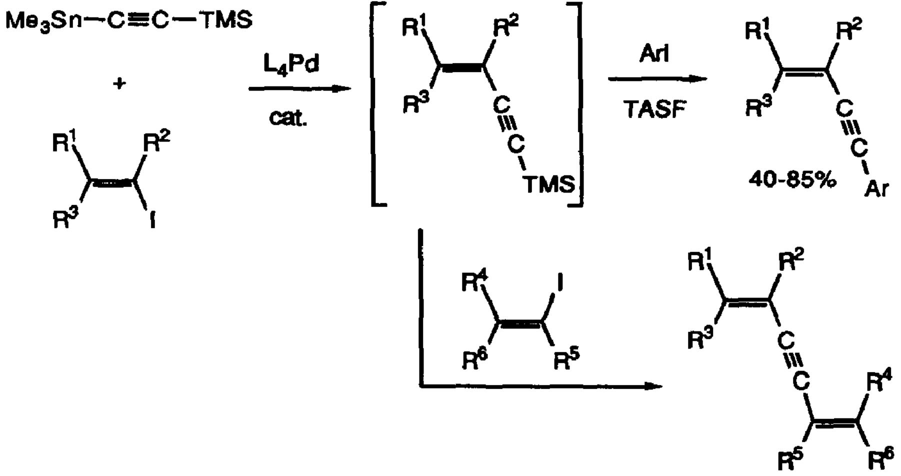
AND

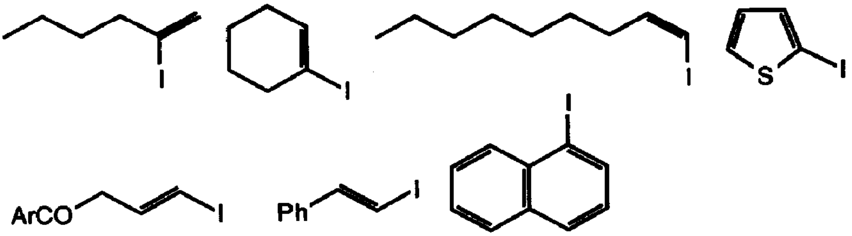

TMS
Bu3Sn
OMe
1) RCOCl, L2PdCl2
2) Bu4NF
O
R
OMe
55%

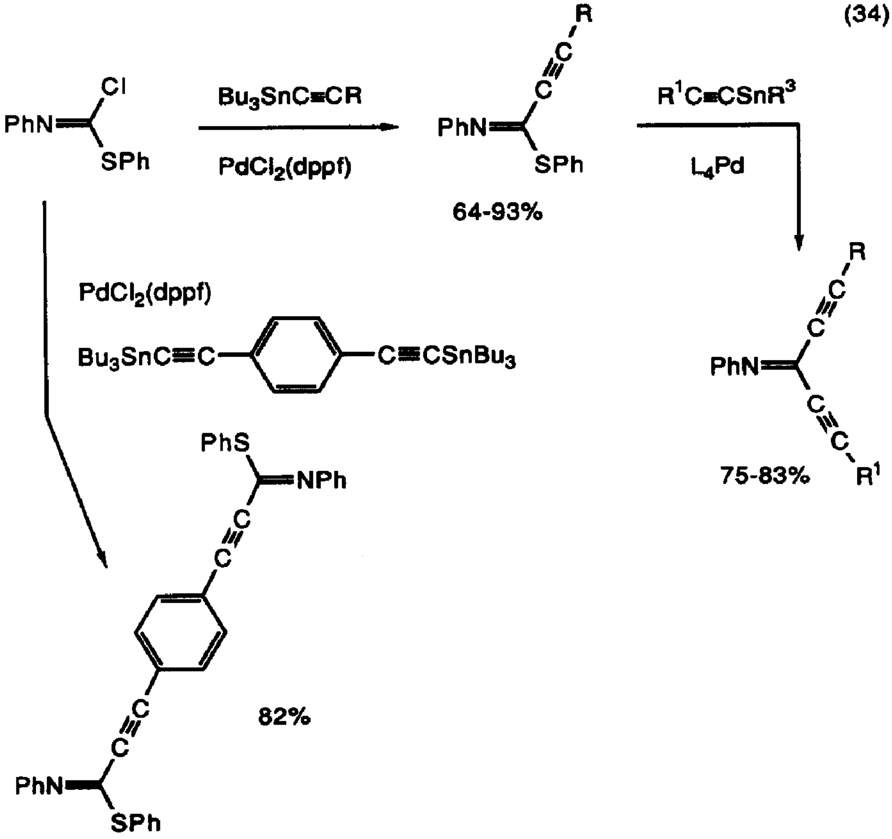
(32)


Br
NHAc
R1
+
R—C≡C—SnMe3
L4Pd, 3%
100°
R1
R
NHAc
PdCl2(MeCN)2
R
R
NHAc
40-90%
35 cases
good yields
20 cases

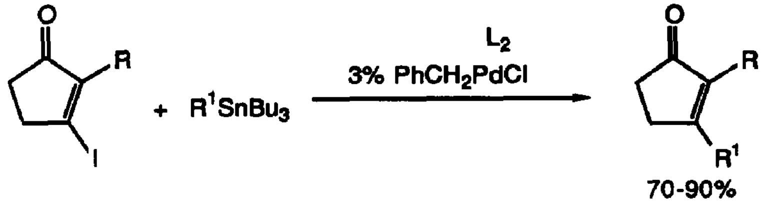
(33)


Me3Sn—C≡C—TMS
+
R1
R2
R3
I
L4Pd
cat.
R1
R2
R3
C
C
TMS
ArI
TASF
R1
R2
R3
C
C
Ar
40-85%
R4
I
R6
R5
R1
R2
R3
C
C
R4
R5
R6


I
I
I
S
I
ArCO
I
Ph
I
I


(34)
PhN
Cl
SPh
Bu$_3$SnC≡CR
PdCl$_2$(dppf)
R
C
C
PhN
SPh
64-93%
R^{1}C≡CSnR^3
L$_4$Pd
R
C
C
PhN
C
C
R^1
75-83%
PdCl$_2$(dppf)
Bu$_3$SnC≡C
C≡CSnBu$_3$
PhS
NPh
C
C
C
C
PhN
SPh
82%

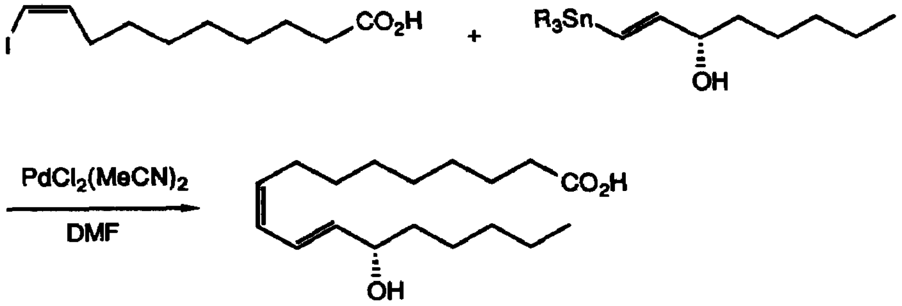
(35)



R = Me, nC_5, $(CH_2)_6CO_2Et$

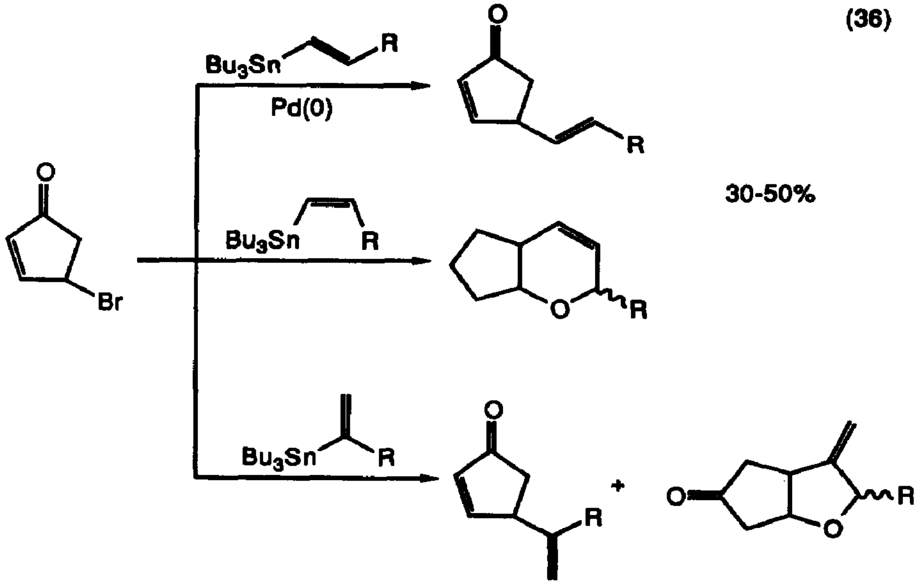
R^1 =

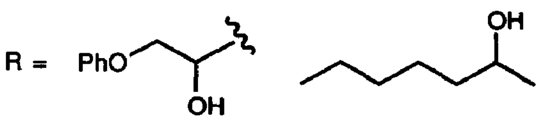
Also



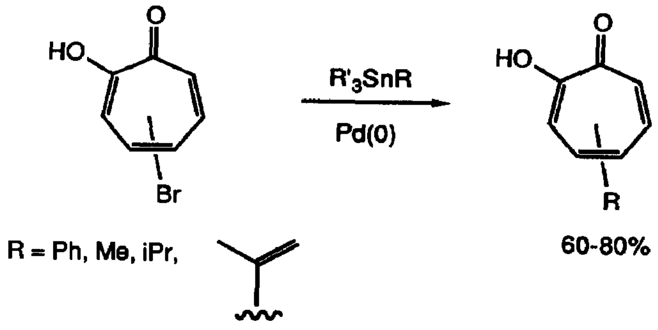
76% coriolic acid

(36)



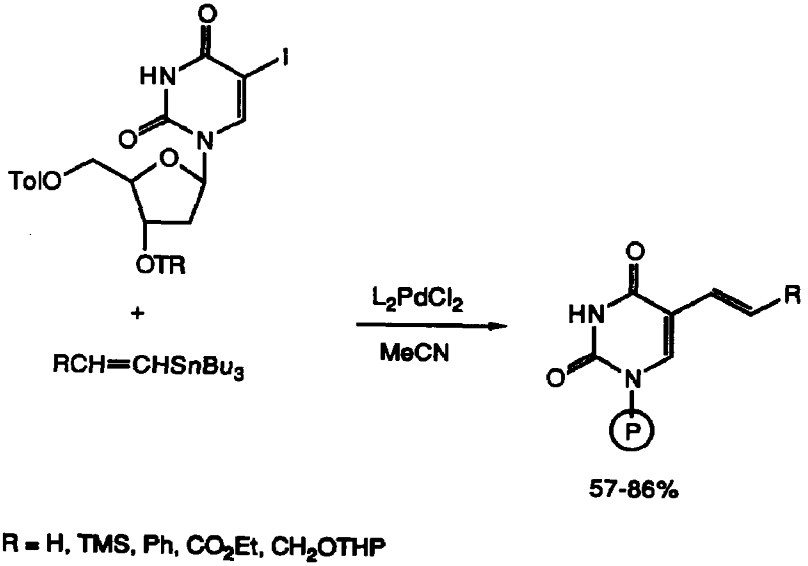


(37)

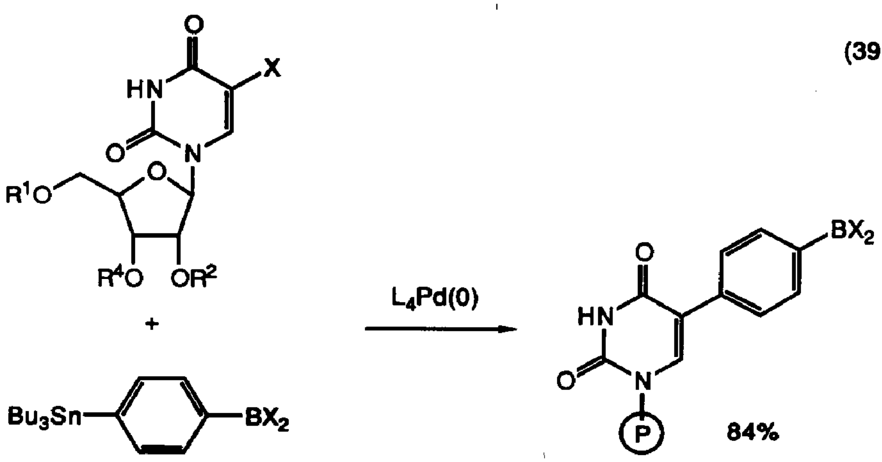


Palladium catalyzed alkylation of halo-pyrimidines (equation 38) [44], (equation 39) [45], and pyridazines (equation 40) [46], (equation 41) [47], (equation 42) [48], (equation 43) [49] by organotin reagents was very popular this year. Palladium catalyzed the alkylation of bicyclic ketals (equation 44) [50].

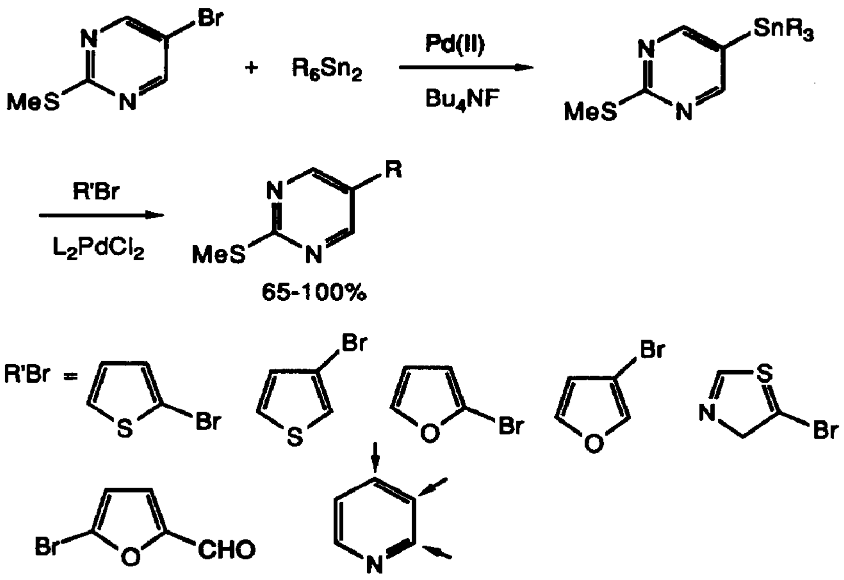
(38)



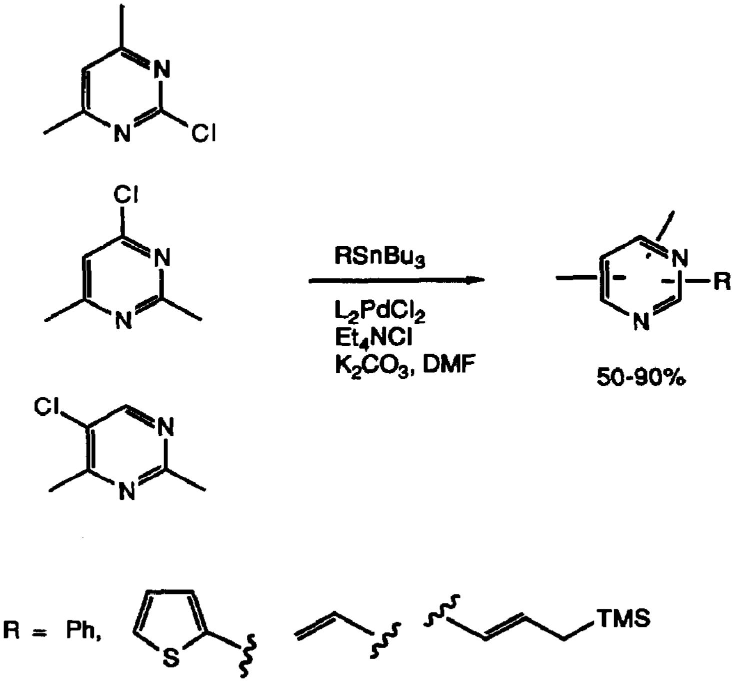
(39)


X
HN
O
N
O
R^{1}O
R^{4}O
OR^2
+
Bu$_3$Sn
BX$_2$
L$_4$Pd(0)
BX$_2$
O
HN
O
N
P
84%

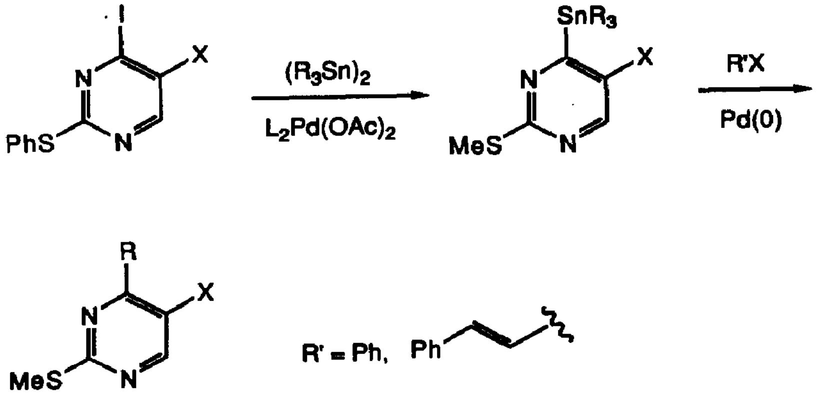
(40)


Br
N
MeS
N
+
R$_6$Sn$_2$
Pd(II)
Bu$_4$NF
SnR$_3$
N
MeS
N
R'Br
L$_2$PdCl$_2$
R
N
MeS
N
65-100%
R'Br =
S
Br
Br
S
O
Br
Br
O
S
N
Br
Br
O
CHO
N

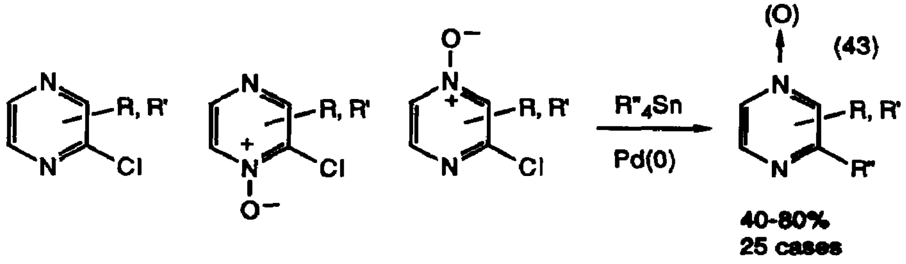
(41)

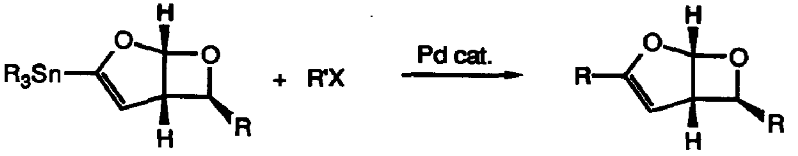

RSnBu3
L2PdCl2
Et4NCl
K2CO3, DMF
50-90%
R = Ph,
TMS

(42)


(R3Sn)2
L2Pd(OAc)2
R'X
Pd(0)
R' = Ph,

(43)

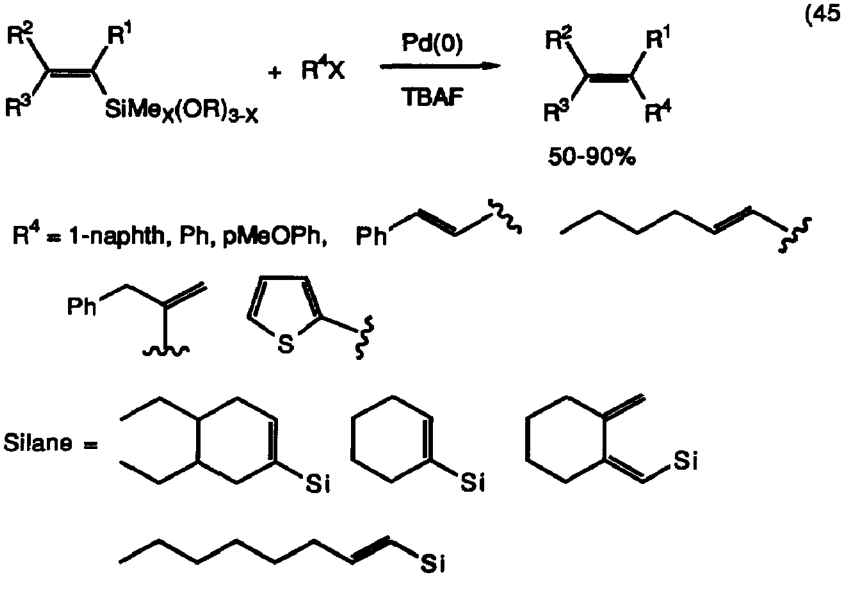

R"4Sn
Pd(0)
40-80%
25 cases



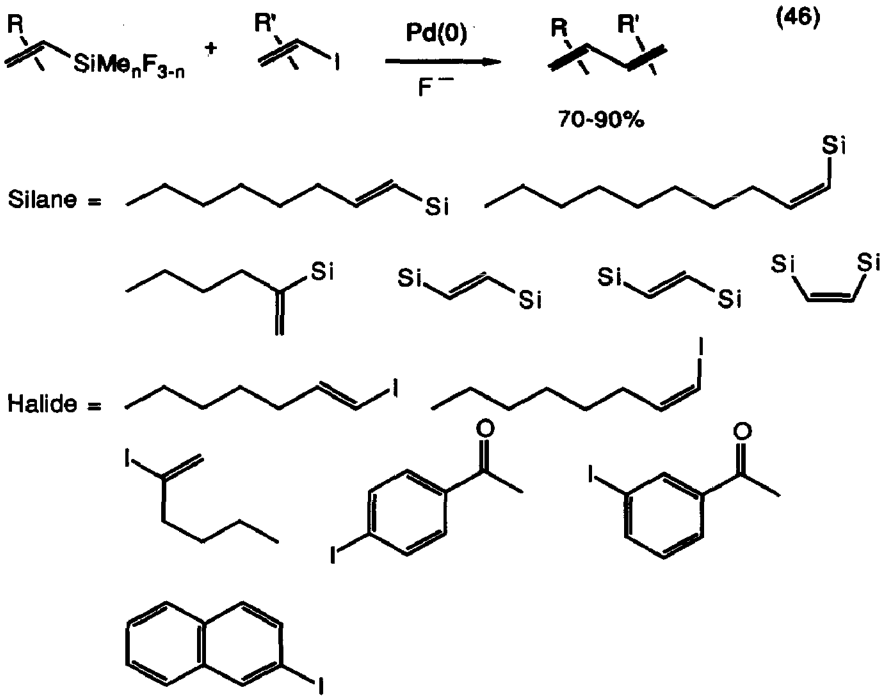
(44)

R' = CH_3CO; R = CO_2Bu, 79%
R' = Ph; R = Ph, 65%
R' = $PhCH_2CO$; R = Ph, 62%

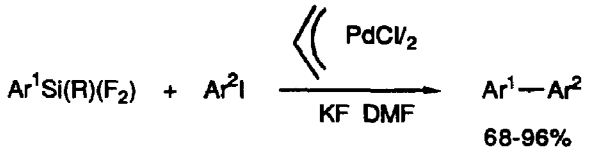
Vinyl (equation 45) [52], (equation 46) [53], and aryl silanes (equation 47) [54] can also transmetallate to palladium in the presence of fluoride ion to result in coupling. Arylmercurials (equation 48) [54] and vinyl alanes from hydroalumination of alkynes [55], also underwent palladium catalyzed coupling reactions with halides.



(45)



(46)



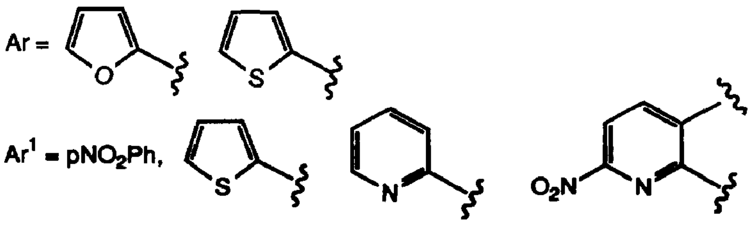
(47)

Ar^1 = Ph, pMePh, pCF$_3$Ph, mClPh, mMeOPh

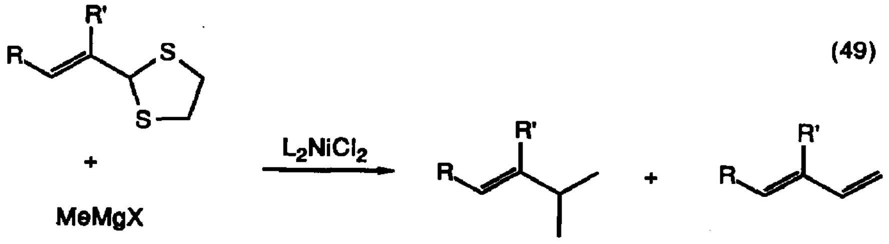
Ar^2 = pEtOPh, pMeOPh, mMeOPh, mHOCH$_2$Ph, MAcOCH$_2$Ph, pNCPh, pAcOPh, pHOPh, pMeCOPh, mHCOPh, 1-naphth

$$Ar_2Hg + Ar^1I \xrightarrow[\text{Pd(0) cat.} \atop \text{NaI}]{\text{DMP}} Ar\text{—}Ar^1 \qquad (48)$$

60-94%



Metal-catalyzed Grignard reactions continue to be exploited for alkylation chemistry. Acrolein dithioacetals were dimethylated by methyl Grignard reagents in the presence of nickel(II) complexes (equation 49) [56]. Vinyl (equation 50) [57] and aryl halides (equation 51) [58] were alkylated in the presence of nickel catalysts. β-Bromostyrenes were arylated by Grignard reagents in the presence of platinum catalysts (equation 52) [59] and aryl bromides were vinylated in the presence of nickel catalysts (equation 53) [60]. Polyunsaturated vinyl silanes were prepared by the coupling of silylmethyl Grignard reagents with vinyl halides (equation 54) [61].

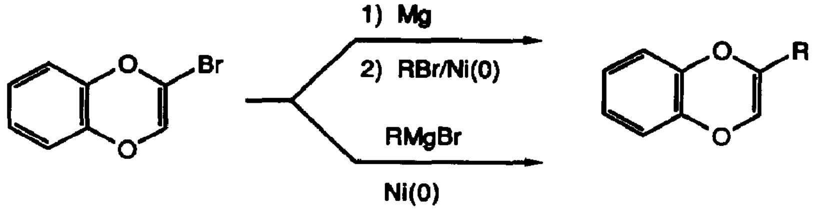


(49)

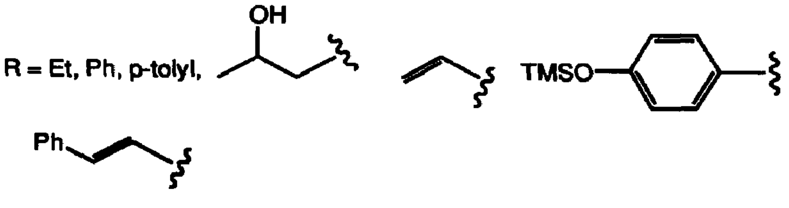
85-98%

R = Ph, pMePh, mMeOPh, 2-Naphth, oMeOPh
R' = H, Me, Et

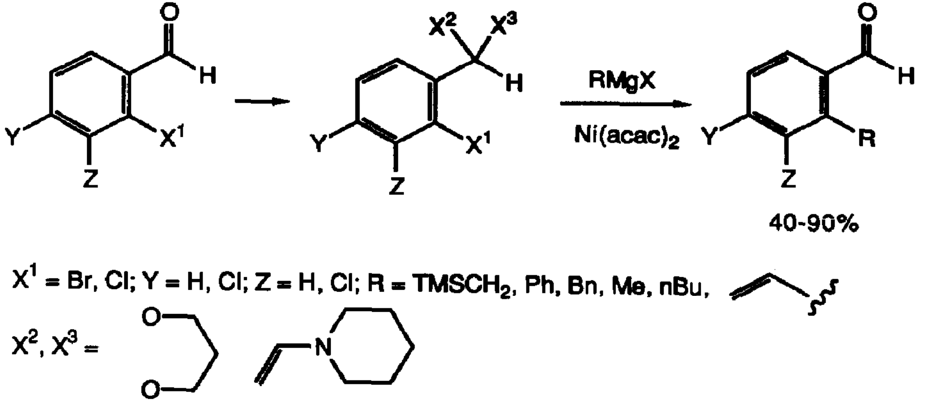
100:1 to 68:32



(50)



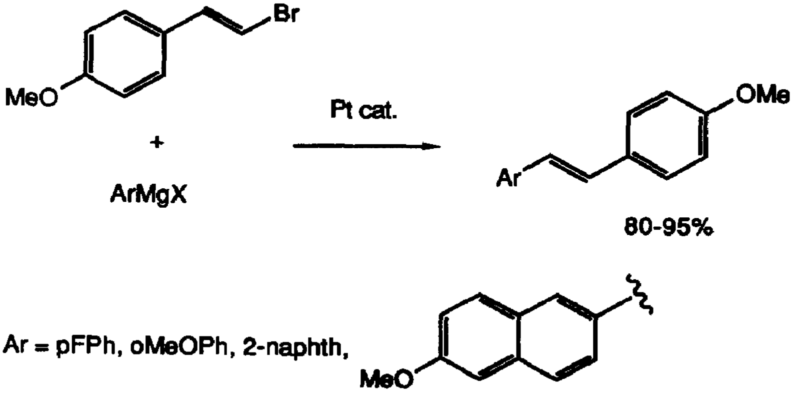
(51)



X^1 = Br, Cl; Y = H, Cl; Z = H, Cl; R = $TMSCH_2$, Ph, Bn, Me, nBu,

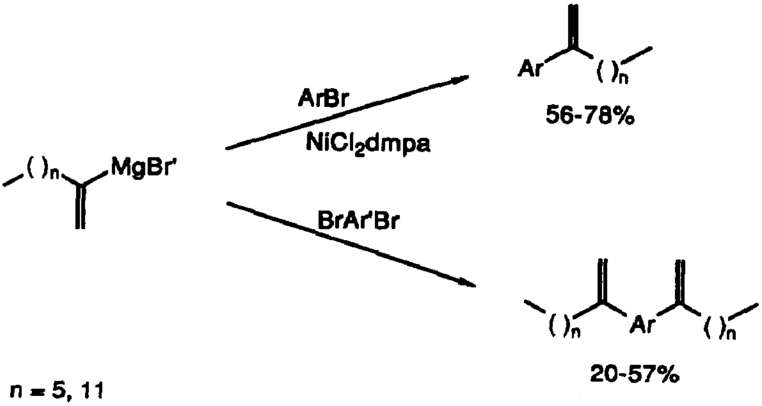
X^2, X^3 =

(52)

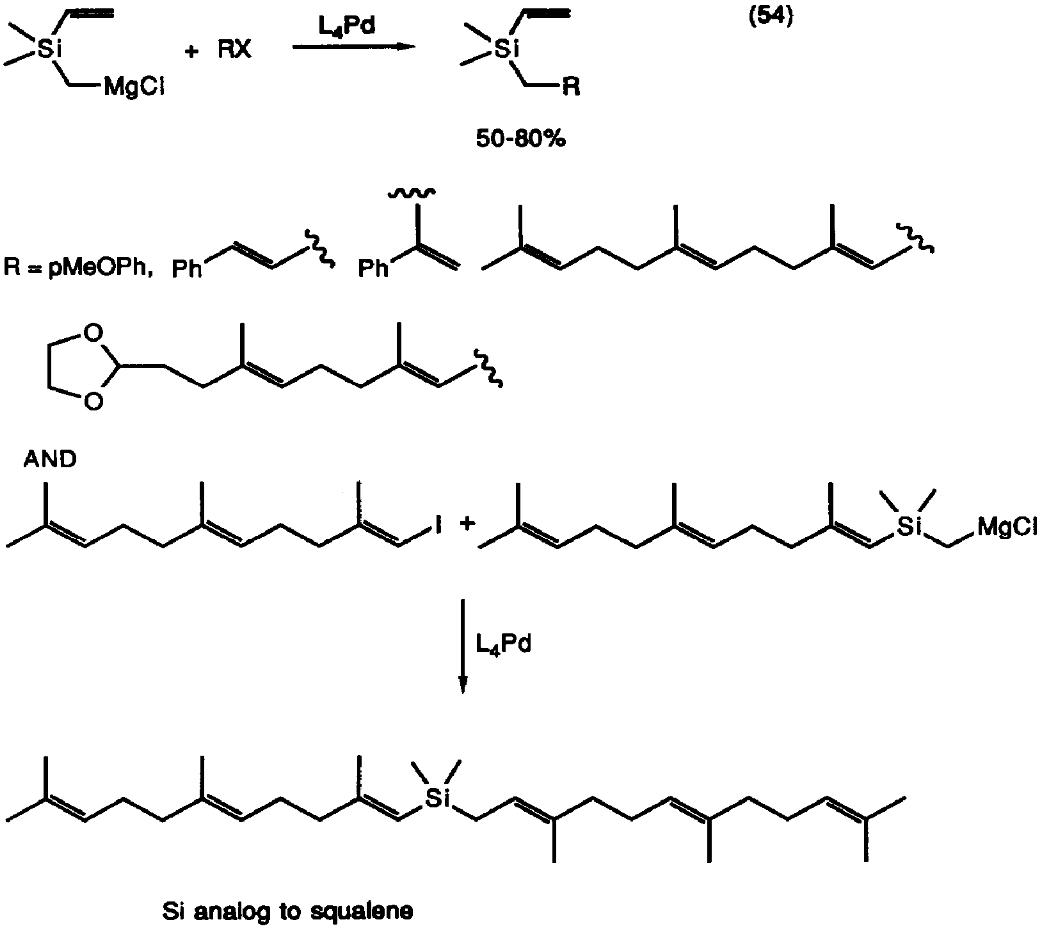


Ar = pFPh, oMeOPh, 2-naphth,

(53)

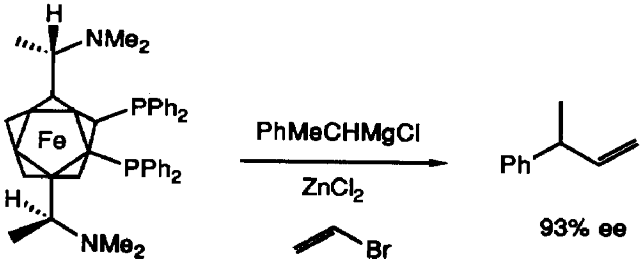


n = 5, 11
Ar = Ph, 1-Naphth, 2-Naphth,
Ar' = 1,2-Ph, 1,4-Ph, 1,5-Naphth, 2,6-Naphth, 4,9-Pyrene, 2,6-Pyrene



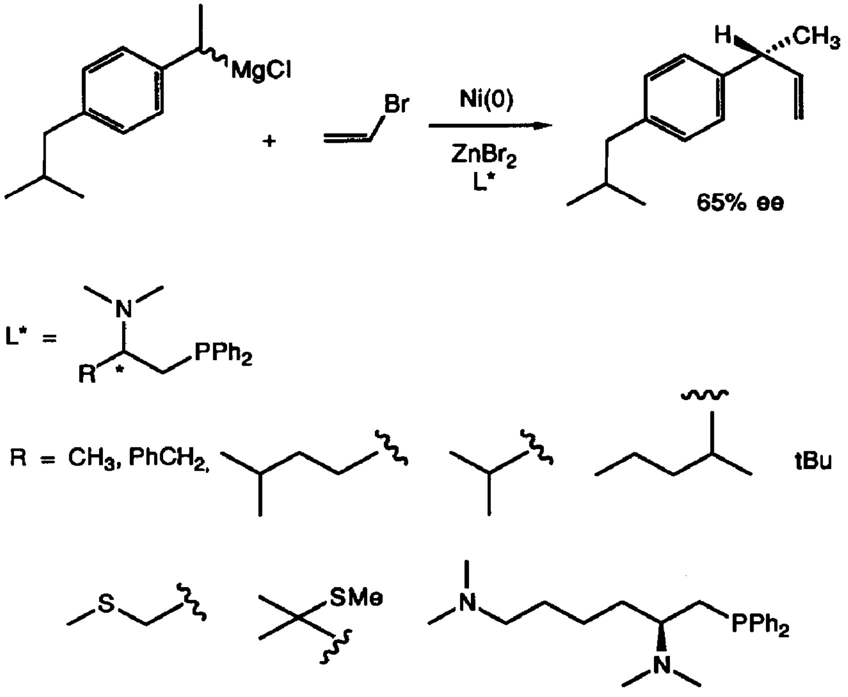
(54)

The use of optically active ligands in the process permitted the metal-catalyzed reactions of Grignard reagents to proceed with high degrees of asymmetric induction (equation 55) [62], (equation 56) [63], (equation 57) [64], (equation 58) [65].

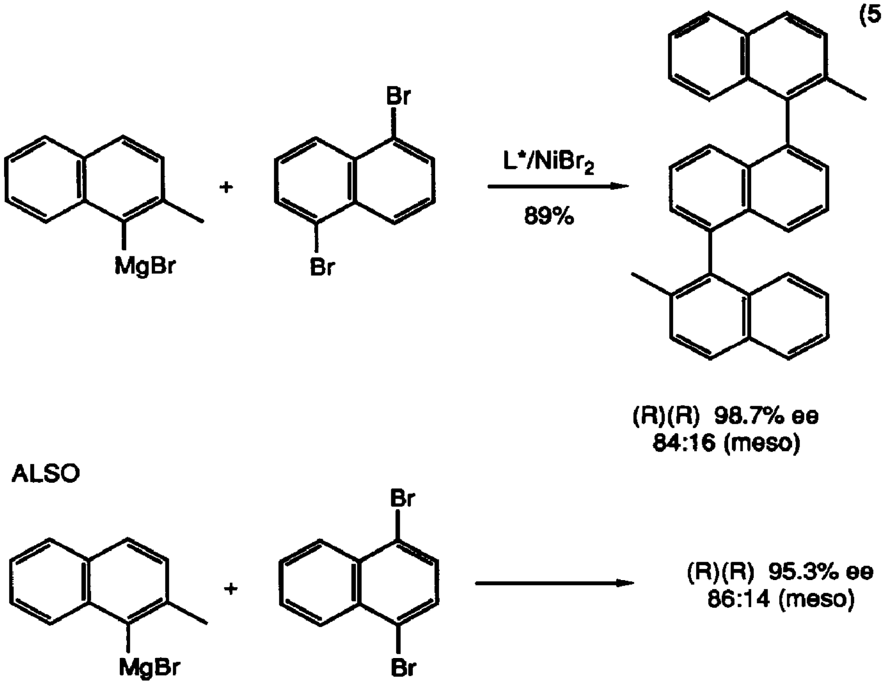

NMe2
PPh2
Fe
PPh2
H
NMe2
PhMeCHMgCl
ZnCl2
Br
Ph
93% ee

(55)

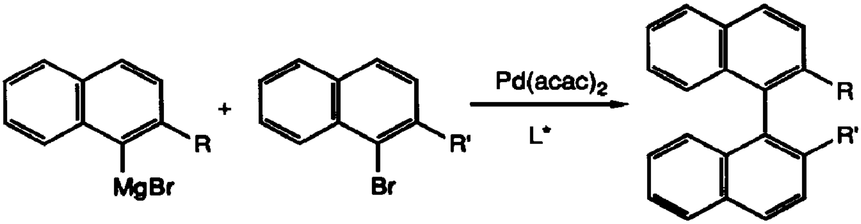
(56)


MgCl
+
Br
Ni(0)
ZnBr2
L*
H
CH3
65% ee
L* =
N
PPh2
R
*
R = CH3, PhCH2,
tBu
S
SMe
N
PPh2
N

(57)

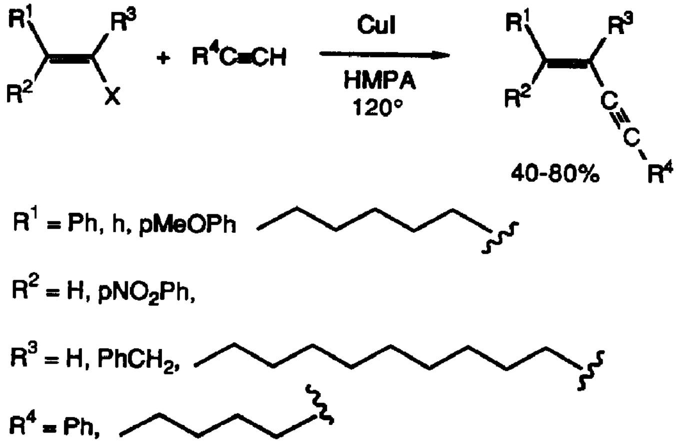


(58)

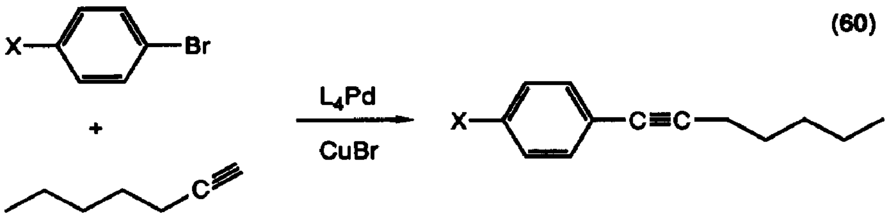


Eneynes were prepared by the copper catalyzed coupling of vinyl halides with terminal alkynes (equation 59) [66]. Palladium(0)/copper(I) catalysts coupled terminal alkynes to aryl halides (equation 60) [67], (equation 61) [68], (equation 62) [69]. Dialkylcuprates alkylated propargyl alcohols and mesylates with high anti stereoselectivity (equation 63) [70]. Fluorinated sugars were arylated by aryl halides (Friedel Crafts) using zirconium/silver systems to promote the reaction (equation 64) [71]. A wide variety of metal alkyls alkylated lithium "carbenoids" (equation 65) [72].

(59)



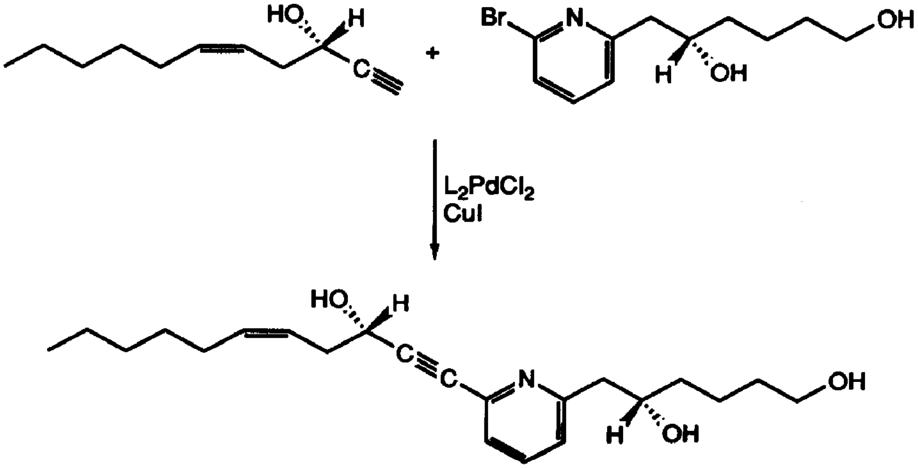
(60)



X = NO$_2$, CN, CHO, CO$_2$Me, CO$_2$H, Cl, Me, H, MeO, NH$_2$

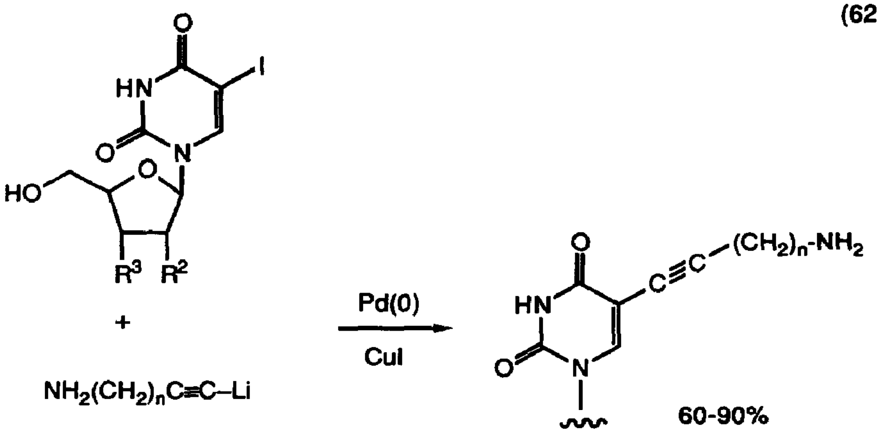
3 min ⟶ 480 min

(61)

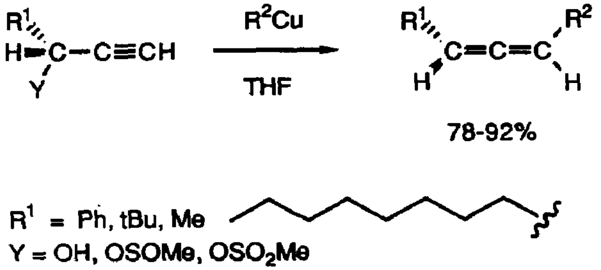


70%

(62)



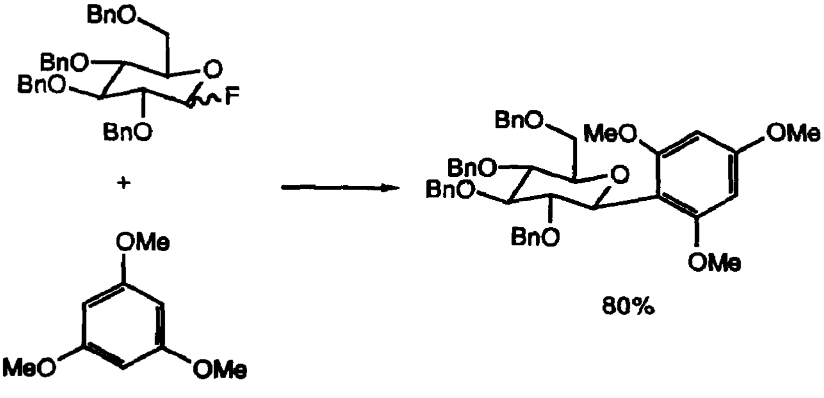
(63)



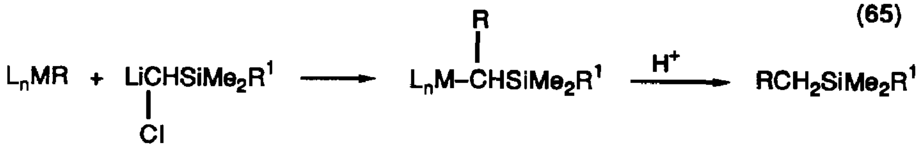
R^1 = Ph, tBu, Me

Y = OH, OSOMe, OSO$_2$Me

(64)



(65)



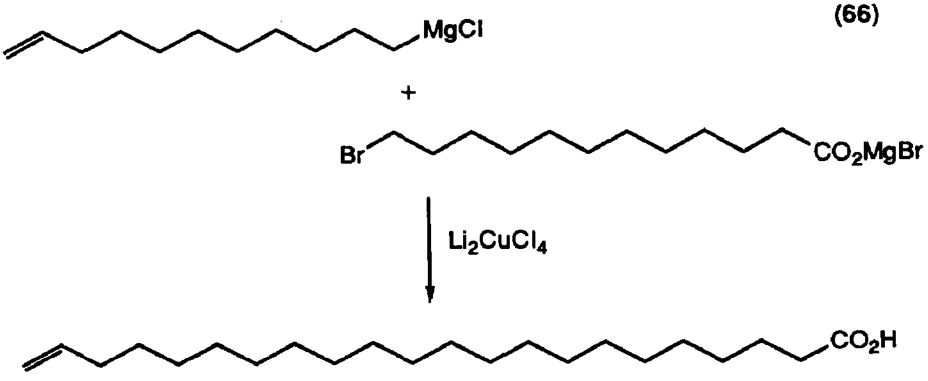
$$L_nMR + \underset{\displaystyle Cl}{LiCHSiMe_2R^1} \longrightarrow L_nM\text{-}\overset{\displaystyle R}{C}HSiMe_2R^1 \xrightarrow{H^+} RCH_2SiMe_2R^1$$

M = Ti, Zr, Hf, V, Cr, Mn, Fe, Co, Ni, Cu

R = nC_4, nC_8, Ph, otol, 

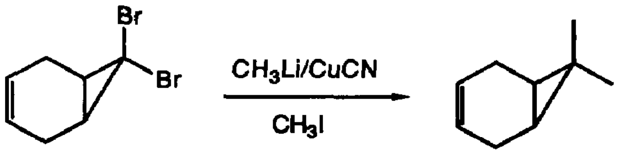
Perfluoroalkyl benzenes were prepared by the coupling of aryl halides with perfluoroalkyl iodides by copper metal [73]. Long chain unsaturated acids were prepared by the copper catalyzed reactions of ω-olefinic Grignard reagents with the MgBr salts of ω-bromo acids (equation 66) [74]. Dibromocyclopropanes were dialkylated by organocopper complexes (equation 67) [75]. Cyclopropyl carbonyl bromides were ring opened by organocopper compounds (equation 68) [76]. Organocopper species arylated ω-iodoamino acids (equation 69) [77]. Functionalized organocuprates were prepared and underwent typical organocopper reactions (eqution 70) [78]. Copper enolates of hydrazones alkylated primary iodides (equation 71) [79]. Organocopper species were used to difunctionalize diiodomethane-derived zinc compounds (equation 72) [80].

(66)

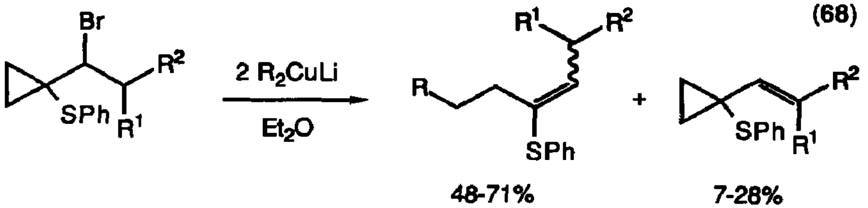


29%

(67)

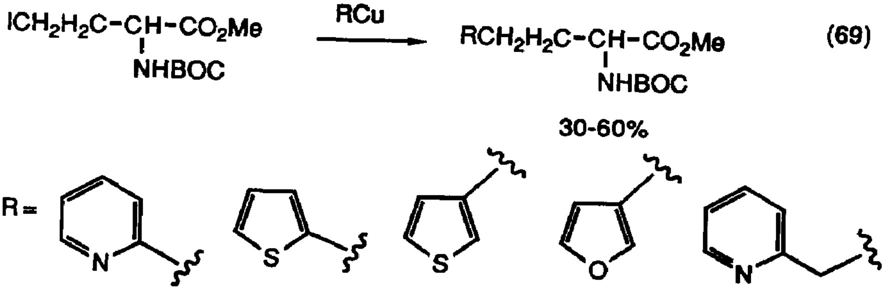


(68)

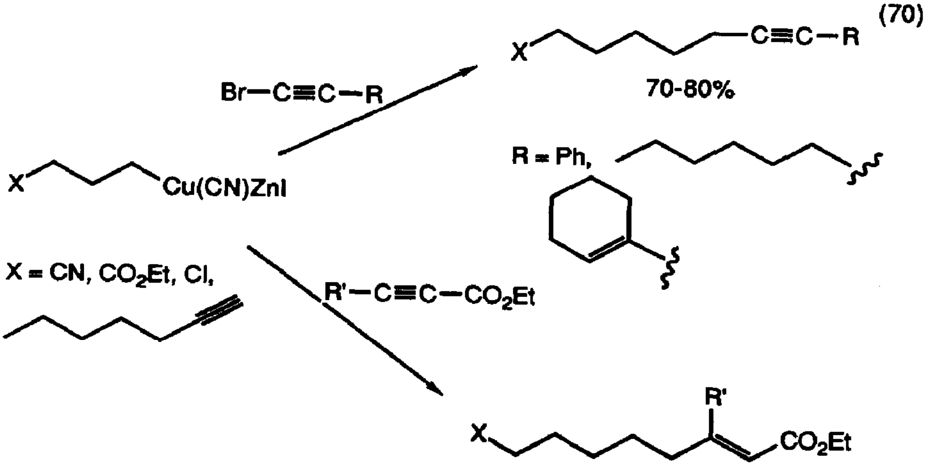


R^1 = H, Me/ R^2 = Me; R = nBu, Me, Ph, tBu

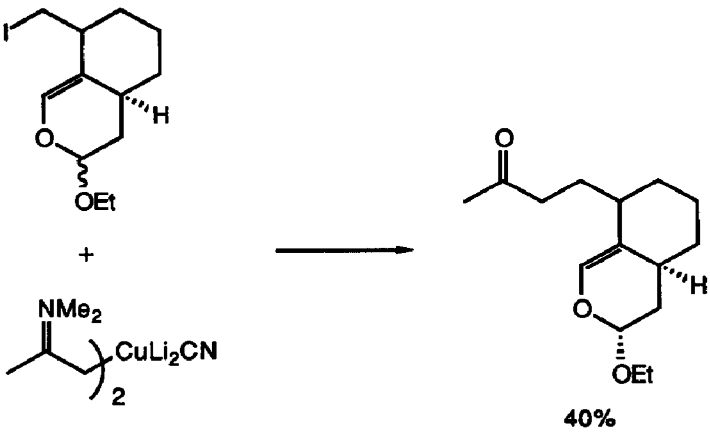
(69)



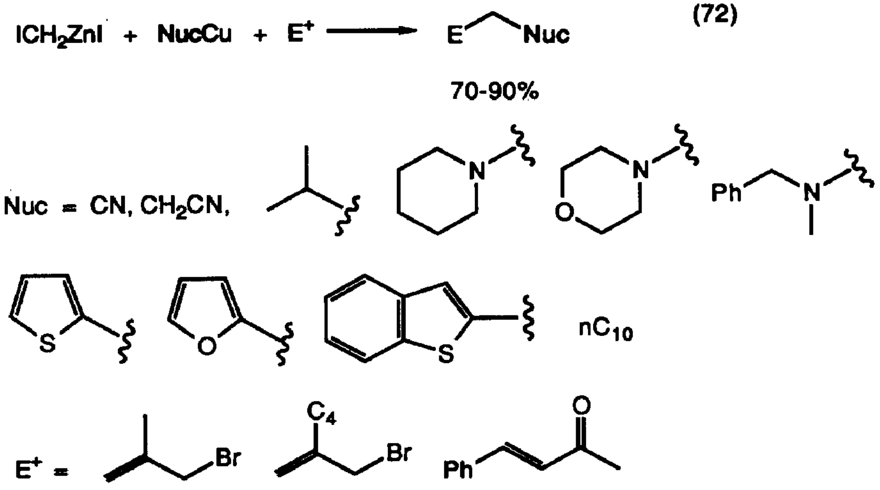
(70)



(71)

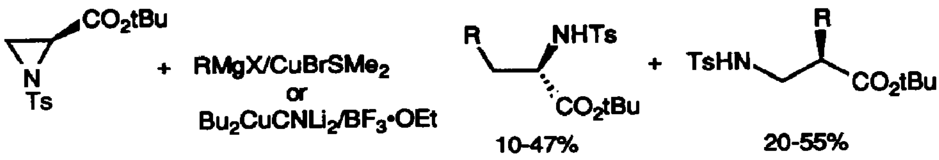


(72)

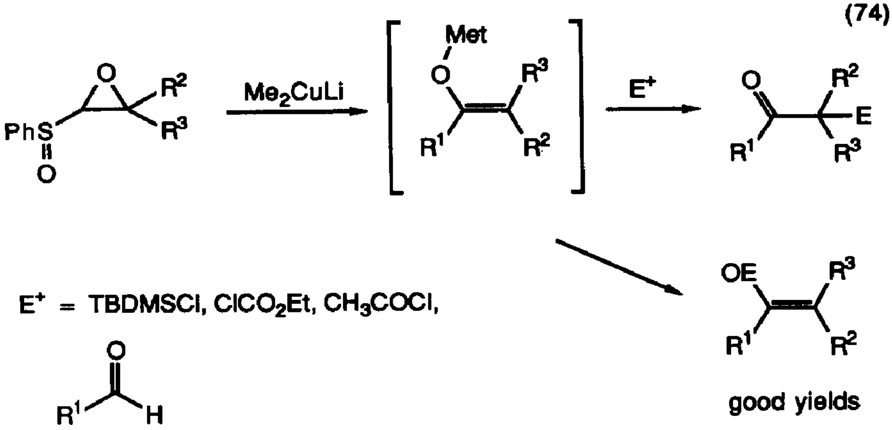


Allylic alcohols, acetates, carbonates and chlorides were directly alkylated by acetylacetone anion in the presence of $Cu(ClO_4)_2$ and copper powder [81]. Organocuprates ring-opened aziridines (equation 73) [82]. S_N2' addition of organocopper reagents to vinyloxiranes was the subject of a review (39 references) [83]. Methylcuprates converted α-epoxysulfoxides to enolates which were then trapped with electrophiles (equation 74) [84]. Organocuprates alkylated epoxy sugars (equation 75) [85] as well as simple epoxides (equation 76) [86]. α-Lithio cyclic enol ethers were ring opened by organocopper species (equation 77) [87]. Grignard reagents deoxygenated ketals in the presence of nickel(II) salts (equation 78) [88]. Ortho-palladated benzaldehyde imines were electrophilically alkylated by methyl iodide (equation 79) [89].

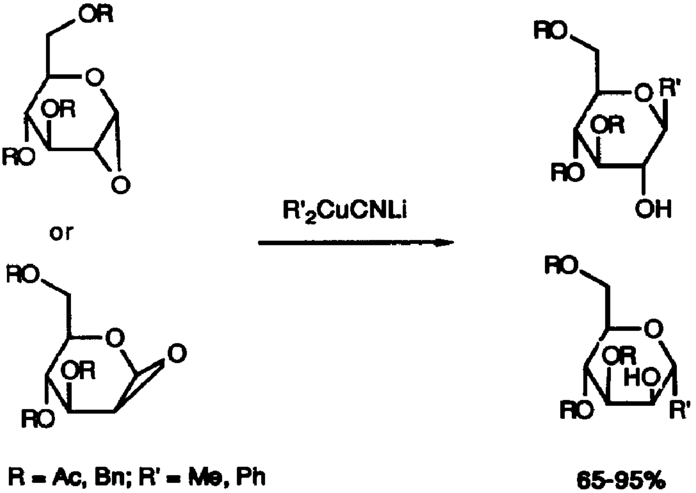
(73)


CO2tBu
N
Ts
+ RMgX/CuBrSMe2
or
Bu2CuCNLi2/BF3•OEt
R
NHTs
CO2tBu
10-47%
+
TsHN
R
CO2tBu
20-55%

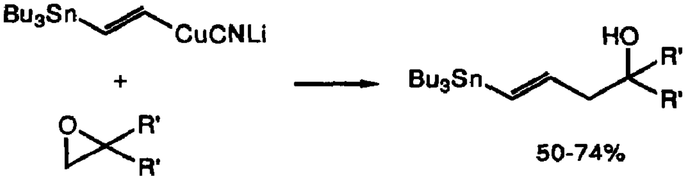
(74)

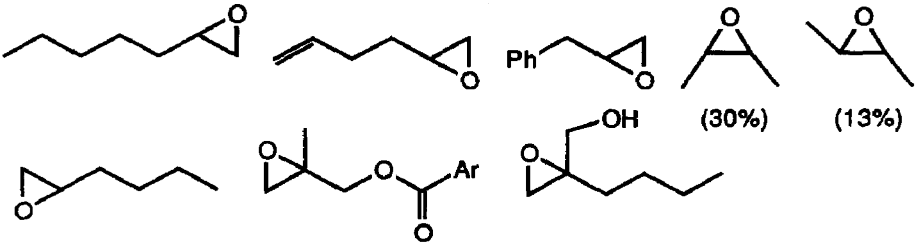

PhS
O
O
R2
R3
Me2CuLi
Met
O
R3
R1
R2
E+
O
R2
E
R1
R3
OE
R3
R1
R2
good yields
E+ = TBDMSCl, ClCO2Et, CH3COCl,
O
R1
H

(75)

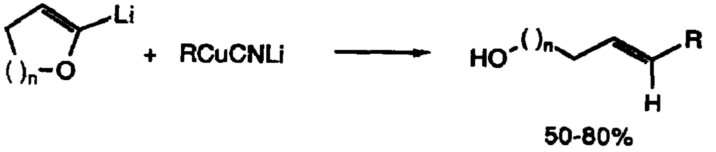

OR
O
OR
RO
O
or
RO
O
O
OR
RO
R'2CuCNLi
RO
O
R'
OR
RO
OH
RO
O
OR
HO
RO
R'
R = Ac, Bn; R' = Me, Ph
65-95%

(76)



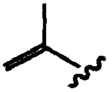


(77)

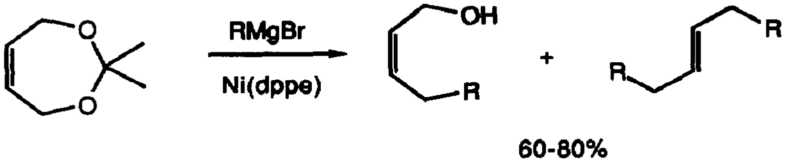


n = 1, 2

R = Bu, secBu, Me, tBu, Bn, Me$_3$Sn,

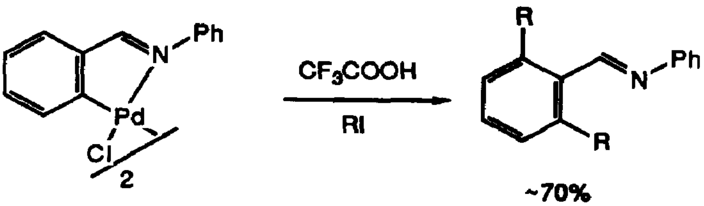


(78)



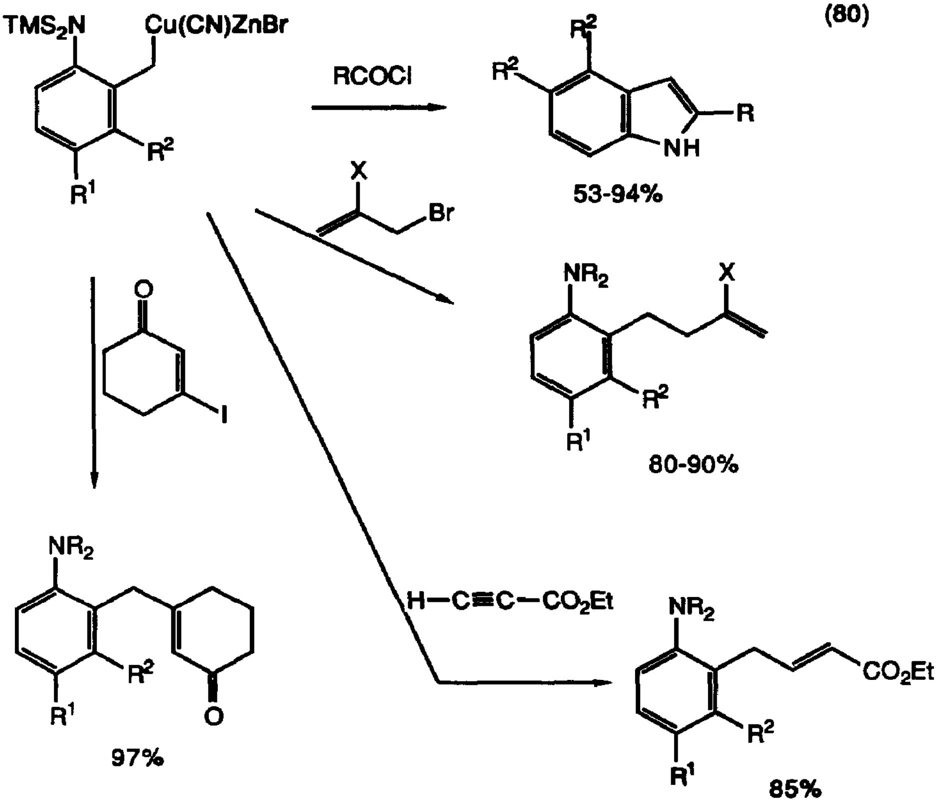
R = Et, nPr, nBu, iBu, iPr, Ph, tBu, Bn,

(79)



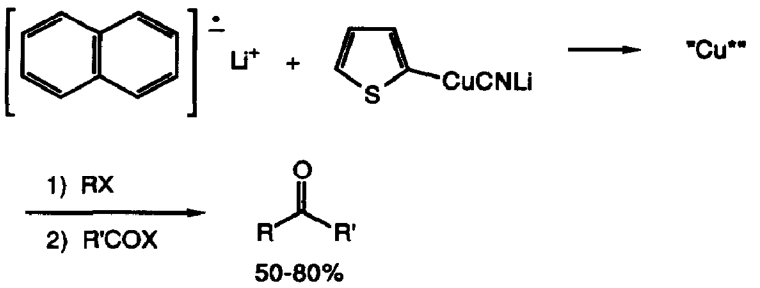
2. Alkylation of Acid Derivatives

Copper complexes catalyzed the coupling of Grignard reagents to acid halides to give straight-chain ketones [90]. Highly functionalized benzyl cuprates alkylated acid halides to give indoles (equation 80) [91]. Activated copper coupled functionalized alkyl halides with acid chlorides to give unsymmetrical ketones (equation 81) [92]. New procedures for the preparation of organomanganese compounds for the conversion of acid chlorides to ketones have been derived [93].

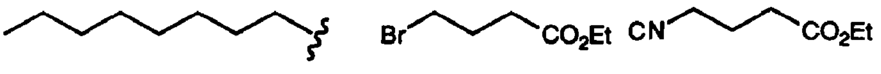


(80)

(81)

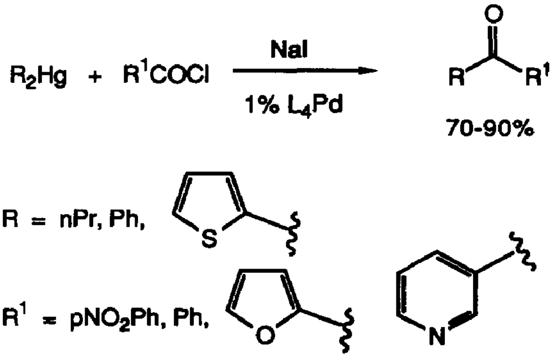


R = pMePhBr, oMeOPhBr, pCNPhBr, mCNPhAr, pClPhI, pFPhBr, pPhCOPhBr

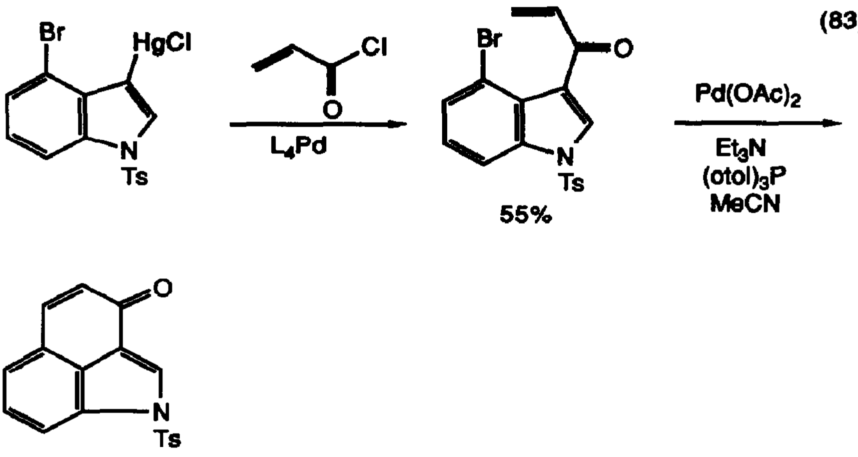


Palladium(0) complexes catalyzed the alkylation of acid halides to ketones by dialkylmercury compounds (equation 82) [94], organomercurial halides (equation 83) [95], sulfonyl chlorides by organostannanes (equation 84) [96], and acid chlorides by organostannanes (equation 85) [97], and organozinc reagents (equation 86) [98]. Electrochemical reduction of nickel(II) bromide (bipy) complexes in the presence of acid halides and organic halides produced ketones (equation 87) [99]. Palladium(0) complexes catalyzed the alkylation of acid chlorides by siloxycyclopropanes (equation 88) [100].

(82)



(83)

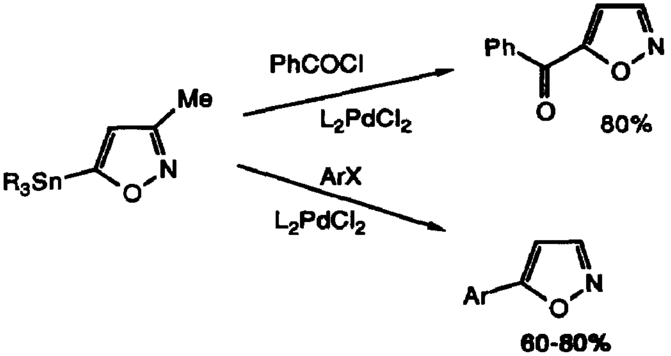


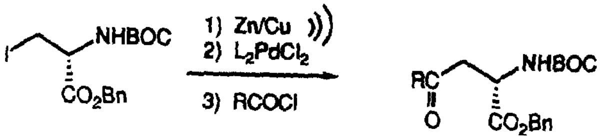
(84)

$$RSO_2Cl + Bu_3SnR' \xrightarrow[THF]{L_4Pd} RSO_2R' + Bu_3SnCl$$

57-90%

(85)

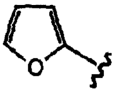


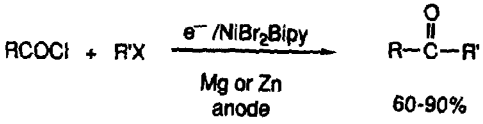


(86)

~100% ee
76-90%

R = Ph, Me, Et, iPrCH$_2$, iBuCH$_2$,





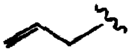
$$\mathrm{RCOCl} + \mathrm{R'X} \xrightarrow[\text{Mg or Zn anode}]{e^{-}/\mathrm{NiBr_2Bipy}} \mathrm{R{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}R'}$$

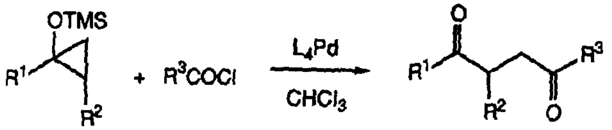
(87)

60-90%

R = Bn, Et, Ph, nPr

R' = Ph, Bn,





(88)

60-90%

R^1 = OEt, OiPr

R^2 = H, Me

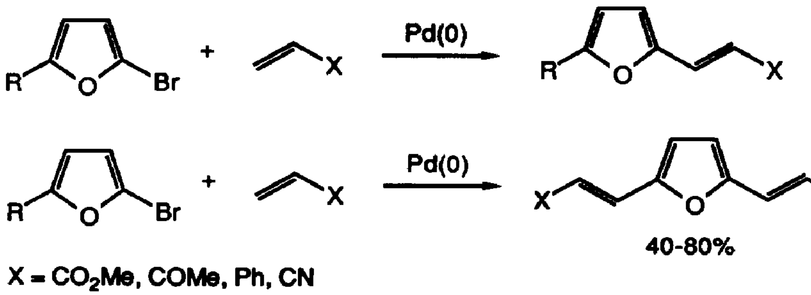
R^3 = Ph, pClPh, pMeOPh, Et,

ALSO

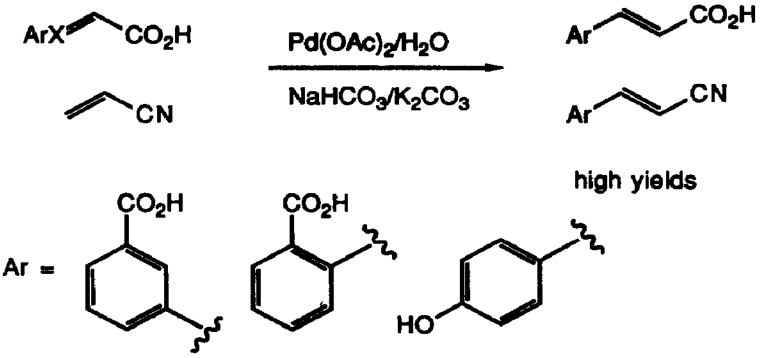
46-70%

3. Alkylation of Olefins

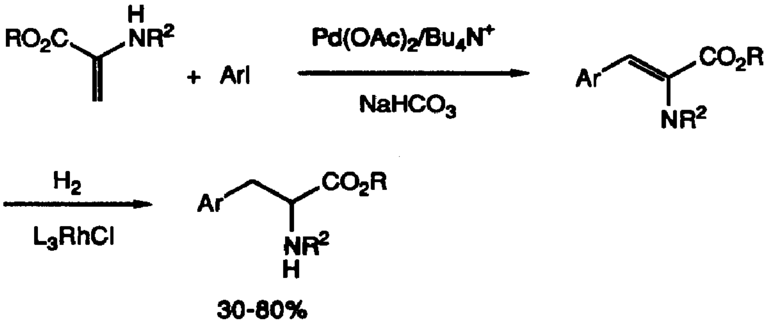
Insertion of olefins into σ-alkylmetal complexes remained the major approach to the alkylation of olefins. Olefin insertion into organyl metal bonds has been reviewed (64 references) [101], as has 1,2-addition to heterosubstituted olefins by organopalladium reagents (114 references) [102]. Palladium(0) catalyzed the alkylation of olefins by bromofurans (equation 89) [103], aryl halides (equation 90) [104], and the arylations of α-acetamidoacrylates (equation 91) [105]. Prostaglandin side chains were appended by palladium(0) catalyzed oxidative addition/insertion processes (equation 92) [106]. Palladium(II) complexes supported on ion exchange resins catalyzed the vinylation of aryl iodides by olefins [107].



(89)



(90)

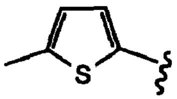


(91)

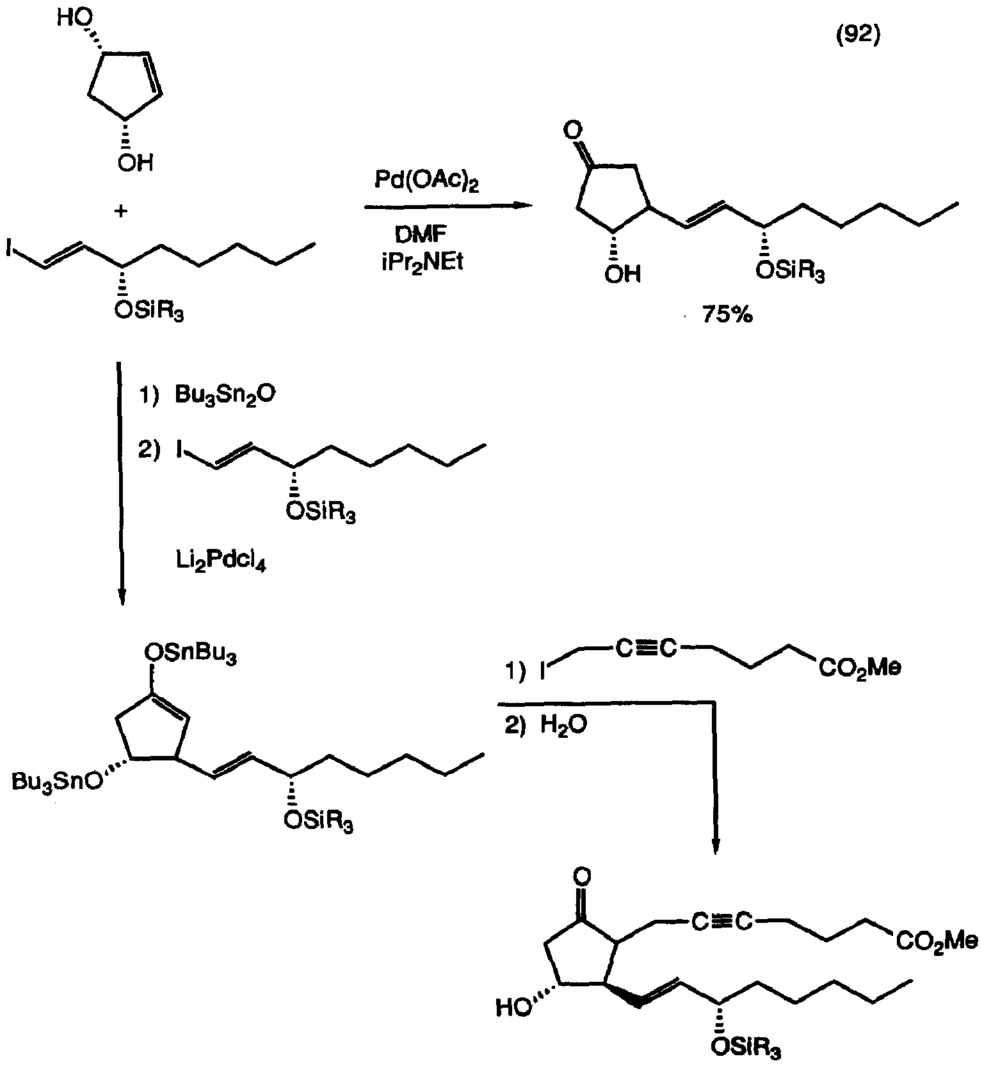
R = H, Me; R^2 = BOC, Bn, Me

Ar = Ph, pMePh, pHOPh, pNO_2Ph, oHOPh, oMeOPh, pMeOPh, $mMeO_2CPh$,

1,1'-ferrocenyl

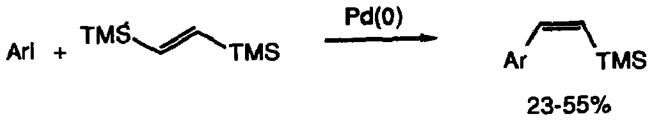


(92)



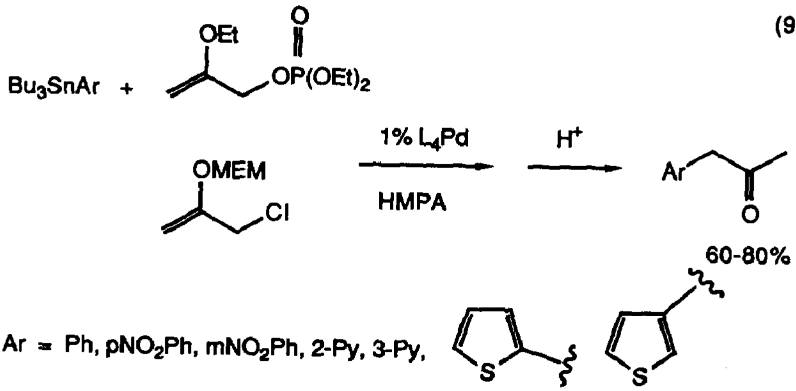
Vinyl silanes were prepared by the palladium(0) catalyzed arylation of 1,2-bis(trimethylsilyl)ethylene (equation 93) [108]. Ketones were prepared by the palladium-catalyzed arylation of allyl phosphonates (equation 94) [109] and ω-olefinic alcohols (equation 95) [110]. Arylsulfonyl chlorides [111] and arylethene sulfonyl chlorides [112] arylated and vinylated olefins in the presence of palladium catalysts. Palladium salts catalyzed the olefination of chloromercuriosydnones (equation 96) [113][114].

(93)



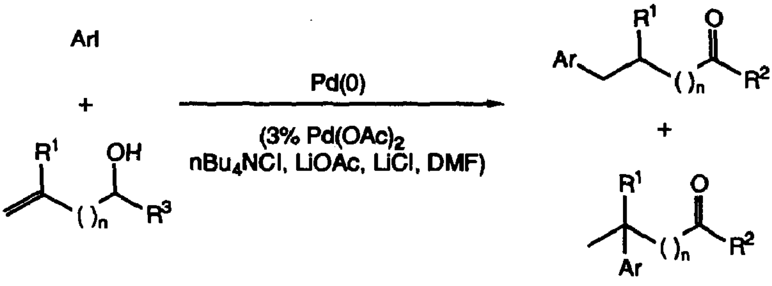
Ar = Ph, pNO_2Ph, pMeOPh, oCO_2MePh, 2-naphth

(94)



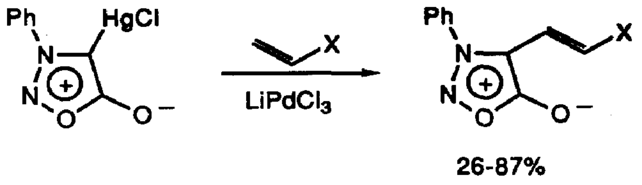
Ar = Ph, pNO_2Ph, mNO_2Ph, 2-Py, 3-Py,

(95)



Ar = Ph, pMePh, pMeOPh, $pEtO_2CPh$, 2-naphth
R^1 = H, Me
R^2 = H, Me
n = 1, 2, 3, 8

69-95%
76:24 to 100:1

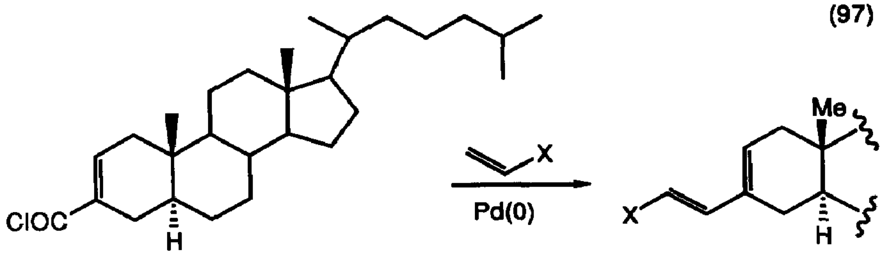


(96)

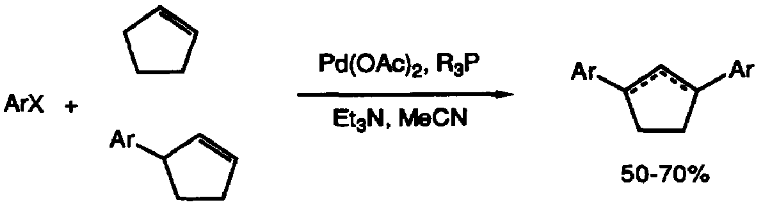
X = COMe, CO_2Me, Ph, CN, $C_2B_{10}H_4$

Cyclic olefins also readily underwent olefination and arylation. The metal-organic approach to the stereoselective synthesis of exocyclic alkenes has been reviewed (75 references) [115], and palladium-catalyzed allylic arylation of cyclic alkenes was the topic of a dissertation [116]. Cholestenyl chlorides were olefinated using palladium catalysts (equation 97) [117]. Simple cyclic olefins underwent diarylation using palladium catalysis (equation 98) [118], or were converted to allyl arenes or dienes under similar conditions (equation 99) [119], (equation 100) [120], (equation 101) [121]. Vinyl triflates alkylated enol ethers to give alkoxydienes in the presence of palladium catalysts (equation 102) [122]. Palladium(0) catalyzed the heteroarylation of indole by chloropyrazines (equation 103) [123].

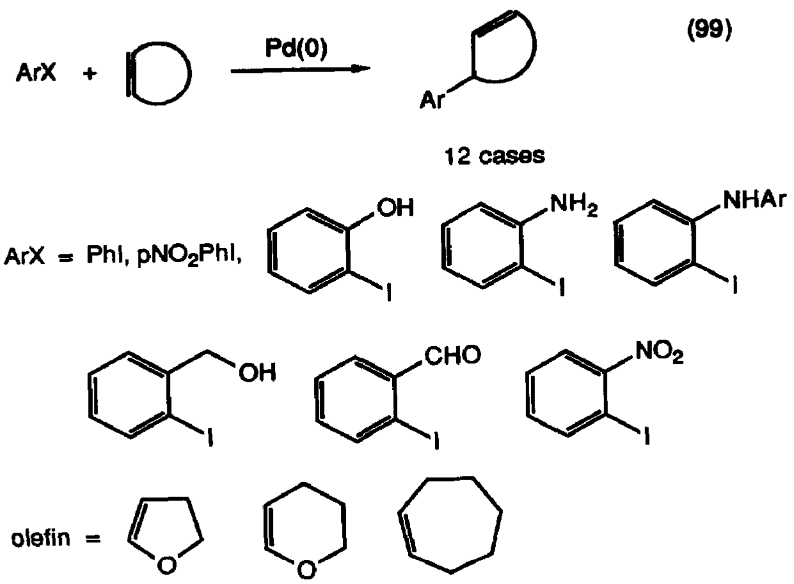
(97)



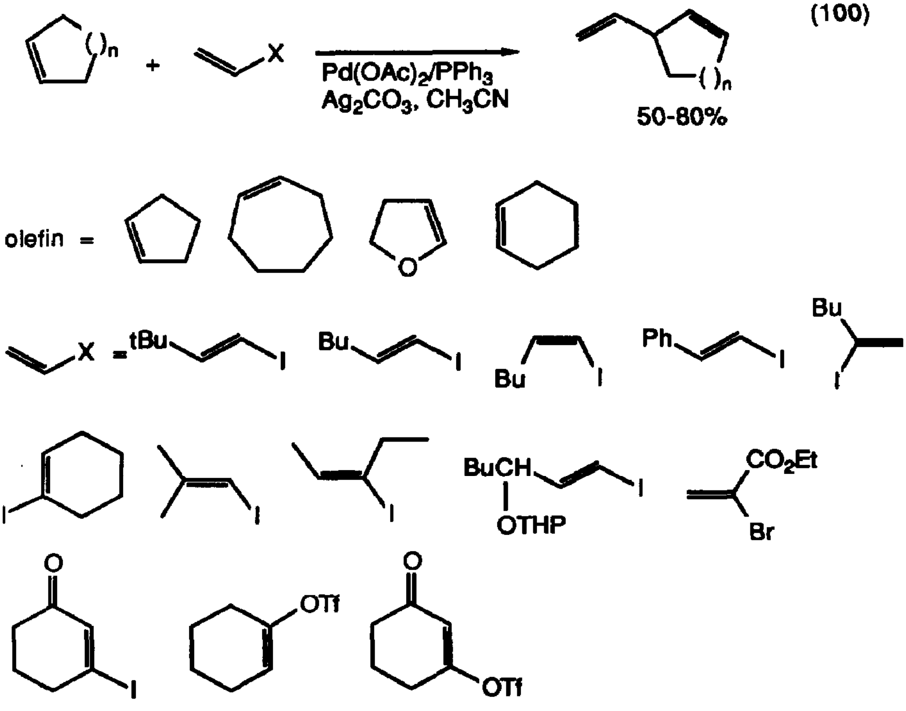
(98)



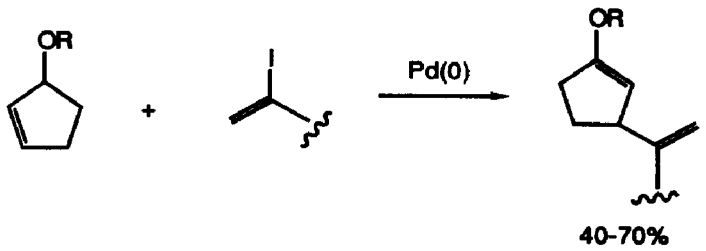
Ar = Ph, pMeOPh, 3,4-(MeO)$_2$Ph, 3,5-(MeO)$_2$Ph, 3,4,5-(MeO)$_3$Ph


ArX
+
Pd(0)
Ar
12 cases
ArX = PhI, pNO₂PhI,
OH
I
NH₂
I
NHAr
I
OH
I
CHO
I
NO₂
I
olefin =
O
O

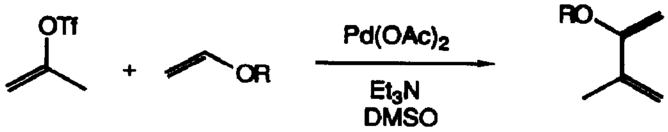
(99)


( )n
+
X
Pd(OAc)₂/PPh₃
Ag₂CO₃, CH₃CN
( )n
50-80%
olefin =
O
X =
tBu
I
Bu
I
Bu
I
Ph
I
Bu
I
I
I
I
BuCH
OTHP
I
CO₂Et
Br
O
I
OTf
O
OTf

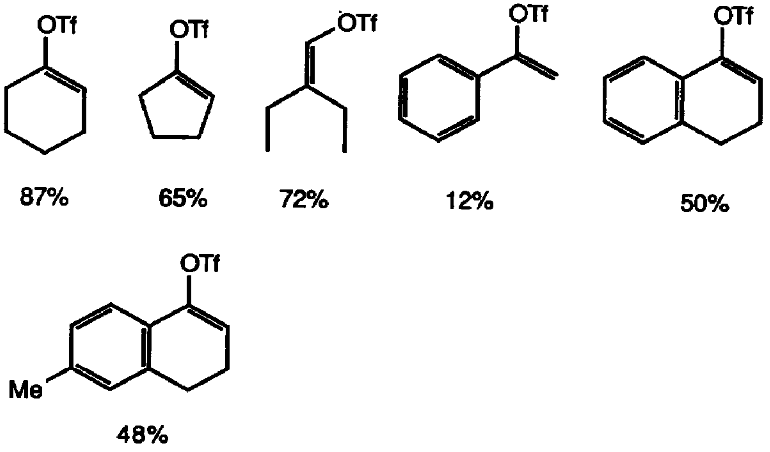
(100)


OR
+
Pd(0)
OR
40-70%

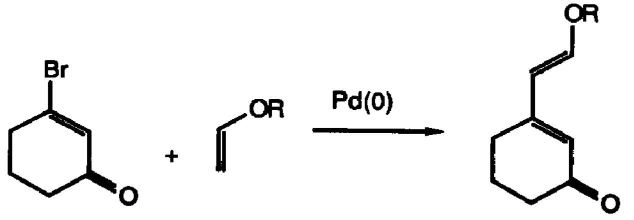
(101)


OTf
+
OR
Pd(OAc)2
Et3N
DMSO
RO

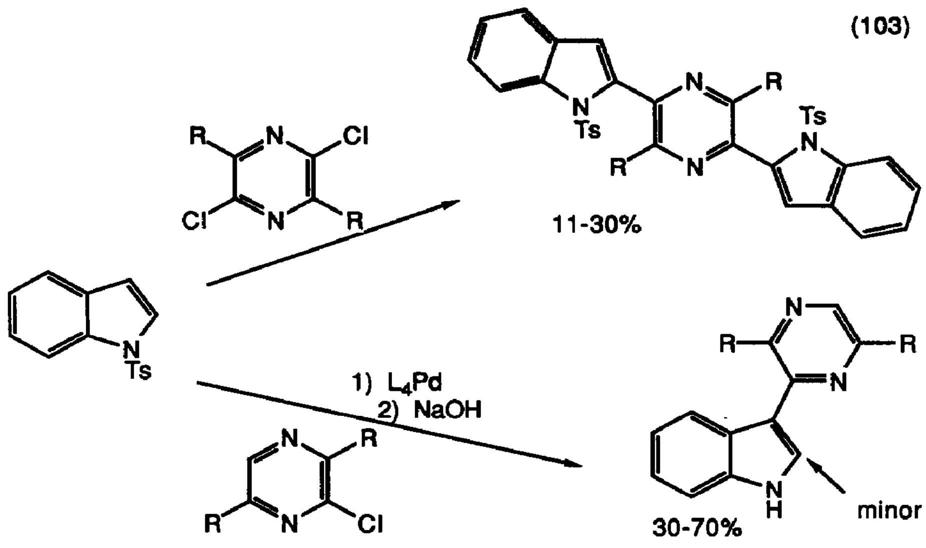
(102)


OTf
87%
OTf
65%
OTf
72%
OTf
12%
OTf
50%
OTf
Me
48%

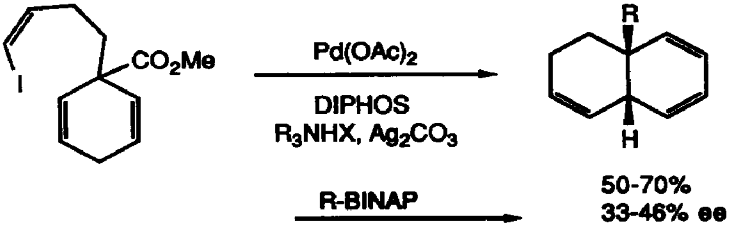
ALSO


Br
O
+
OR
Pd(0)
OR
O

(103)

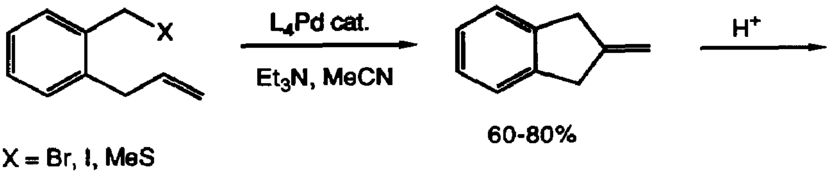


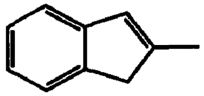
Intramolecular versions of the above processes have been extensively developed (equation 104) [124], (equation 105) [125], (equation 106) [126], (equation 107) [127], (equation 108) [128], (equation 109) [129]. Related intramolecular arylation of olefins involving direct palladation of arenes has also been developed (equation 110) [130], (equation 111) [131].



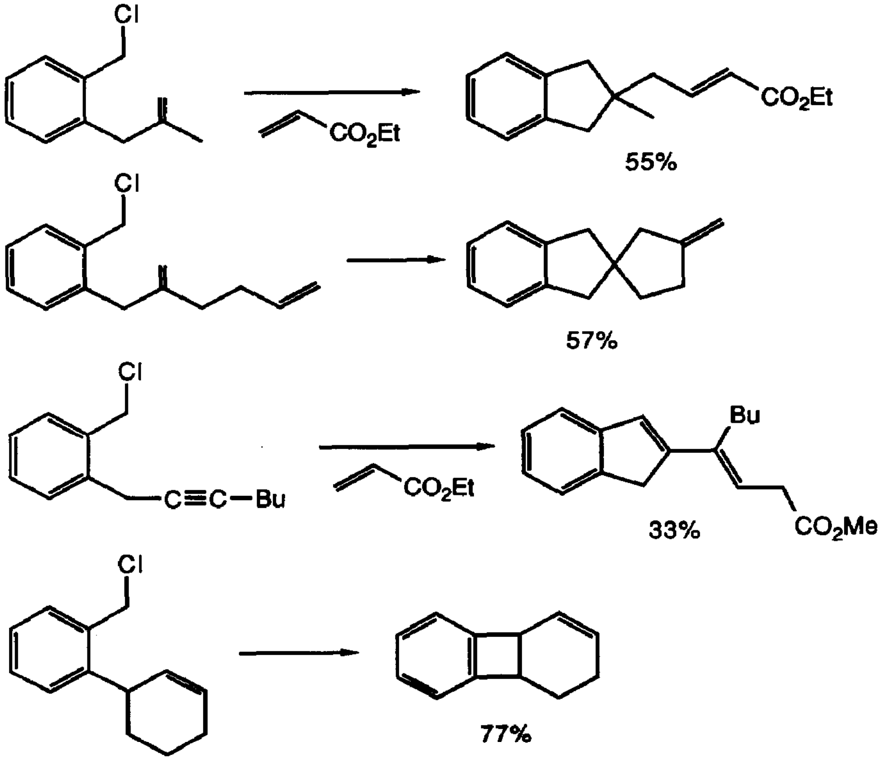
(104)

(105)

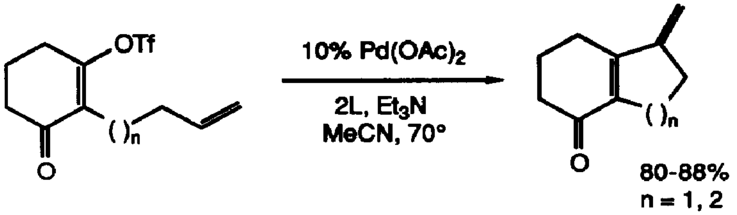




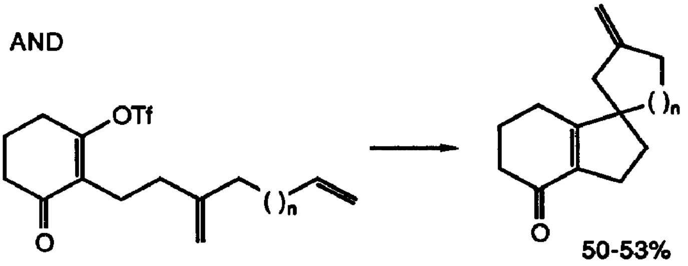
Many examples, e.g.:



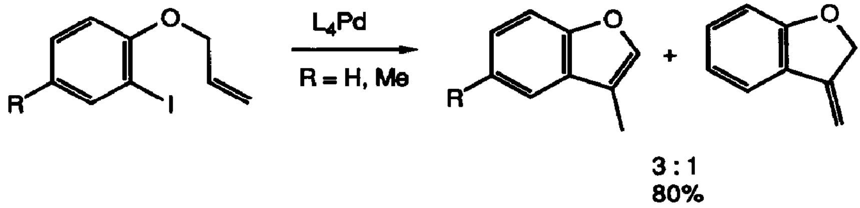
(106)

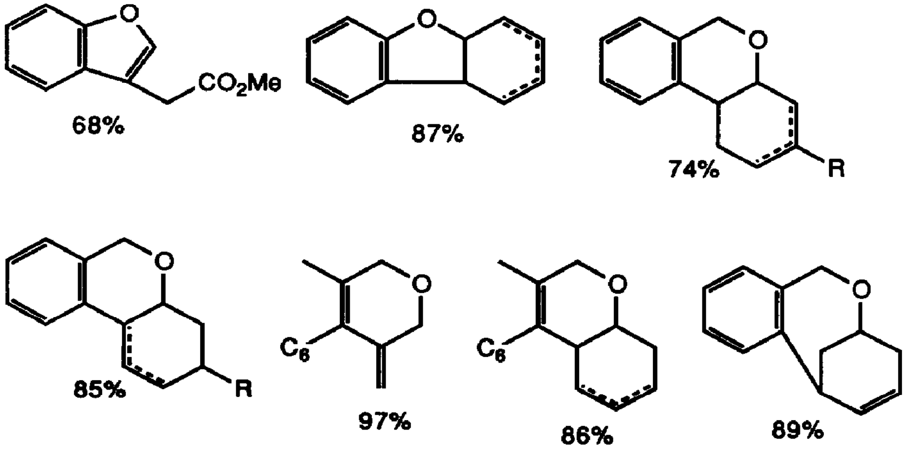

OTf
( )n
O
10% Pd(OAc)2
2L, Et3N
MeCN, 70°
( )n
O
80-88%
n = 1, 2

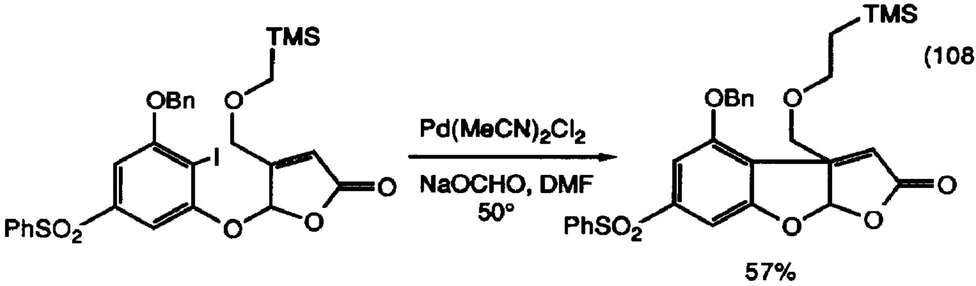
AND


OTf
O
( )n
( )n
O
50-53%

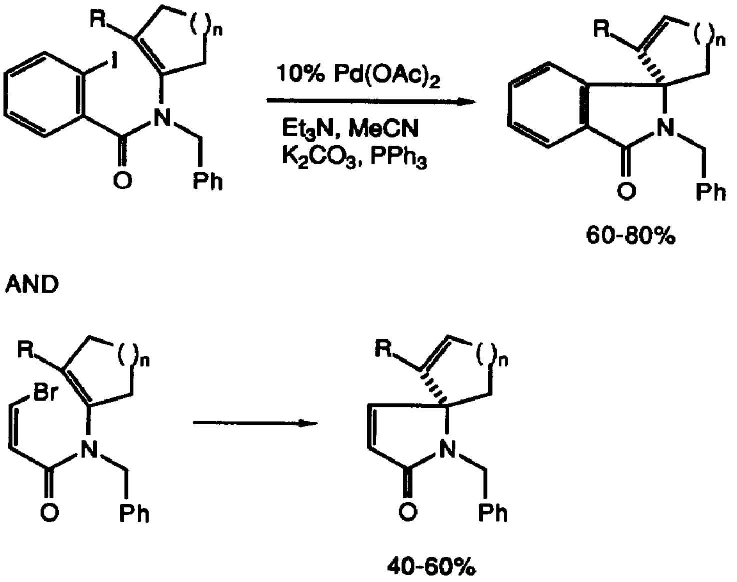
(107)


O
R
I
L4Pd
R = H, Me
O
R
+
O
3 : 1
80%

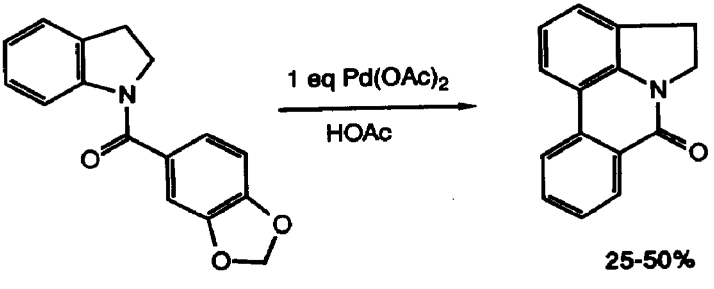

O
CO2Me
68%
O
87%
O
74%
R
O
85%
R
O
C6
97%
O
C6
86%
O
89%


TMS
OBn
I
O
PhSO2
O
O
=O
Pd(MeCN)2Cl2
NaOCHO, DMF
50°
57%

(108)

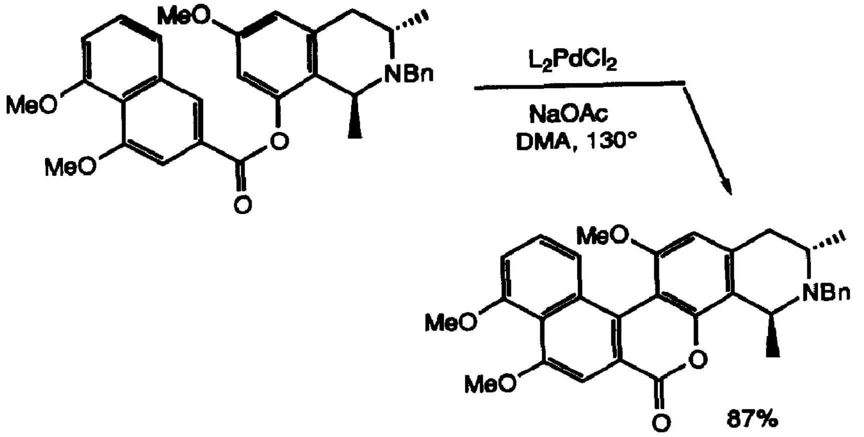

R
( )n
I
N
O
Ph
10% Pd(OAc)2
Et3N, MeCN
K2CO3, PPh3
60-80%
AND
Br
40-60%

(109)


N
O
1 eq Pd(OAc)2
HOAc
25-50%

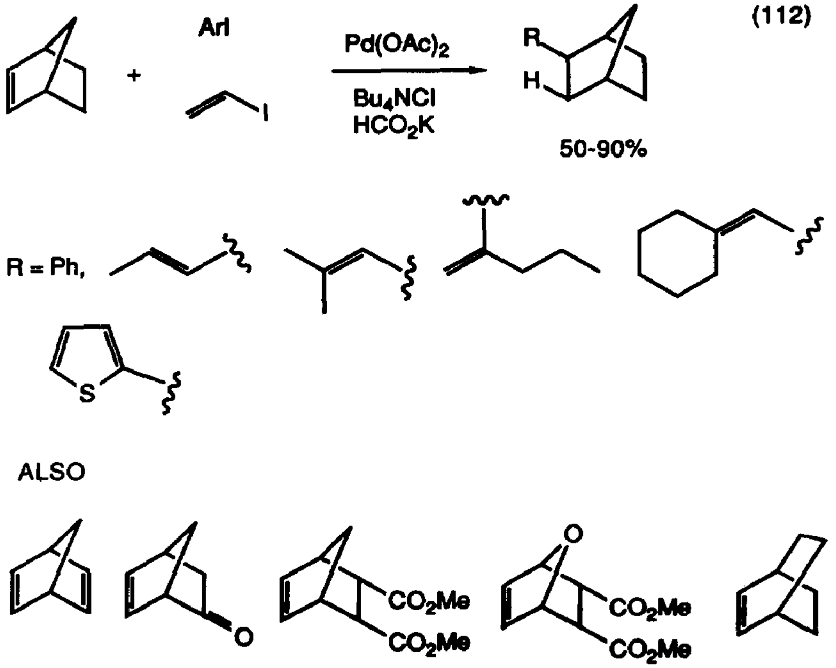
(110)

(111)



As in some of the examples cited above, palladium can be used several times in a sequence to effect polyfunctionalization. Recent examples of this include olefin arylation or vinylation/reduction (equation 112) [132], arylation/alkylation (equation 113) [133], diarylation (equation 114) [134], arylation/cyclization (equation 115) [135], and arylation/elimination (equation 116) [136].

(112)



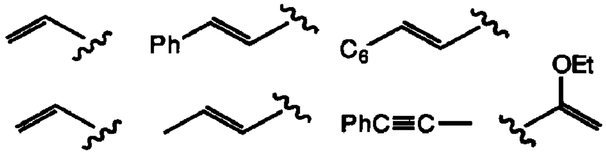
56-99%

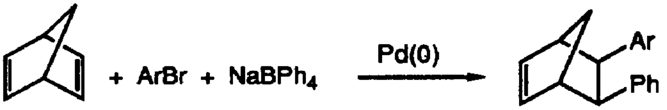
(113)

Ar = Ph, pMeOPh, oMePh, pAcOPh, pCNPh

ALSO

R' = Ph,

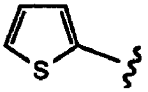




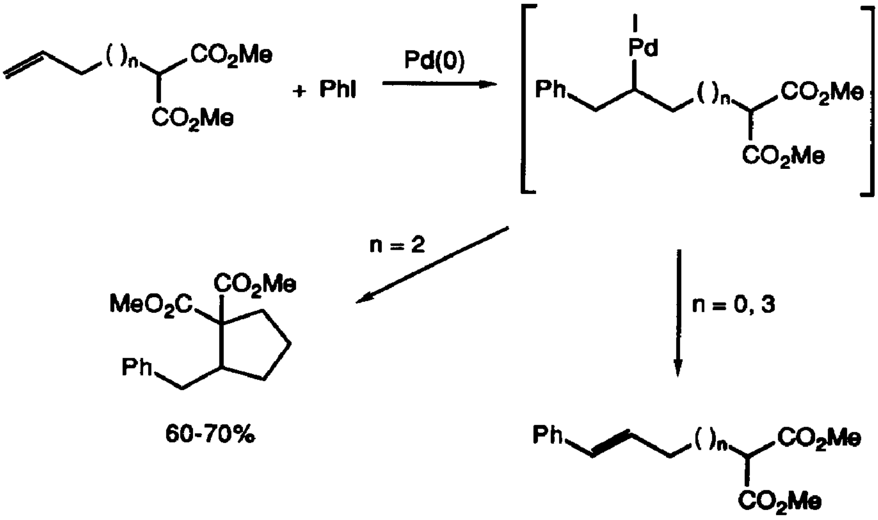
70-95%

(114)

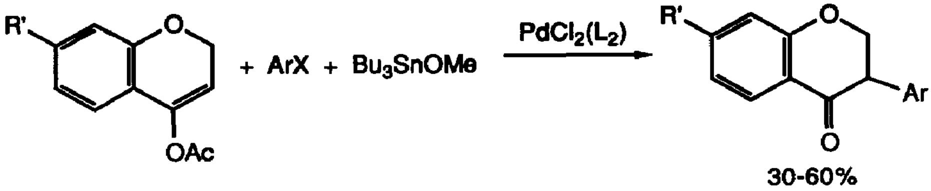
Ar = Ph, oMePh, mMePh, pMePh, pMeOPh, pNO$_2$Ph,



(115)

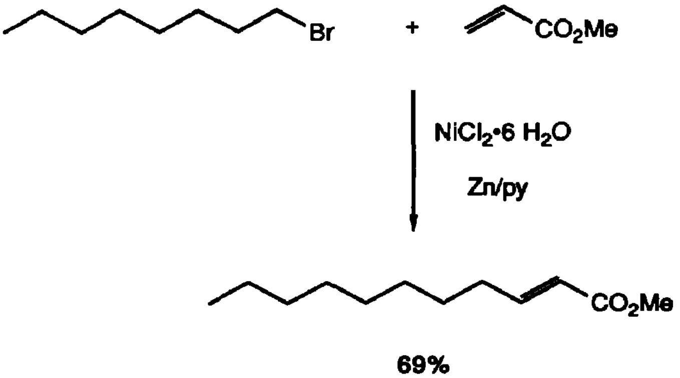


(116)

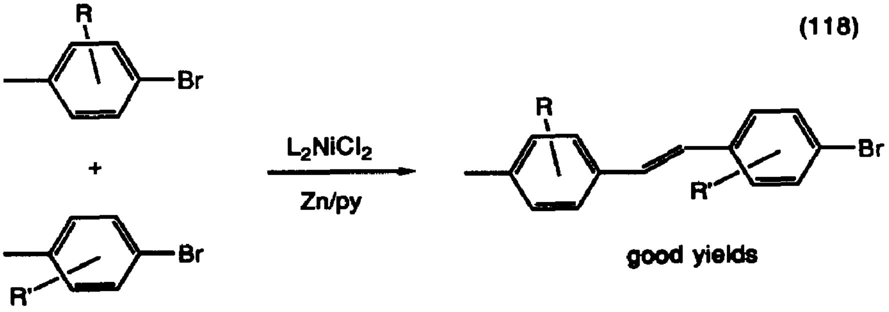


Electron deficient olefins were alkylated by alkyl halides in the presence of $NiCl_2{\bullet}6H_2O$/zinc in pyridine (equation 117) [137]. Similar catalyst systems resulted in the condensation of aryl halides with styrenes to give substituted stilbenes (equation 118) [138]. Acrolein ketals were difunctionalized by organozinc halides in the presence of nickel(II) phosphine complexes (equation 119) [139], while Grignard reagents ring opened dihydropyran (equation 120) [140]. Vinyl sulfoxamines were substituted by organozinc or magnesium species in the presence of nickel(II) phosphine complexes (equation 121) [141].

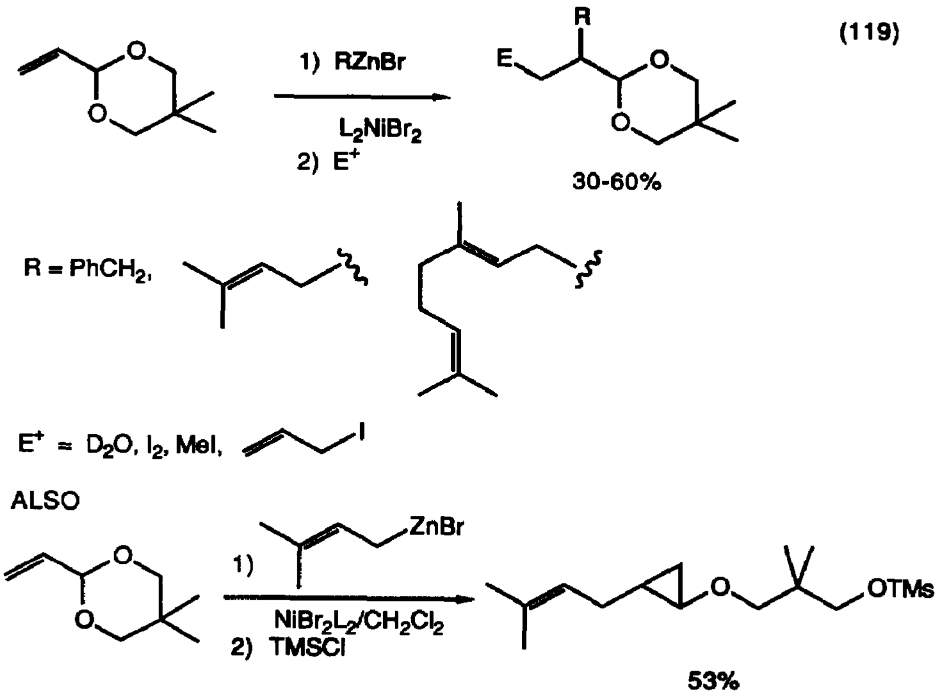
(117)



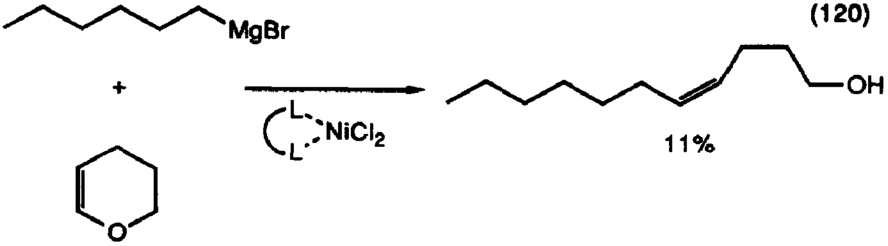
(118)



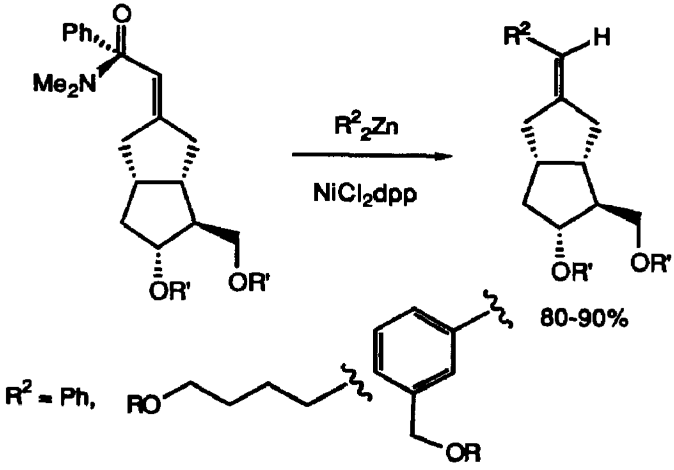
(119)


1) RZnBr
L2NiBr2
2) E+
E
R
30-60%
R = PhCH2,
E+ = D2O, I2, MeI,
ALSO
ZnBr
1)
NiBr2L2/CH2Cl2
2) TMSCl
OTMs
53%

(120)

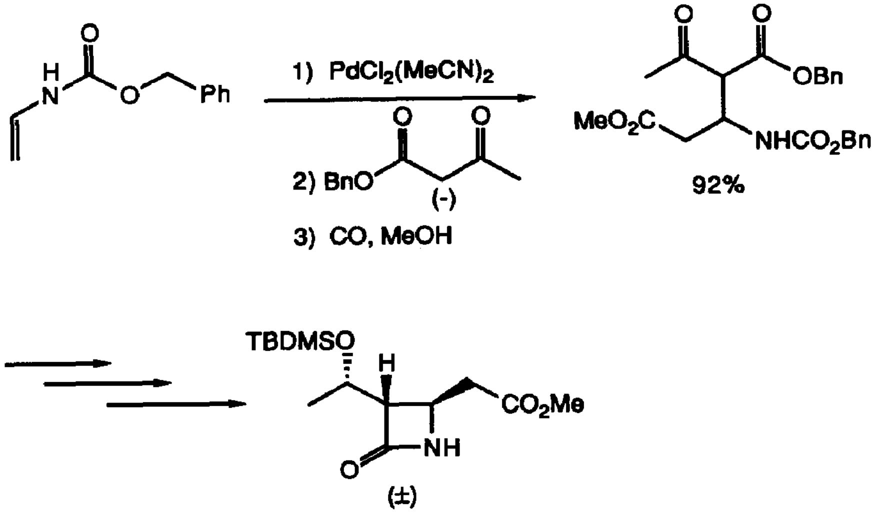

MgBr
+
NiCl2
L
L
OH
11%

(121)

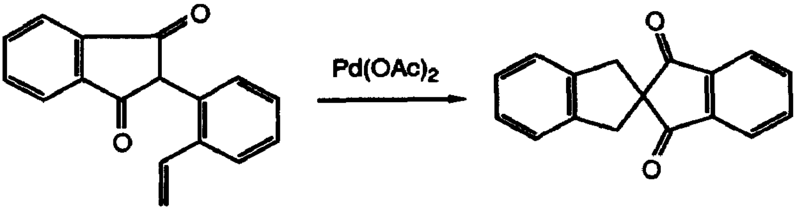

Ph
Me2N
R2
H
R22Zn
NiCl2dpp
OR'
OR'
80-90%
R2 = Ph,
RO
OR

A relay to (±)-thienamycin was synthesized using a palladium(II) catalyzed alkylation of an olefin (equation 122) [142], as was a relay to fredericamycin A (equation 123) [143]. Allyl oxetanes were ring opened by malonate in the presence of palladium(0) catalysts (equation 124) [144], as were cationic iron complexes of dihydrofurans (equation 125) [45]. Intramolecular coupling reactions between dieneiron tricarbonyl complexes and pendent olefins was the subject of a dissertation [146].

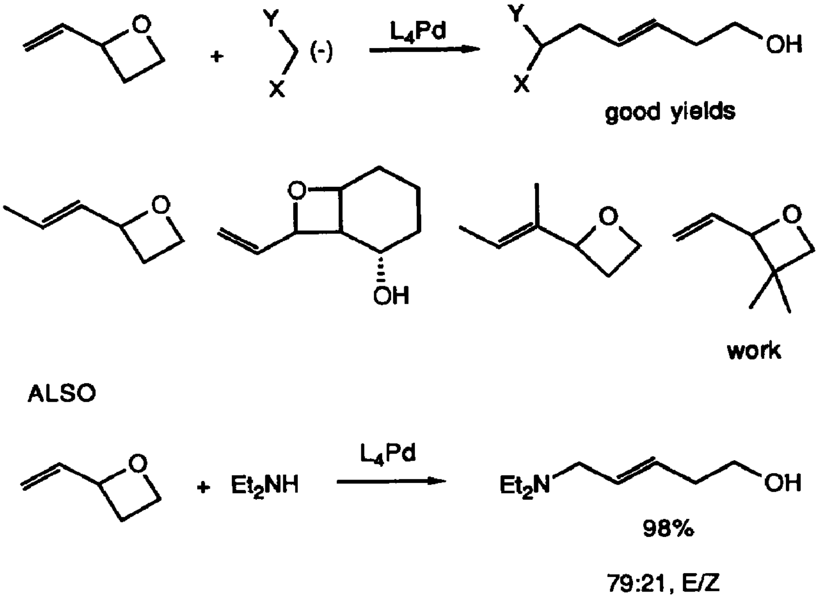
(122)



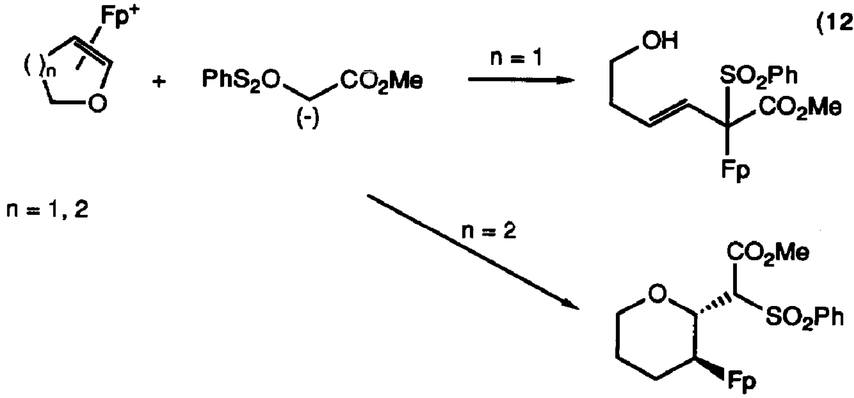
(123)



(124)

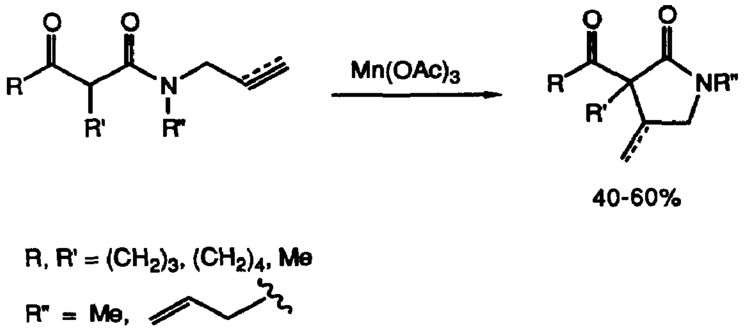


(125)

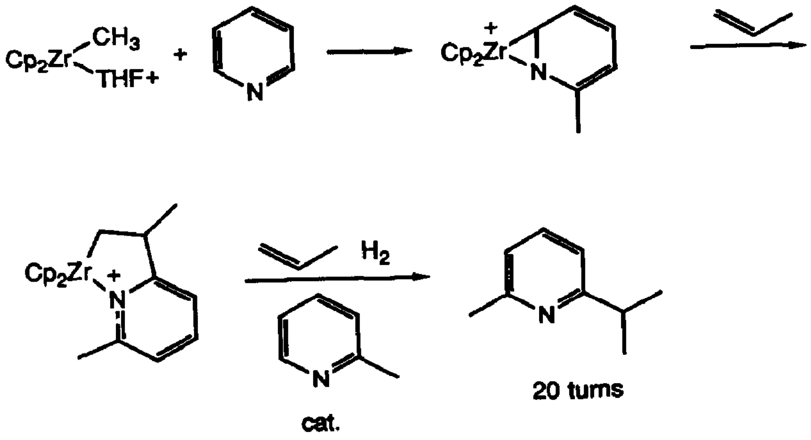


Manganese(III) acetate cyclized N-allyl acetoacetamides (equation 126) [147]. Zirconium complex chemistry was used to alkylate pyridines with olefins (equation 127) [148]. "Photochemically induced C-C bond formation in the coordination sphere of transition metals" was the title of a published lecture [149].

(126)



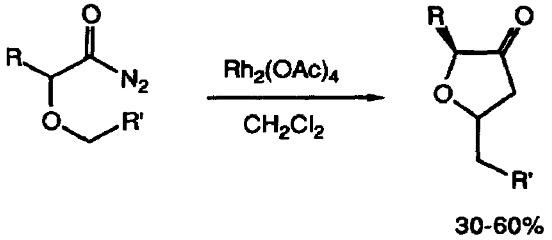
(127)



4. Decomposition of Diazoalkanes and Other Cyclopropanations

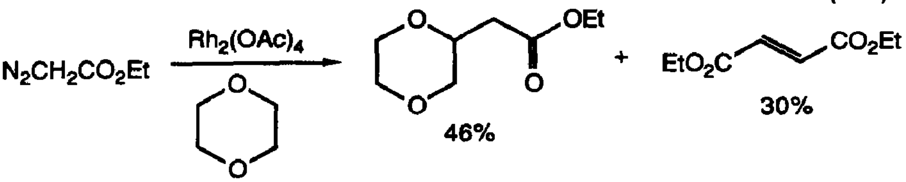
Metal catalyzed diazoalkane decomposition has been "discovered" by synthetic chemists and has been extensively exploited this year. Carbene insertions into C-H bonds α- to oxygen (equation 128) [150] [151], (equation 129) [152], nitrogen (equation 130) [153], carbonyl groups (equation 131) [154], aryl CH bonds (equation 132) [155], and aliphatic C-H bonds (equation 133) [156], (equation 134) [157] have all been reported.

(128)

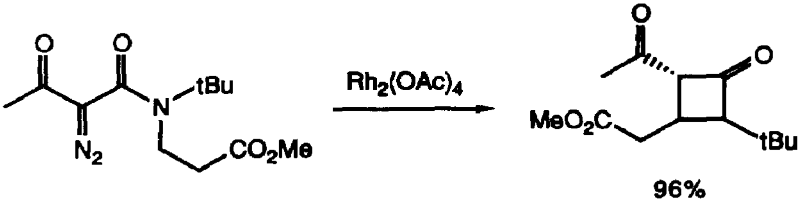


R = Me, nPr, iBu, Me$_2$
R' = Ph, Me, Me$_2$, CH$_2$OBn

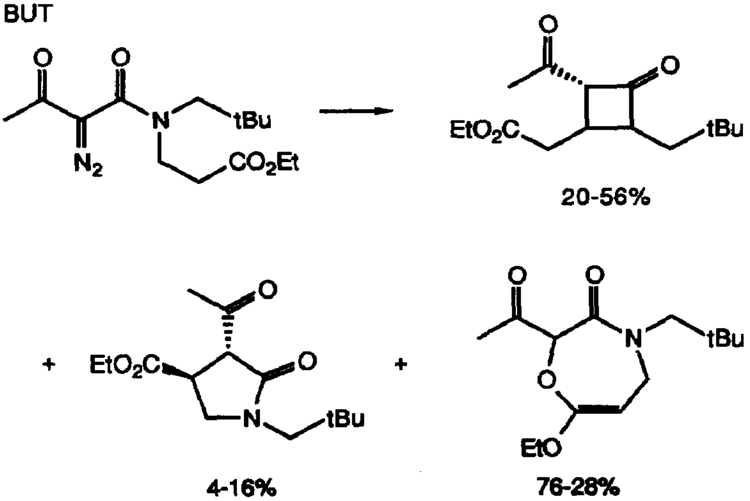
(129)



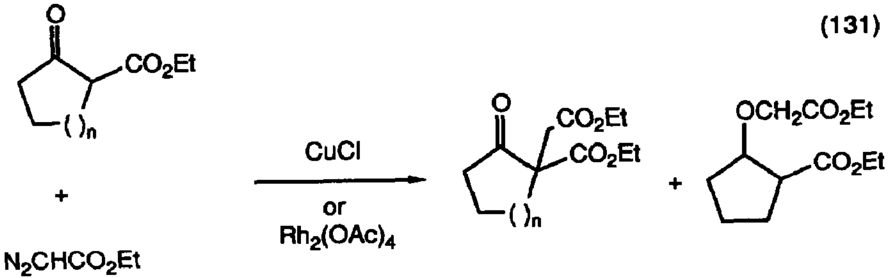
(130)



BUT

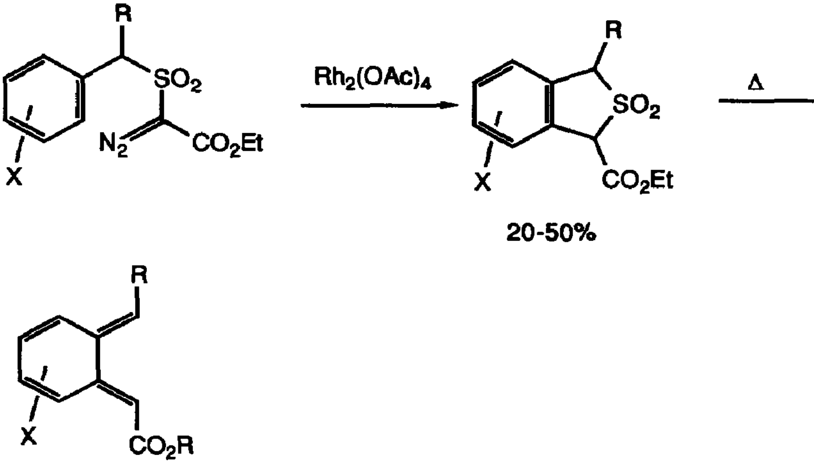


(131)



distribution depends on catalysts

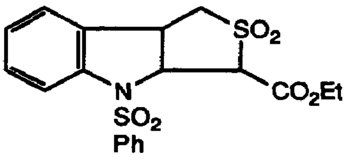
(132)



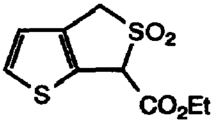
20-50%

X = H, 2-Br, 2-Me, 2,5-(MeO)$_2$, 3,4- O⌒O, 4-MeO, 3-MeO
R = H, Ph

ALSO

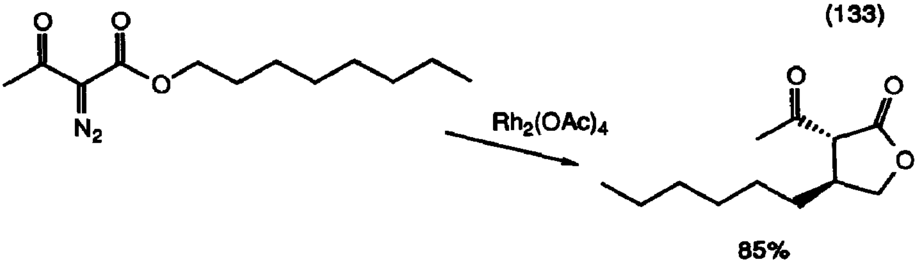


24%

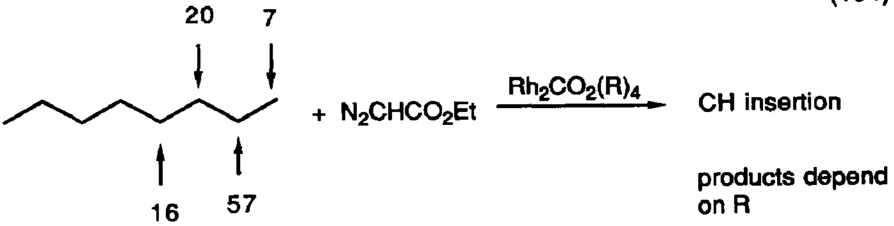


50%

(133)



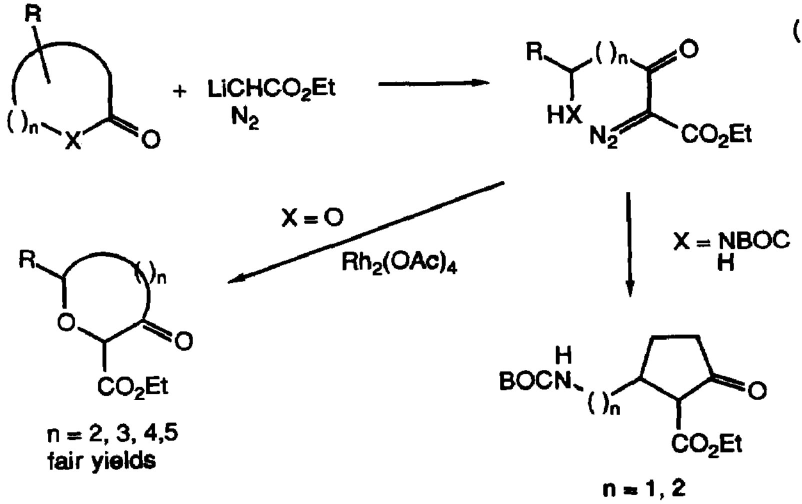
(134)



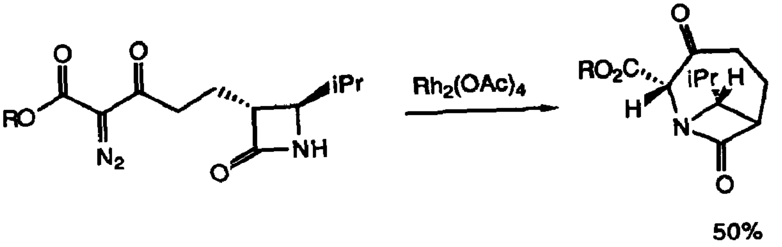
for R = H but 19:53:14:14 for R = 9-triptycenyl

Metals also catalyzed the insertion of diazoalkanes into OH bonds (equation 135) [158], and amidic NH bonds (equation 136) [159]. More complex insertions (equation 137) [160], (equation 138) [161], (equation 139) [162] have also been observed.

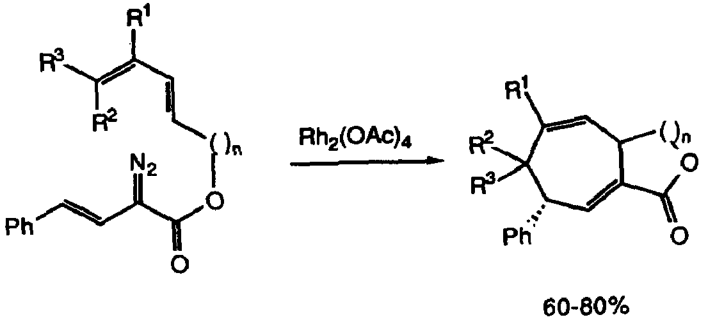
(135)



(136)

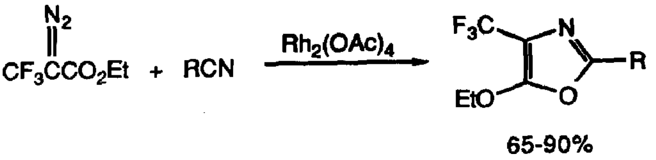


(137)



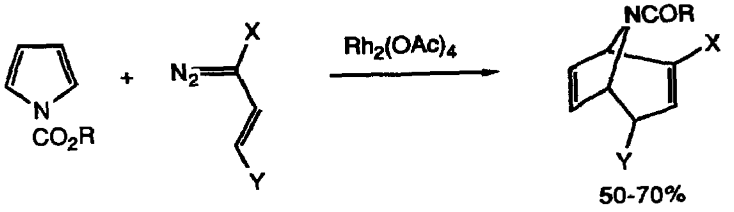
n = 1, 2; R's = H, Me

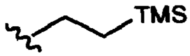
(138)



R = Ph, pClPh, Bn, 2-py, Me, Et$_2$OCCH$_2$-, 

(139)

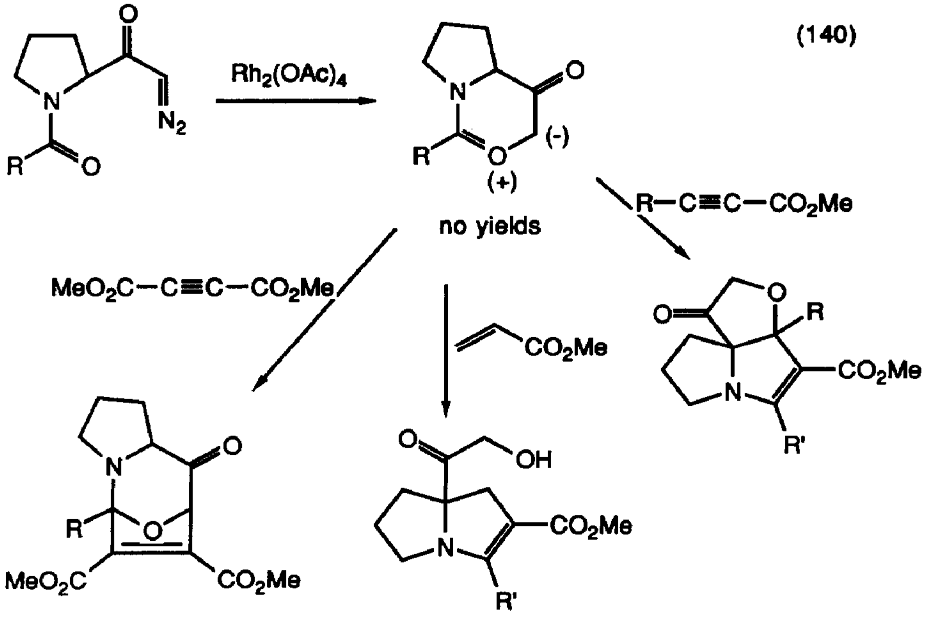


R = Me, Et, 

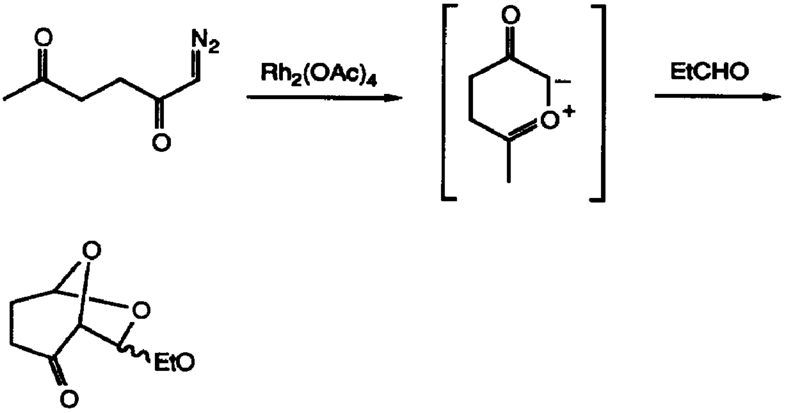
X = CO$_2$Me; Y = CO$_2$Et, SO$_2$Ph, Ph

Transition metals also catalyzed the decomposition of diazo compounds to add to carbonyl oxygens to give oxonium ylids which underwent a bewildering array of rearrangements and additions to acceptors which, none the less were synthetically useful (equation 140) [163][164], (equation 141) [165], (equation 142) [166], (equation 143) [167][168], (equation 144) [169], (equation 145) [170], (equation 146) [171], (equation 147) [172], (equation 148) [173].

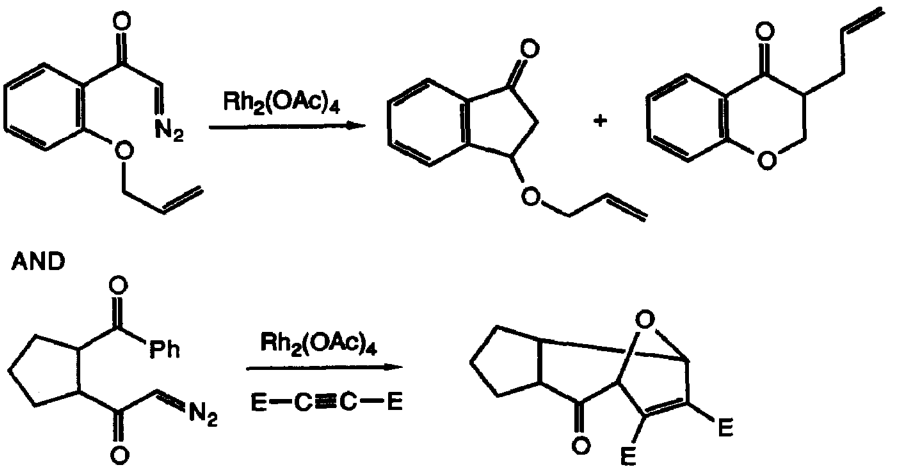
(140)



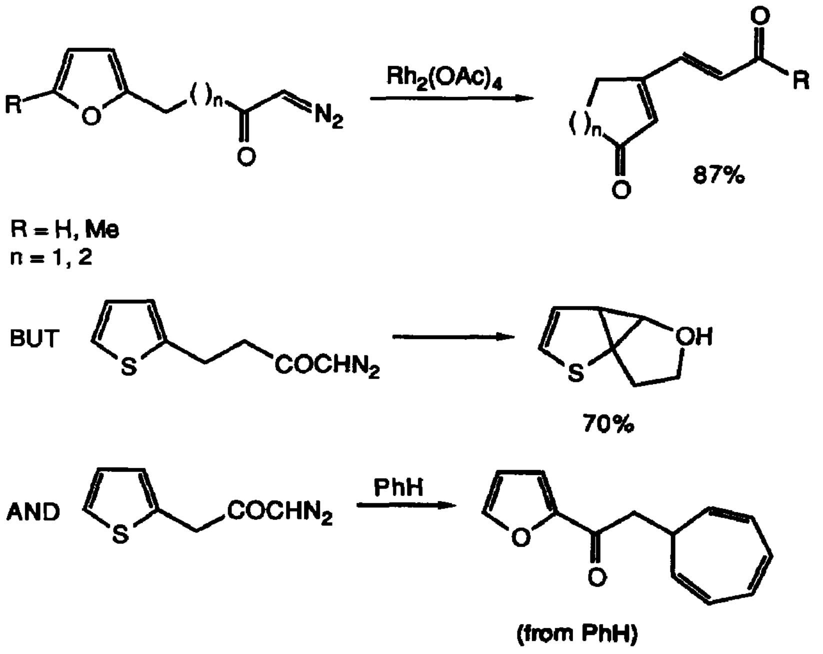
(141)



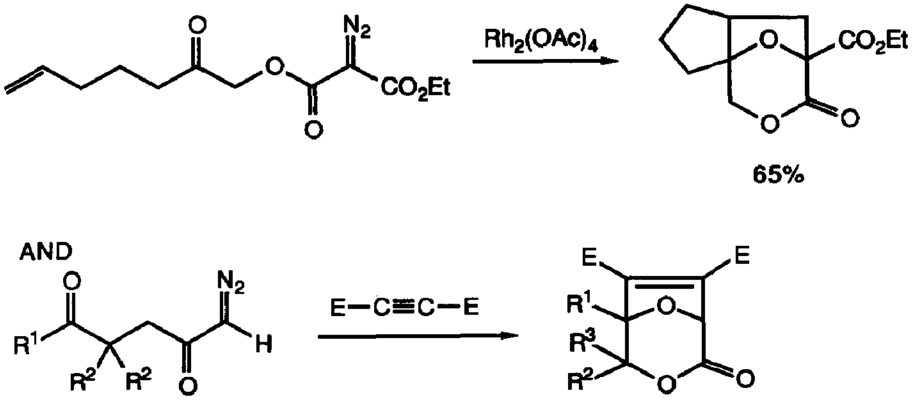
(142)


Rh$_2$(OAc)$_4$
+
AND
Ph
N$_2$
Rh$_2$(OAc)$_4$
E—C≡C—E
E
E

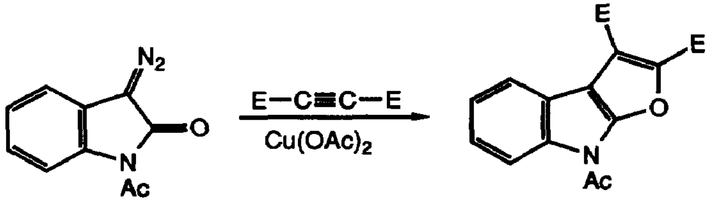
(143)


R
N$_2$
Rh$_2$(OAc)$_4$
( )$_n$
R
87%
R = H, Me
n = 1, 2
BUT
COCHN$_2$
OH
70%
AND
COCHN$_2$
PhH
(from PhH)

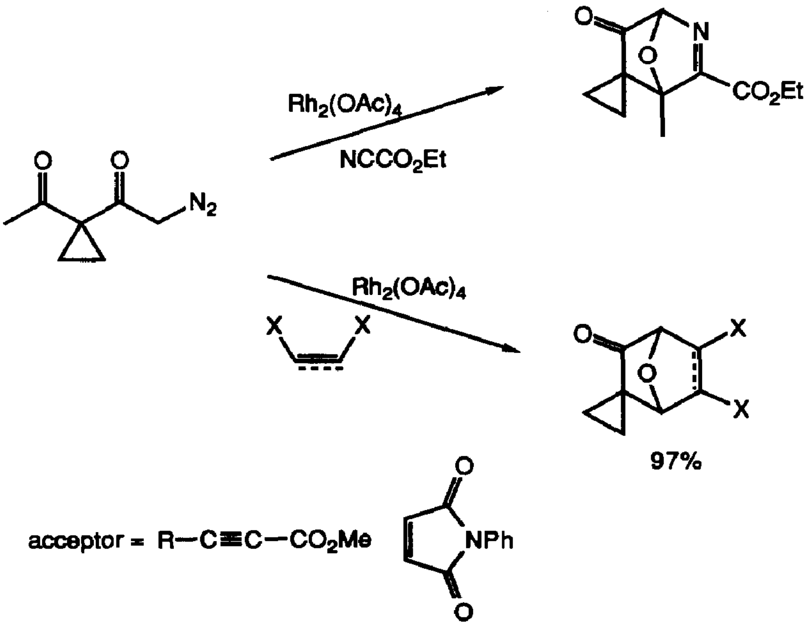
(144)

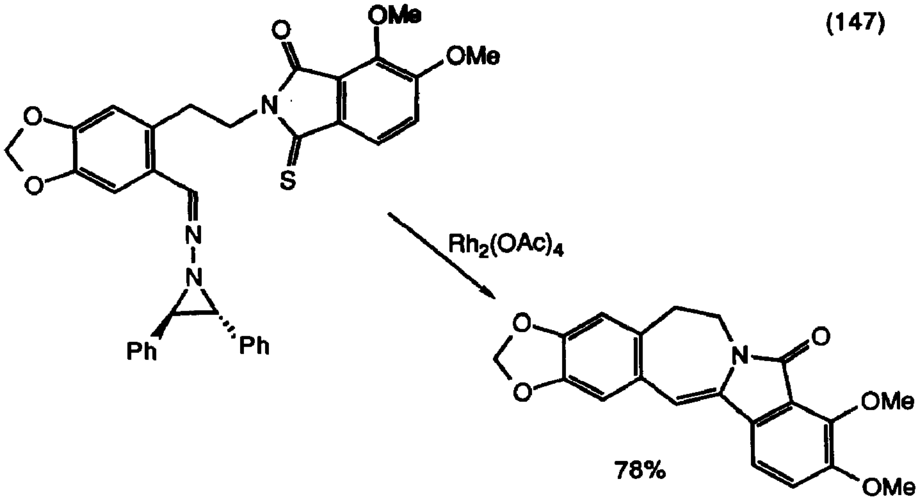

N2
CO2Et
Rh2(OAc)4
CO2Et
65%
AND
N2
R1
R2
R2
H
E—C≡C—E
E
E
R1
R3
R2

(145)

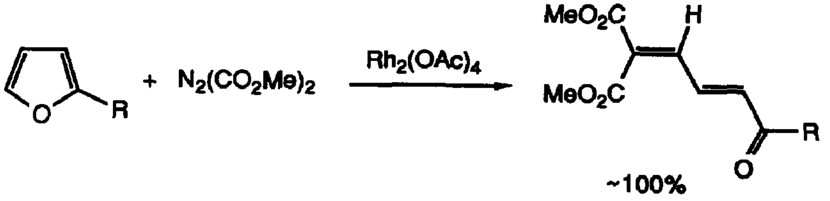

N2
N
Ac
E—C≡C—E
Cu(OAc)2
E
E
N
Ac

(146)


Rh2(OAc)4
NCCO2Et
N
CO2Et
N2
Rh2(OAc)4
X
X
X
X
97%
acceptor = R—C≡C—CO2Me
NPh

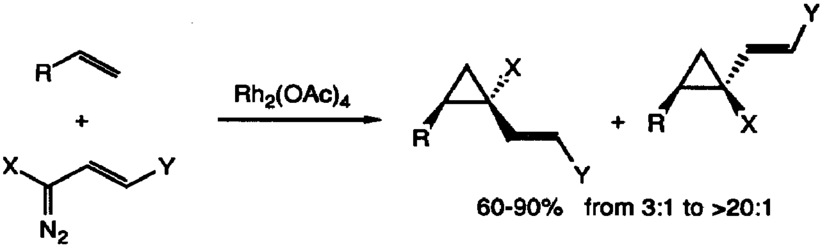


(147)



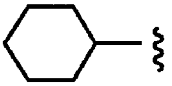
(148)

Rhodium(II) acetate and copper acetate also catalyzed the cyclopropanation of olefins by diazo compounds (equation 149) [174], (equation 150) [175], (equation 151) [176], (equation 152) [177], (equation 153) [178], (equation 154) [179][180].



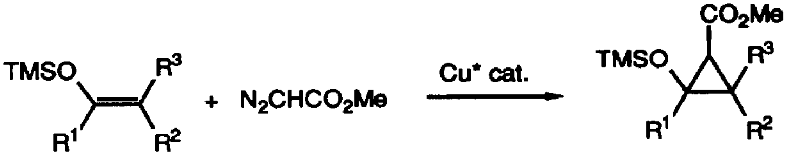
(149)

60-90% from 3:1 to >20:1

R = Ph, pMeOPh, AcO, BuO, tBu, nBn, $ClCH_2$, $BrCH_2$, $EtOCH_2$,


X = CO_2Et

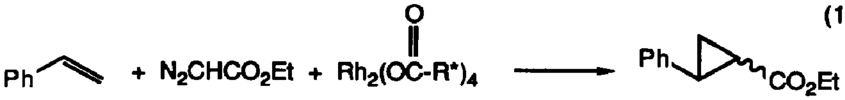
Y = CO_2Et, Ph,

(150)

40-70%
30-40% ee

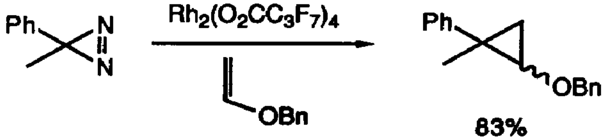
R^1 = H, Ph
R^2 = H
R^3 = H, iPr
$-(CH_2)_3-$



(151)

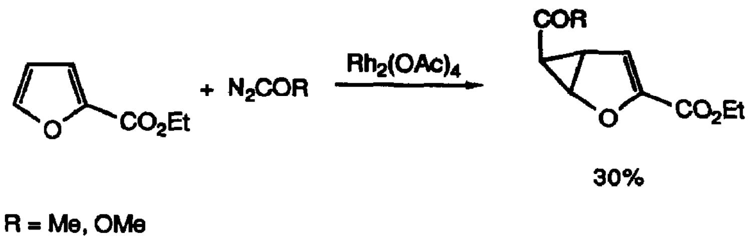
good yields
maximum 12% ee

R* = 13 different chiral acids

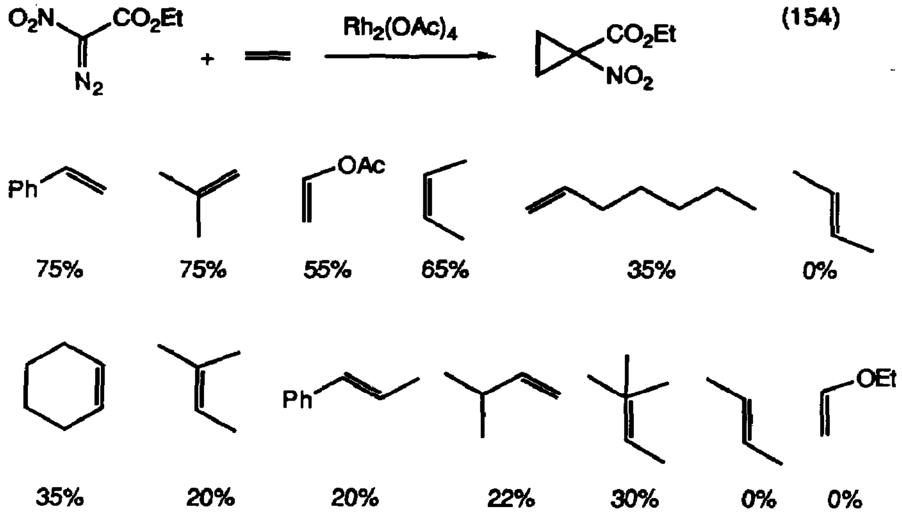


(152)

83%



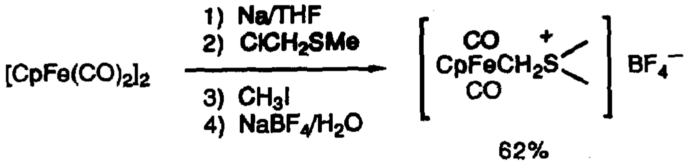
(153)



(154)

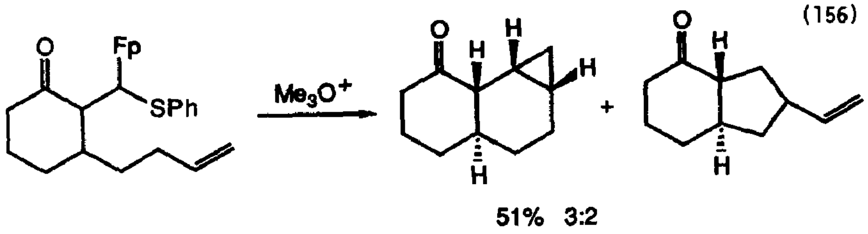
A large scale preparation for the FpCH$_2$ cyclopropanating agent precursor has been developed (equation 155) [181]. Using similar iron cyclopropanating agents intramolecularly resulted in some CH insertion products (equation 156) [182]. Optically active iron carbene species cyclopropanated olefins with good enantiomeric excess (equation 157) [183], (equation 158) [184]. The cis/trans ratios of cyclopropanes from iron phenyl carbenes was determined (equation 159) [185]. Titanium(IV) greatly accelerated the cyclopropanation of olefins by dihalomethanes and zinc/copper couple (equation 160) [186]. Allylmanganese derivatives reacted with γ-bromocrotonates to give cyclopropanes (equation 161) [187].

(155)



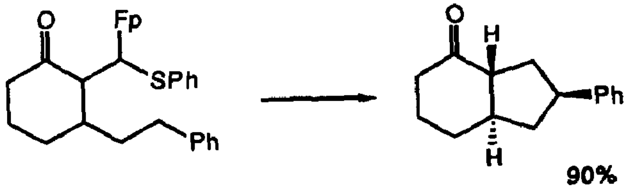
Also made [CP*Fe(CO)$_2$CH$_2\overset{+}{S}$Me$_2$] BF$_4^-$ in 92% yield.

(156)

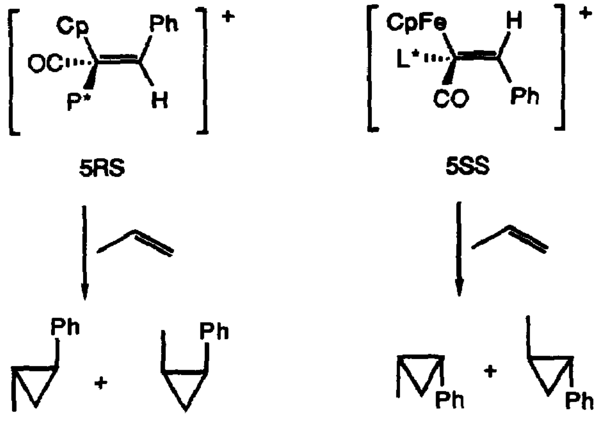


51% 3:2

BUT



(157)

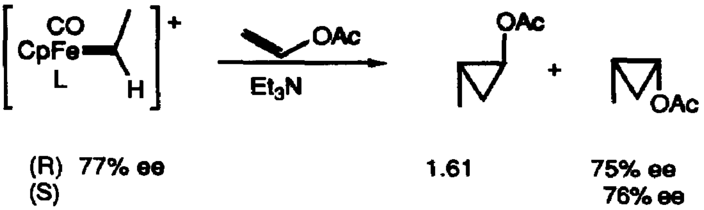


5RS

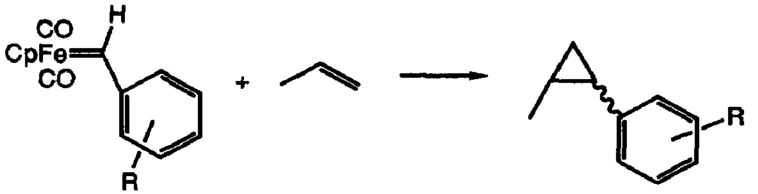
5SS

1S2R 3:2 1R2S
67% o.p. 43% o.p.

2:3
53% o.p. 76% o.p.

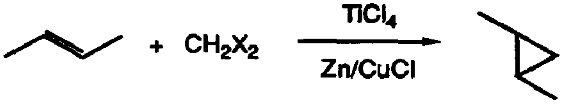


(158)

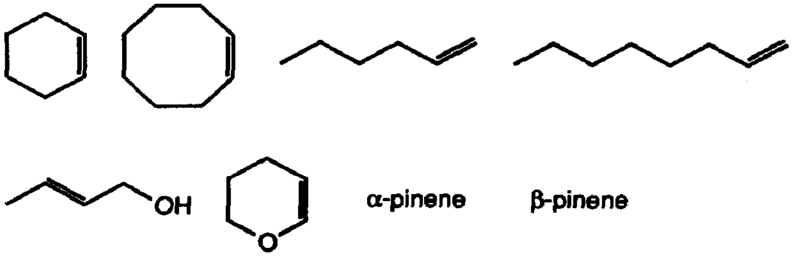


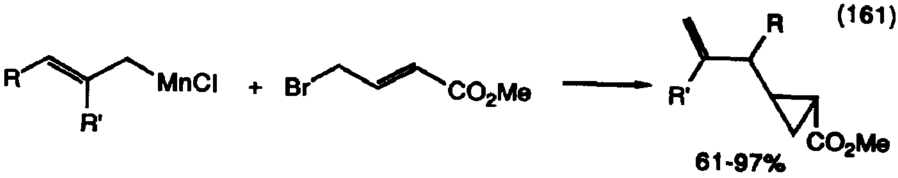
(159)

R =		
H	90%	7.8 c/t
pF	80	9.4
pMe	60	9.0
pMeO	85	2.0
mMeO	81	9.6
pCF$_3$	67	7.3



(160)

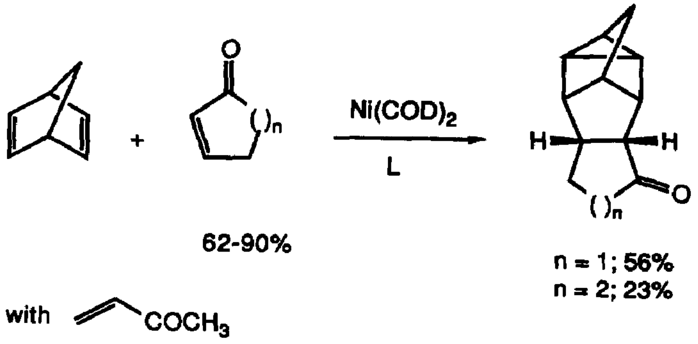




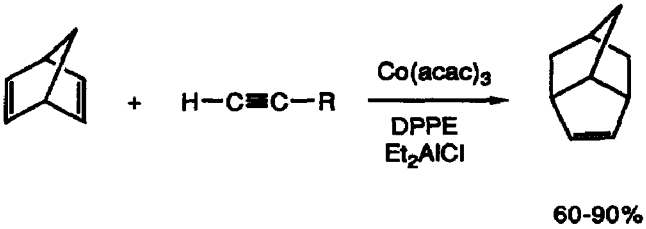
(161)

5. Cycloaddition Reactions

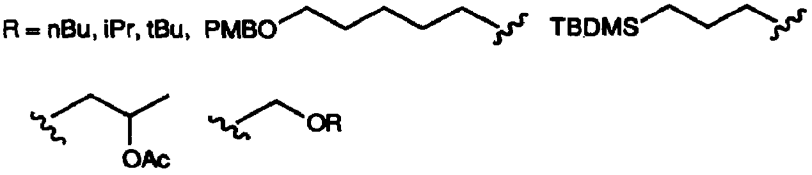
Nickel(0) (equation 162) [188] and cobalt(0) (equation 163) [189] catalyzed homo Diels Alder cycloadditions. Iron carbonyl effected the complex cycloaddition shown in equation 164 [190].

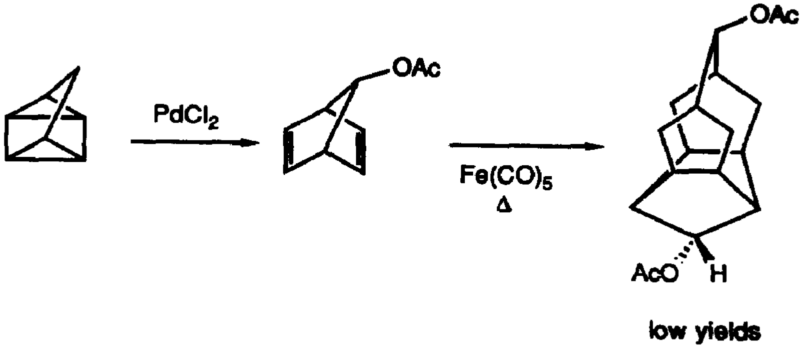


(162)



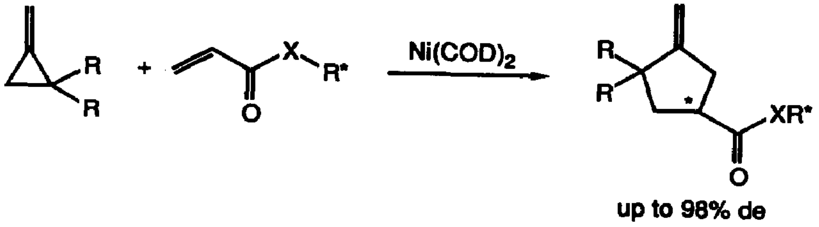
(163)



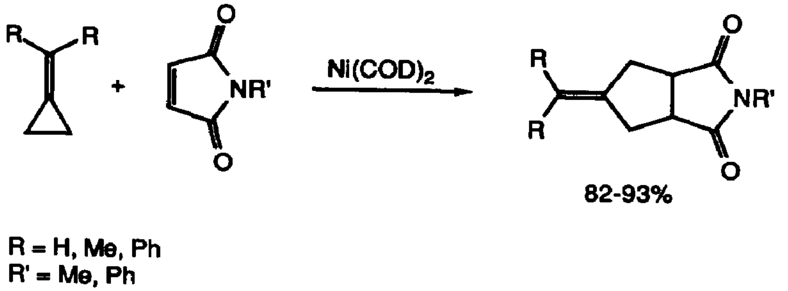


(164)

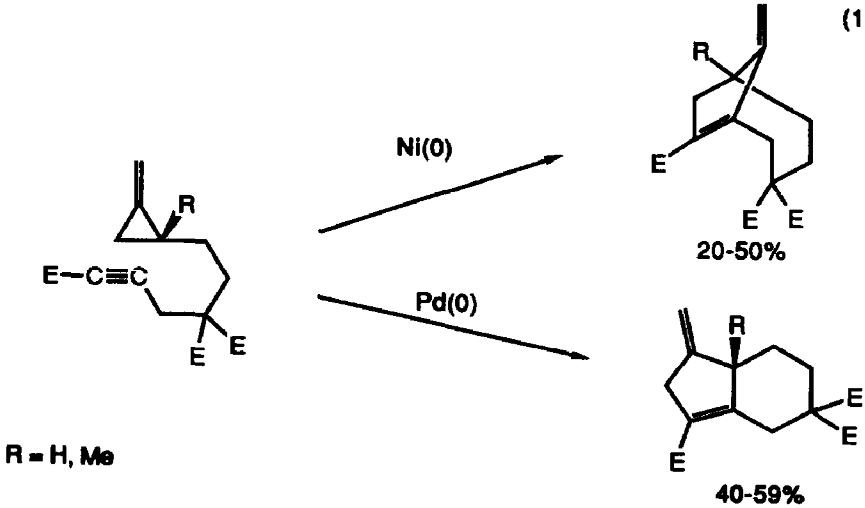
Nickel(0) (equation 165) [191], (equation 166) [192], and palladium(0) (equation 167) [193] catalyzed the cycloaddition of methylenecyclopropanes to alkenes and alkynes.



(165)



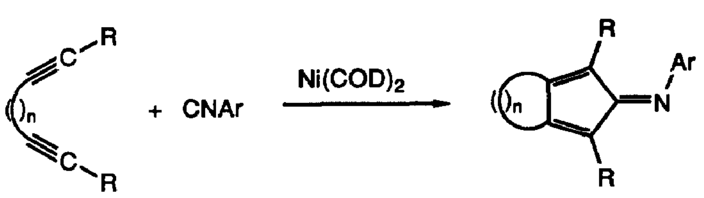
(166)



(167)

Nickel(0) complexes catalyzed the cycloaddition of diynes to isonitriles (equation 168) [194], while cobalt catalyzed the cycloaddition of diynes to ketones (equation 169) [195]. Nickel(0) complexes catalyzed the intramolecular 4+2 cycloaddition of dienes to alkynes (equation 170) [196]. Nickel-catalyzed intramolecular [4+4] cycloaddition methodology: asymmetric total synthesis of (+)-asteriscanolide was the subject of a dissertation [197].

(168)

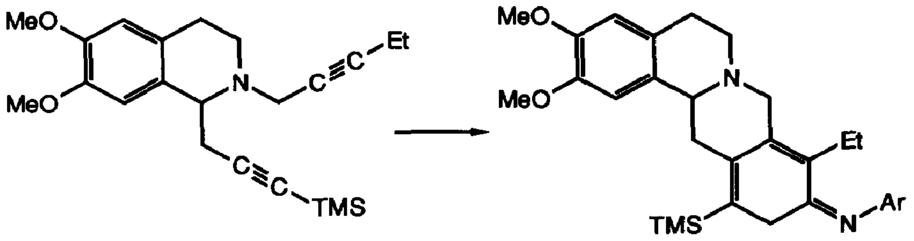


60-80%

R = Ph, TMS, Et

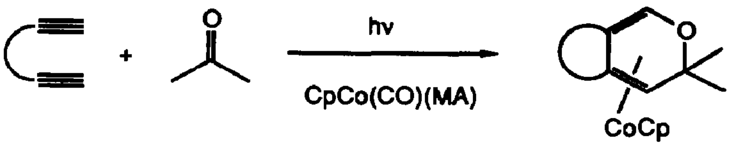
( )$_n$ = $(CH_2)_3$, $(CH_2)_4$, $(CH_2)_5$, CH_2OCH_2

ALSO



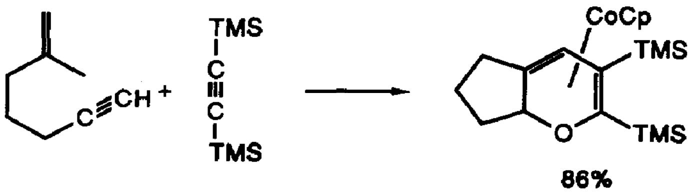
56%

(169)



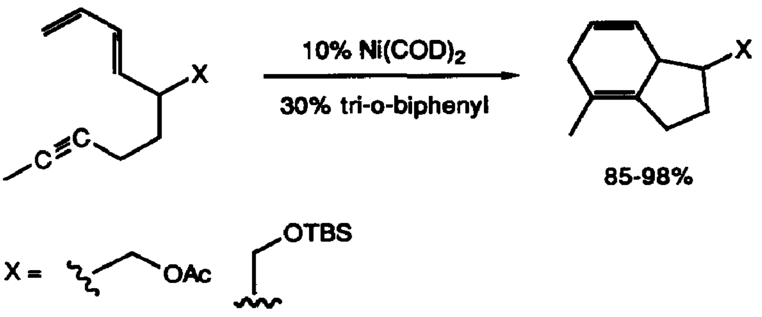
~40%

AND



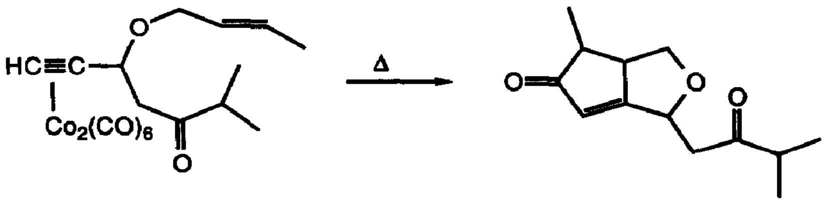
86%

(170)

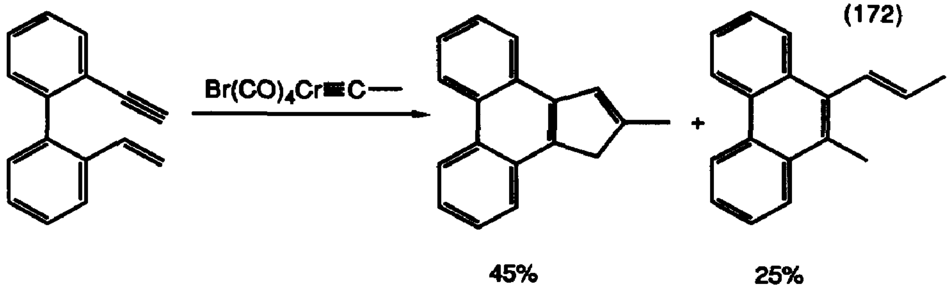


Cobalt alkyne complexes underwent 2+2+1 cycloaddition (equation 171) [198]. Chromium carbyne (equation 172) and chromium carbene complexes (equation 173) effected enyne cycloadditions [199]. Cp_2TiCl_2 catalyzed the cycloaddition of epoxides to acrylates (equation 174) [200], while manganese aryl complexes cycloadded to alkynes (equation 175) [201]. Allyl stannanes underwent reaction with α,β-unsaturated acyliron complexes to give cyclopentane (equation 176) [202].

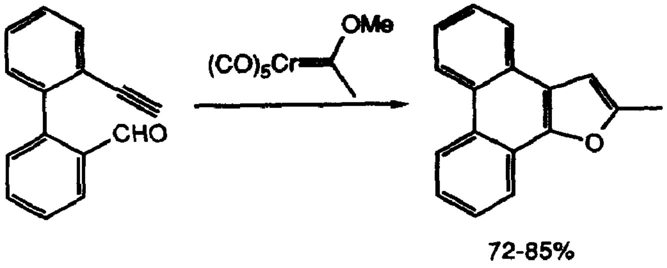
(171)



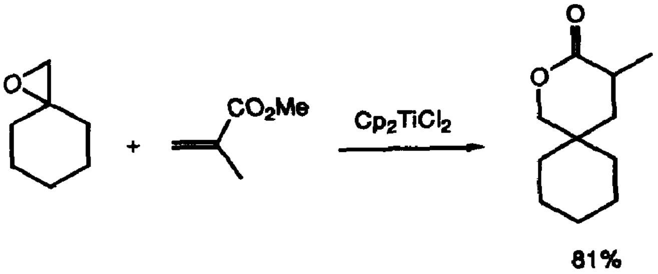
(172)



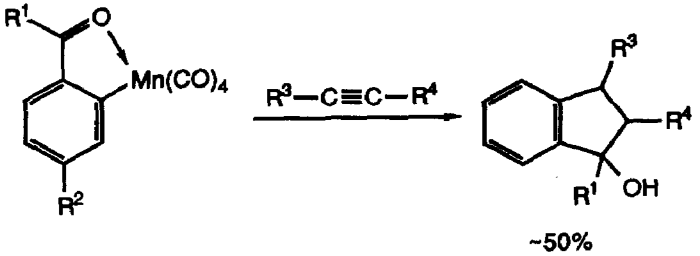
(173)



(174)

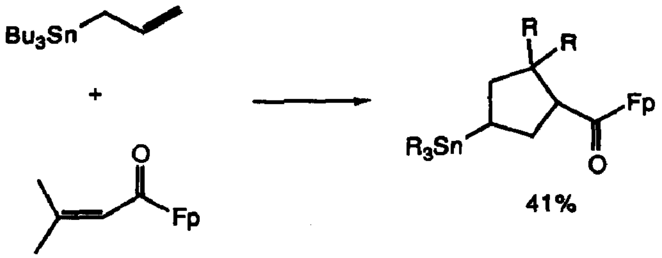


(175)



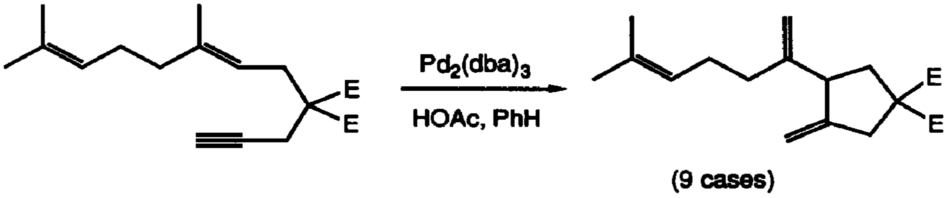
R^1 = H, Me; R^2 = H, NMe_2
R^3 = H, Ph, TMS; R^4 = H, Ph, TMS

(176)

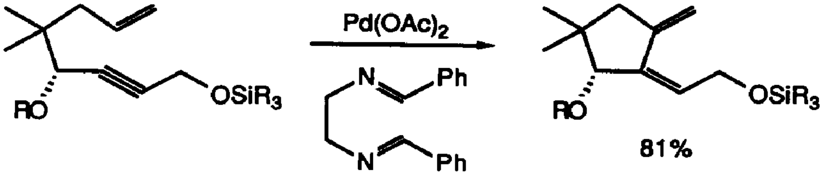


Palladium catalyzed cyclizations of enynes and diynes was the topic of a dissertation [203]. This process was useful for the synthesis of highly functionalized cyclopentyl systems (equation 177) [204], (equation 178) [205], (equation 179) [206]. The resulting dienes were intramolecularly trapped in Diels-Alder reactions to give polycyclic systems (equation 180) [207]. α,ε-Diynes cyclized under palladium catalysis (equation 181) [208]. Cyclopentyl systems were also prepared via π-allylpalladium intermediates (equation 182) [209], (equation 183) [210].

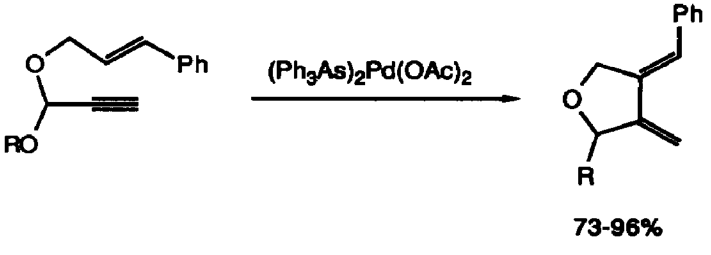
(177)

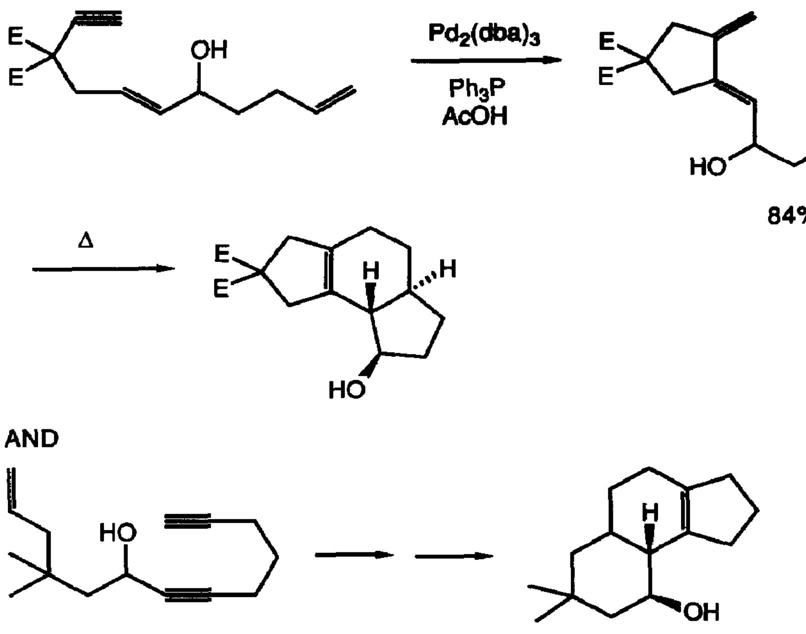


(178)

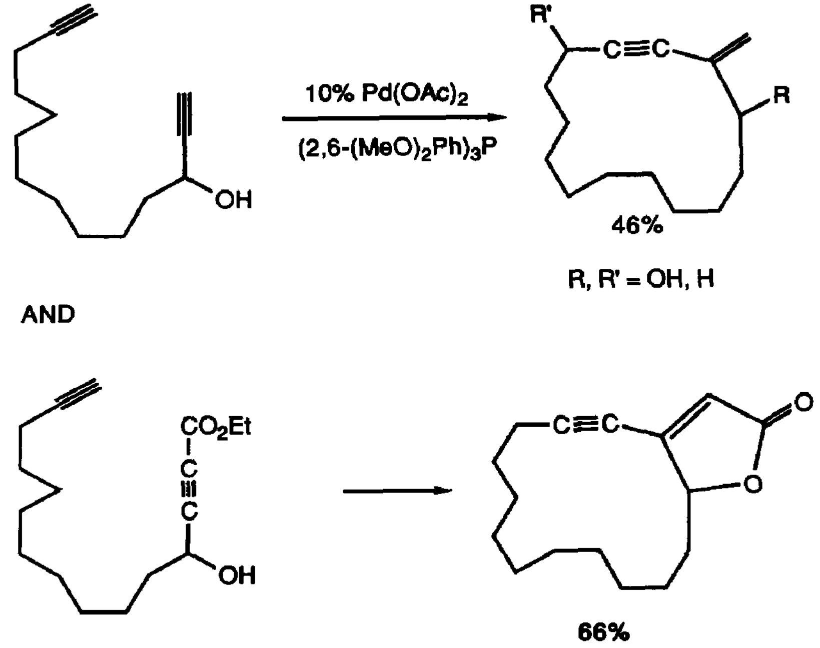


(179)



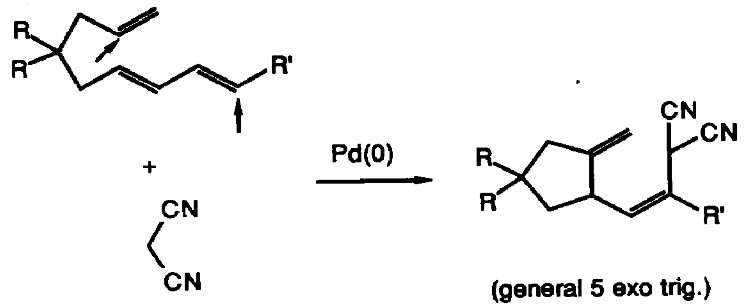

E
E
OH
Pd2(dba)3
Ph3P
AcOH
HO
84%
Δ
H
H
HO
AND
HO
H
OH

(180)

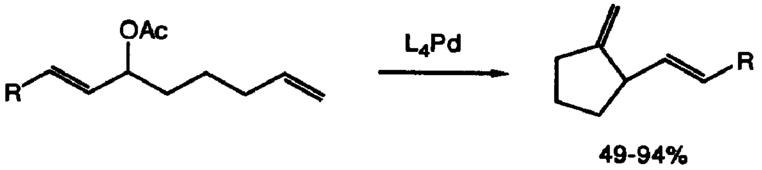

OH
10% Pd(OAc)2
(2,6-(MeO)2Ph)3P
R'
C≡C
R
46%
R, R' = OH, H
AND
CO2Et
C
C
OH
C≡C
O
O
66%

(181)

(182)

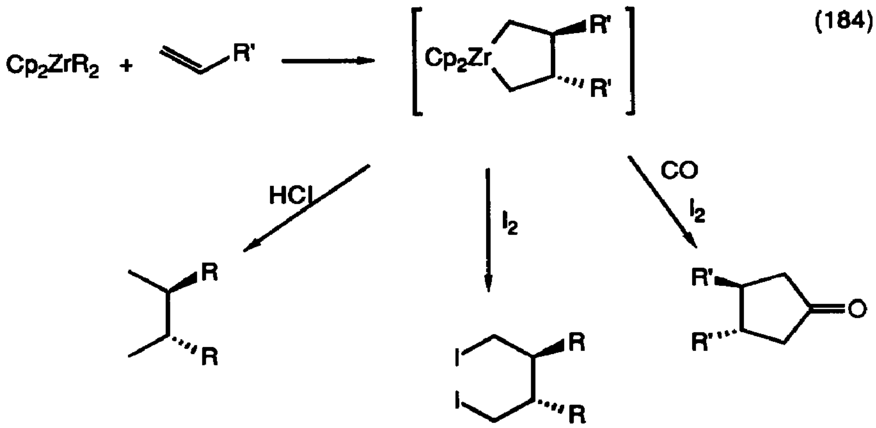


(183)

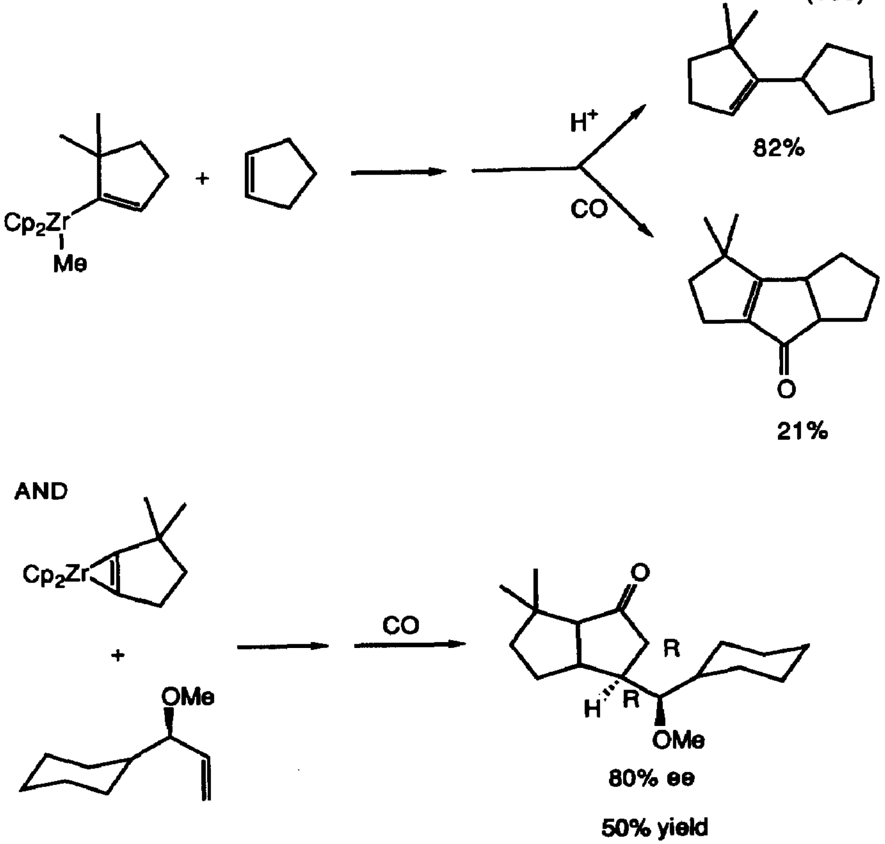


Cyclopentadienylzirconium complexes cyclodimerized alkenes (equation 184) [211], (equation 185) [212], alkynes (equation 186) [213], as well as enynes (equation 187) [214], (equation 188) [215], and alkyne-hydrazones (equation 189) [216] to give zirconacyclopentyl systems which were further homologated.

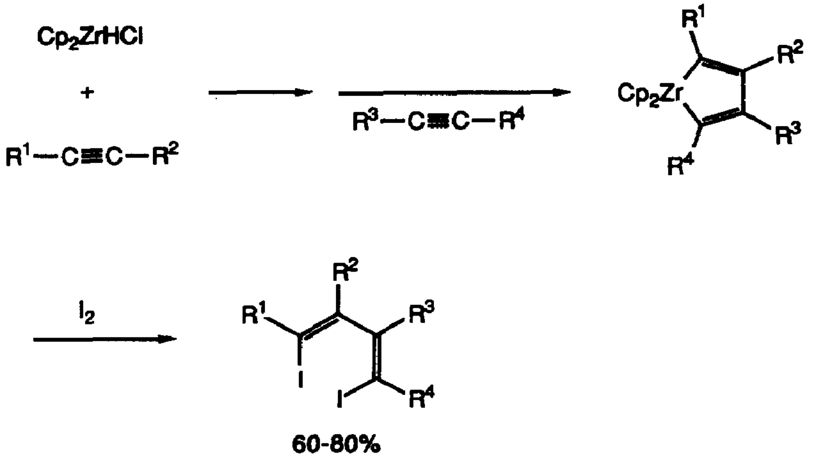
(184)



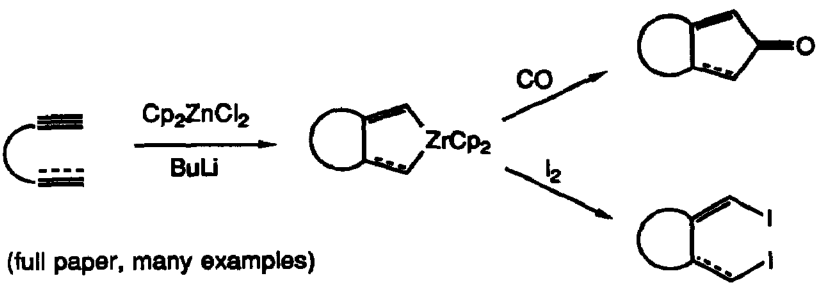
(185)


Cp₂Zr
Me
+
H⁺
CO
82%
21%
AND
Cp₂Zr
+
OMe
CO
O
R
H
R
OMe
80% ee
50% yield

(186)

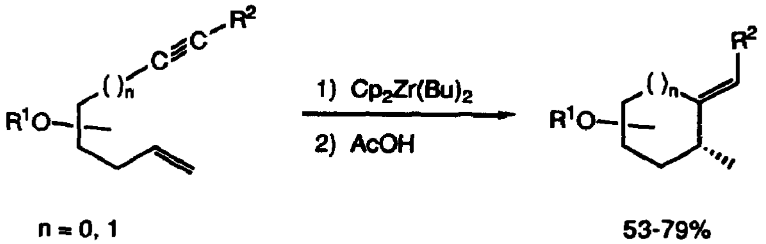

Cp₂ZrHCl
+
R¹—C≡C—R²
R³—C≡C—R⁴
R¹
R²
Cp₂Zr
R³
R⁴
I₂
R²
R¹
R³
I
I
R⁴
60-80%

(187)



(full paper, many examples)

(188)

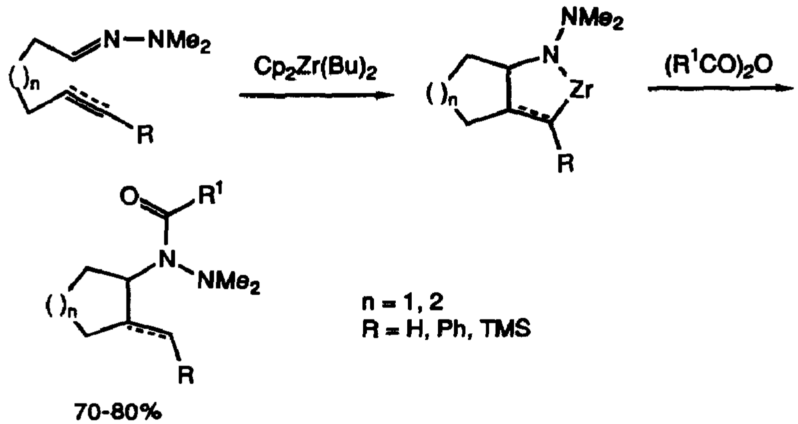


n = 0, 1

53-79%

R^1 = TBDMS, H; R^2 = TMS, SMe

(189)

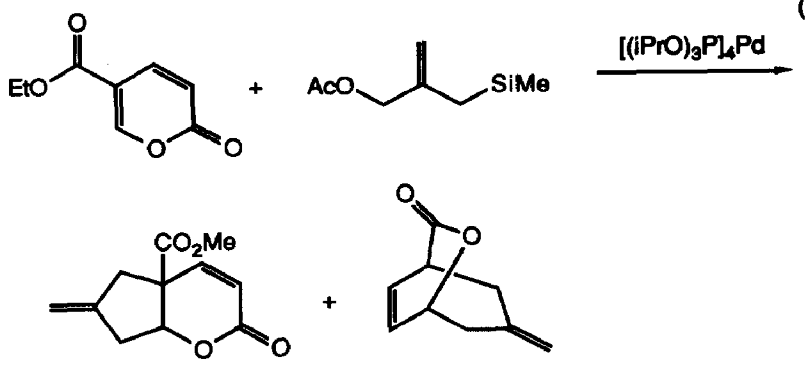


n = 1, 2
R = H, Ph, TMS

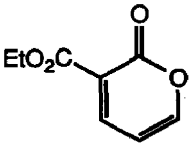
70-80%

Transition metal templates as guides for cycloadditions was the subject of a review (36 references) [217]. The stereochemical aspects of the trimethylenemethane palladium catalyzed cycloaddition to olefins was the topic of a dissertation [218]. A wide range of acceptor molecules including pyrones (equation 190) [219] vinyl sulfones (equation 191) [220], methylenemalonates (equation 192) [221], and functionalized cyclohexenones (equation 193) [222] underwent this reaction. Aldehydes also added (equation 194) [222]. Use of chiral vinyl sulfoxides (equation 195) [223] or chiral ligands (equation 196) led to reasonable asymmetric induction [224]. This chemistry was used to construct the five-membered rings in an attempted steroid synthesis (equation 197) [225]. With strained olefins, the trimethylenemethane precursor underwent a cyclopropanation reaction (equation 198) [226].

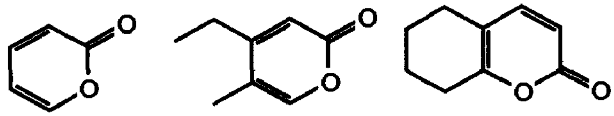
(190)



87% 1:1.2

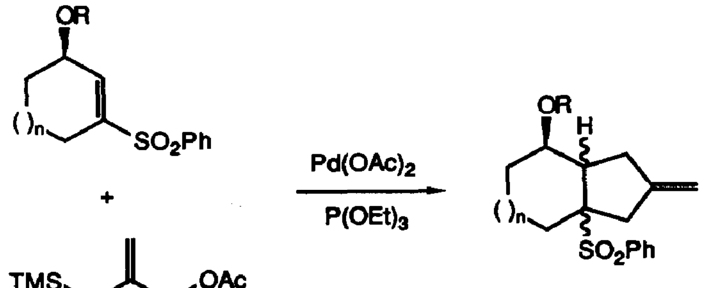


4+2 71%



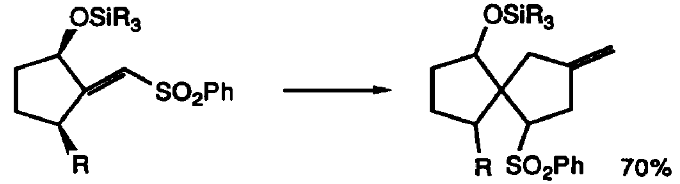
all gave 4+3

(191)

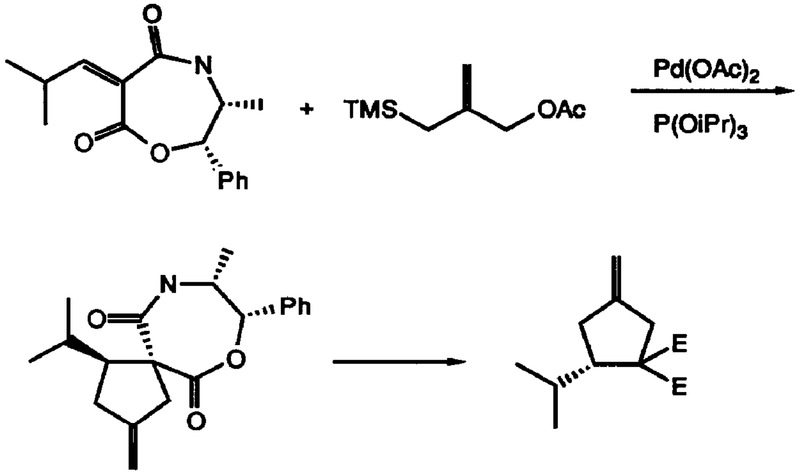


40-70% n = 1, 2

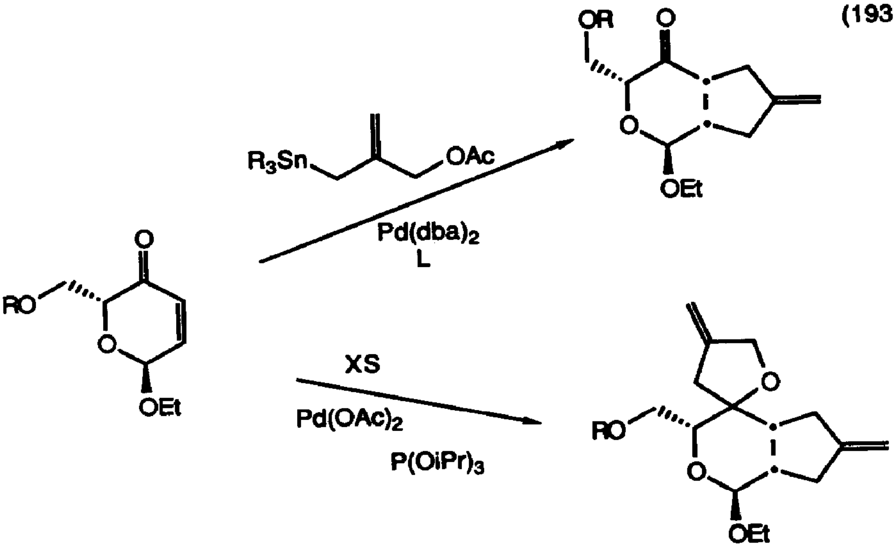
AND


OSiR3
SO2Ph
R
OSiR3
R SO2Ph
70%

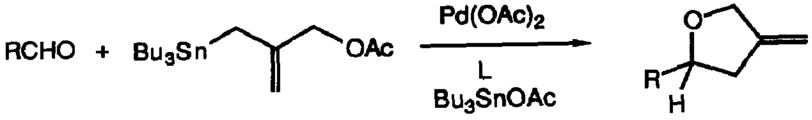
(192)


O
N
O
O
Ph
+
TMS
OAc
Pd(OAc)2
P(OiPr)3
N
Ph
O
O
O
E
E

(193)

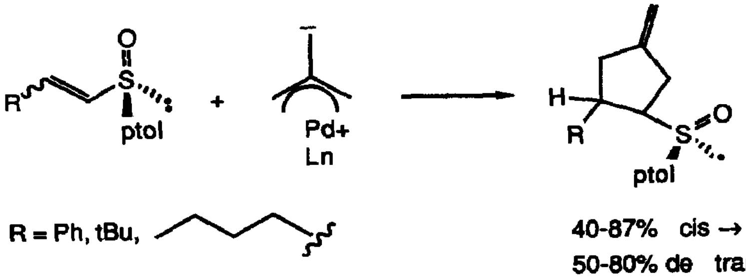

OR
O
O
OEt
R3Sn
OAc
Pd(dba)2
L
O
RO
O
OEt
XS
Pd(OAc)2
P(OiPr)3
O
RO
O
OEt

(194)



good yields

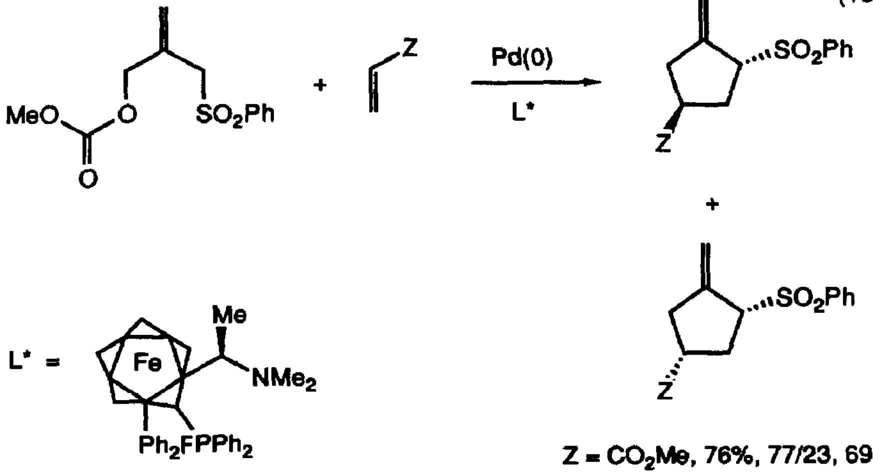
(195)



40-87% cis → cis

50-80% de trans → trans

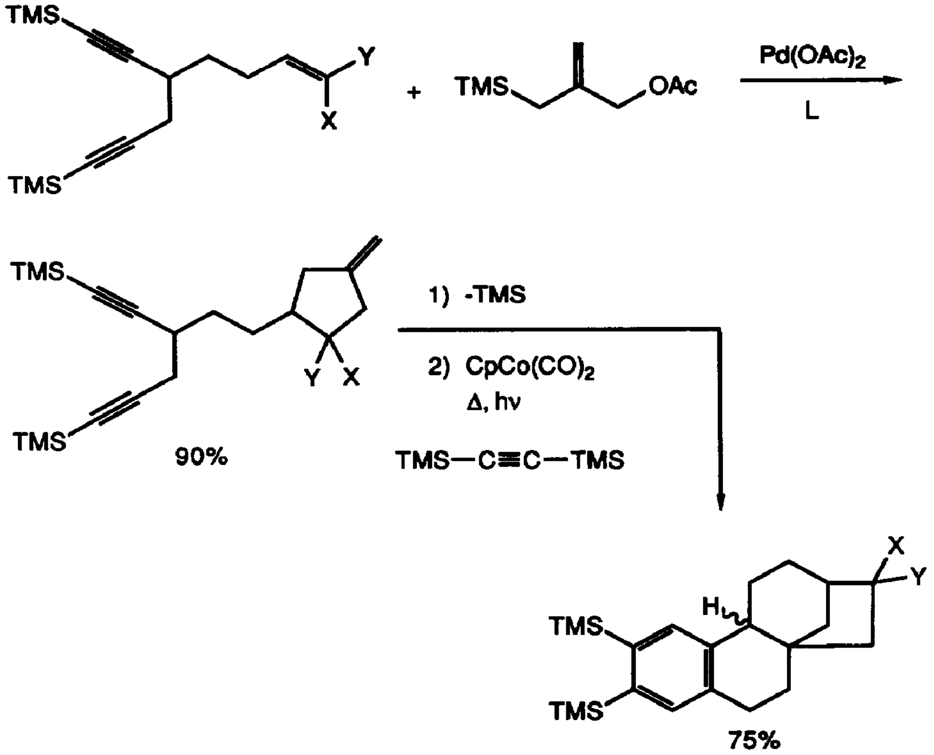
(196)



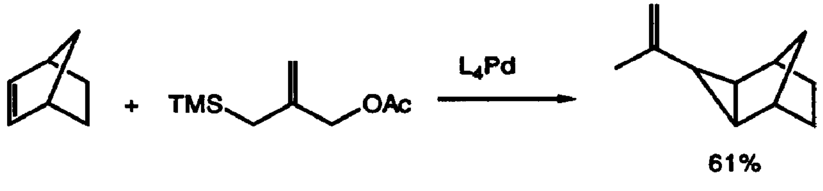
Z = CO_2Me, 76%, 77/23, 69%33

Z = COMe, 91%

(197)



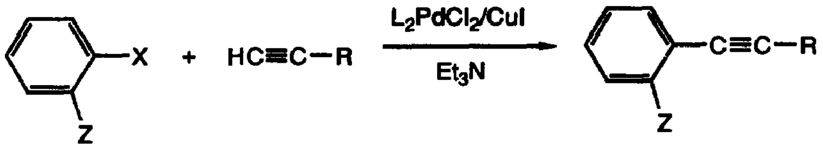
(198)



6. Alkylation of Alkynes

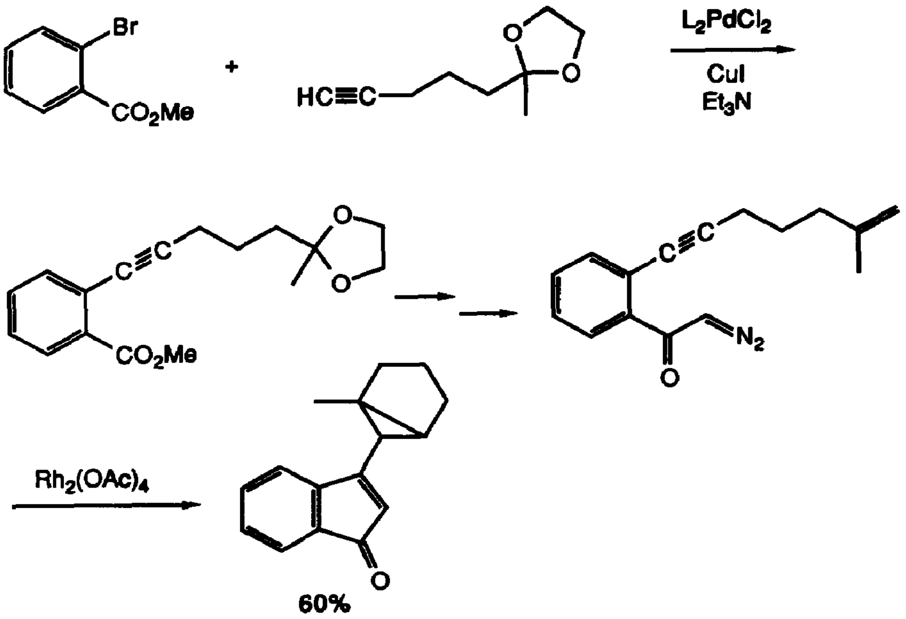
The palladium(II)/copper(I) catalyzed arylation of alkynes by aryl halides (equation 199) [227] has been used extensively to synthesize a variety of natural products (equation 200) [228], (equation 201) [229], (equation 202) [230]. Heterocyclic aryl iodides also were efficiently alkynylated by this catalyst system (equation 203) [231], (equation 204) [232]. Alkynes were acylated by imidoyl chlorides by the same catalyst system (equation 205) [233]. Alkynes were vinylated by vinyl halides using the same catalyst (equation 206) [234].

(199)

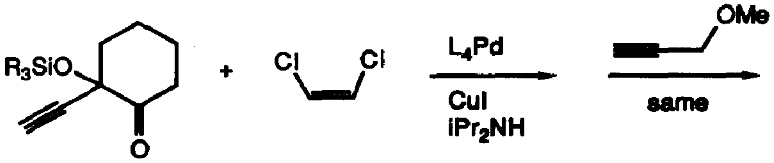


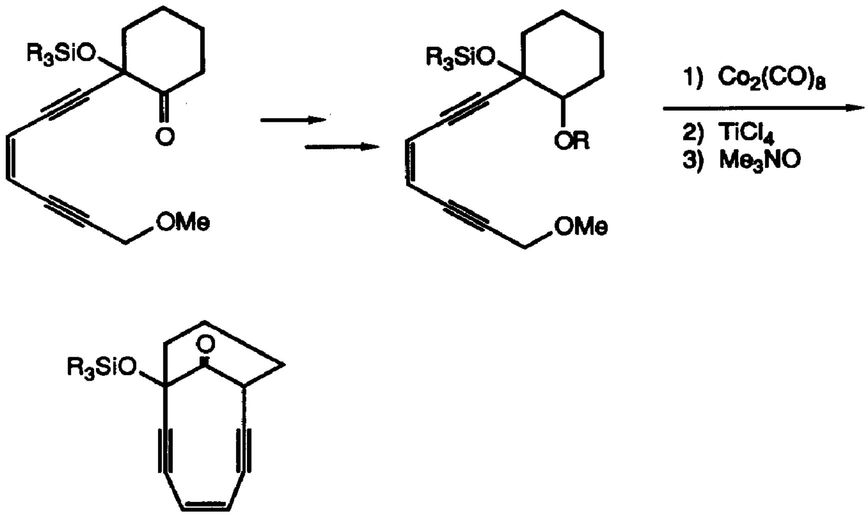
X = Br, I; Z = NH_2, NHAc; R = Ph, TMS, CH_2OH, CO_2Me, CO_2H

(200)

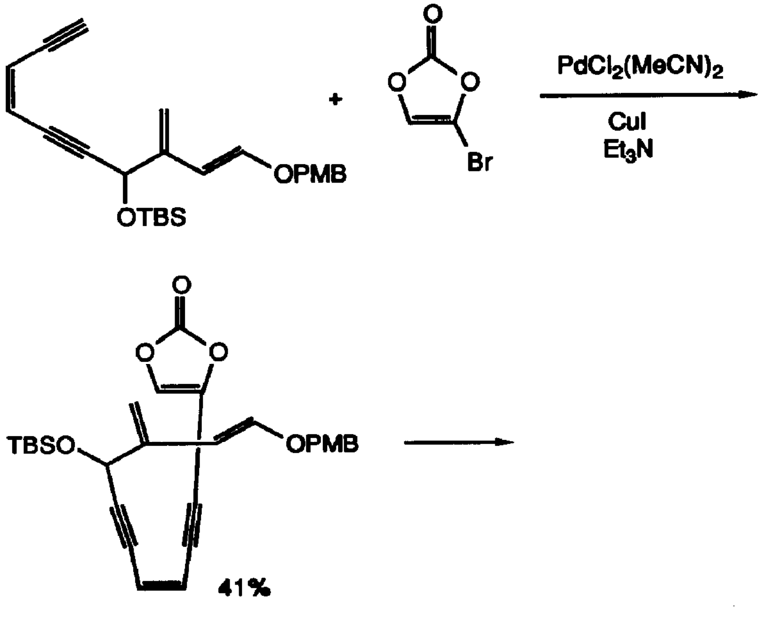


(201)

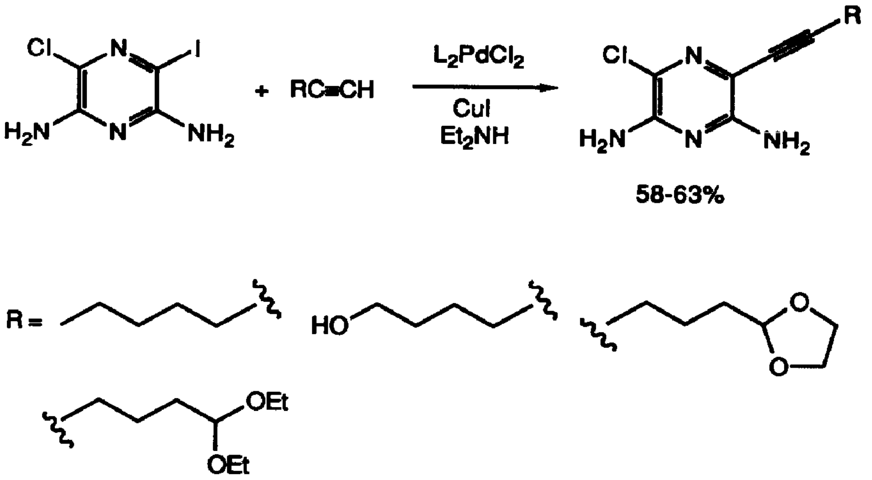



R₃SiO
O
OMe
R₃SiO
OR
OMe
1) Co₂(CO)₈
2) TiCl₄
3) Me₃NO
R₃SiO
O

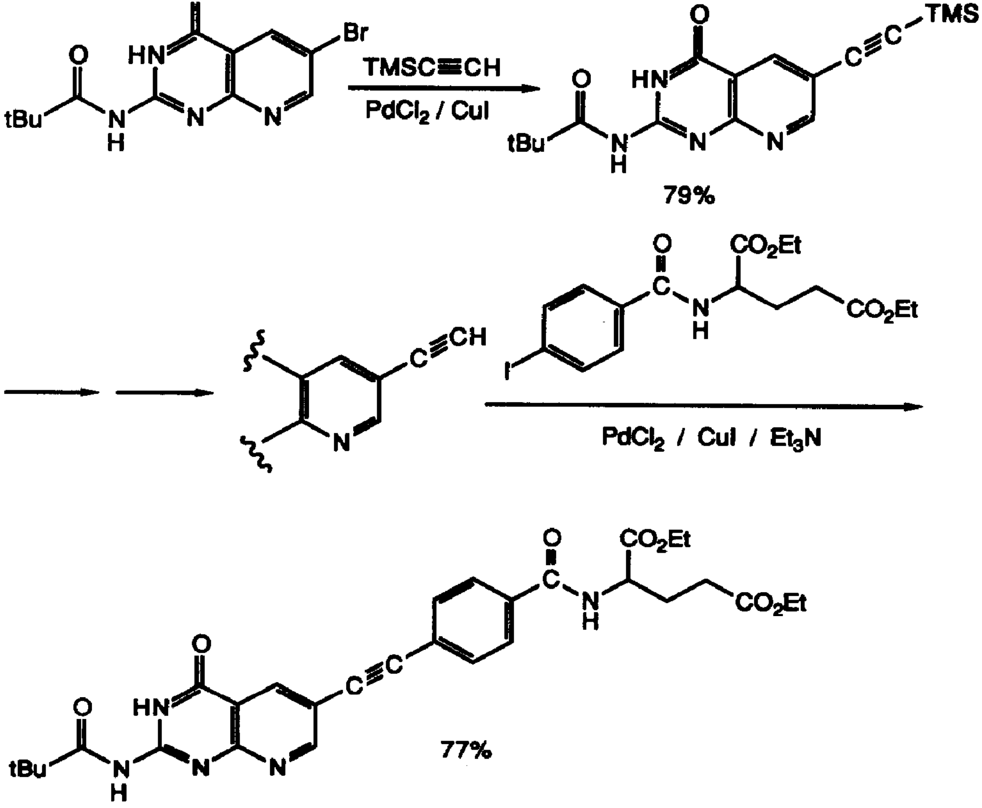
(202)


OTBS
OPMB
+
O
O
O
Br
PdCl₂(MeCN)₂
CuI
Et₃N
O
O
O
TBSO
OPMB
41%

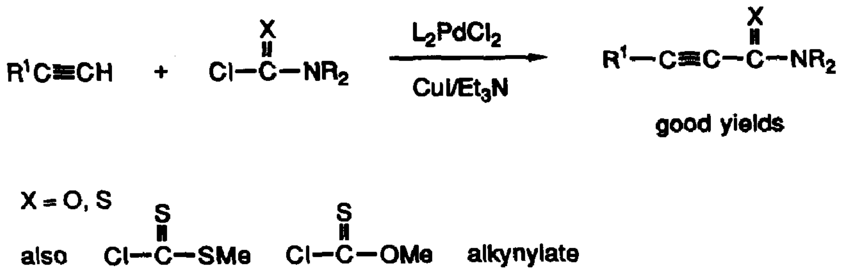
(203)


Cl
N
I
H$_2$N
N
NH$_2$
+ RC≡CH
L$_2$PdCl$_2$
CuI
Et$_2$NH
R
Cl
N
H$_2$N
N
NH$_2$
58-63%
R =
HO
O
O
OEt
OEt

(204


O
Br
O
HN
tBu
N
H
N
N
TMSC≡CH
PdCl$_2$ / CuI
O
C≡C
TMS
O
HN
tBu
N
H
N
N
79%
O
CO$_2$Et
C
N
H
CO$_2$Et
I
C≡CH
N
PdCl$_2$ / CuI / Et$_3$N
O
CO$_2$Et
C
N
H
CO$_2$Et
C≡C
O
O
HN
tBu
N
H
N
N
77%

(205)



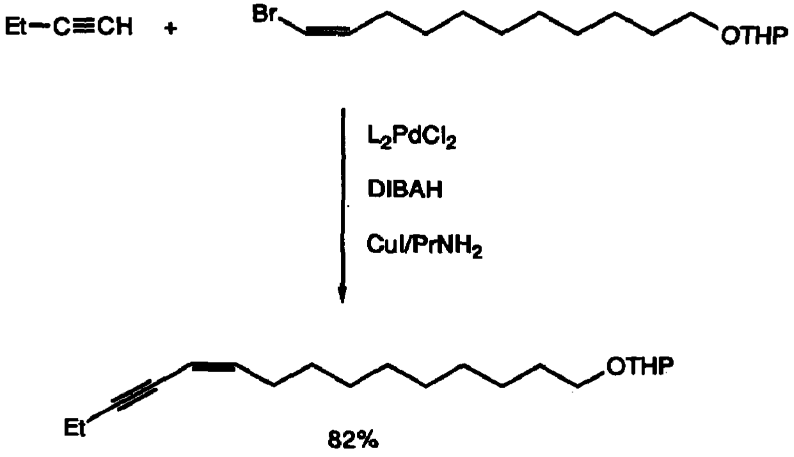
$$R^1C\equiv CH + Cl-\overset{\overset{X}{\|}}{C}-NR_2 \xrightarrow[CuI/Et_3N]{L_2PdCl_2} R^1-C\equiv C-\overset{\overset{X}{\|}}{C}-NR_2$$

good yields

X = O, S

also $Cl-\overset{\overset{S}{\|}}{C}-SMe$ $Cl-\overset{\overset{S}{\|}}{C}-OMe$ alkynylate

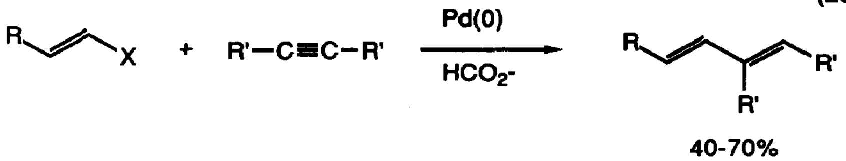
(206)



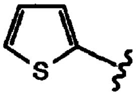
82%

Palladium(0) complexes catalyzed the reductive vinylation of alkynes by vinyl halides (equation 207) [235]. Alkynes were arylated then vinylated using palladium(0) catalysts (equation 208) [236]. Alkyl and vinyl halides underwent similar reactions (equation 209) [237]. Phenyl acetylene was arylated by o-palladated acetanilides (equation 210) [238].

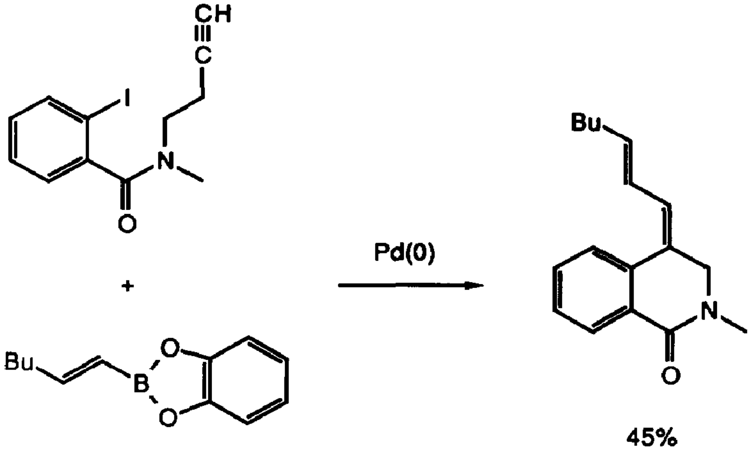
(207)



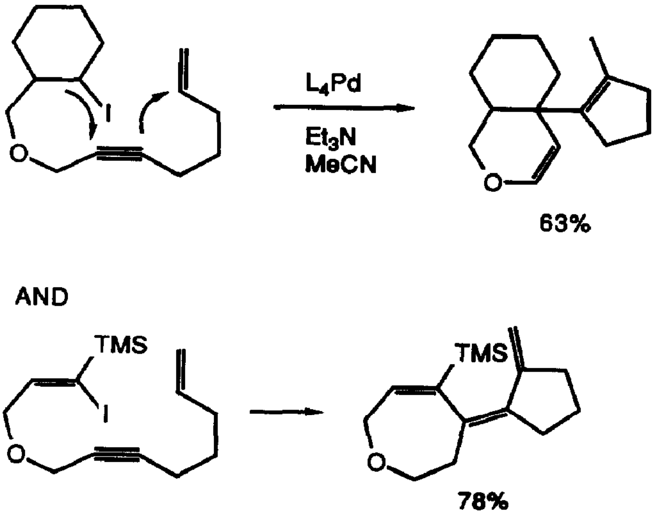
R = Bu, Ph
R' = Ph, pMeOPh, nPr, steroidal alkynes,



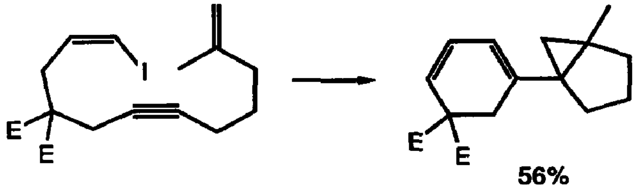
(208)



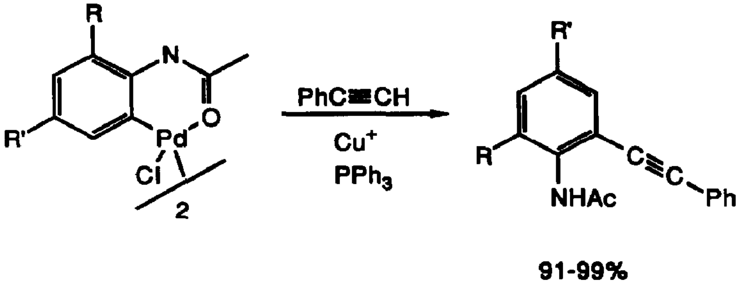
(209)



AND

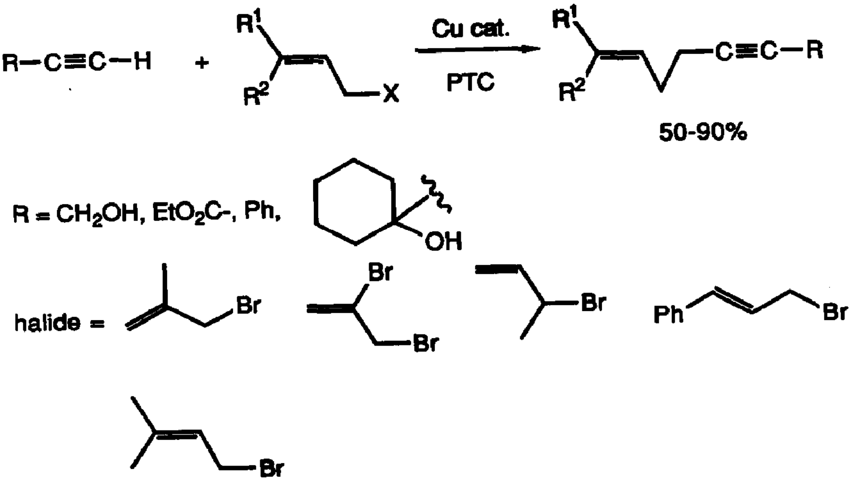


(210)

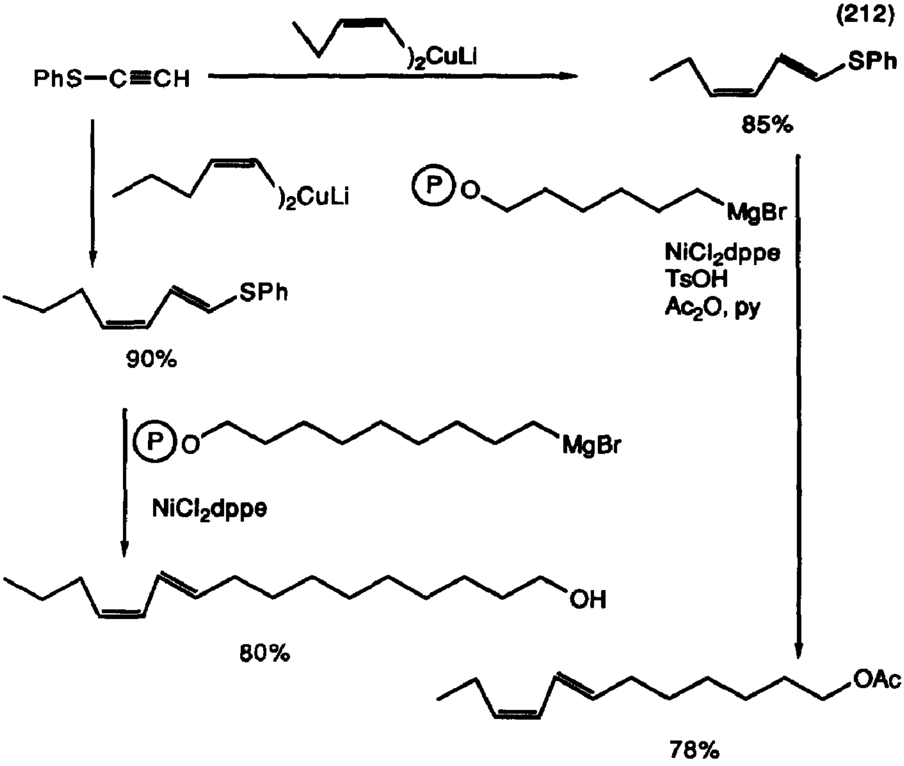


Copper catalyzed the alkylation of alkynes by allylic halides under phase transfer conditions (equation 211) [239]. Vinyl cuprates alkylated phenylthioacetylene (equation 212) [240]. Propargyl epoxides were alkylated by copper catalyzed Grignard reactions to give allenes (equation 213) [241], (equation 214) [242].

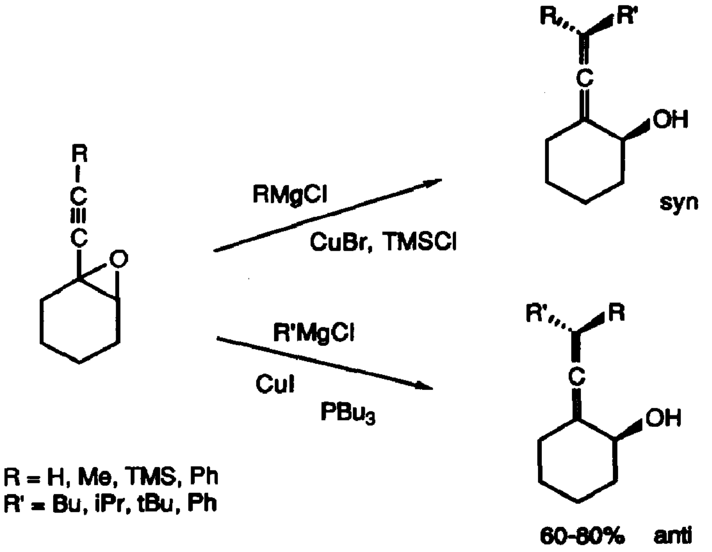
(211)



(212)

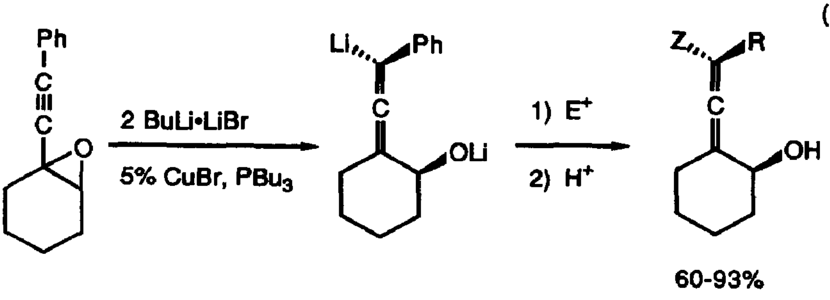


(213)



R = H, Me, TMS, Ph
R' = Bu, iPr, tBu, Ph

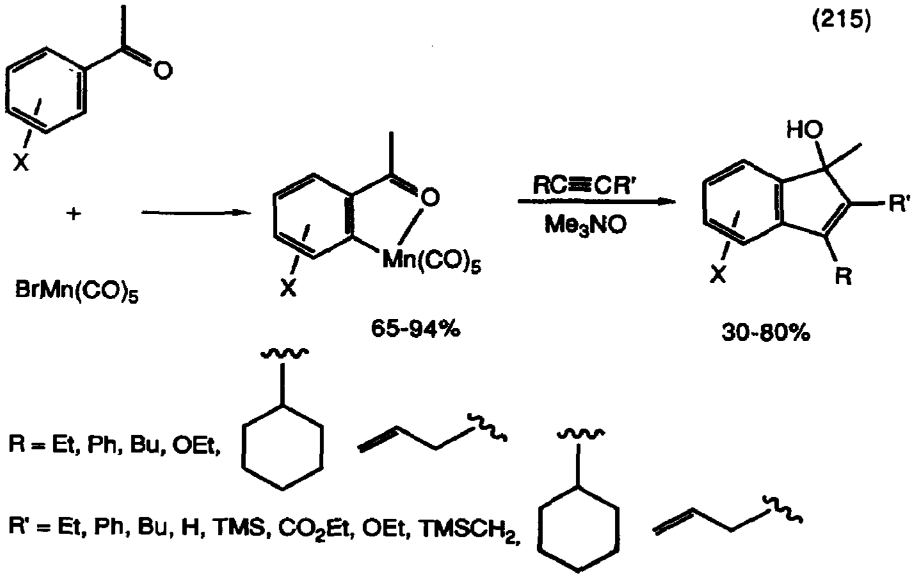
(214)



E^+ = MeI, MeSSMe, PhCHO, CO_2

Orthomanganated acetophenones inserted alkynes (equation 215) [243]. "Highly selective syntheses of naturally occurring acetylenes and their structural analogs by palladium-catalyzed carbon-carbon bond forming reactions" was the title of a review with 39 references [244]. Ziegler-Natta type nickel complexes catalyzed the hydromethylation of acetylene [245]. Alkynes were oxidatively coupled to polyalkynes via (bis) alkynylnickel complexes [246].

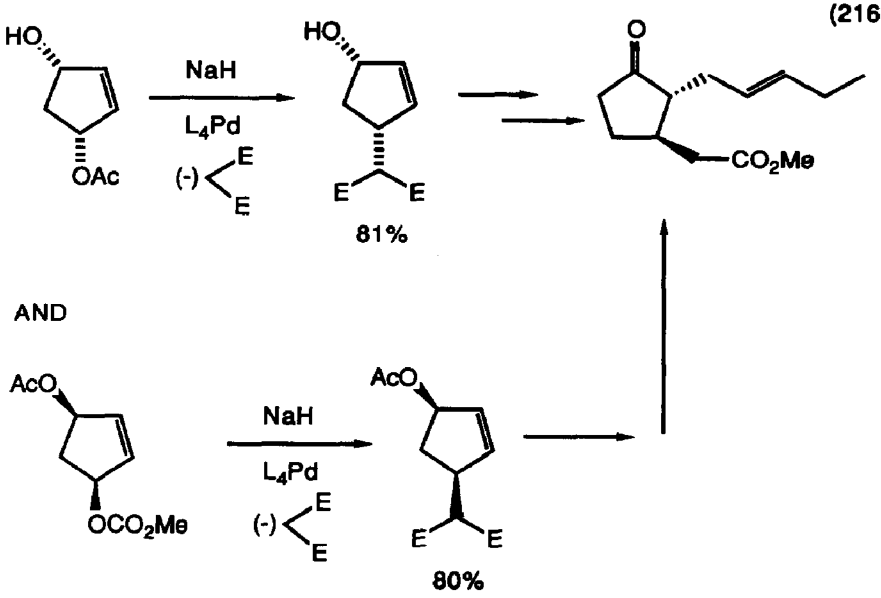
(215)



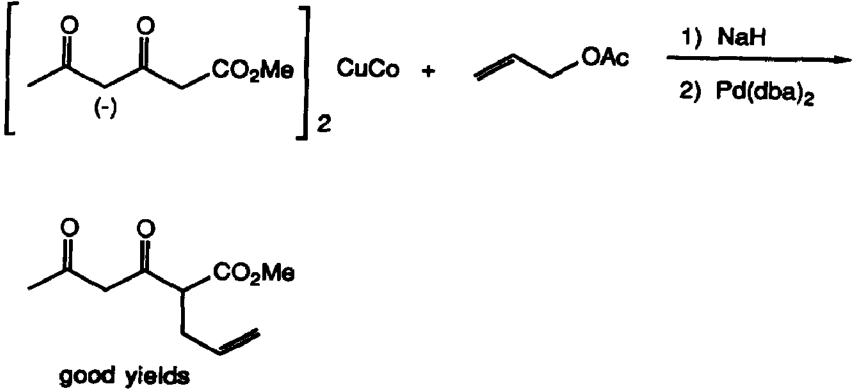
7. Alkylation of Allyl, Propargyl and Allenyl Systems

Alkylation of allylic systems via π-allylpalladium complex intermediate continues to be extensively exploited in synthesis. Cyclizations via palladium catalyzed allylic alkylation has been reviewed (135 reference) [247]. Prostaglandin intermediates were prepared using multiple palladium catalyzed allylic alkylation (equation 216) [248]. Copper and cobalt salts of β,δ-diketo esters alkylated allyl acetates exclusively at the α-position in the presence of palladium catalysts (equation 217) [249]. Other useful alkylations are presented in equation 218 [250], equation 219 [251], and equation 220 [252].

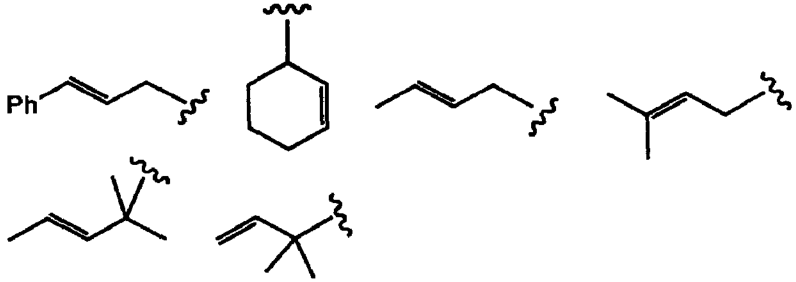
(216)



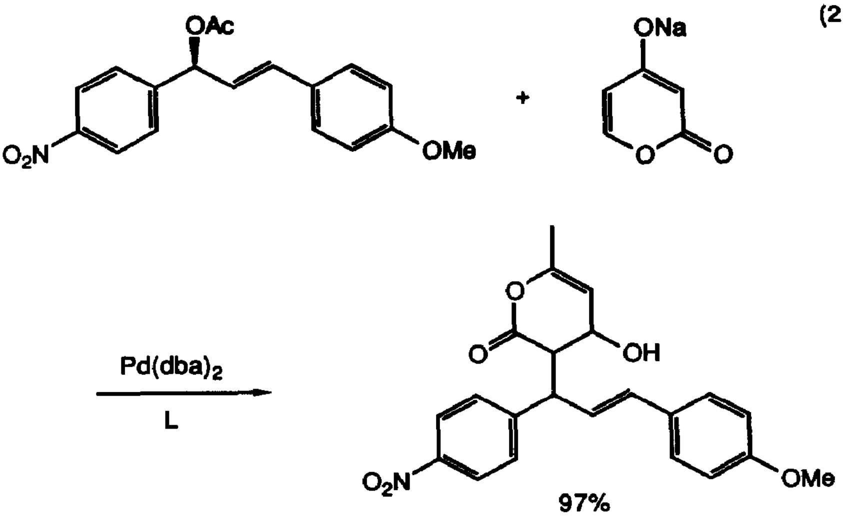
(217)



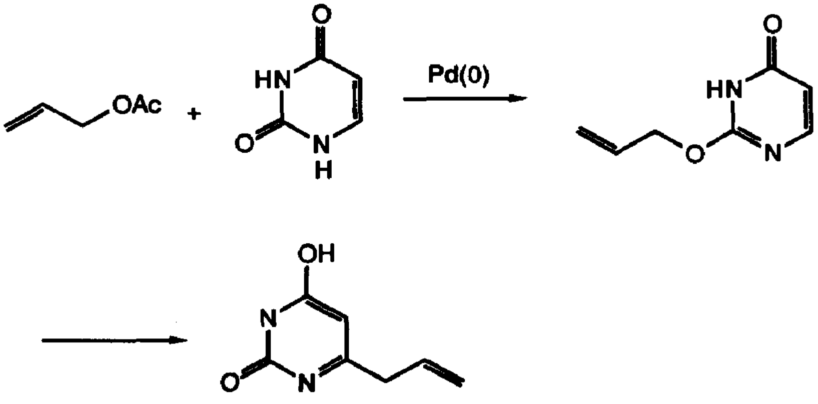
allyl =



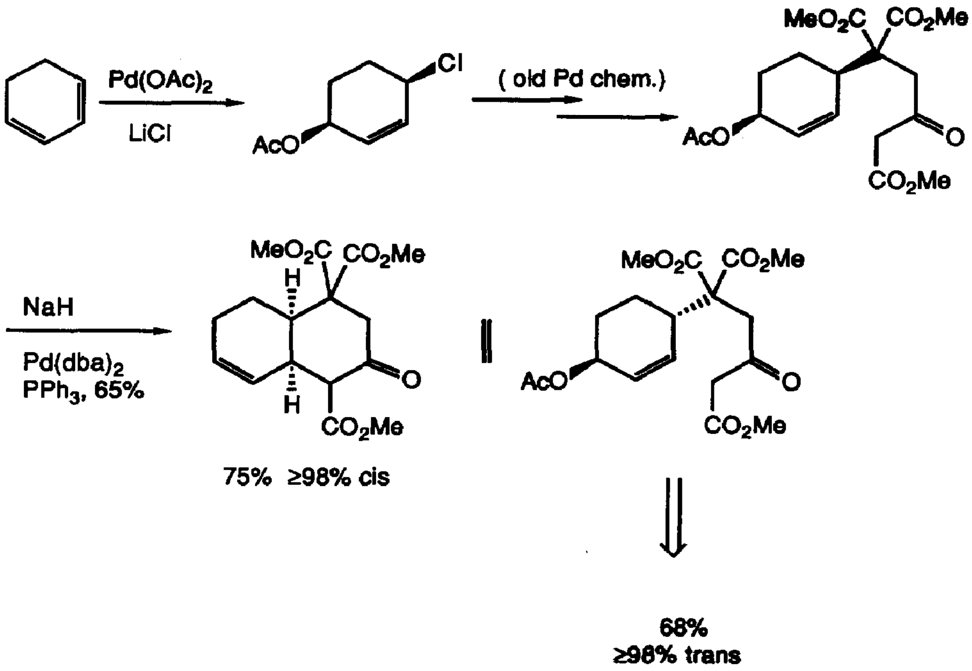
(218)



(219)

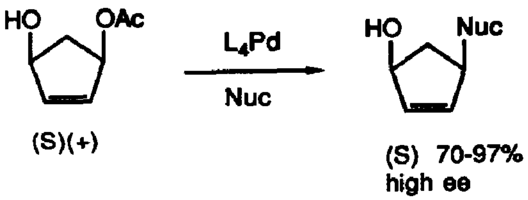


(220)



Asymmetric induction is high in these π-allylpalladium alkylation reactions, and has been achieved by using optically active allyl acetates (equation 221) [253], nucleophiles (equation 222) [254], or ligands (equation 223) [255]. Intramolecular versions were also highly stereoselective (equation 224) [256]. Allylic 1,1-diol diacetates were alkylated by malonates in the presence of palladium catalysts [257].

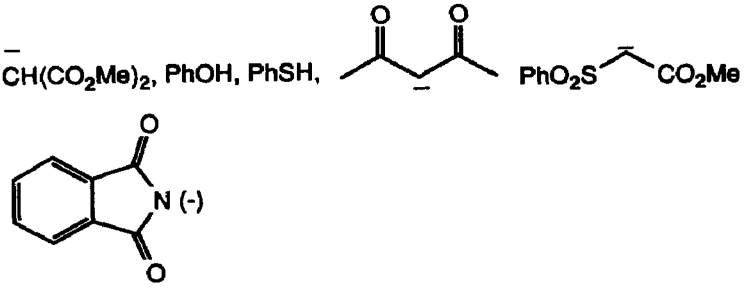
(221)



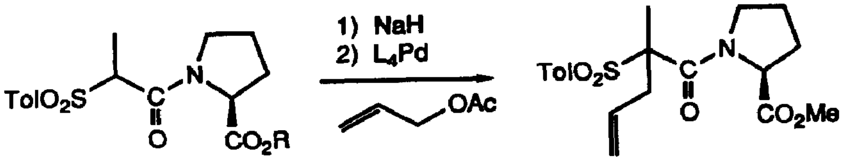
(S)(+)

(S) 70-97%
high ee

Nuc = $\bar{C}H(CO_2Me)_2$, PhOH, PhSH,

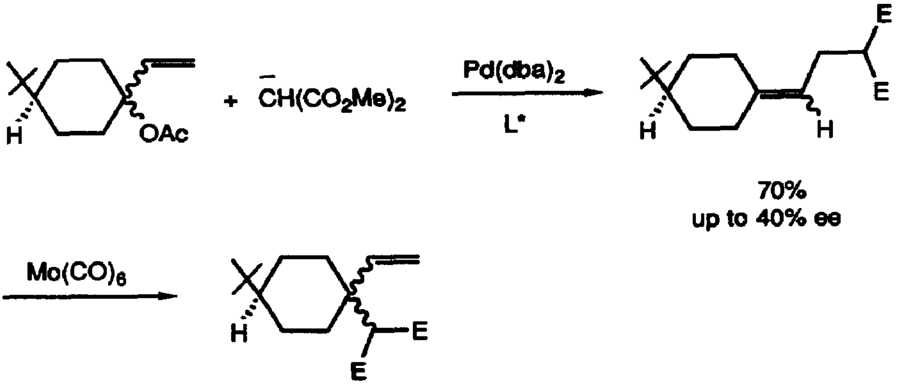


(222)

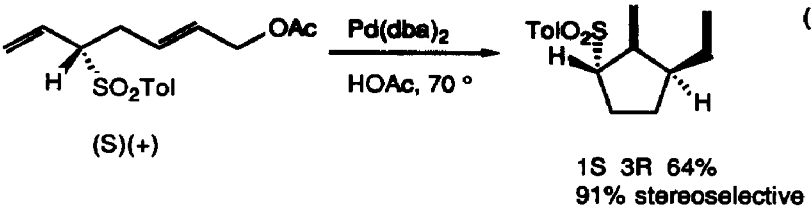


70-97%
40-86% de

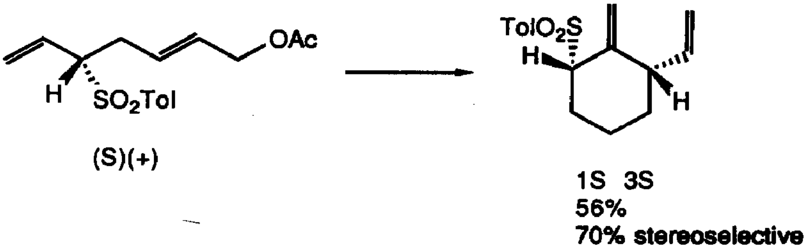
(223)



70%
up to 40% ee

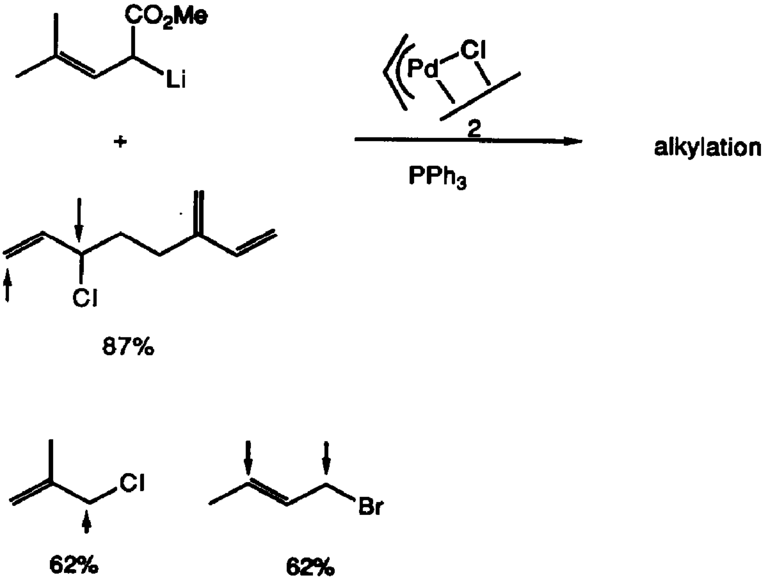


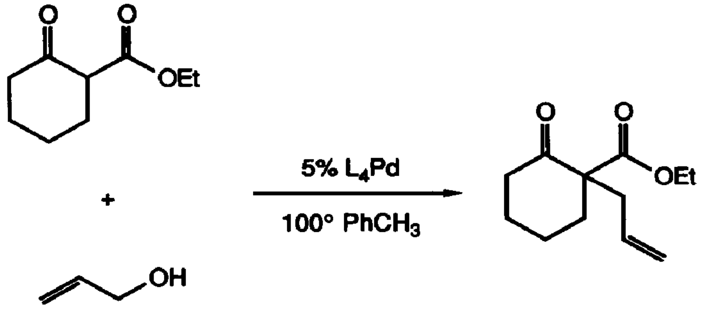
(224)



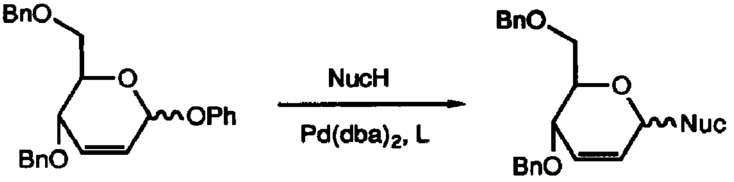
Crotonate anions alkylated allylic halides in the presence of π-allyl palladium halide catalyst (equation 225) [258]. Palladium also catalyzed the alkylation of allylic alcohols (equation 226) [259], allylic phenyl ethers (equation 227) [260], (equation 228) [261], and o-allylimidates (equation 229) [262]. Palladium(0) complexes catalyzed the alkylation of allyl epoxides by alkyltin reagents (equation 230) [263].

(225)

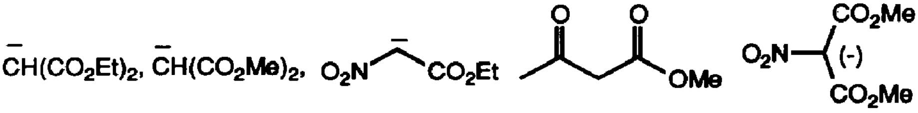


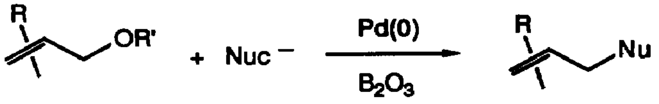


(226)



(227)

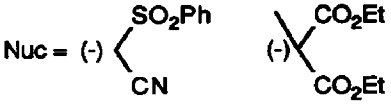


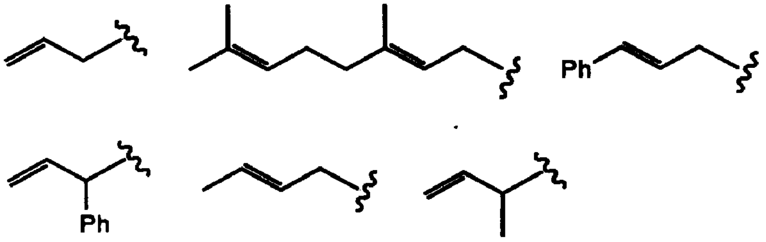


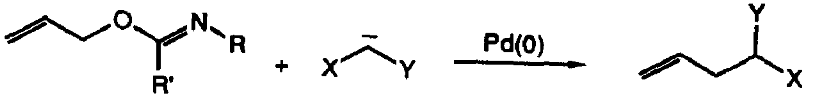
(228)

good yields

R = $PhCH_2$, nBu, Me, nPr



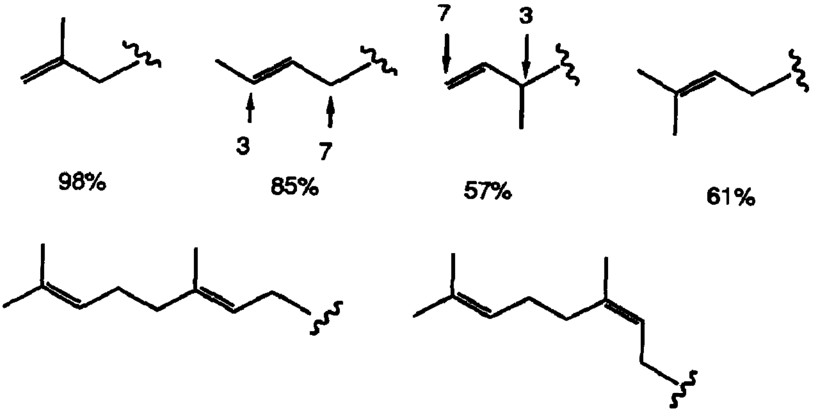


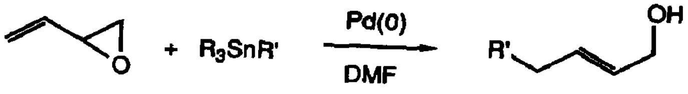


(229)

X = CO_2Me, COMe, Ph, $PhSO_2$

Y = COMe, CO_2Et, PhCO



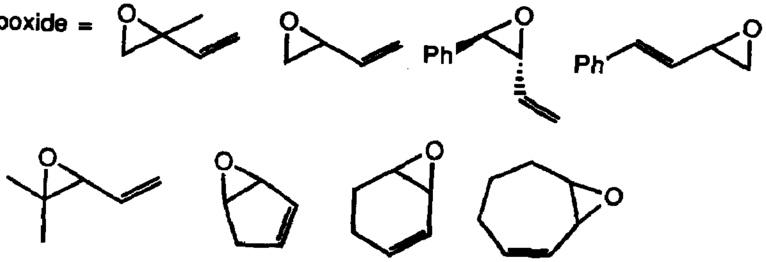


(230)

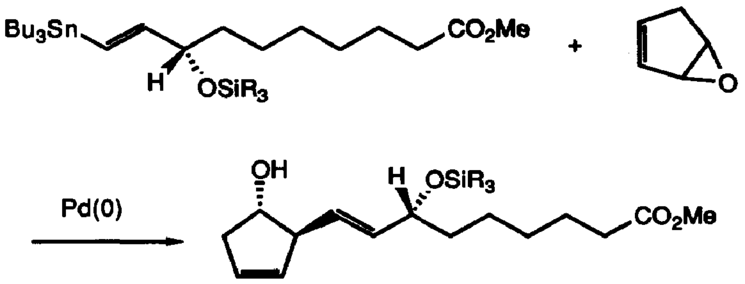
good yields

R' = Ph,

epoxide =

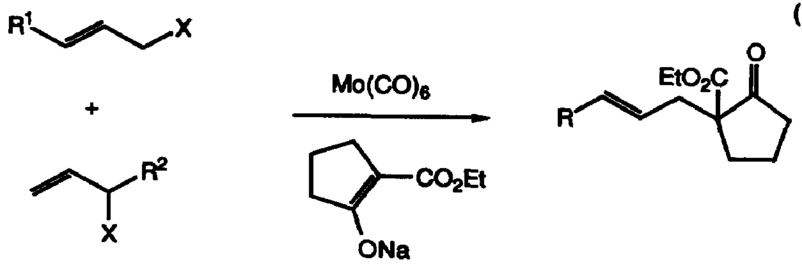


AND



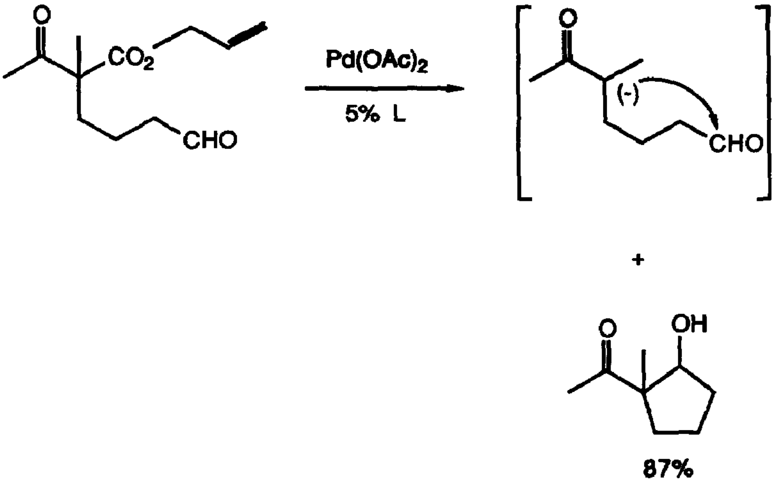
Molybdenum catalyzed allylic alkylations were the topic of a dissertation [264] (equation 231) [265]. Palladium(0) complexes were highly reactive toward allylic carbonates, extruding CO_2 and generating enolates (equation 232) [266][267], undergoing allylic alkylation (equation 233) [268], (equation 234) [269], or nucleophilic attack (equation 235) [270].

(231)

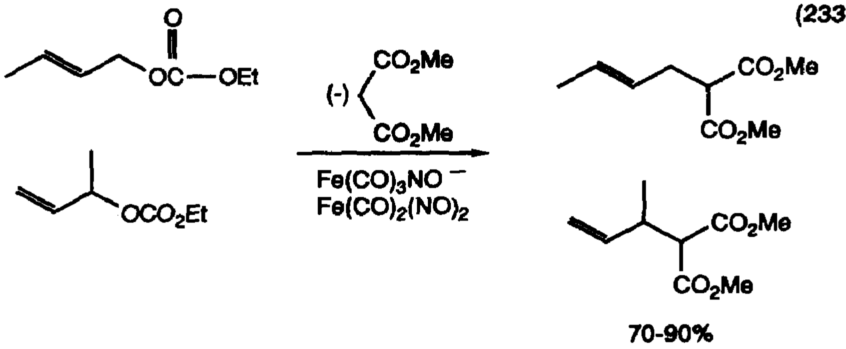


X = SR', SeR', SO_2R'

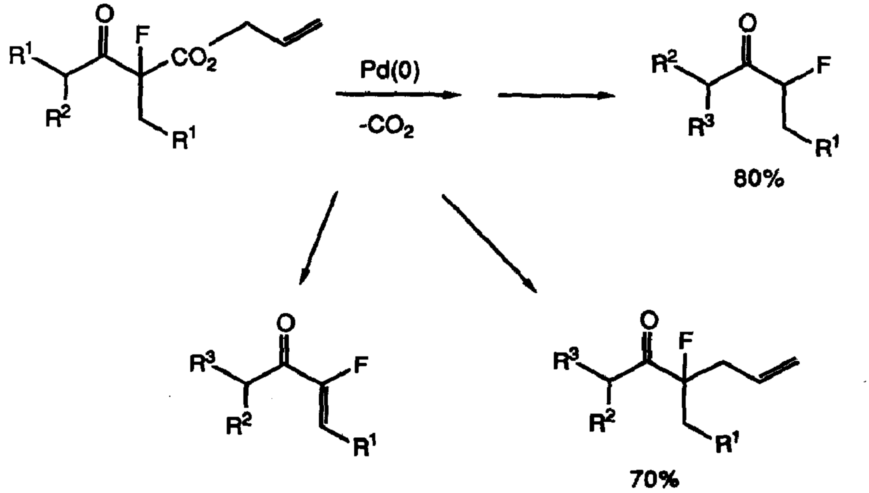
(232)

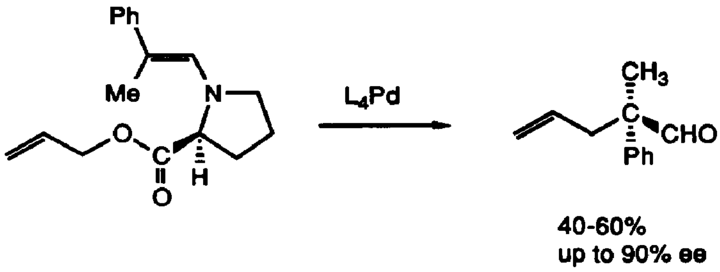

CO2
CHO
Pd(OAc)2
5% L
(-)
CHO
+
O
OH
87%

(233)


O
OC—OEt
OCO2Et
CO2Me
(-)
CO2Me
Fe(CO)3NO⁻
Fe(CO)2(NO)2
CO2Me
CO2Me
CO2Me
CO2Me
70-90%

(234)

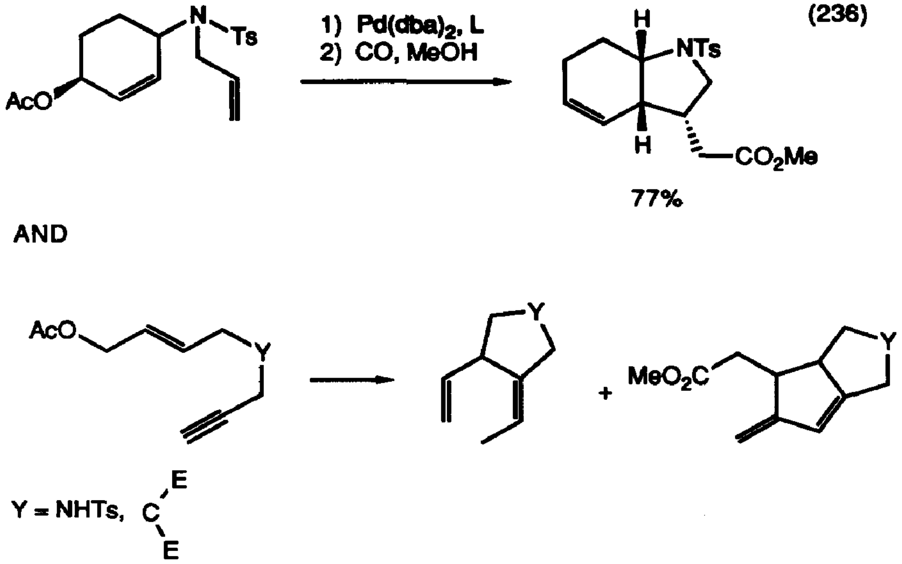

R1
O
F
CO2
R2
R1
Pd(0)
-CO2
R2
O
F
R3
R1
80%
O
R3
F
R2
R1
O
F
R3
R2
R1
70%



(235)

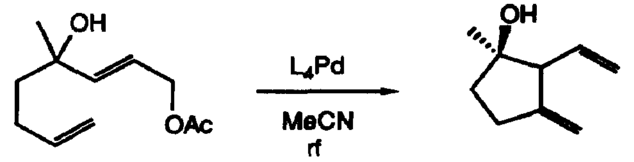
Allylic acetates have been bifunctionalized via π-allylpalladium complex chemistry by olefin insertion (equation 236) [271], (equation 237a) [272a], (equation 237b) [272b], (equation 238) [273], by nucleophilic attack (equation 239) [274] and by transmetallation (equation 240) [275], (equation 241) [276].

Dienes were dialkylated by iron nitrosyl dicarbonyl complexes (equation 242) [277]. Trimethylene methane iron complexes alkylated allylic halides (equation 243) [278].



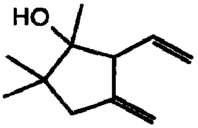
(236)

(237a)

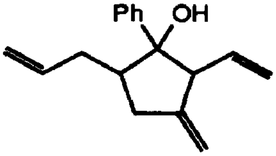


75% 4:1 Z:E

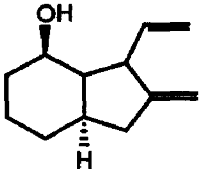
ALSO



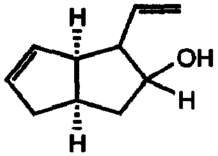
82%



80%

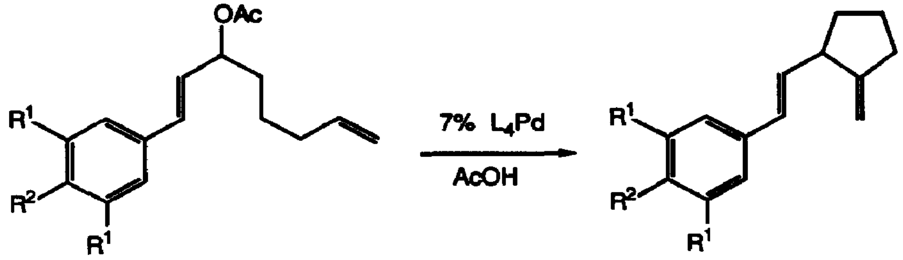


75%



73%

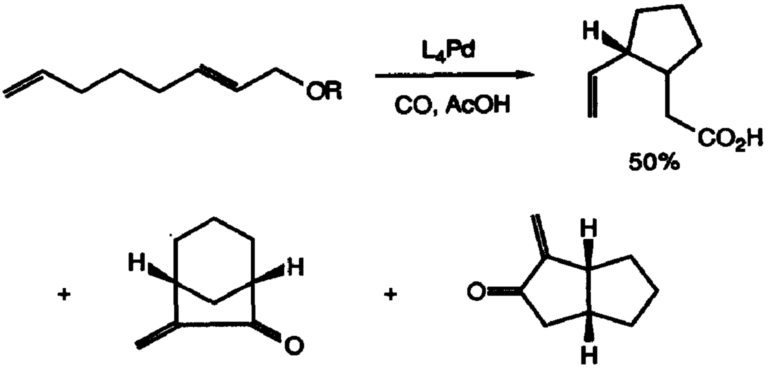
(237b)



49-97%

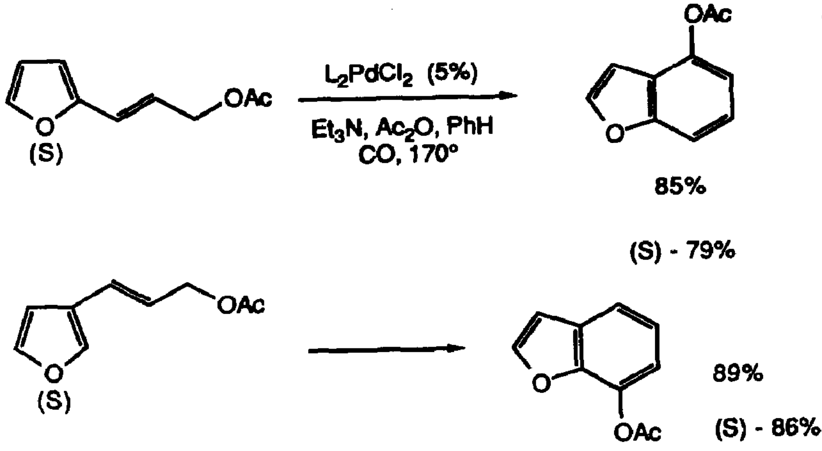
R^1 = H, OMe
R^2 = H, CF_3

(238)



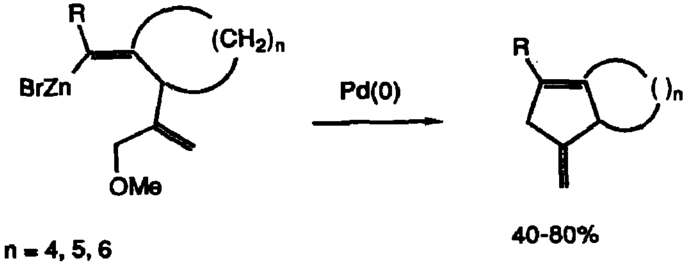
20%   10%

(239)



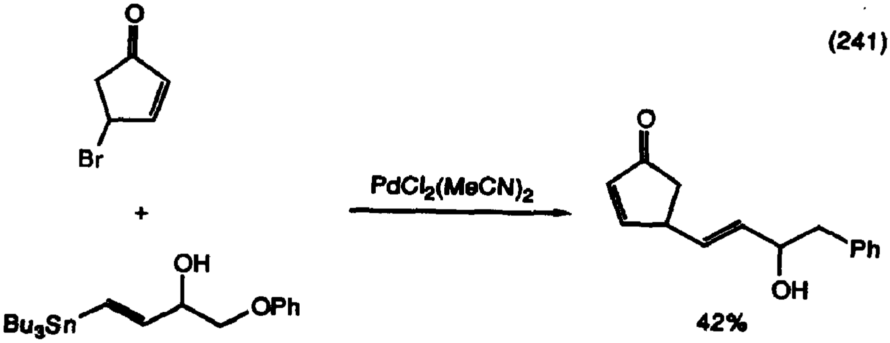
(S)
OAc
L2PdCl2 (5%)
Et3N, Ac2O, PhH
CO, 170°
OAc
O
85%
(S) - 79%
OAc
(S)
O
89%
OAc
(S) - 86%

(240)



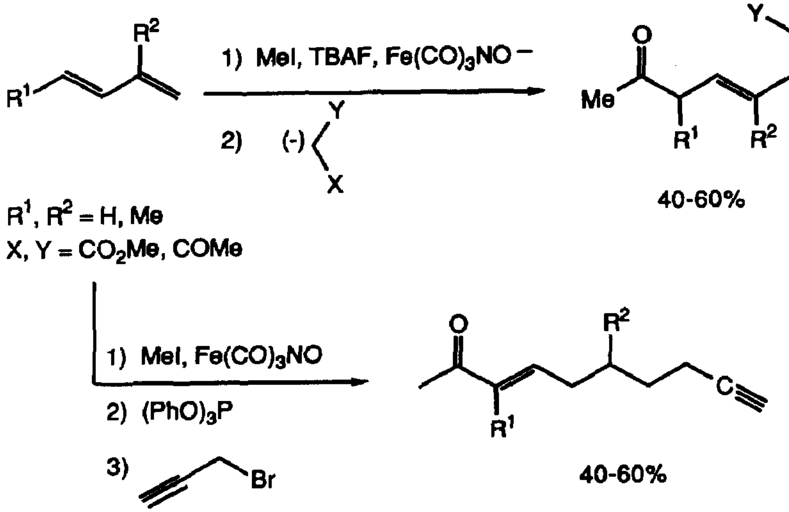
R
(CH2)n
BrZn
OMe
n = 4, 5, 6
Pd(0)
R
( )n
40-80%

(241)

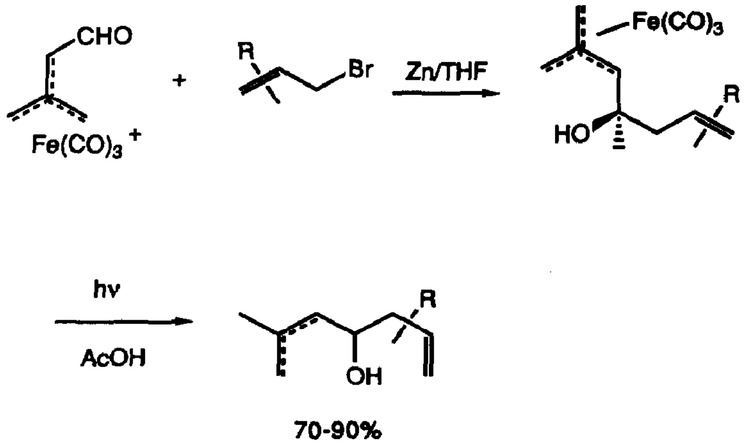


O
Br
+
OH
Bu3Sn
OPh
PdCl2(MeCN)2
O
Ph
OH
42%

(242)

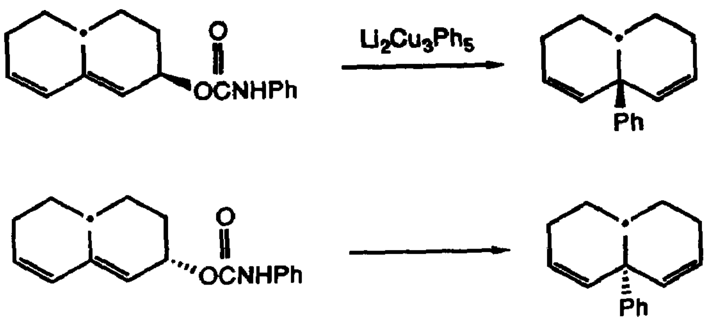


(243)



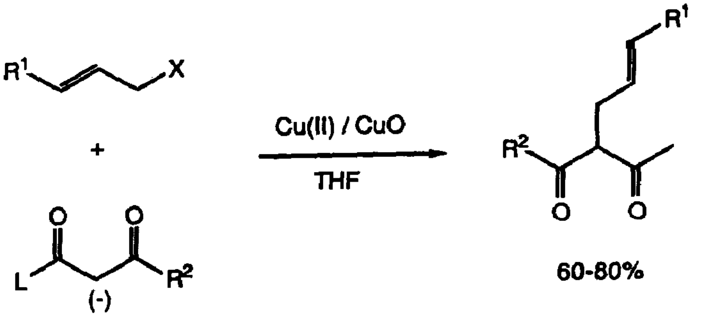
Cross coupling reactions of allylic and dienyl allylic carboxylates with organocopper and Grignard reagents was the topic of a dissertation [279]. Allyl carbamates (equation 244) [280], carbonates and halides (equation 245) [281], allyl ethers (equation 246) [282], (equation 247) [283], and allyl pivalates (equation 248) [284] were alkylated by organocopper or nickel complexes .

(244)



syn γ exclusively

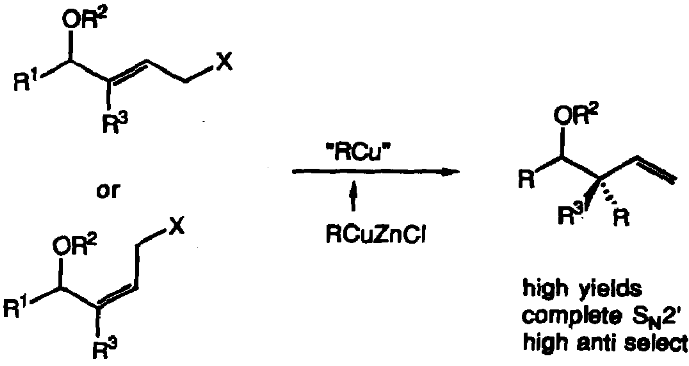
(245)



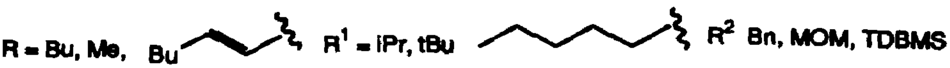
60-80%

R^1 = H, Me, Ph; R^2 = Me, Ph, OMe, OEt
X = Cl, OAc, OCO_2Et, $OCOCF_3$, OH

(246)

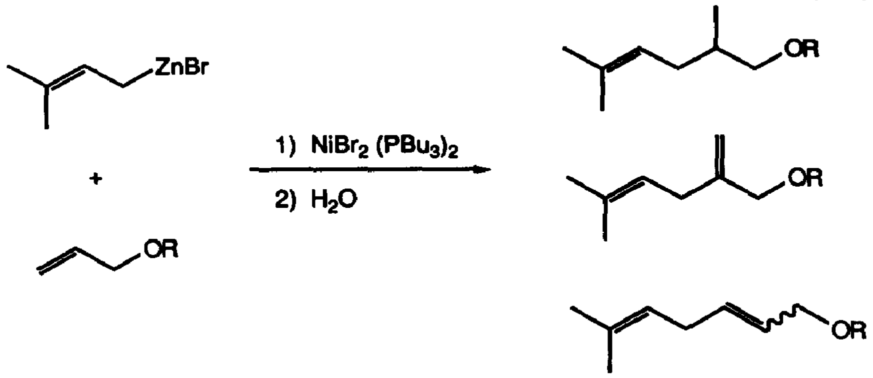


high yields
complete S_N2'
high anti selectivity

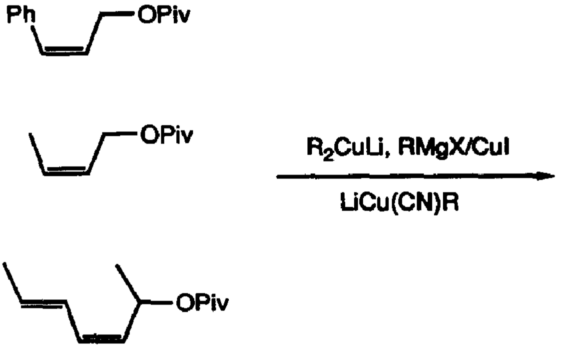


R = Bu, Me, Bu, R^1 = iPr, tBu, R^2 Bn, MOM, TDBMS

(247)



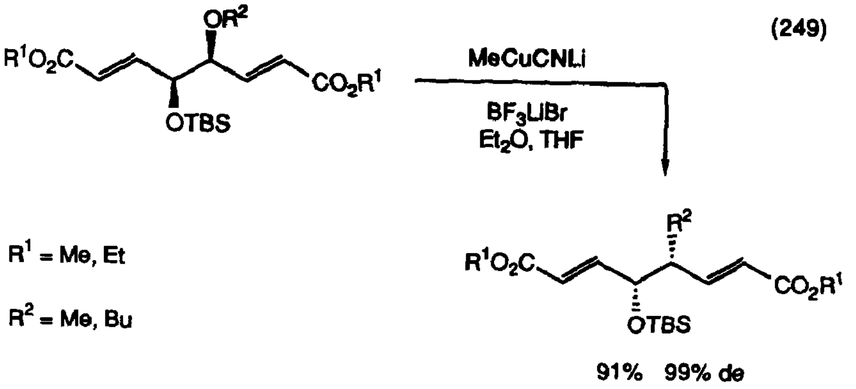
(248)



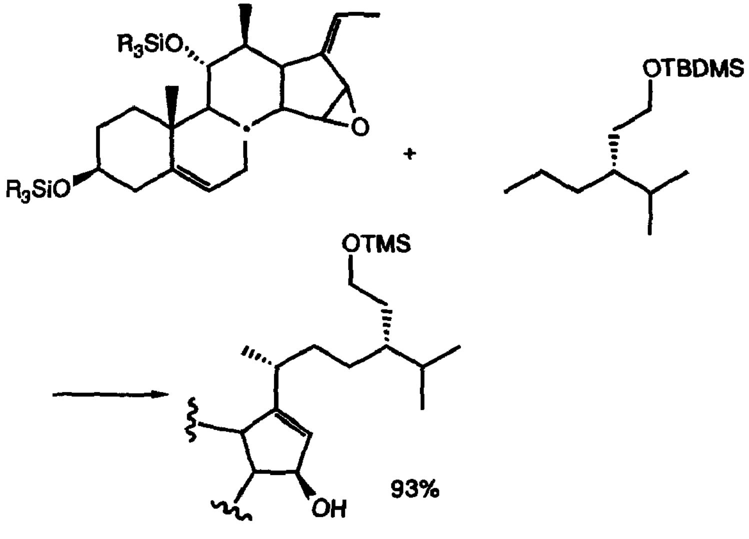
Effects of reagents in stereochemistry and regiochemistry Sp^3 coppers lead to loss of configuration. Sp^2 preserves configuration of olefin.

Allylic ethers and mesylates γ- to the ester group of acrylates were alkylated with S_N2' regiochemistry and high diastereoselectivity (equation 249) [285][286] by cyanocuprates. Steroidal allyl epoxides were alkylated by Grignard reagents in the presence of copper catalysts (equation 250) [287].

(249)

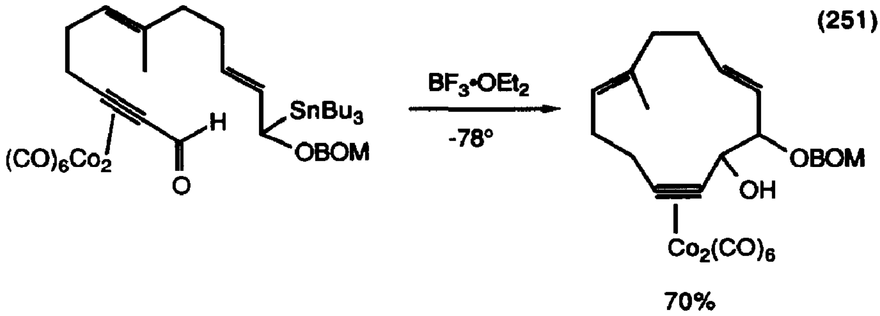


(250)

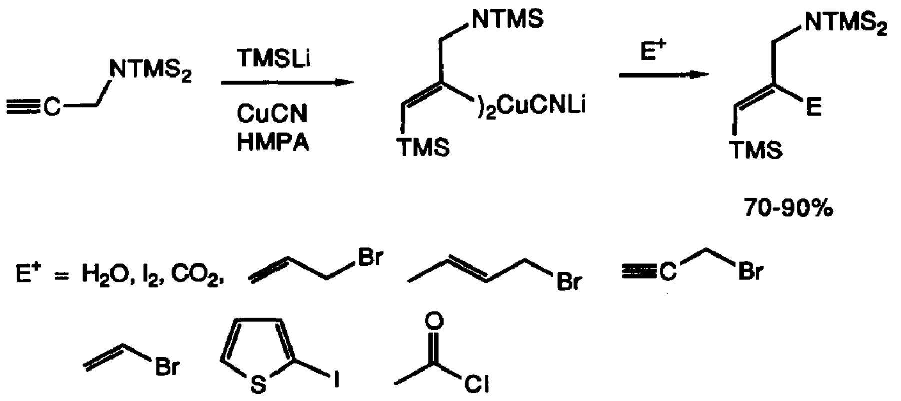


Cobalt-stabilized propargyl aldehydes underwent efficient 1,2-addition with tin reagents (equation 251) [288]. Propargyl amines were C-silylated and alkylated by silyl cuprates (equation 252) [289].

(251)

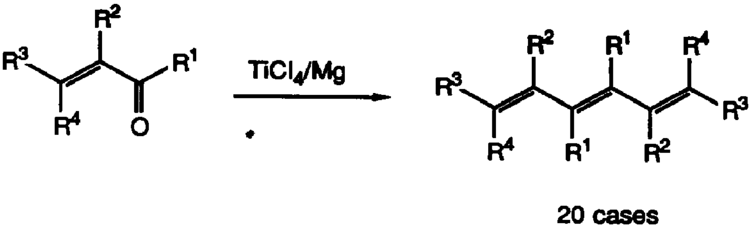


(252)



8. Coupling Reactions

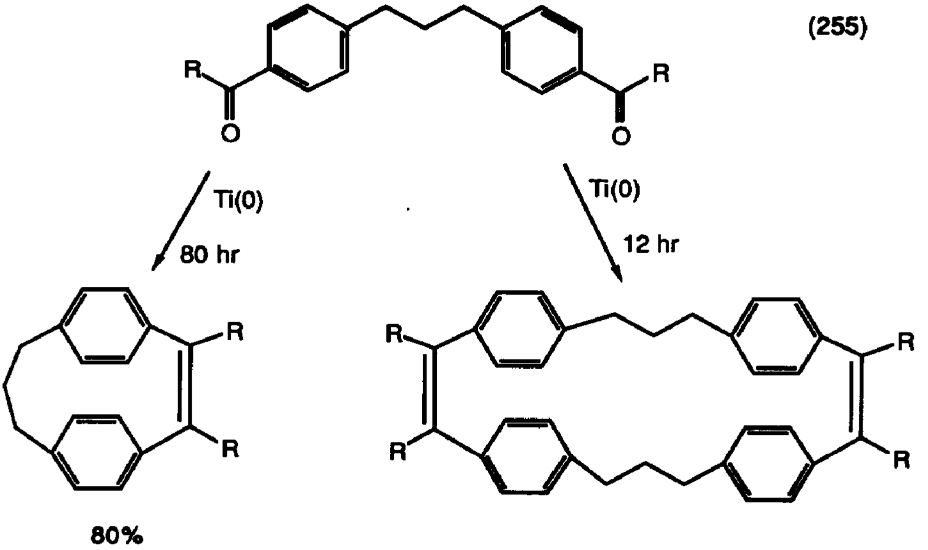
Carbonyl coupling via low valent titanium reagents has been further developed and applied this year, and several reviews on the subject have appeared [290] (124 references), [291] (132 references, [292] (110 references). The reaction has been optimized by using recrystallized $TiCl_3 \cdot 1.5$ DME/Zn/Cu to generate the titanium(0) reagent [293]. α,β-Unsaturated ketones were coupled to trienes (equation 253) [294]. Unusual cyclic olefin (equation 254) [295] and diolefin (equation 255) [296] were prepared by this method. Ketones bearing remote quarternary ammonium salts were efficiently dimerized (equation 256) [297]. High valent tungsten complexes also coupled ketones to give olefins (equation 257) [298]. Isoprene was coupled by "titanocene" (equation 258) [299].


R2
R3
R1
R4
O
TiCl4/Mg
R2
R1
R4
R3
R3
R4
R1
R2
20 cases

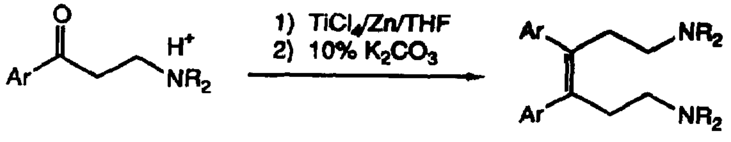
(253)


O
CHO
TiCl3/Zn/Cu
30%

(254)


R
O
R
O
Ti(0)
80 hr
Ti(0)
12 hr
R
R
80%
R
R
R
R

(255)

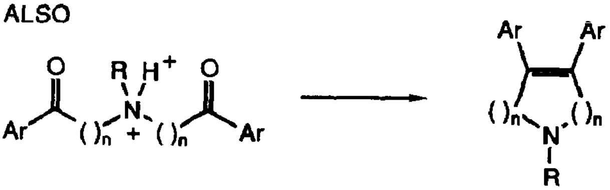


(256)

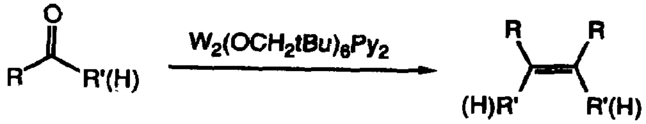
33-81%

Ar = Ph, pBrPh, pMePh, pMeOPh
R = Me, $(CH_2)_5$, $-CH_2CH_2OCH_2CH_2-$

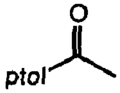
ALSO

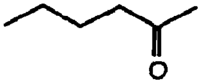


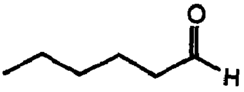
n = 1, 2



(257)



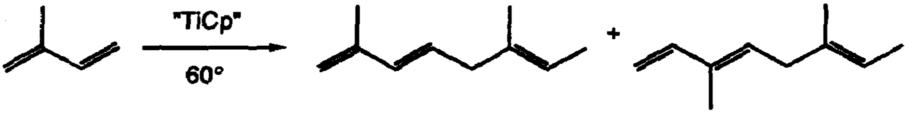




PhCHO

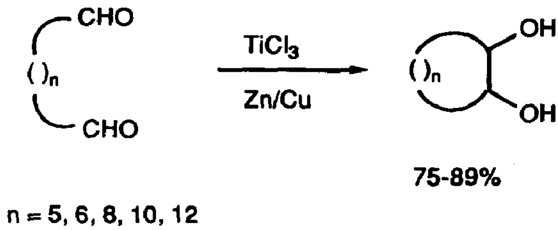
51% 21% 18% 34%

(258)



depending on cat. up to 99% up to 49%

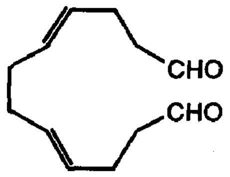
This titanium coupling can be stopped at the pinacol stage, and this process has also been exploited for the formation of both cyclic (equation 259) [300], (equation 260) [301], (equation 261) [302] and acyclic (equation 262) [303], (equation 263a) [304a] pinacols. Reduced vanadium complexes cross-couple aldehydes to pinacols (equation 263b) [304b]. Trichloroethanes were coupled to disubstituted olefins by low-valent titanium complexes [305].



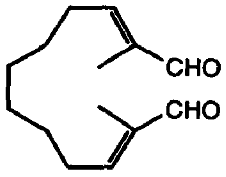
(259)

75-89%

n = 5, 6, 8, 10, 12

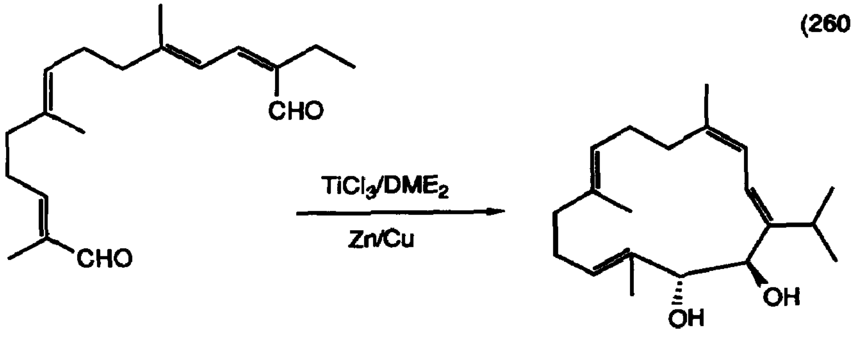


83%



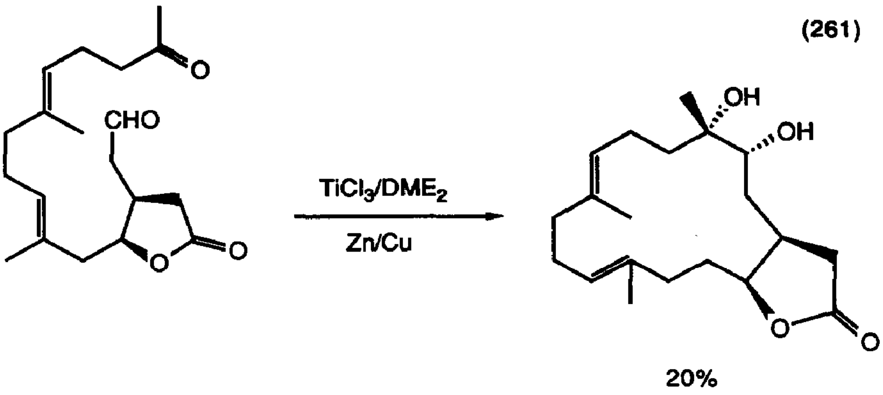
84%

(260)



46%
Sacrophytol

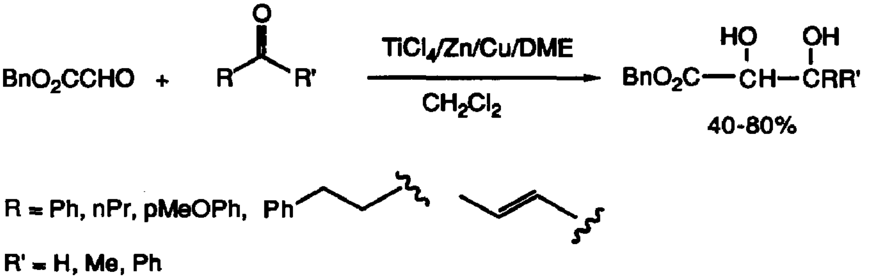
(261)



$\longrightarrow$ $\longrightarrow$ (±)-Crassin

$$\mathrm{RCHO} \xrightarrow{\mathrm{CpTiCl_3/LiAlH_4}} \mathrm{R\overset{HO}{C}H{-}\overset{OH}{C}HR} \qquad (262)$$

(263a)



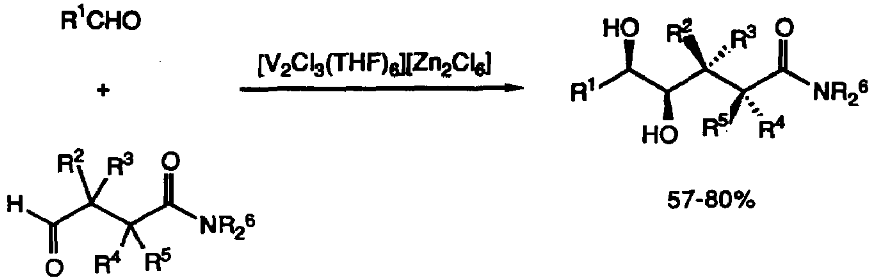
$$\mathrm{BnO_2CCHO} + \mathrm{RC(O)R'} \xrightarrow[\mathrm{CH_2Cl_2}]{\mathrm{TiCl_4/Zn/Cu/DME}} \mathrm{BnO_2C{-}\overset{HO}{C}H{-}\overset{OH}{C}RR'}$$

40-80%

R = Ph, nPr, pMeOPh,

R' = H, Me, Ph

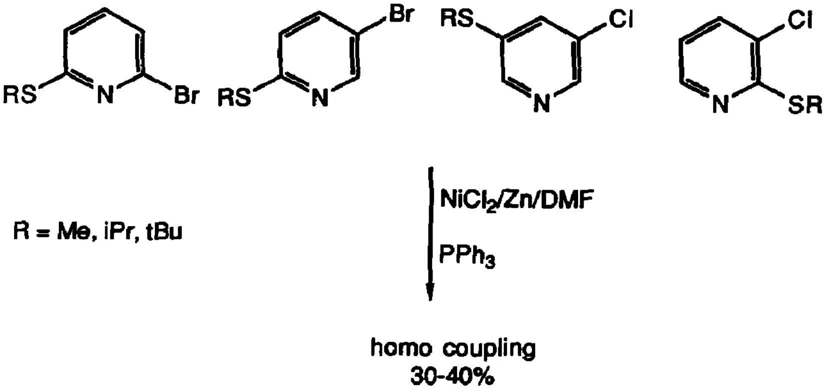
(263b)



$$Ar_2CHCCl_3 \xrightarrow{TiCl_4/LiAlH_4} E\ ArCH{=}CHAr \qquad (264)$$

Low valent nickel reagents reductively coupled heteroaromatic halides (equation 265) [306] and aryl halides (equation 266) [307], (equation 267) [308][309]. Ullmann couplings of bromobenzaldehydes went at room temperature (equation 268) [310]. Low valent nickel complexes also coupled vinyl halides (equation 269) [311] and acetylides (equation 270) [312].

(265)



$$\mathrm{ArX}\ \xrightarrow[\substack{4:2:1:2:1\\ \mathrm{PhH}}]{\mathrm{NiCRA/bpy/KT}}\ \mathrm{Ar{-}Ar} \qquad (266)$$

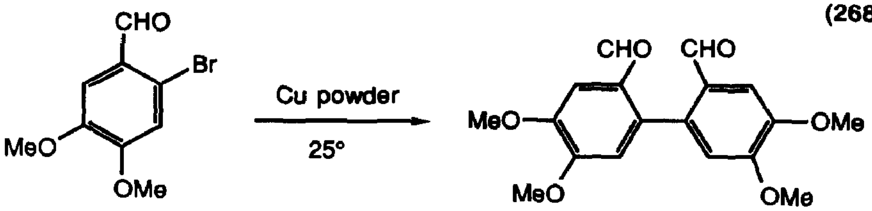
70-90%   60 cases!

(CRA = complex reducing agent - see paper for details)

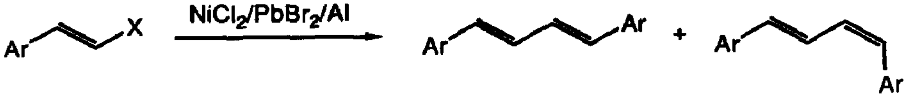
$$\mathrm{ArX} + \mathrm{Ar'X}\ \xrightarrow[\mathrm{Bpy}\quad 60^\circ]{4:2:1:2\quad \mathrm{NiCRA}}\ \mathrm{ArAr} + \mathrm{Ar'Ar} + \mathrm{Ar'Ar'} \qquad (267)$$

modest yields
all possible products

(268)

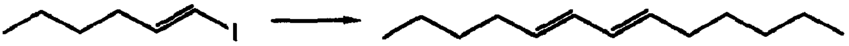


(269)

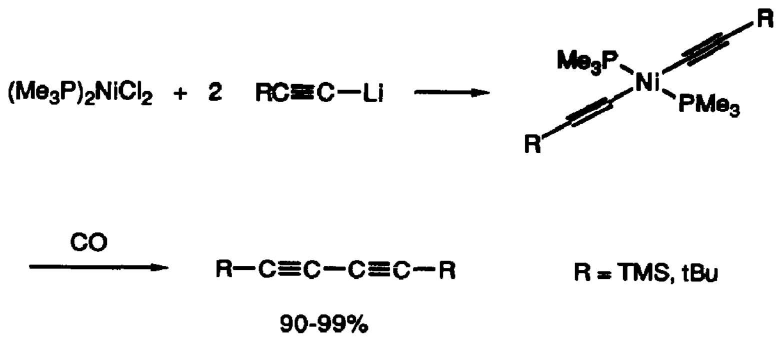


70-80%

AND



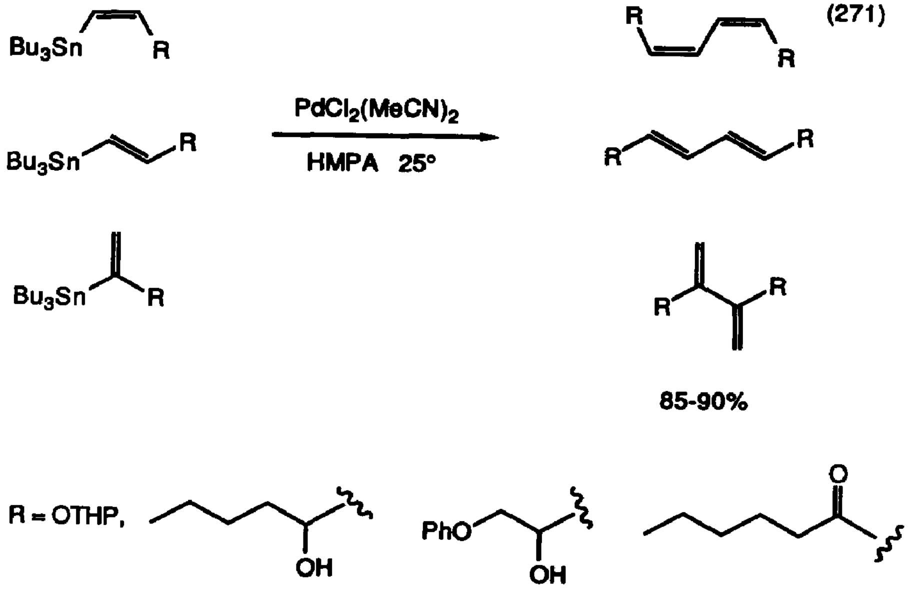
77%

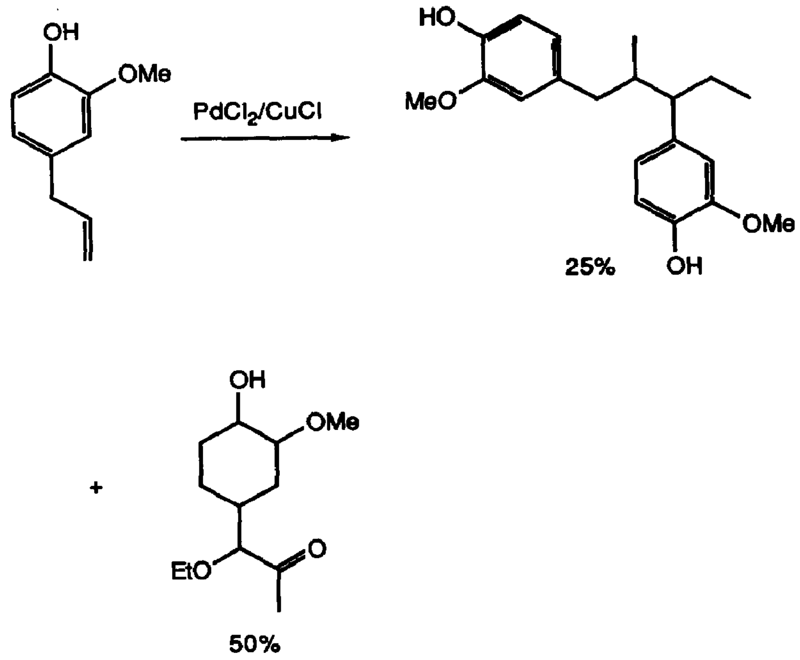


(270)

Coupling of olefins by Group(VIII) transition metals was the topic of a dissertation [313]. Chiral phosphine palladium complexes coupled 1-bromo-2-benzyloxynaphthalene to the binaph with 2 to 23% ee [314]. Palladium acetate/copper acetate coupled alkenylboronates to dienes [315], while palladium chloride coupled vinyl tin reagent to dienes (equation 271) [316]. Palladium/copper salts dimerized an allyl phenol (equation 272) [317].

(271)



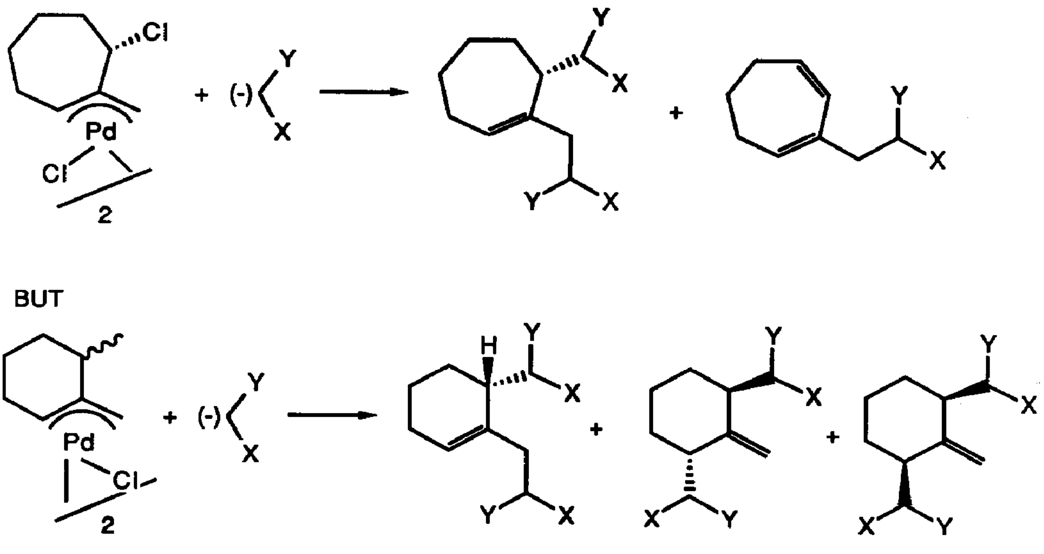


(272)

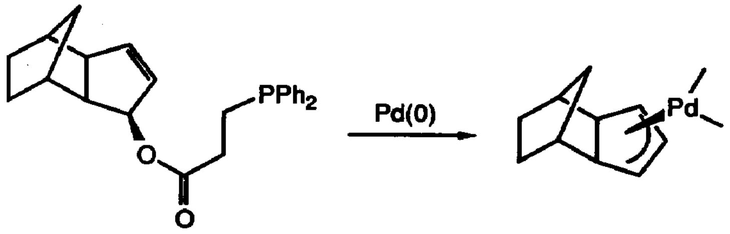
9. Alkylation of π-Allyl Complexes

Selectivity in organic reactions via π-allylpalladium complexes has been reviewed (24 references) [318]. Nucleophilic attack on π-allylpalladium complexes having 2-chloromethyl substitutions was studied (equation 273) [319]. By proper construction of the substrate/*syn* addition of palladium(0) to an allyl acetate was achieved (equation 274) [320]. π-Allyl iron complexes underwent alkylation/acylation (equation 275) [321] and acted as nucleophiles to σ-chloro tropones (equation 276) [322]. Functionalized π-allylnickel complexes were prepared by the insertion of allene into amidonickel complexes (equation 277) [323]. π-Allylmolybdenum complexes were α-alkylated under basic conditions (equation 278) [324]. π-Allyltitanium(III) complexes reacted with cinnamoyl bromide to give cross and homo coupled products [325].

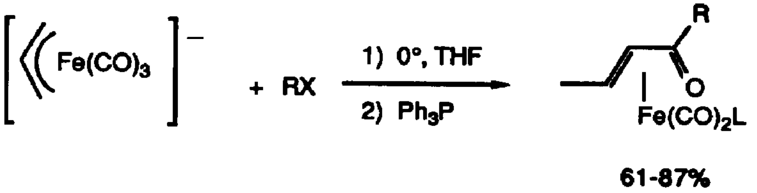
(273)


Cl
(-)
Y
X
Pd
Cl
2
BUT
H

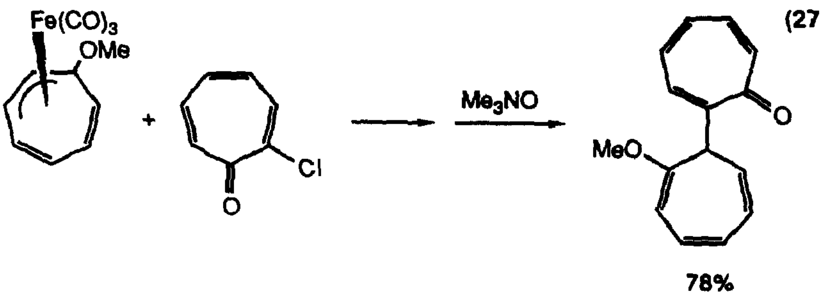
(274)


PPh$_2$
O
Pd(0)
Pd

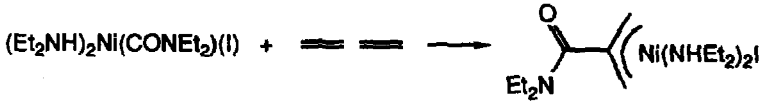
(275)


Fe(CO)$_3$
+ RX
1) 0°, THF
2) Ph$_3$P
R
O
Fe(CO)$_2$L
61-87%

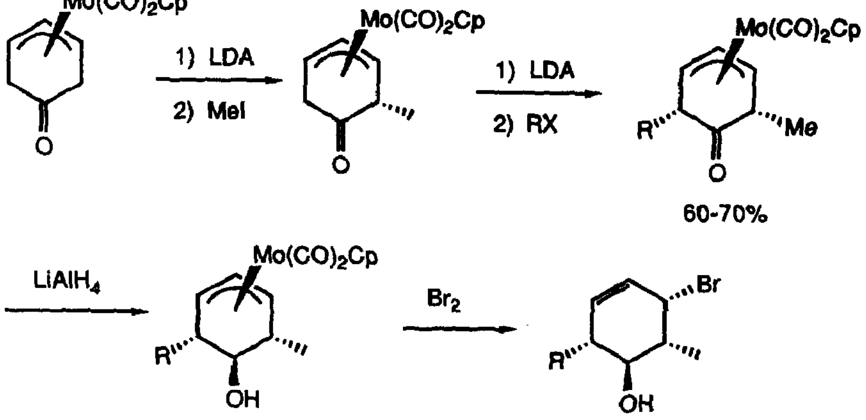
(276)



(277)



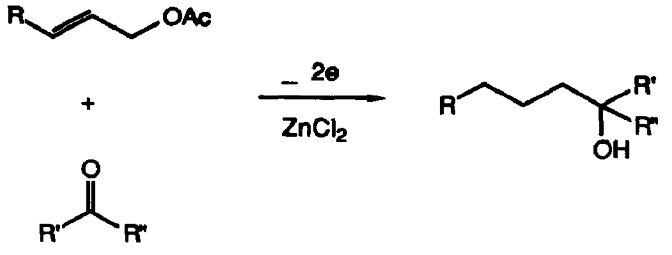
(278)



10. Alkylation of Carbonyl Compounds

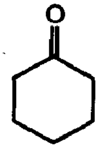
The reductive coupling of aldehydes and ketones to allylic substrates has been developed for several systems including electrochemical coupling with allyl acetates (equation 279) [326] and allyl chlorides (equation 280) [327], chromium(II) coupling with allylic chlorides (equation 281) [328] and alkyl iodides (equation 282) [329]. A variety of allylmetal complexes alkylated aldehydes (equation 283) [330].

(279)

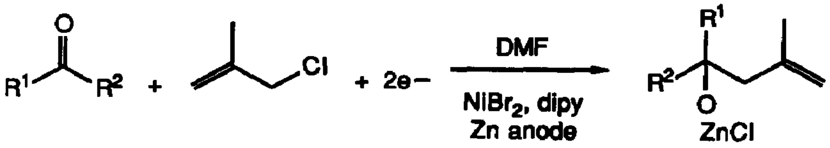


R = H, Ph, Me,
R' = Ph, pMeOPh, pClPh
R" = H

ALSO

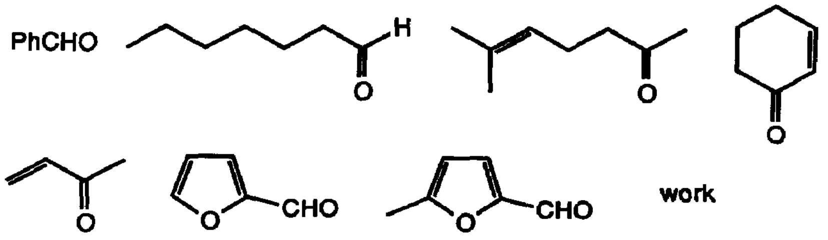


(280)

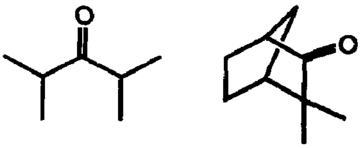


60-80%

PhCHO

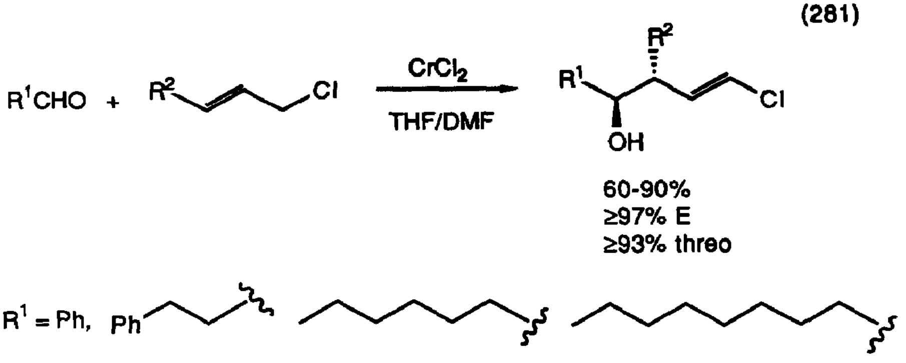


work



fail

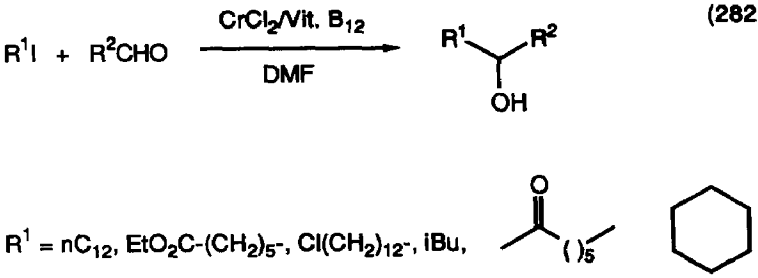
(281)



60-90%
≥97% E
≥93% threo

R^2 = Me, nPr

(282)

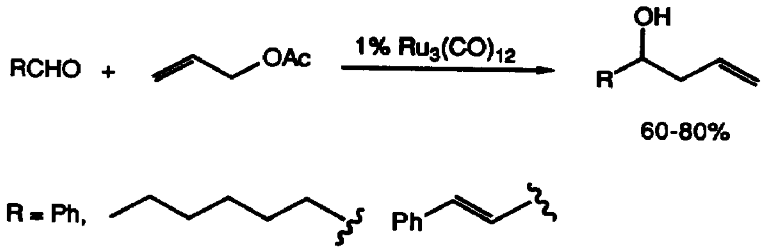


(283)

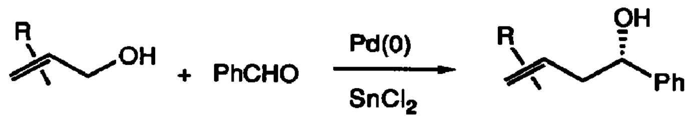
R = Bn, $BnOCH_2$, TBDMS, MeM, Ph_3C

M = MgCl, Cp_2TiCl, Cp_2ZrCl, $Ti(OPh)_3$, CrCl, $CrCl_2$

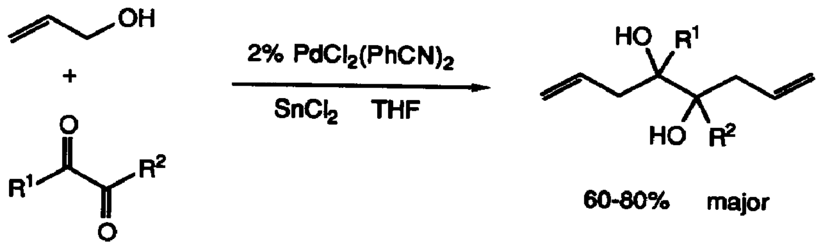
Ruthenium(0) (equation 284) [331], palladium(0) (equation 285) [332], palladium(II) (equation 286) [333] and iron(II) (equation 287) [334] complexes catalyzed the alkylation of aldehydes by allyl systems. Optically active π-allyl molybdenum complexes alkylated aldehydes with high stereoselectivity (equation 288) [335][336].



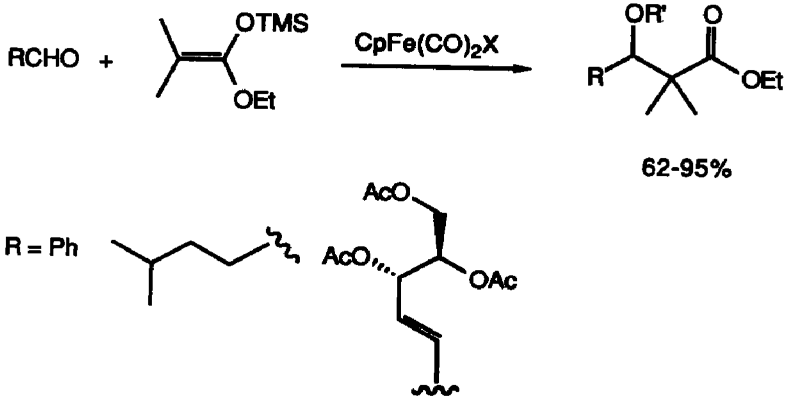
(284)



(285)

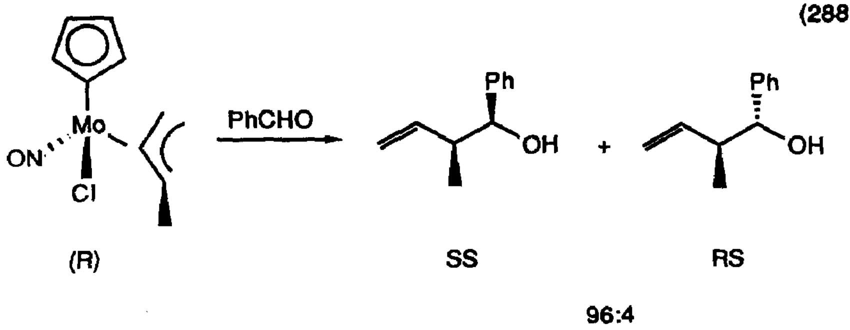


(286)



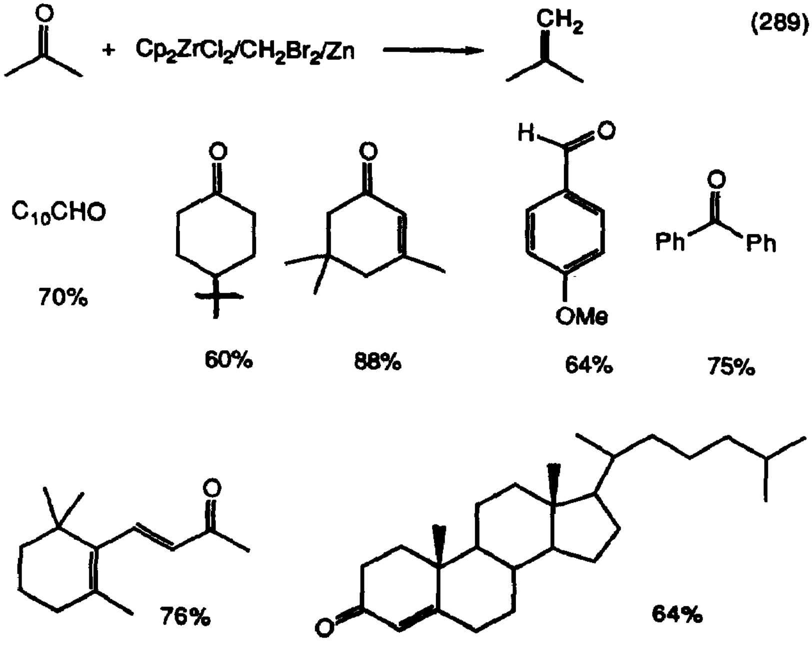
(287)

(288)

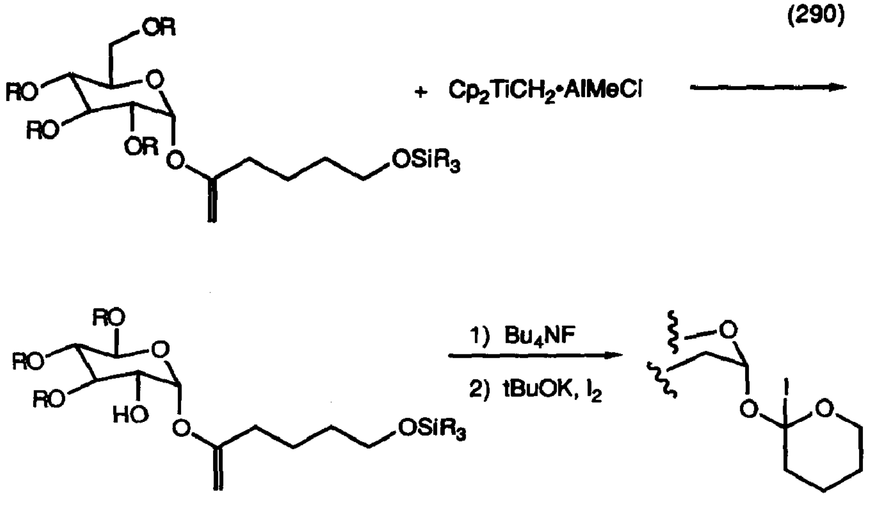


Zirconium (equation 289) [337] and titanium (equation 290) [338] carbenes methylenated carbonyl compounds.

(289)

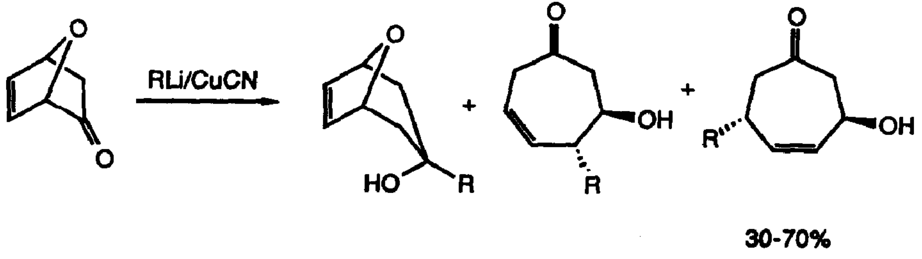


(290)

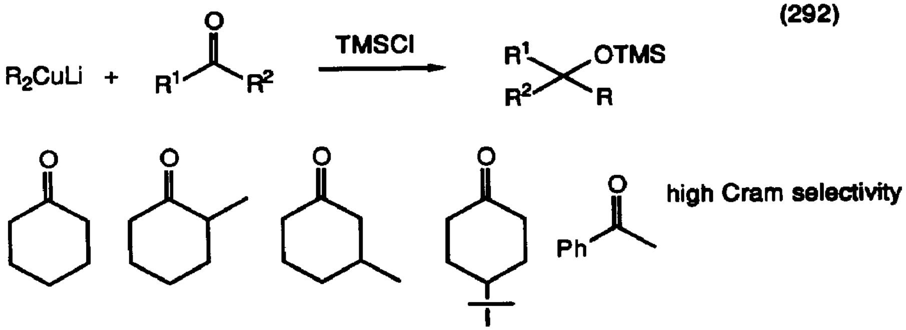


Organocopper complexes alkylated ketones (equation 291) [339], (equation 292) [340], aldehydes (equation 293) [341] and THP esters (equation 294) [342], and condensed α-epoxysulfoxides with ketones (equation 295) [343]. α-Stannylenolates were alkylated by cyanocuprates (equation 296) [344].

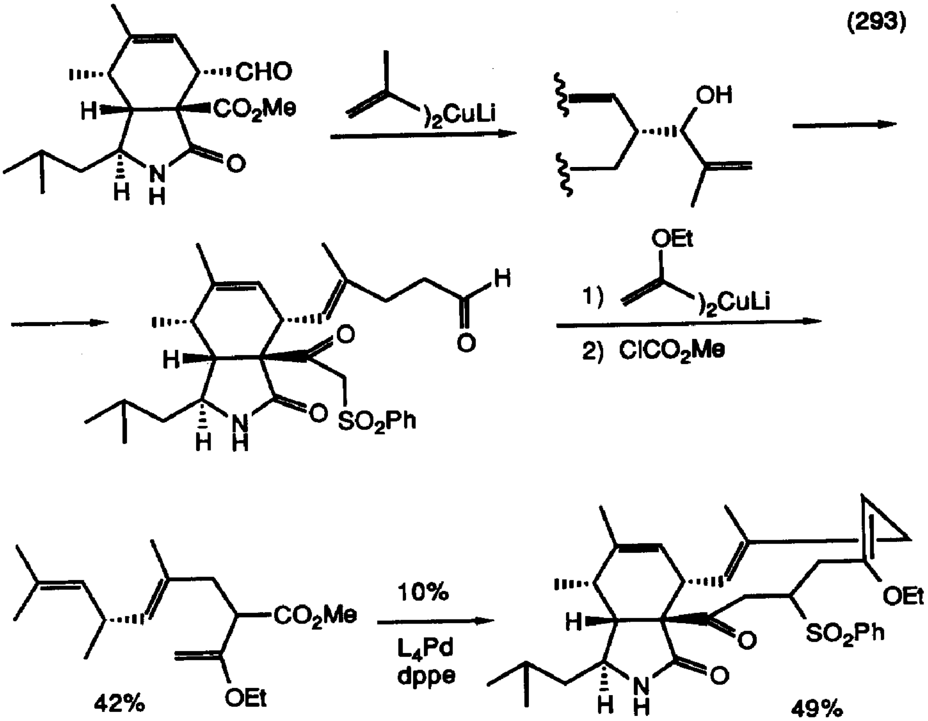
(291)



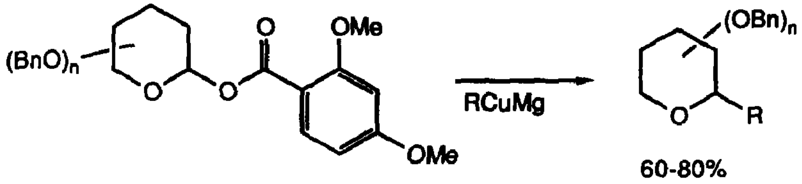
(292)


R₂CuLi
+
R¹
R²
O
TMSCl
R¹
OTMS
R²
R
Ph
high Cram selectivity

(293)

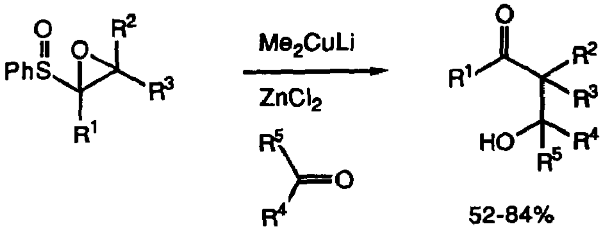

CHO
CO₂Me
H
H
N
H
O
)₂CuLi
OH
O
H
SO₂Ph
OEt
1)
)₂CuLi
2) ClCO₂Me
CO₂Me
42%
10%
L₄Pd
dppe
49%

(294)



R = Bu, nC_8, Ph, Bn

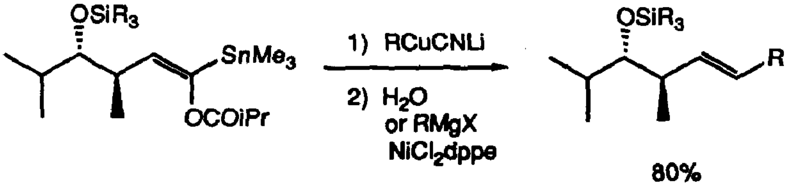
(295)



R^1 = Bn, nC_7, nPr; R^2 = Me, Et, nC_6, nC_5, Ph

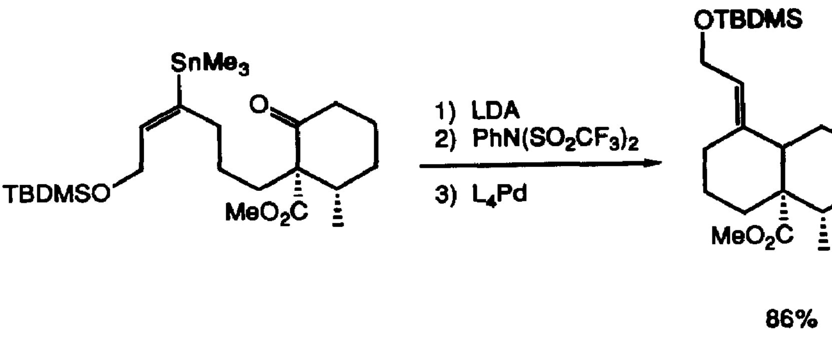
R^3 = H; R^4 = Ph, Me, Et; R^5 = H, Me

(296)

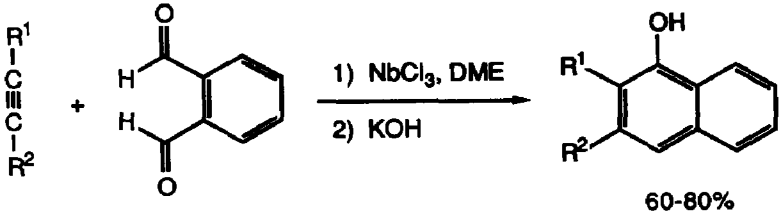


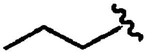
Palladium(0) catalyzed the cyclization shown in equation 297 [345]. Niobium complexes condensed dialdehydes with alkynes (equation 298) [346]. Rhodium enolate complexes catalyzed aldol reactions (equation 299) [347]. Acetylenic aldehydes complexed to cobalt were alkylated by TMS enol ether in the presence of BF_3•etherate (equation 300) [348] [349] [350].

(297)



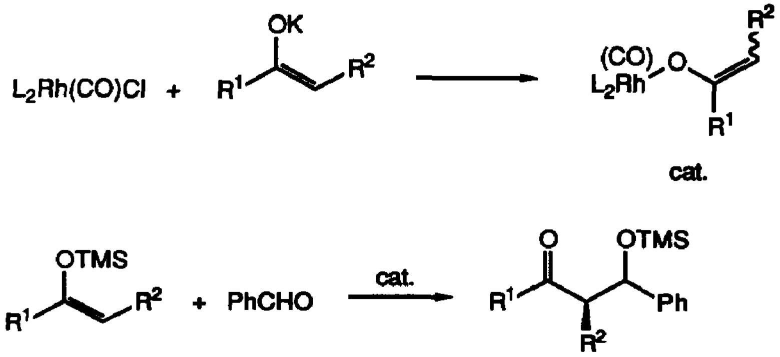
(298)



R^1 = Ph, nPr, pMeOPh, MeO, 

R^2 = Ph, nPr, tBu, TMS

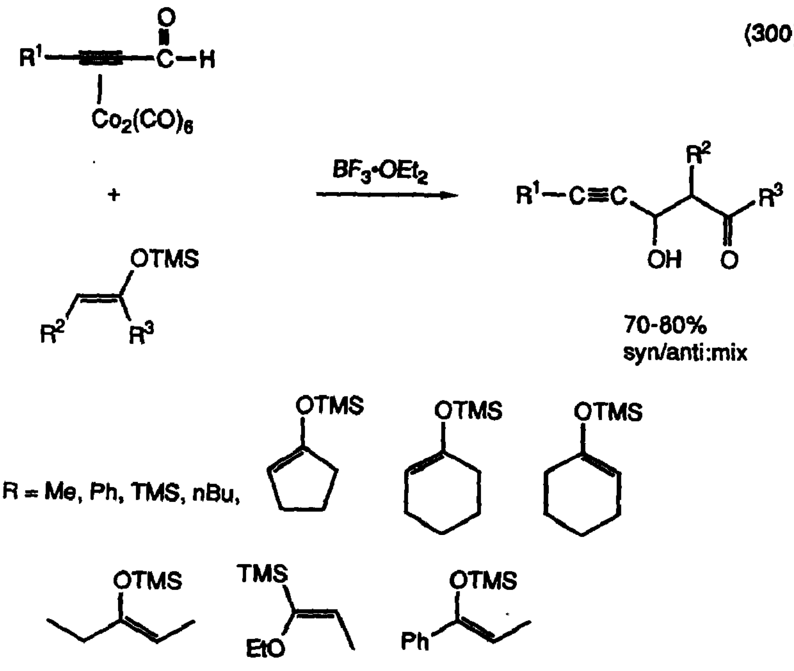
(299)



R^1 = Ph, tBu, OEt

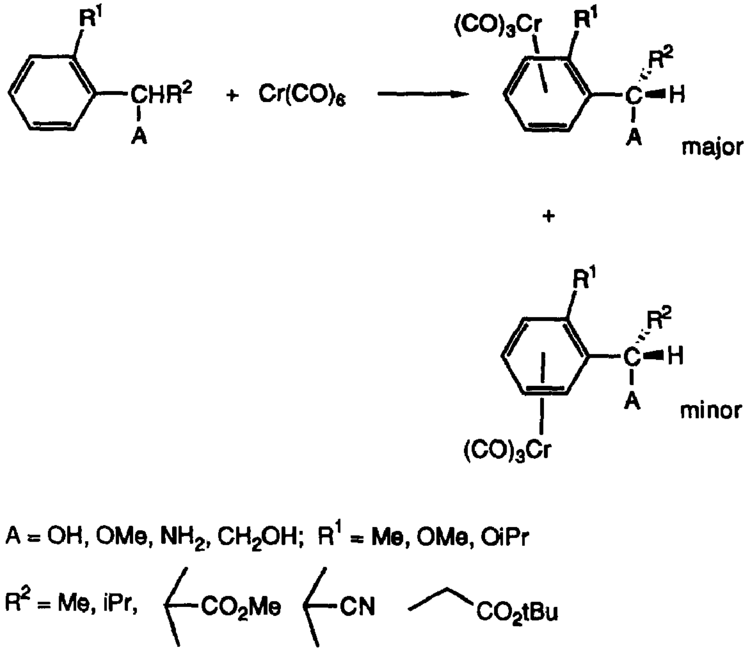
R^2 = H, Me

(300)



11. Alkylation of Aromatic Compounds

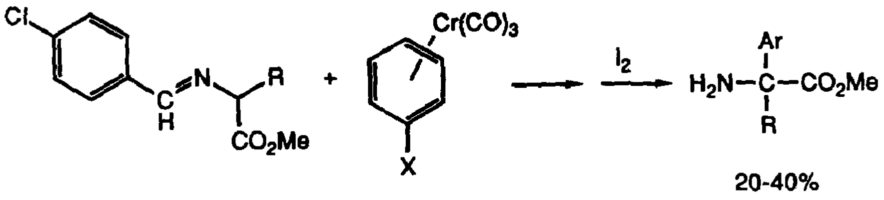
Reviews entitled "Selective reactions on arene chromium complexes" (55 references) [351], "Application of arene chromium tricarbonyl complexes in organic synthesis" (22 references) [352], "Chromium arene carbonyl complexes in asymmetric synthesis" (65 references) [353] and "Arene displacement reactions of [Δ^6-arene] chromium(0) carbonyl complexes" (33 references) [354] have appeared. The diastereoselective complexation of 1,2-disubstituted arenes to chromium has been studied (equation 301) [355].



(301)

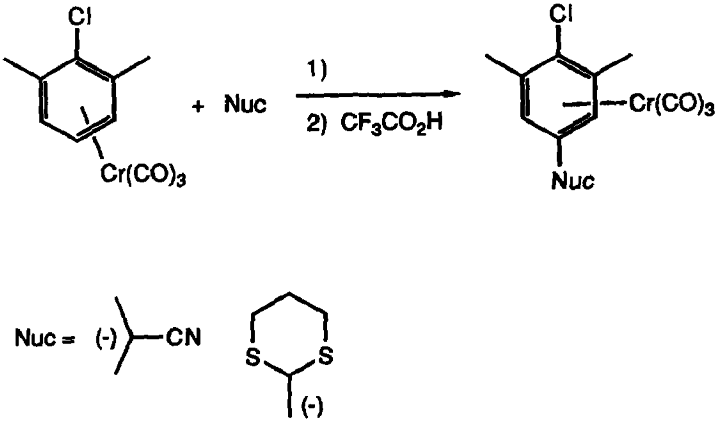
The effect of substituents on the rate of chloride displacement from chlorobenzene chromium tricarbonyl complexes by methoxides was studied [356]. The rate is inversely related to the donating ability of the substituents. Chromium carbonyl complexed aryl halides were alkylated by amino acid enolate equivalent (equation 302) [357]. SNAr nucleophilic aromatic substitution on chromium complexed aryl halides was studied [358] (equation 303) [359], (equation 304) [360]. Palladium catalyzed the alkynylation of dichlorobenzenechromium tricarbonyl by alkynyl stannanes (equation 305) [361].

(302)

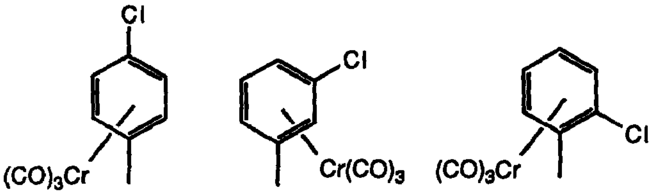


X = F, Cl; R = H, Me, Bn

(303)

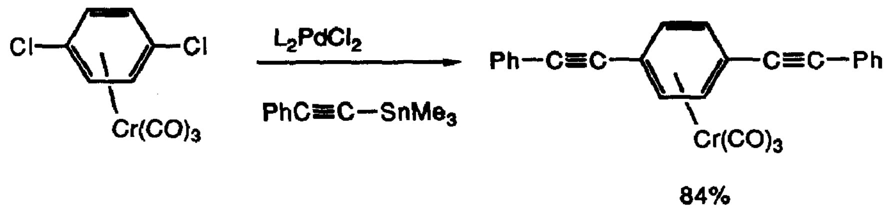


(304)



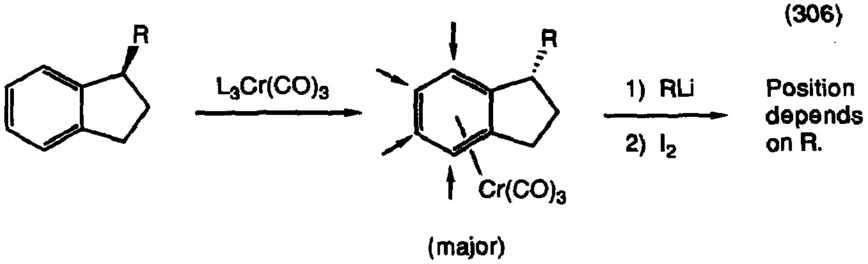
Nucleophile attack at C eclipsed by Cr-CO bond in most stable conformer.

(305)



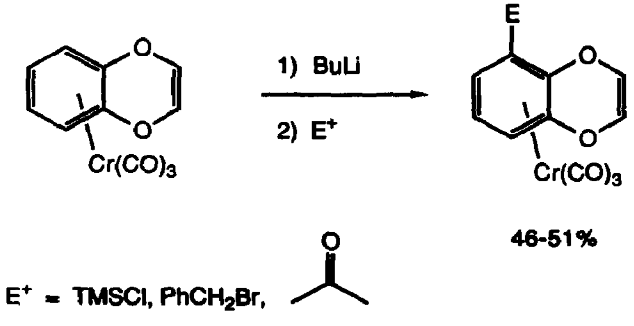
The ortho directing ability for o-lithiation of free arenes was compared to that of chromium tricarbonyl complexed arenes [362]. The order for free arenes was: $CONR_2 > CO_2NHR > NHCOR > CH_2NR_2 > OMe > NMe_2 \sim F$; for complexed: $F > CONHR > NHCOR > CH_2NR_2 = OMe >> CH_2OMe$. The site of alkylation of chromium complexed indones depended on conditions and substituents (equation 306) [363]. Complexed, protected o-hydroquinones were lithiated and allylated (equation 307) [364]. Complexed indole was lithiated and alkylated first in the 2-position then in the 7-position (equation 308) [365]. Chromium-complexed pyridine, prepared in a sneaky way, underwent lithiation and alkylation (equation 309) [366].

(306)

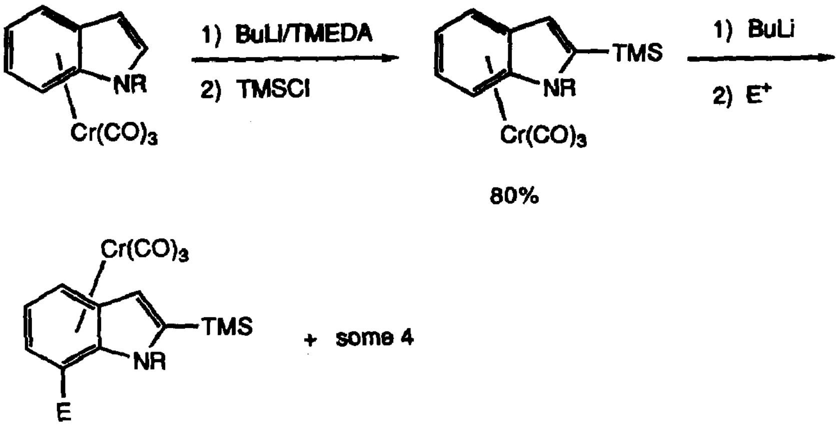


$R = OH, OMe, OSiR_3$

(307)



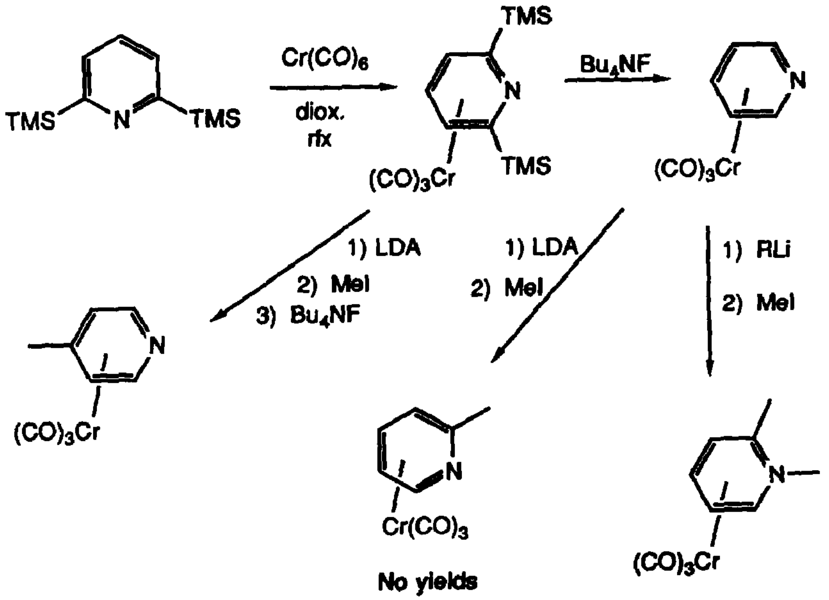
(308)



R^1 = TMS, H

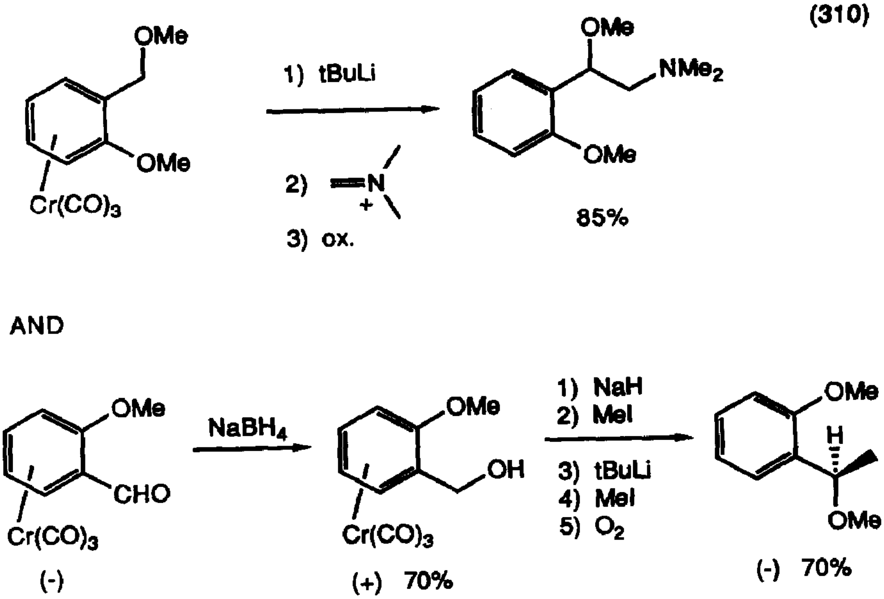
E$^+$ = MeI, DMF, TMSCl, EtO$_2$CCl, Me$_3$SnCl, 

(309)

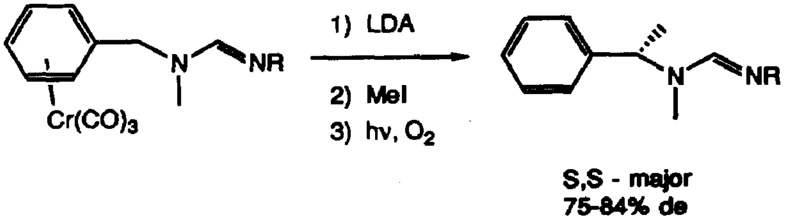


Side chain reactions of complexed, substituted arenes was very popular this year. The reactions studied included benzylic alkylation (equation 310) [367], (equation 311) [368], (equation 312) [369], conjugate addition (equation 313) [370], allylic rearrangement (equation 314) [371], Grignard reactions (equation 315) [372], Friedel Crafts alkylation (equation 316) [373], benzylic displacement (equation 317) [374], and remote side chain stereocontrol (equation 319) [375] [376].

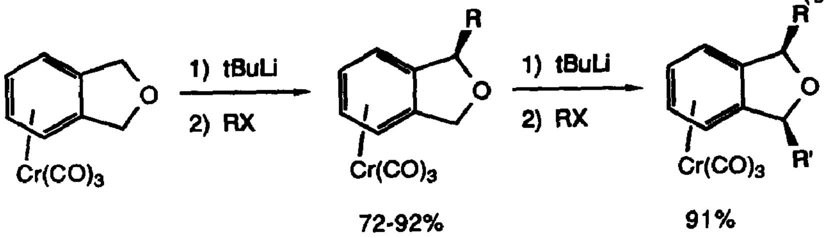
(310)



(311)



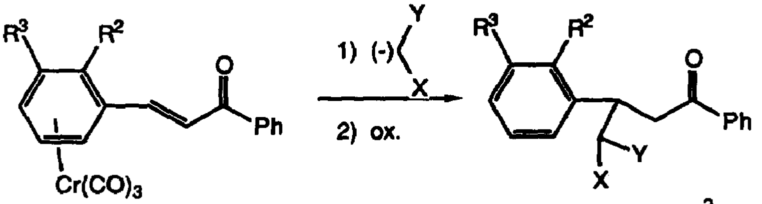
(312)



R = Me, Et, Bn, D

R' = Me, D

(313)

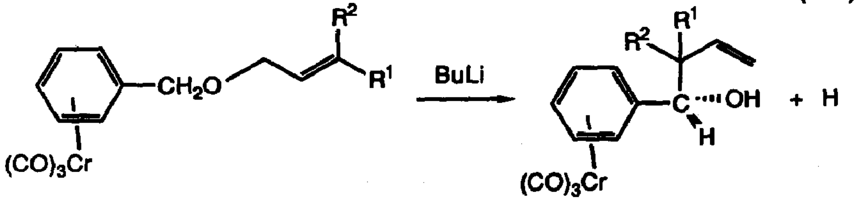


(enantiomers separated)

~100% de when $R^2 \neq H$

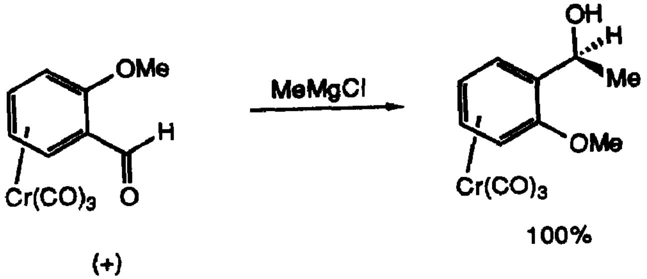
R^1 = H, Me, OMe, NMe$_2$, Cl
R^2 = Me, OMe, OEt, NMe$_2$, Cl, H

(314)

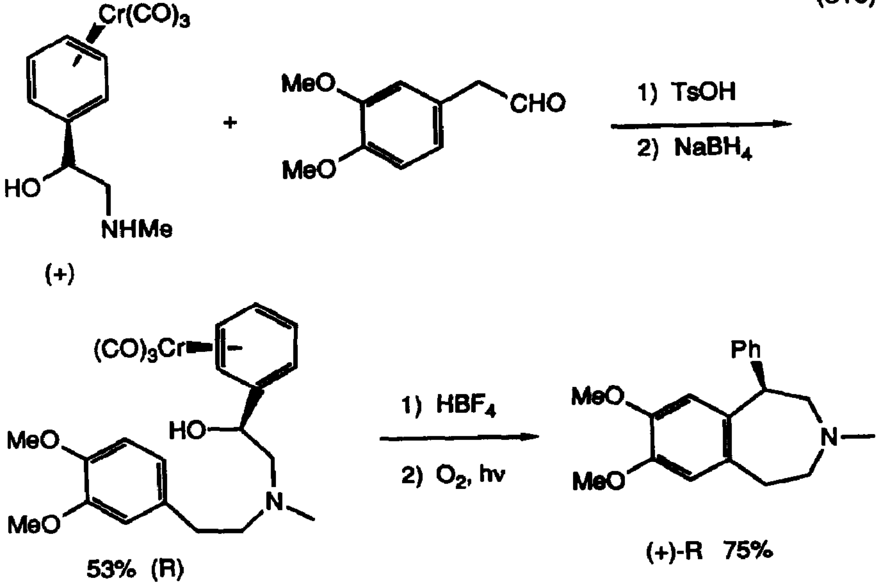


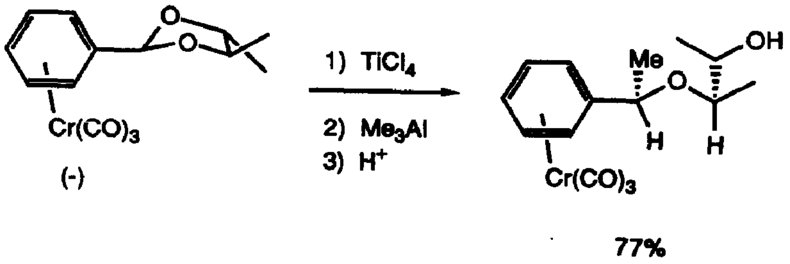
2-erythro
A, R^1, R^2 = H, Me 82-90%
A = OMe; R^1, R^2 = H 16-30%

(315)

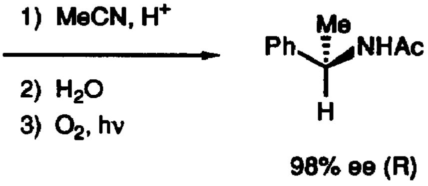

OMe
H
Cr(CO)3
O
(+)
MeMgCl
OH
H
Me
OMe
Cr(CO)3
100%

(316)

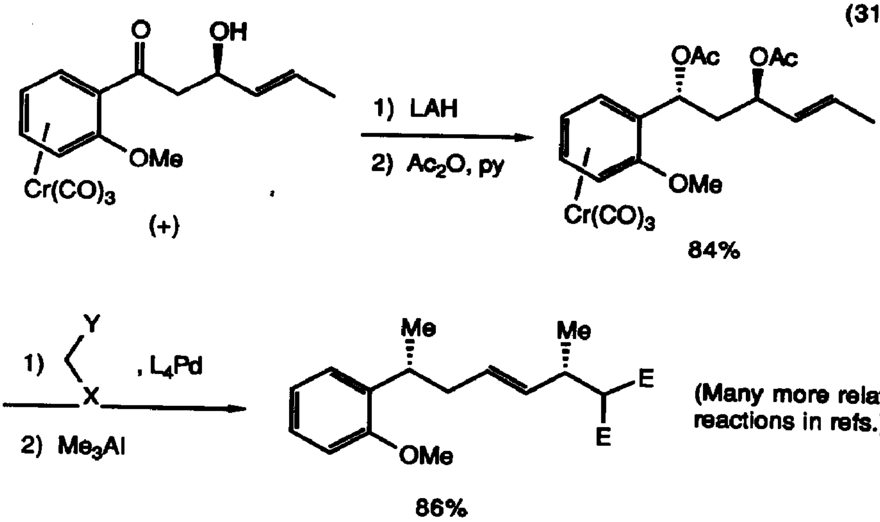

Cr(CO)3
HO
NHMe
(+)
+
MeO
MeO
CHO
1) TsOH
2) NaBH4
(CO)3Cr
HO
MeO
MeO
N
53% (R)
1) HBF4
2) O2, hv
Ph
MeO
MeO
N
(+)-R 75%


O
O
Cr(CO)3
(-)
1) TiCl4
2) Me3Al
3) H+
Me
O
OH
H
H
Cr(CO)3
77%

(317)

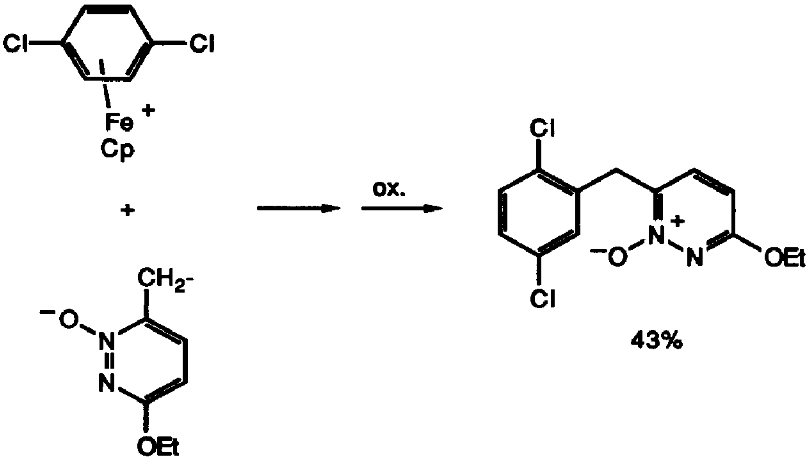


(318)

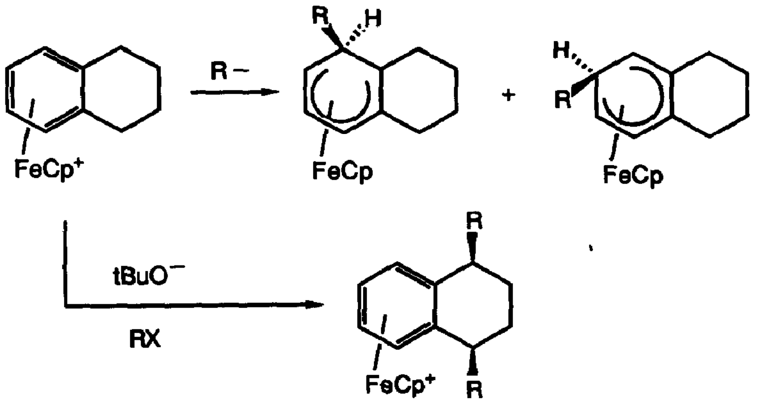


(Many more related reactions in refs.)

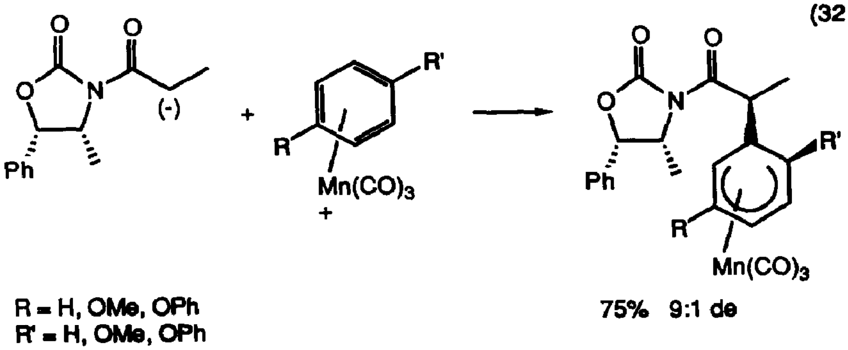
(319)



Iron arene complexes also underwent nucleophilic attack (equation 319) [377] and lithiation (equation 320) [378]. Cationic manganese arene complexes were similarly alkylated by anions (equation 321) [379], (equation 322) [380]. Manganese(III) acetate promoted a radical alkylation of an arene (equation 323) [381]. Indole cuprates were alkylated by a variety of electrophiles (equation 324) [382].

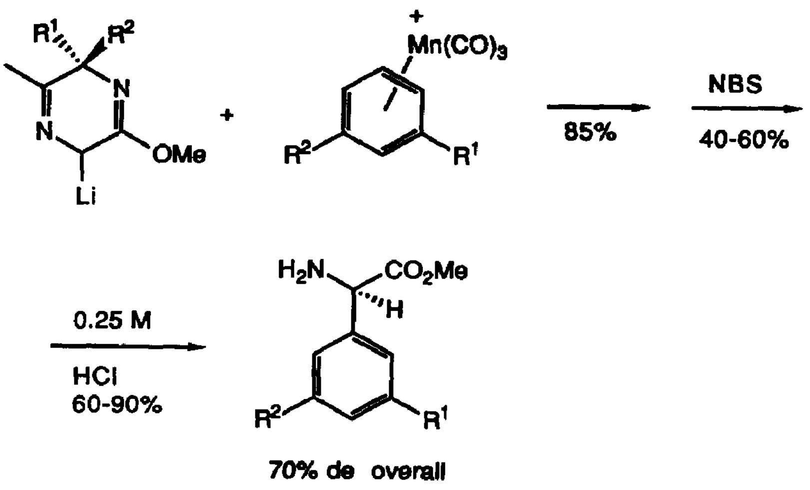


(320)

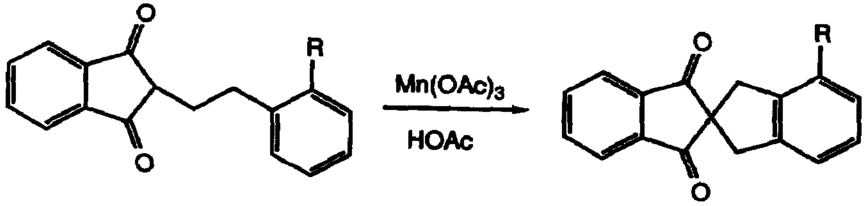


(321)

(322)

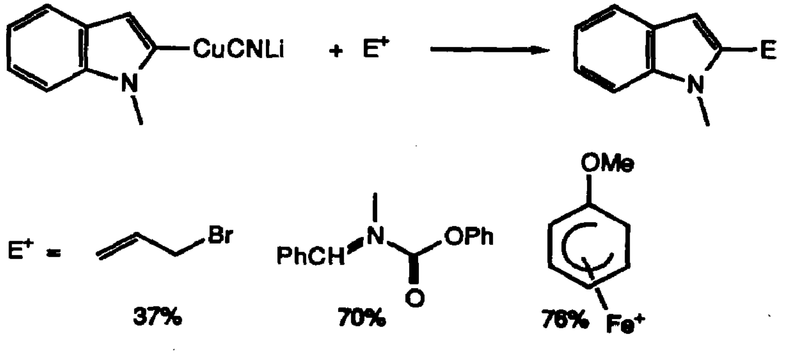


(323)



R = H 0%
R = OMe 33%
R = OBn 25%

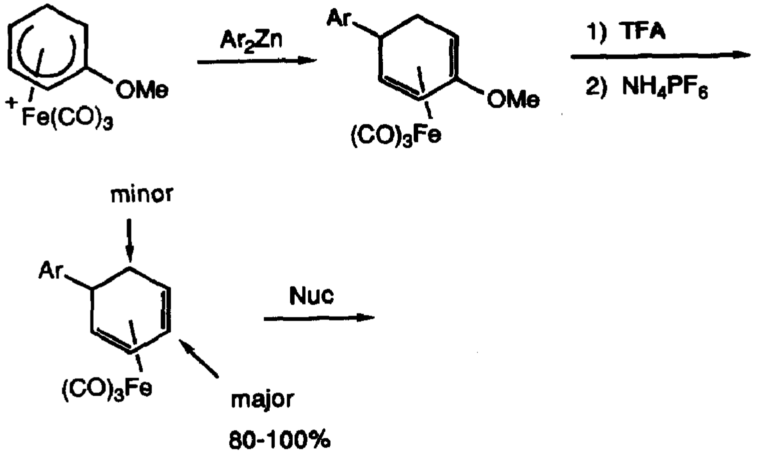
(324)



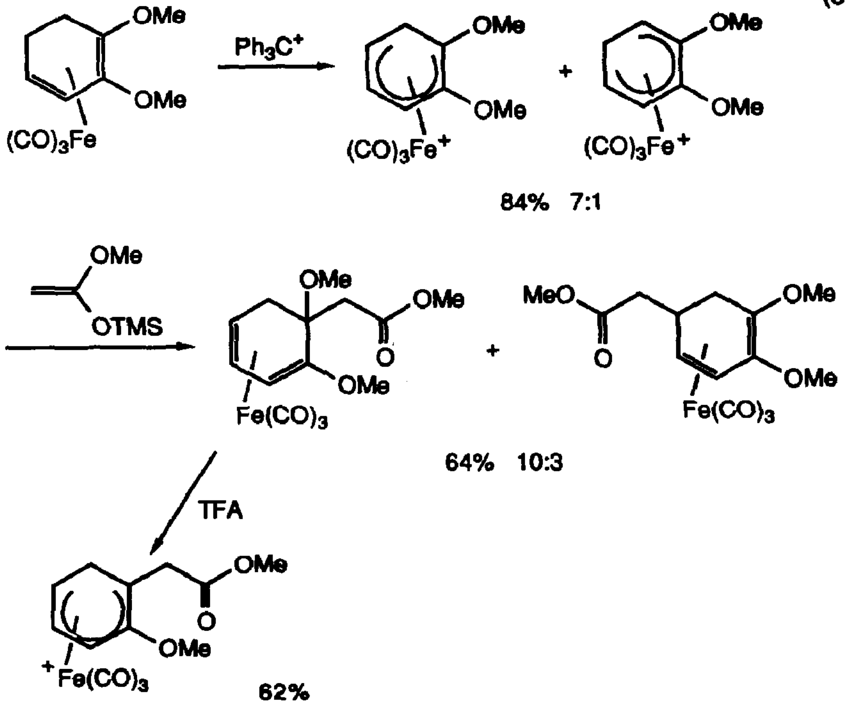
12. Alkylation of Dienyl and Diene Complexes

Alkylation of cationic cyclohexadienyliron continues to provide regioselective approaches to cyclohexyl systems (equation 325) [383], (equation 326) [384], (equation 327) [385], (equation 328) [386] and has been used in the total synthesis of several polycyclic natural products (equation 329) [387], (equation 330) [388], (equation 331) [389].

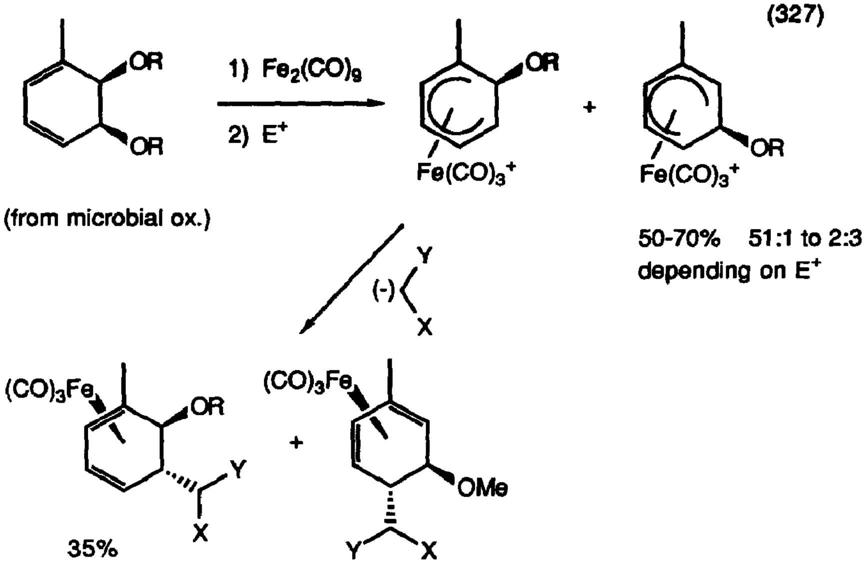
(325)



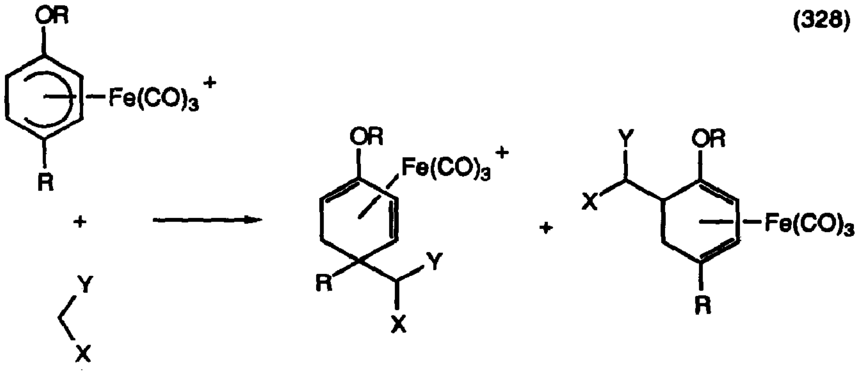
(326)



(327)

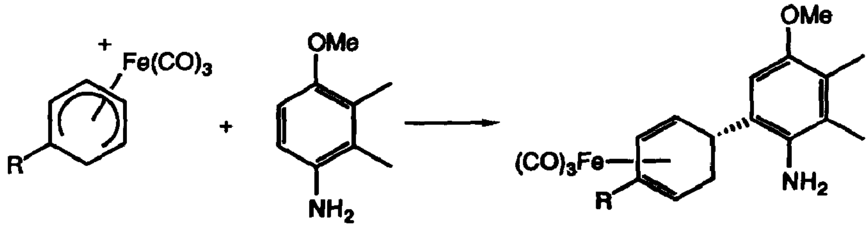


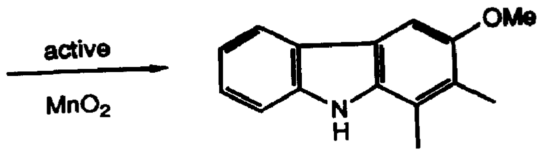
(328)



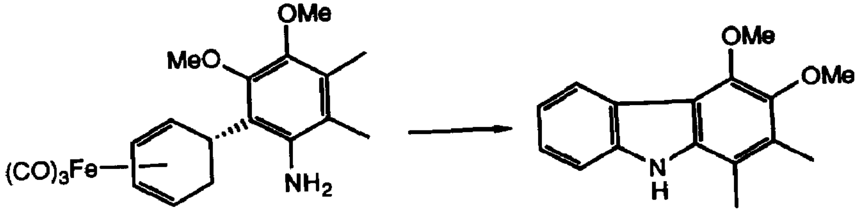
(Studied factors controlling regiochemistry.)

(329)

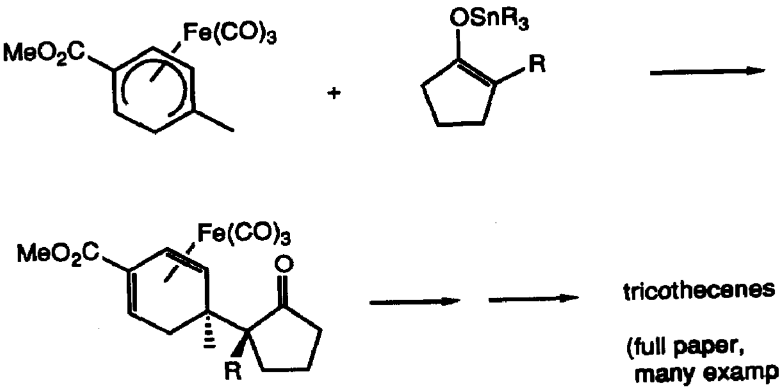




AND



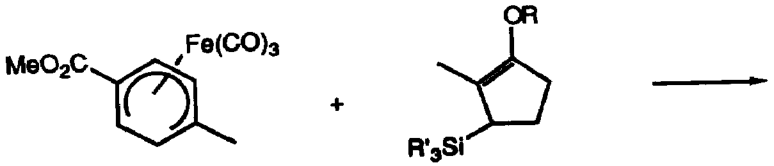
(330)

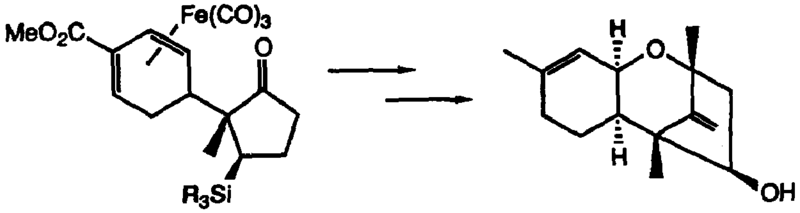


tricothecenes

(full paper,
many examples)

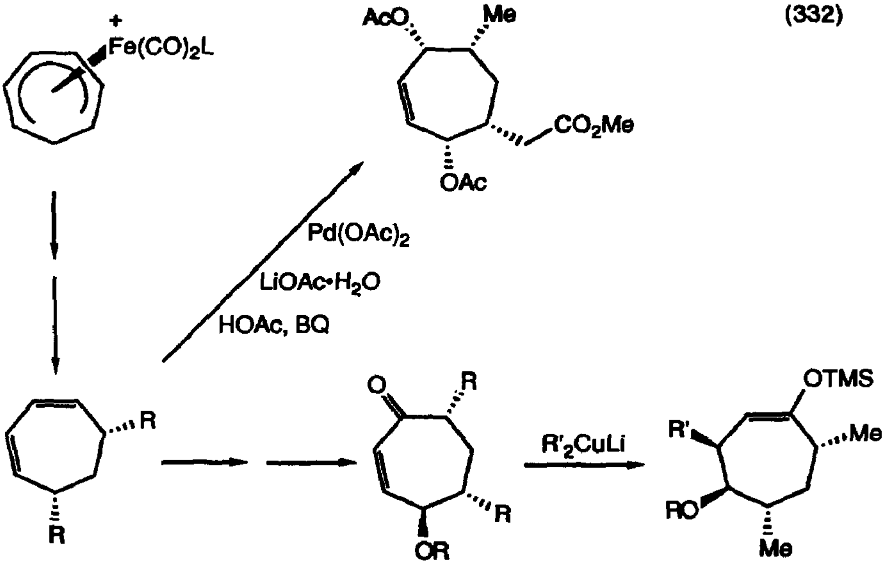
(331)



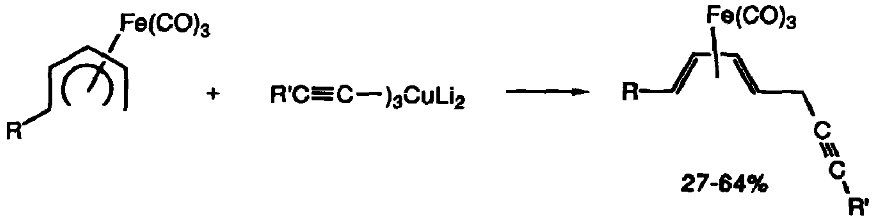


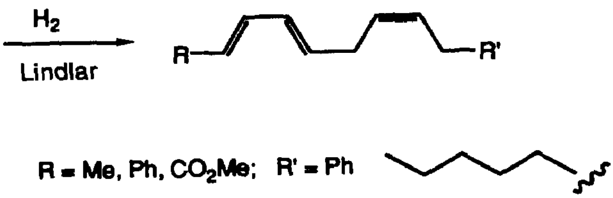
These reactions were not restricted to cyclohexadienyl systems. Cycloheptadienyl (equation 332) [390] and open chain hexadienyl (equation 333) [391], (equation 334) [392] also underwent alkylation.

(332)

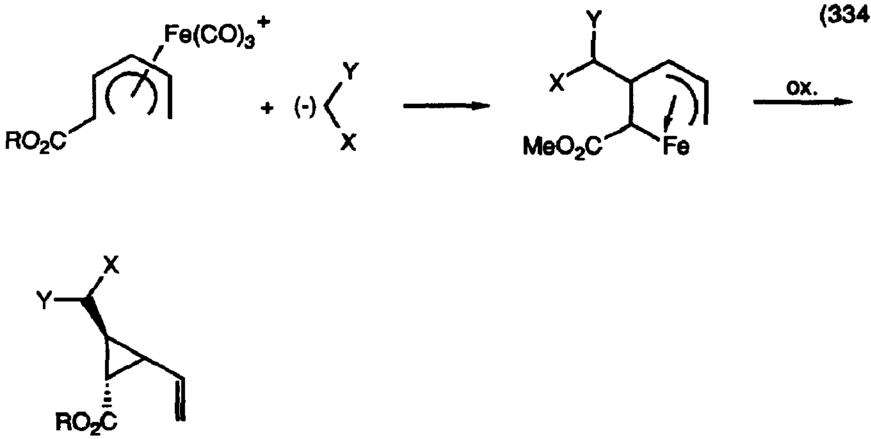


(333)



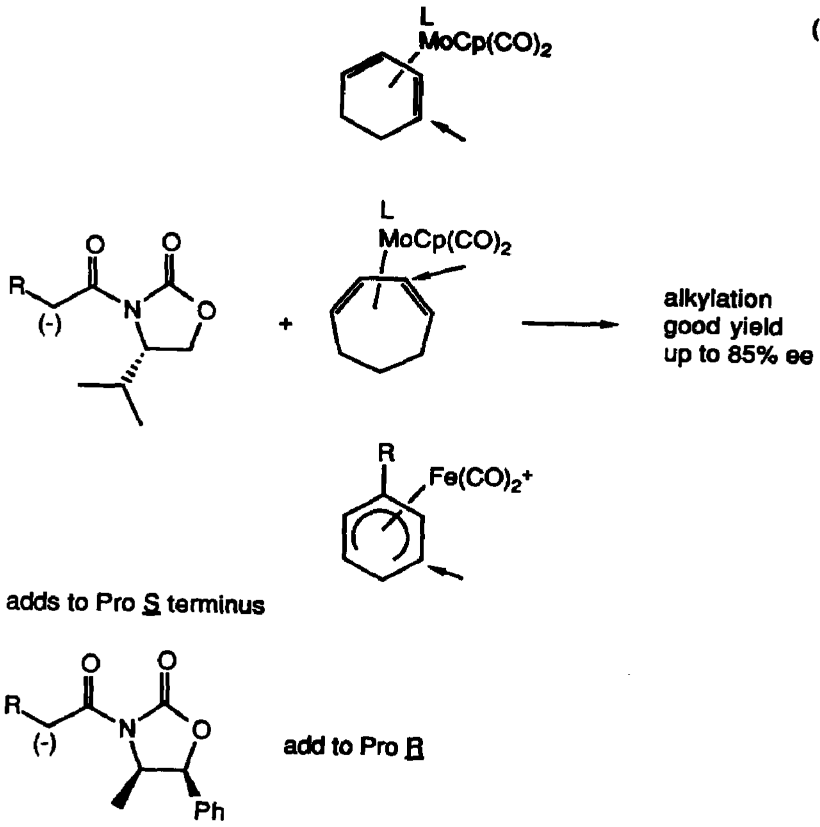


(334)

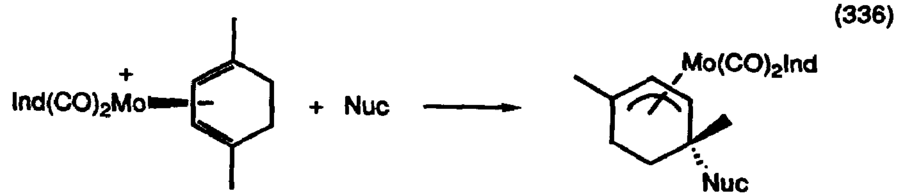


Cationic molybdenum diene complexes were similarly alkylated (equation 335) [393], (equation 336) [394], (equation 337) [395]. In contrast, deprotonation led to η^3-allyl complexes which were lithiated then alkylated (equation 338) [396]. Acyclic cationic molybdenum diene complexes also underwent alkylation (euqation 339) [397].

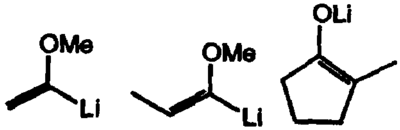
(335)



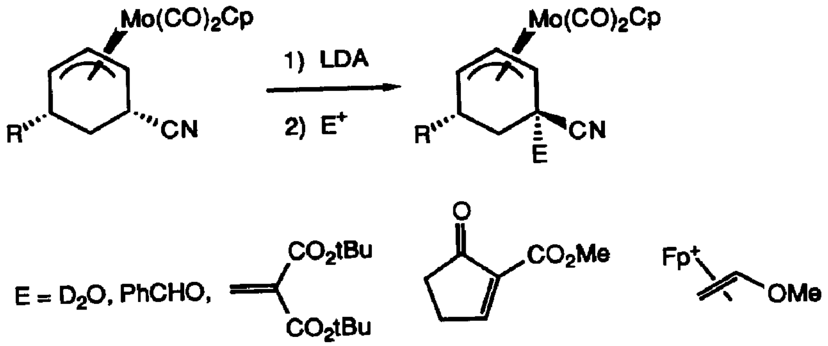
(336)



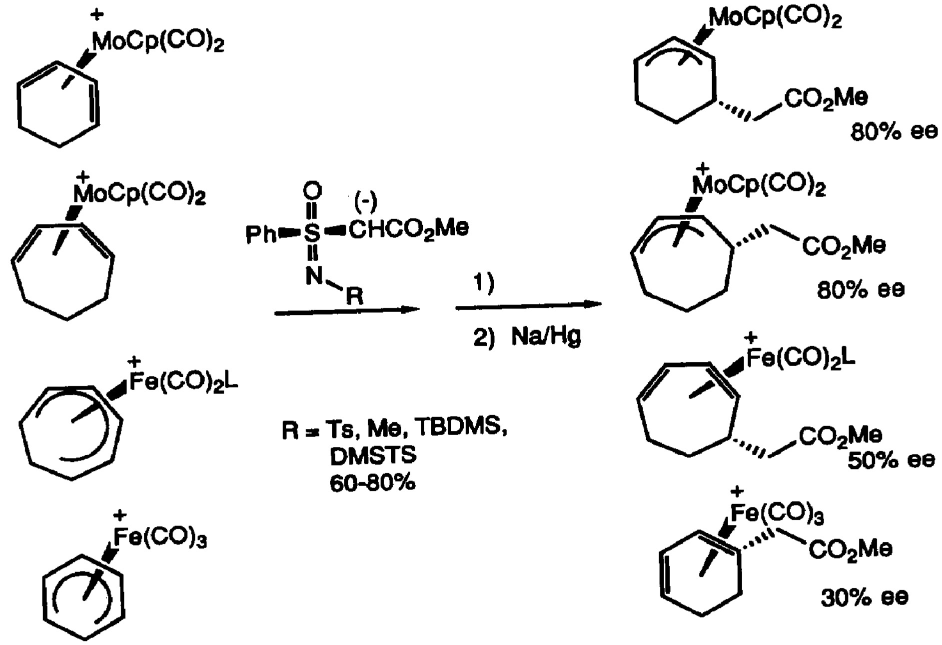
Nuc^- = CN, $CH(CO_2Me)_2$, MeMgBr, PhMgBr,



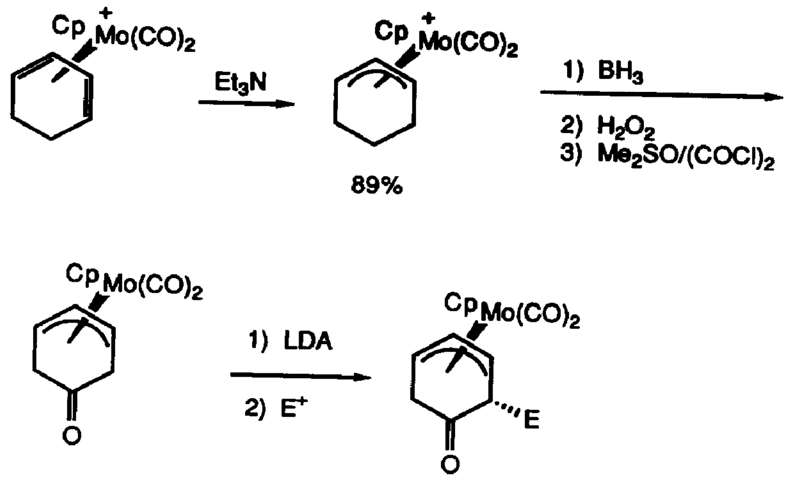
AND


Mo(CO)2Cp
R
CN
1) LDA
2) E+
Mo(CO)2Cp
R
CN
E
E = D2O, PhCHO,
CO2tBu
CO2tBu
O
CO2Me
Fp+
OMe

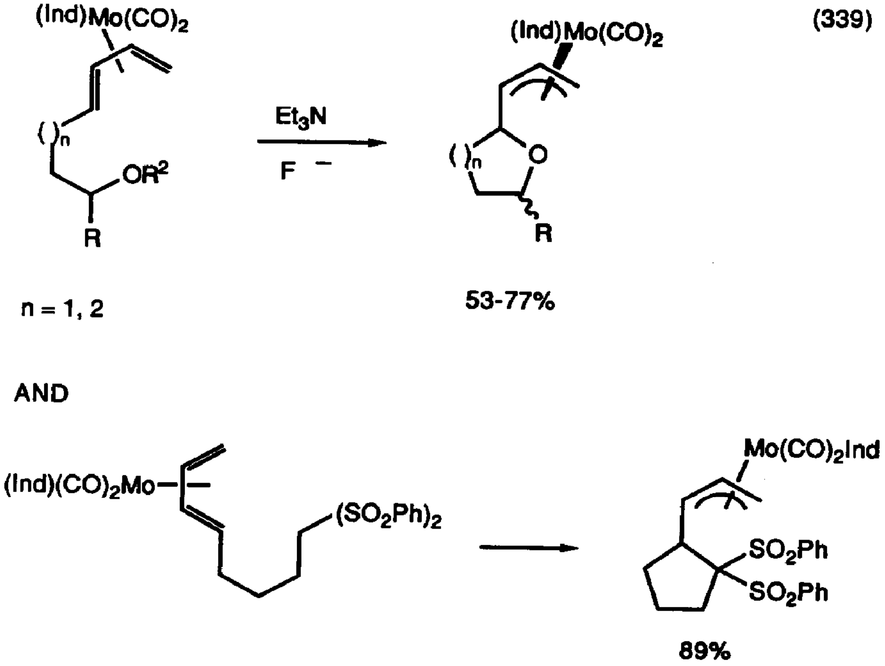
(337)


+ MoCp(CO)2
+ MoCp(CO)2
+ Fe(CO)2L
+ Fe(CO)3
O
Ph S (-) CHCO2Me
N R
1)
2) Na/Hg
R = Ts, Me, TBDMS, DMSTS
60-80%
+ MoCp(CO)2
CO2Me
80% ee
+ MoCp(CO)2
CO2Me
80% ee
+ Fe(CO)2L
CO2Me
50% ee
+ Fe(CO)3
CO2Me
30% ee

(338)

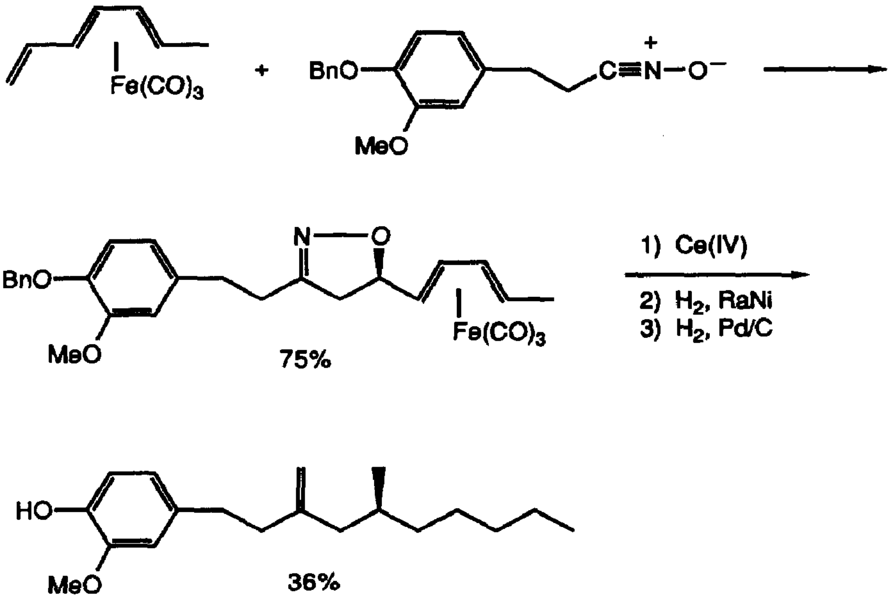

Cp
Mo(CO)₂
Et₃N
89%
1) BH₃
2) H₂O₂
3) Me₂SO/(COCl)₂
1) LDA
2) E⁺
O
E

(339)

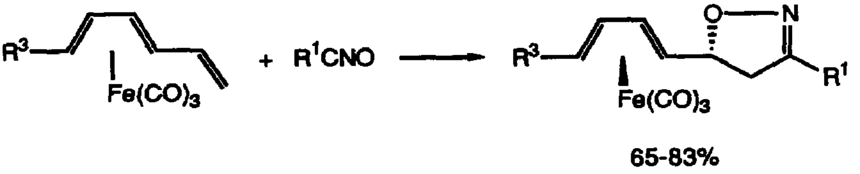

(Ind)Mo(CO)₂
( )n
OR²
R
Et₃N
F ⁻
O
n = 1, 2
53-77%
AND
(Ind)(CO)₂Mo
(SO₂Ph)₂
Mo(CO)₂Ind
SO₂Ph
SO₂Ph
89%

Functionalized 1,3-dienes were protected in their Fe(CO)$_3$ complexes, permitting 1,3-dipolar cycloaddition reactions (equation 340) [398], (equation 341) [399], acylation (equation 342) [400] and Wittig reactions (equation 343) [401], (equation 344) [402] to ensue without complication from the diene. Thermolysis of unsaturated iron diene complexes led to cyclization (equation 345) [403].

(340)



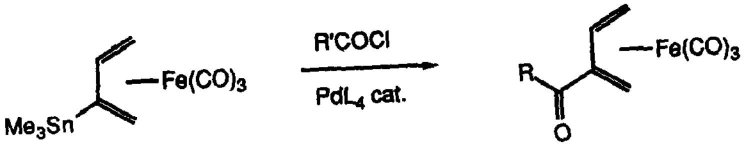
(341)



R^1 = Me, Et, tBu, Ph

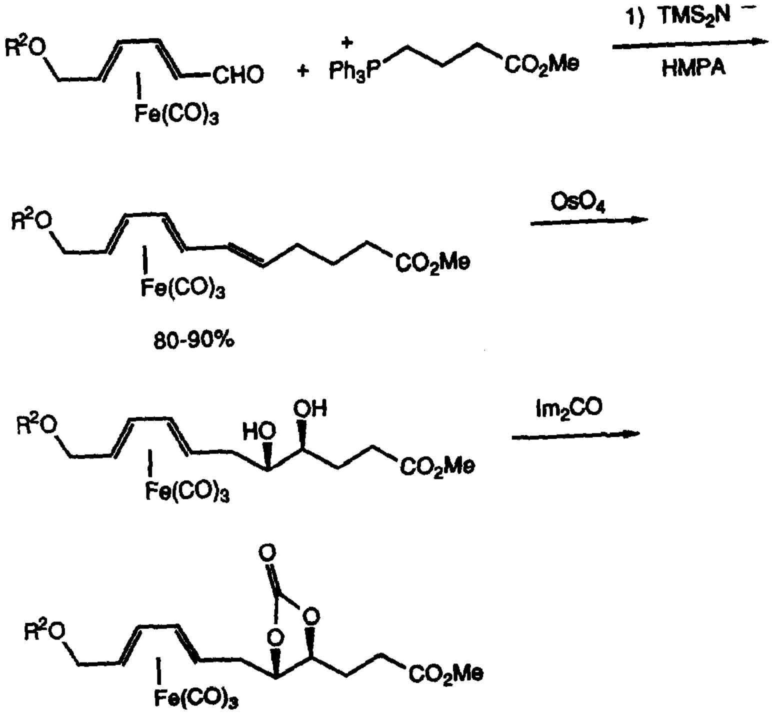
R^3 = Me, R_3SiOCH_2, CO_2Me

(342)

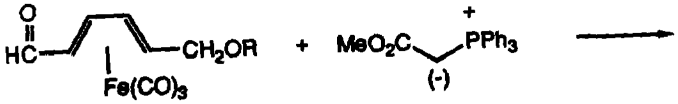


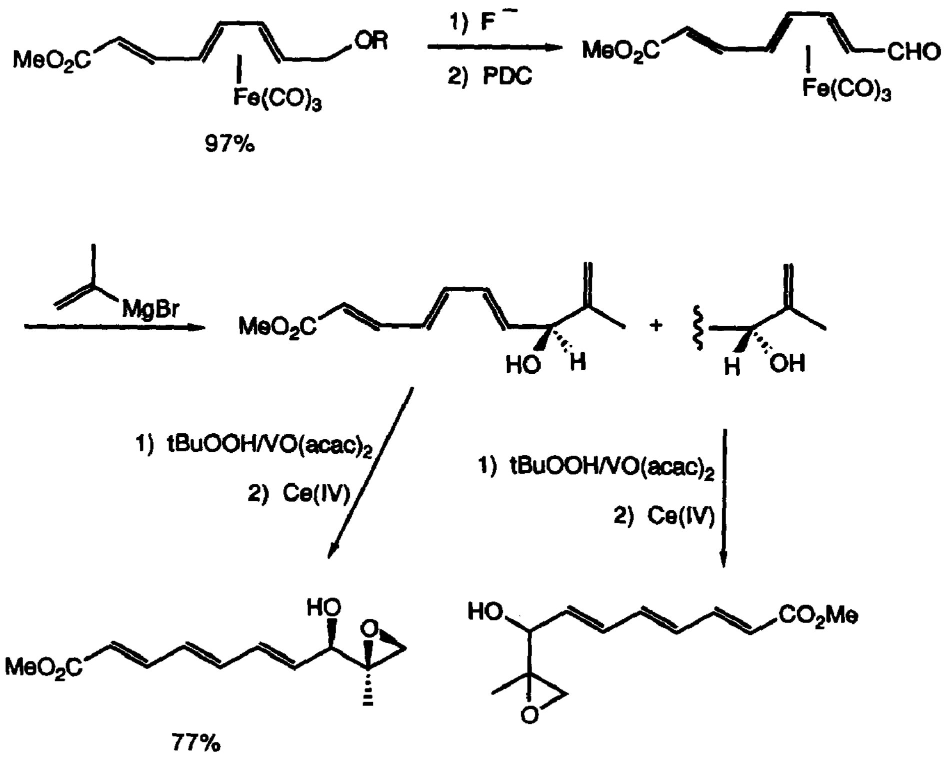
30-60%

(343)

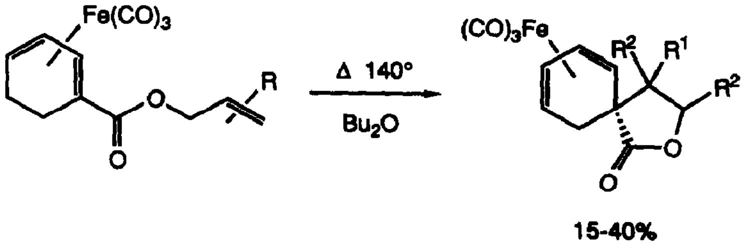


(344)

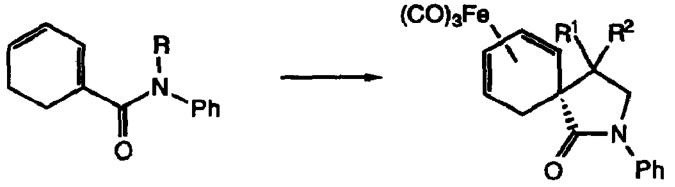



MeO$_2$C
OR
Fe(CO)$_3$
97%
1) F$^-$
2) PDC
MeO$_2$C
CHO
Fe(CO)$_3$
MgBr
MeO$_2$C
HO
H
+
H
OH
1) tBuOOH/VO(acac)$_2$
2) Ce(IV)
1) tBuOOH/VO(acac)$_2$
2) Ce(IV)
HO
MeO$_2$C
77%
HO
CO$_2$Me
O
O

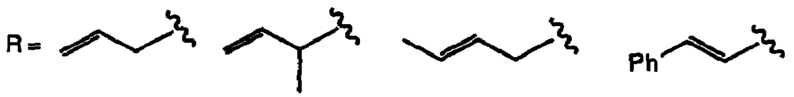
(345)


Fe(CO)$_3$
R
O
O
Δ 140°
Bu$_2$O
(CO)$_3$Fe
R^2
R^1
R^2
O
O
15-40%

AND

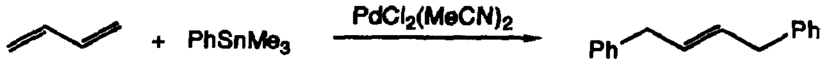


80-90%

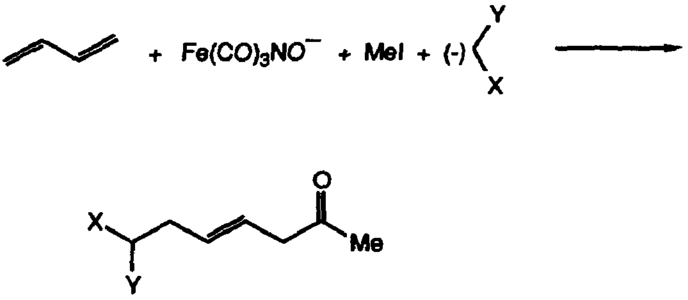


Palladium(II) chloride catalyzed the 1,4-diarylation of butadiene by phenyl tin reagents (equation 346) [404]. Iron nitrosyl tricarbonyl was used to 1,4-functionalize 1,3-dienes (equation 347) [405]. The bis-chromium arene complex of 1,4-diphenylbutadiene was dialkylated by reductive processes (equation 348) [406]. Cationic cobalt diene complexes were also difunctionalized (equation 349) [407].

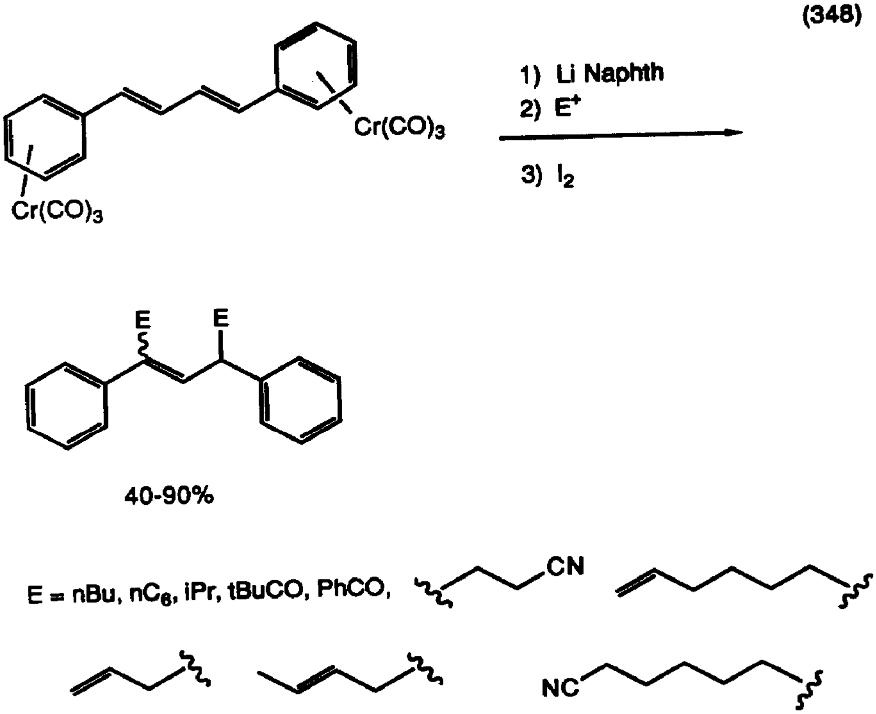
(346)



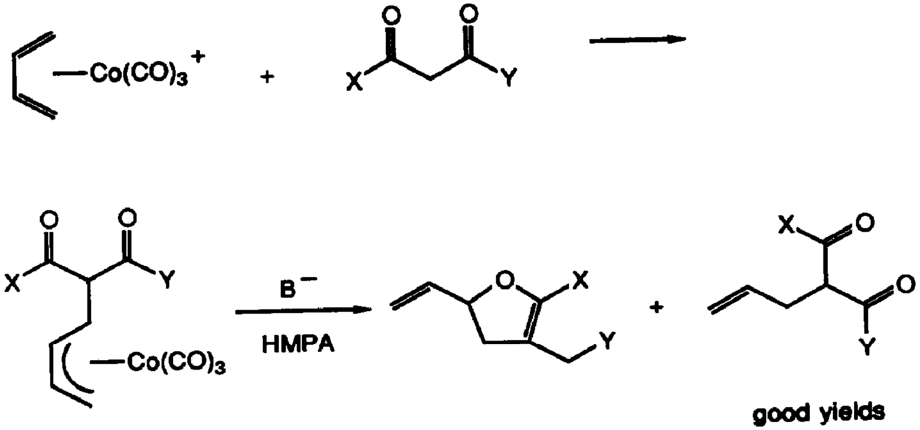
(347)



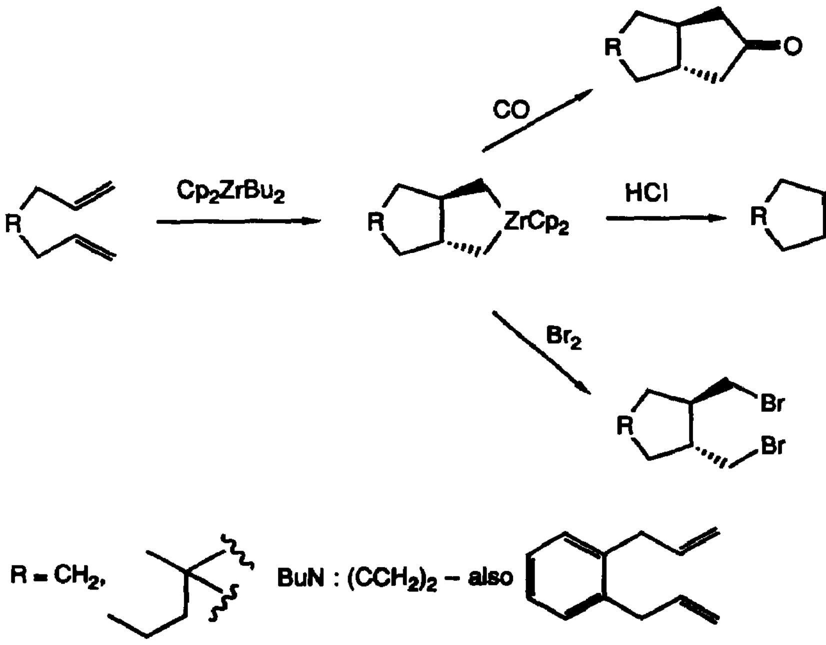
(348)


Cr(CO)3
Cr(CO)3
1) Li Naphth
2) E+
3) I2
E
E
40-90%
E = nBu, nC6, iPr, tBuCO, PhCO,
CN
NC

(349)

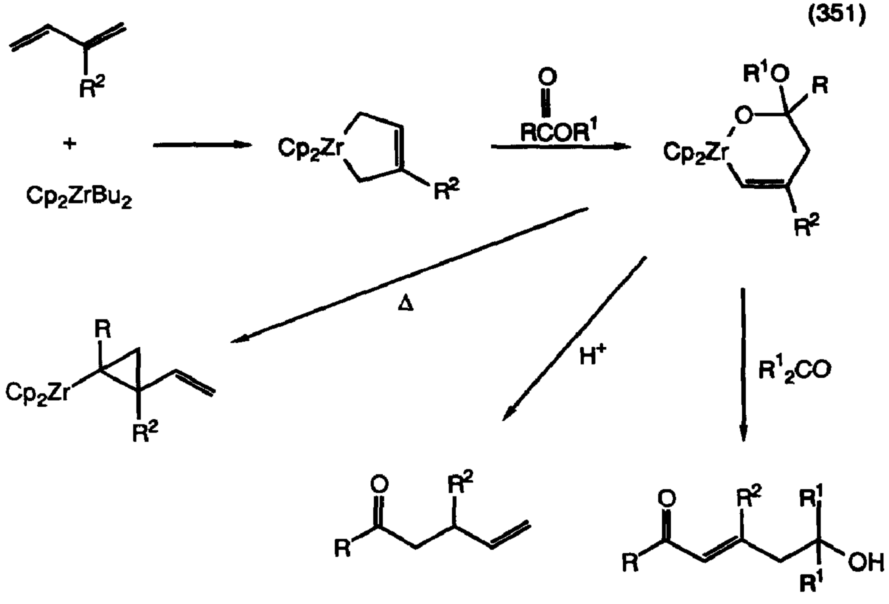

Co(CO)3+
+
O
O
X
Y
O
O
X
Y
Co(CO)3
B−
HMPA
O
X
Y
+
X
O
O
Y
good yields

Dicyclopentadienylzirconium complexes underwent reaction with dienes to give metallocycles which were further elaborated (equation 350) [408], (equation 351), (equation 352), (equation 353) [409], (equation 354) [410]. Palladium(II) complexes catalyzed the cyclofunctionalization of tetraenes (equation 355) [411]. Nickel(0) complexes catalyzed the alkylation of dienoic esters with butadienes (equation 356) [412]. Palladium(0) complexes catalyzed the coreaction of allenes, vinyl halides and stabilized carbanions (equation 357) [413].

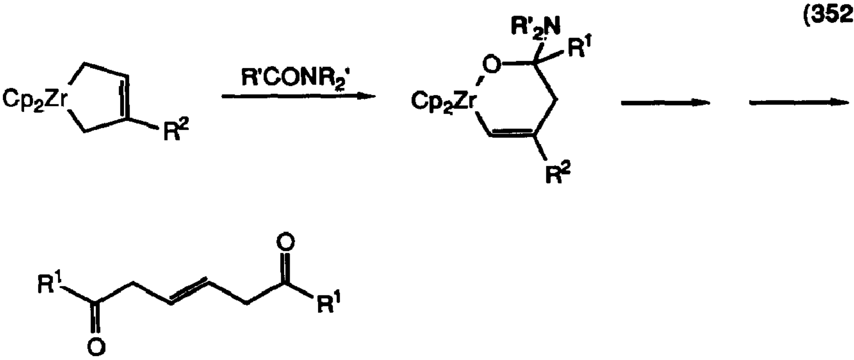


(350)

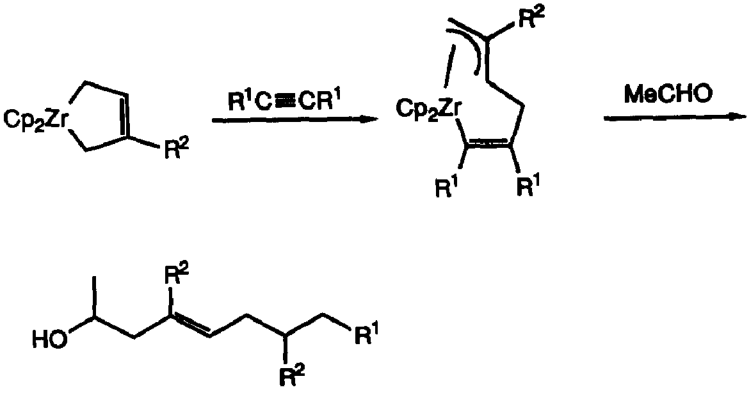
(351)


R^2
+
Cp$_2$ZrBu$_2$
Cp$_2$Zr
R^2
RCOR^1
R^{1}O
R
O
Cp$_2$Zr
R^2
Δ
H$^+$
R$^1_2$CO
R
Cp$_2$Zr
R^2
O
R^2
R
O
R^2
R^1
R
OH
R^1

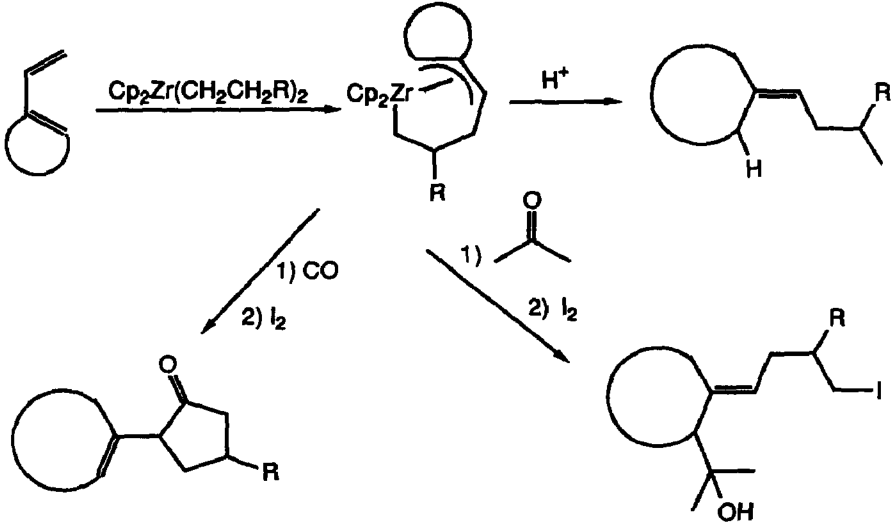
(352)


Cp$_2$Zr
R^2
R'CONR$_2$'
R'$_2$N
R^1
O
Cp$_2$Zr
R^2
R^1
O
O
R^1

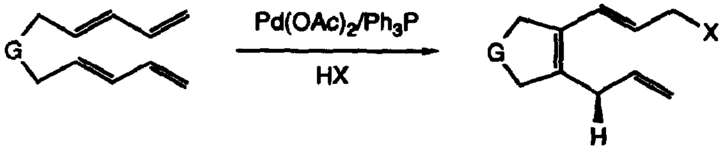
(353)


Cp$_2$Zr
R^2
R^{1}C≡CR^1
R^2
Cp$_2$Zr
R^1
R^1
MeCHO
R^2
HO
R^1
R^2

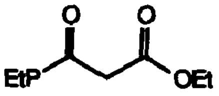
(354)



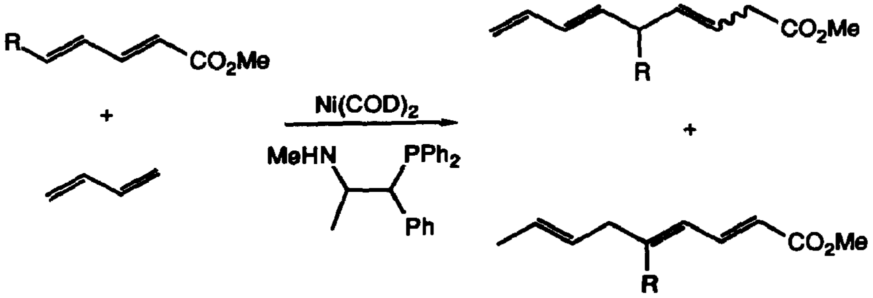
(355)



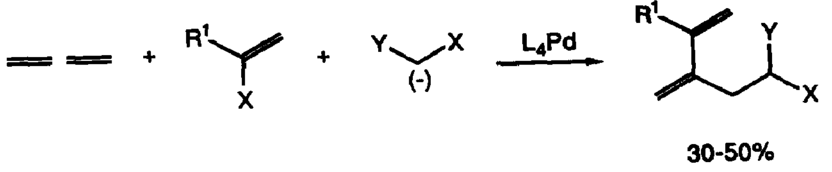
G = NCOPh, $(PhSO_2)_2C$; HX = PhOH, $PhCH_2OH$, Et_2NH, CH_3NO_2,



(356)



(357)

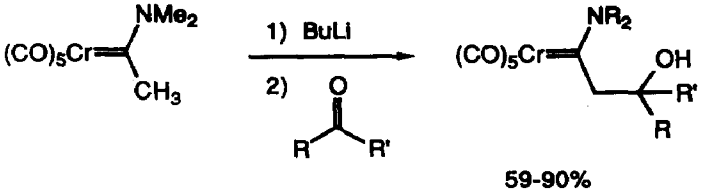


13. Metal-Carbene Reactions

Synthetic methodology involving metal carbene complex chemistry has become a growth industry, and a number of reviews on the subject have appeared. These include "Transition metal carbene complexes in organic synthesis" (190 reference) [414]. "Transition metal carbene complexes and their applications in organic synthesis" (32 references) [415], "Advances in chemical reaction using metal carbene complexes" (10 references) [416], "Studies on the synthesis, structure and reactivity of carbene and related ligands on transition metal clusters (dissertation) [417], "Carbene complexes with Group(IV) metallocene reagents" (62 references) [418], "Reactions of benzylidene (pentacarbonyl) complexes" (15 references) [419], and "Formation of carbon-carbon double bonds by novel insertion reactions of allenes, heterocumulenes and acid amides into metal-carbon double bonds of Fischer carbene complexes" (24 references) [420].

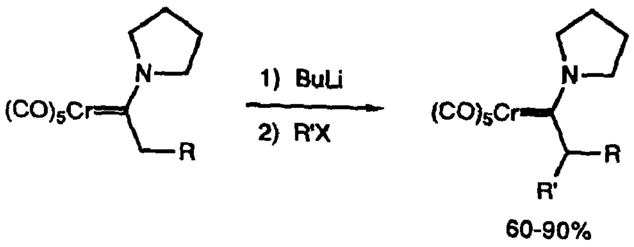
Because of their utility in synthesis, a variety of methods to elaborate carbenes have been developed, including α-acylation (equation 358) [421], α-alkylation (equation 359) [423], N-diacylation (equation 360) [423], (equation 361) [424], olefin insertion (equation 362) [425], acrylonitrile insertion (equation 363) [426], and allene and isonitrile insertion (equation 364) [427], (equation 365) [428].

(358)



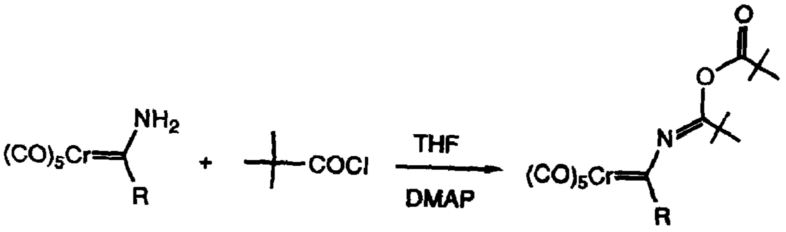
R = Ph, nPr, Me, iPr

R' = H, Me, $(CH_2)_5$



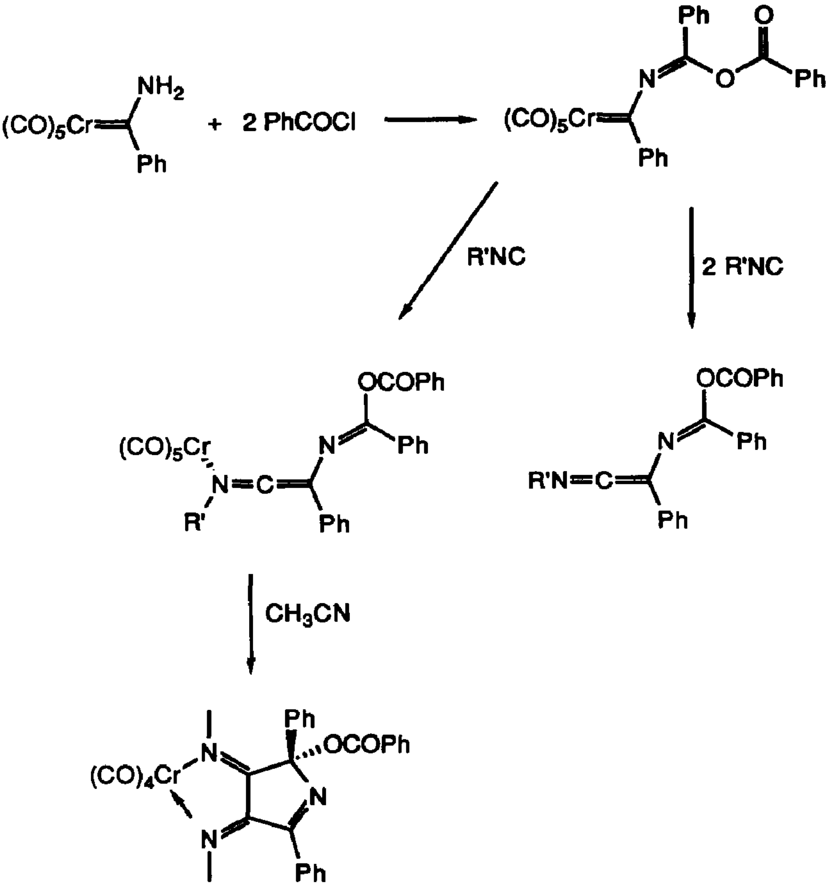
(359)

R = H, Me; R' = MeOTf, MeI, EtI, EtBr, EtOTs, nPrI, BnBr, 

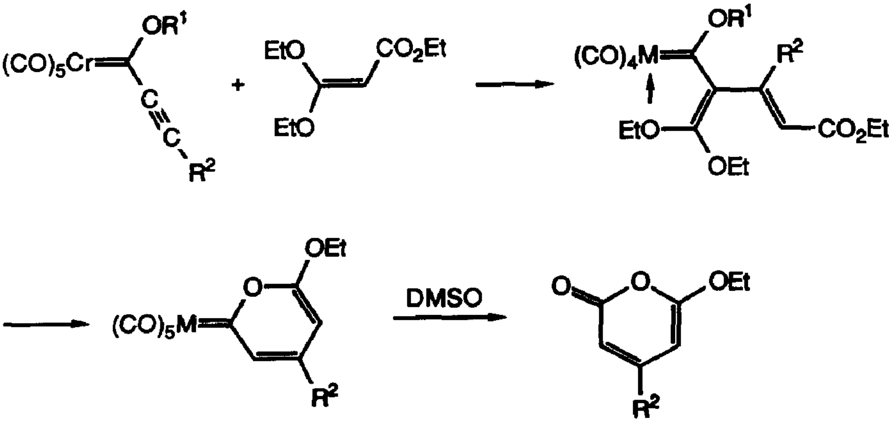


(360)

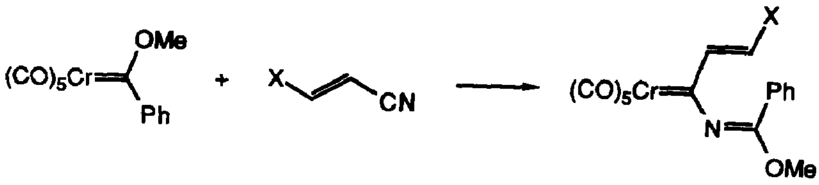
(361)


(CO)5Cr
NH2
Ph
+ 2 PhCOCl
R'NC
2 R'NC
OCOPh
R'N=C
CH3CN
(CO)4Cr

(362)

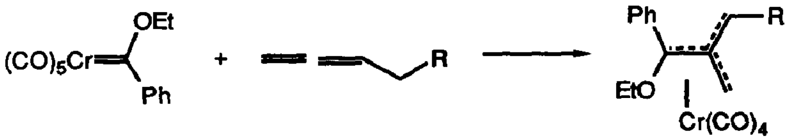

(CO)5Cr
OR1
R2
EtO
CO2Et
(CO)4M
OEt
(CO)5M
DMSO

(363)



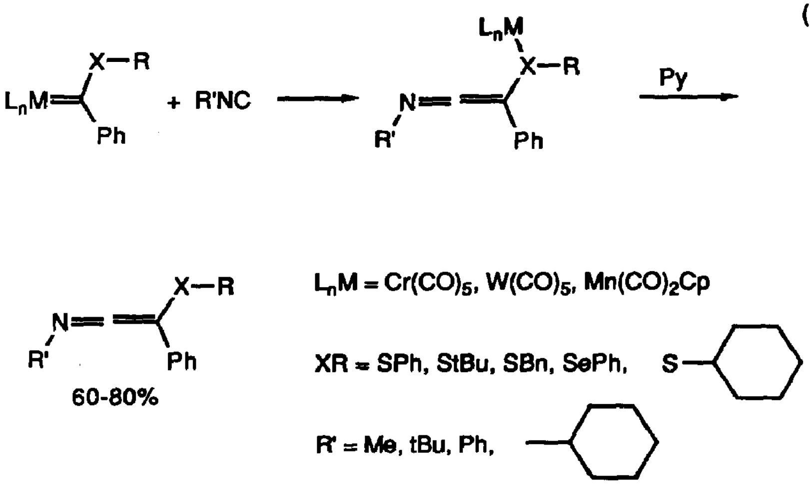
X = NMe$_2$, OMe

(364)



R = OH, CO$_2$Et, CH$_2$OH

(365)



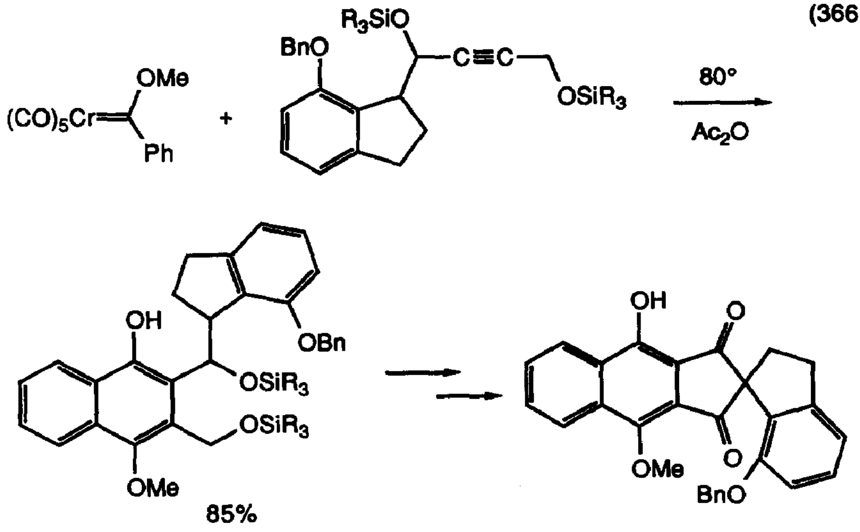
60-80%

L$_n$M = Cr(CO)$_5$, W(CO)$_5$, Mn(CO)$_2$Cp

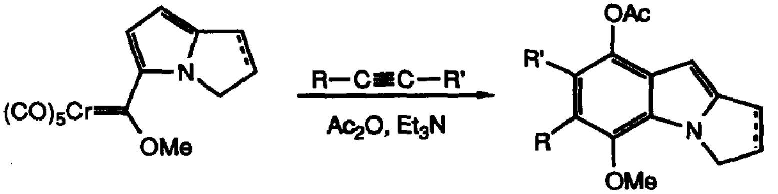
XR = SPh, StBu, SBn, SePh,

R' = Me, tBu, Ph,

"Carbene complexes in selective organic synthesis: novel applications of carbene annulation reactions" was the subject of a review (21 references) [429]. Calculations on the mechanism of the Dötz annulation reaction indicated the intermediate was not a metallocyclobutene, but more nearly an allyl carbene complex [430]. Whatever the mechanism, it is a useful reaction for the formation of naphthohydroquinones (equation 366) [431], (equation 367) [432], (equation 368) [433], (equation 369) [434], cyclohexadienones (equation 370) [435], cyclopentandiones (equation 371) [436], aminoindenes (equation 372) [437], thionaphthoquinones (equation 373) [438], and phosphorous heterocycles (equation 374) [439].



(366)

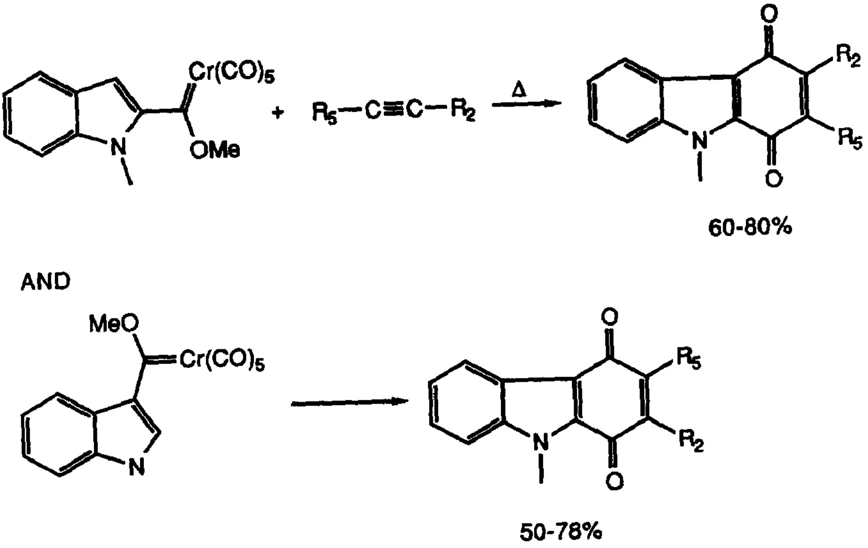


(367)

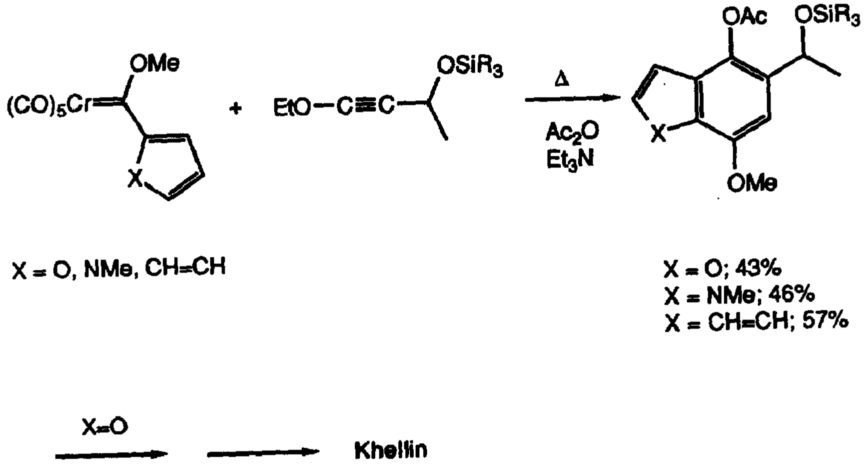
R = Me, nBu, TMS
R' = OMe, OEt, H, Me, TMS

30-60%

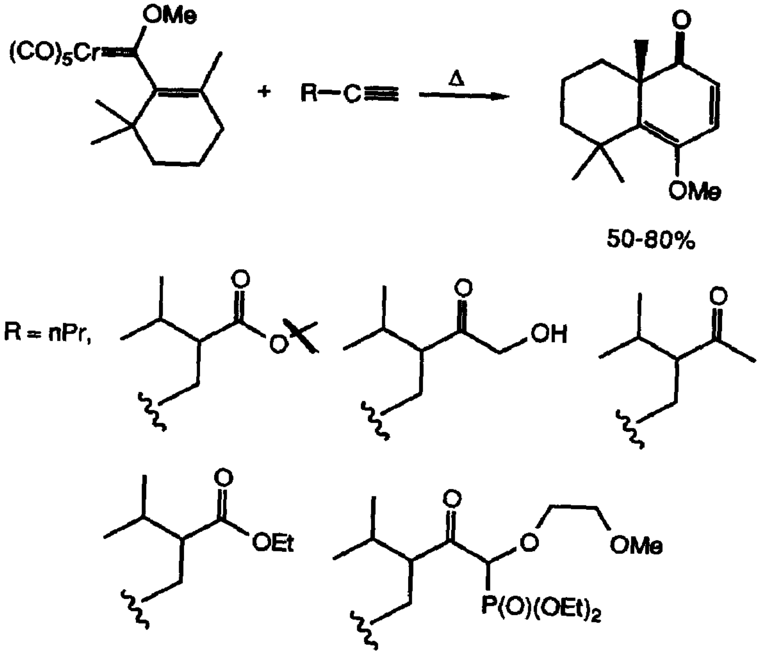
(368)


Cr(CO)$_5$
OMe
N
+
$R_5-C\equiv C-R_2$
Δ
O
R_2
R_5
N
O
60-80%
AND
MeO
Cr(CO)$_5$
N
O
R_5
R_2
N
O
50-78%

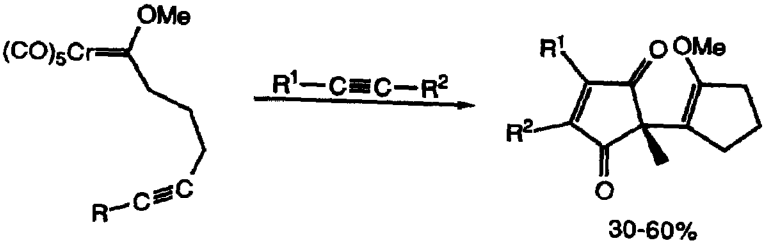
(369)


OMe
(CO)$_5$Cr
X
+
EtO—C≡C
OSiR$_3$
Δ
Ac$_2$O
Et$_3$N
OAc
OSiR$_3$
X
OMe
X = O, NMe, CH=CH
X = O; 43%
X = NMe; 46%
X = CH=CH; 57%
X=O
Khellin

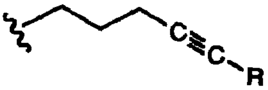
(370)



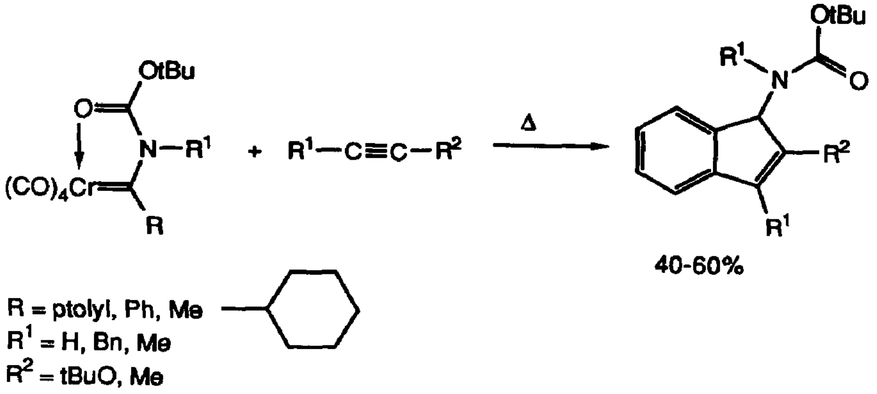
(371)



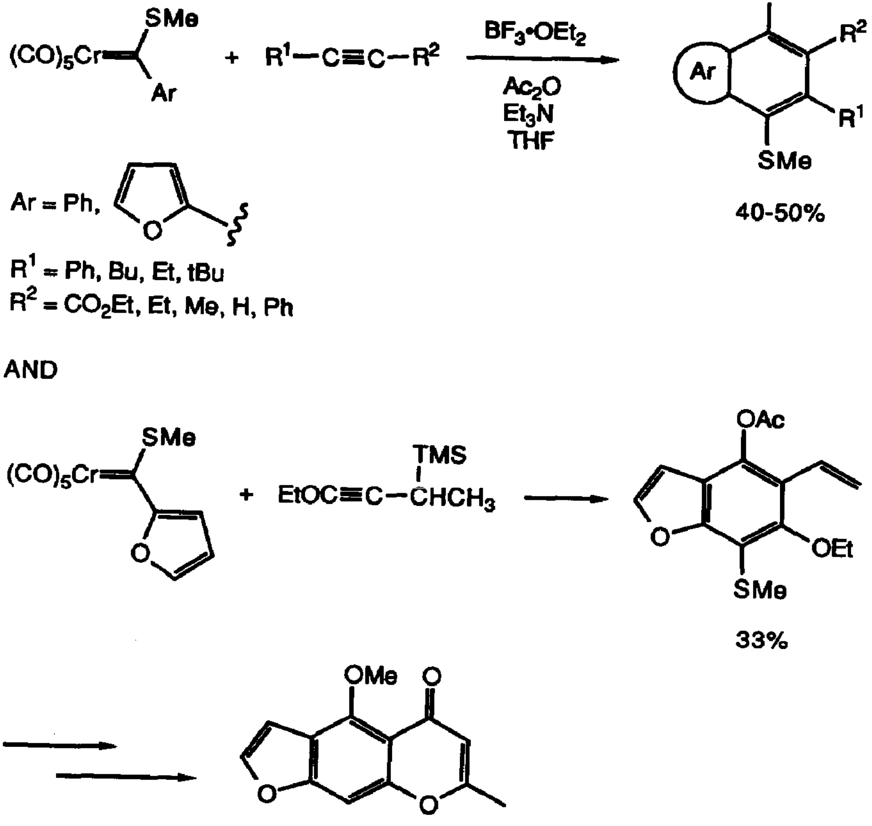
R^1 = H, Et

R^2 = Ph, nPr, Me, TMS, $(CH_2)_3CO_2tBu$, 

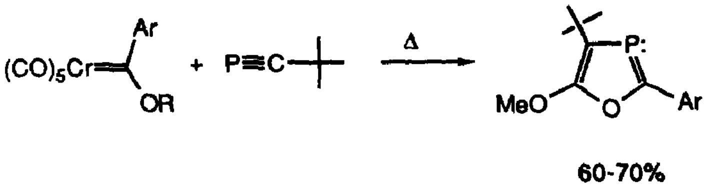
(372)



(373)

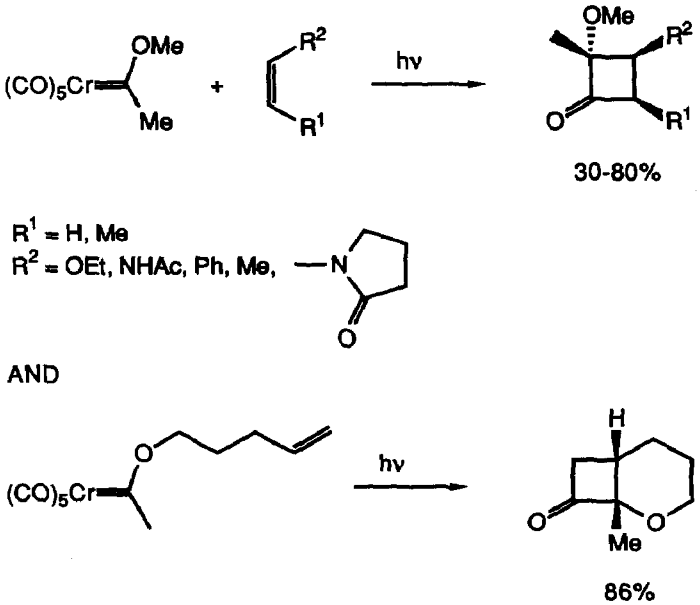


Visnagan



(374)

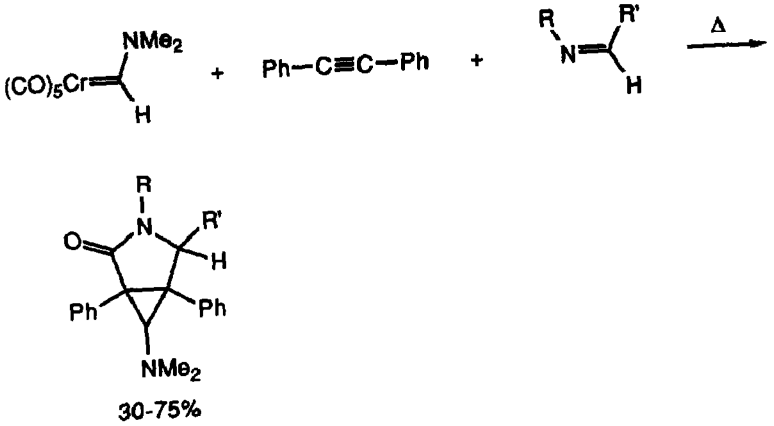
Photolytic reactions of chromium carbene complexes in the synthesis of optically active biologically active compounds has been reviewed (16 references) [440]. Photolysis of alkoxycarbene complexes with olefins produced cyclobutanones (equation 375) [441].



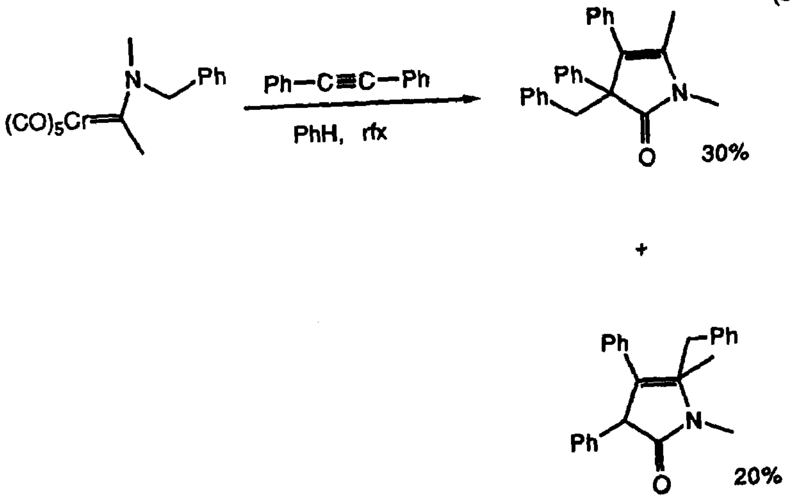
(375)

Thermal reactions of aminocarbenes with alkynes and imines (equation 376) [442], with alkynes alone (equation 377) [443], as well as the thermal reaction of N-allylamino carbenes with alkynes (equation 378) [444], (equation 379) [445], led to unusual ring systems. S-Allylthiocarbenes behaved in a similar manner (equation 380) [446].

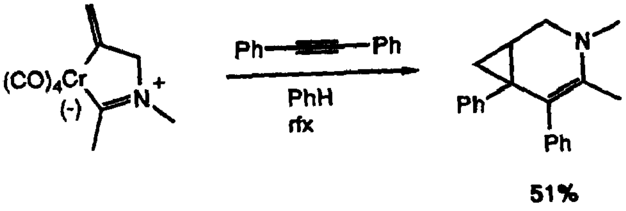
(376)

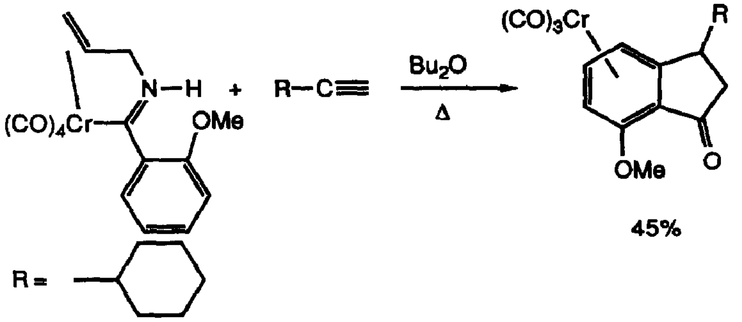

(CO)$_5$Cr
NMe$_2$
H
+
Ph—C≡C—Ph
+
R
N
R'
H
Δ
R
N
O
R'
H
Ph
Ph
NMe$_2$
30-75%

(377)

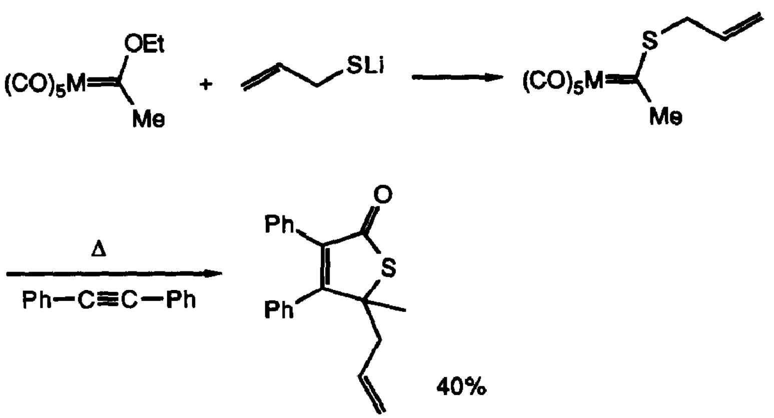

(CO)$_5$Cr
N
Ph
Ph—C≡C—Ph
PhH, rfx
Ph
Ph
Ph
N
O
30%
+
Ph
Ph
Ph
N
O
20%

(378)


(CO)$_4$Cr
(-)
N
+
Ph—≡—Ph
PhH
rfx
N
Ph
Ph
51%

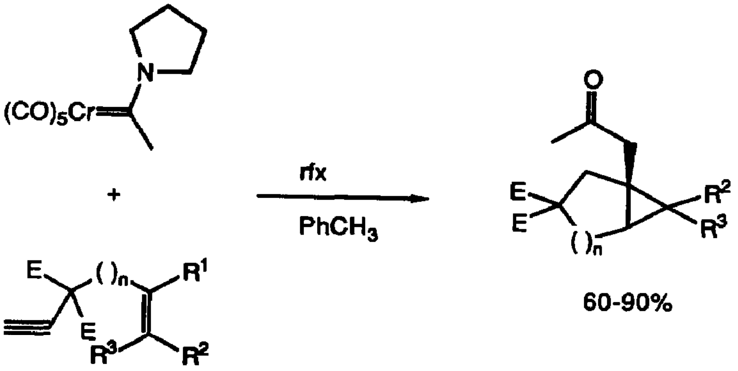


(379)

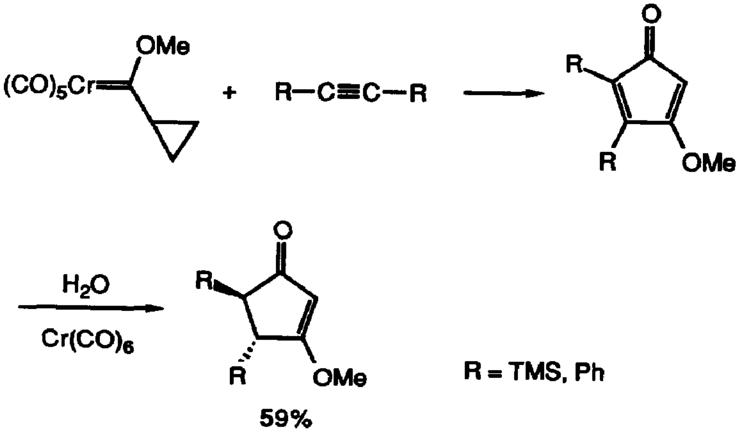


(380)

Eneynes underwent a complex cascade of cycloaddition, metathesis, cycloaddition and reductive elimination with aminocarbenes (equation 381) [447]. Cyclopropylcarbenes gave cyclopentenones with alkynes (equation 382) [448], and cyclopropylcyclopropanes with alkynes (equation 383) [449]. Tungsten carbynes also homologated alkynes to naphthols (equation 384) [450]. Ketenyl complexes in organic synthesis has been reviewed (33 references) [451].

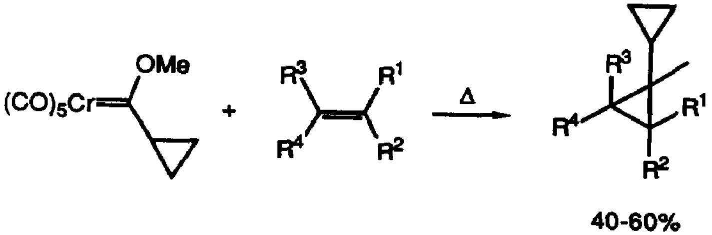


(381)



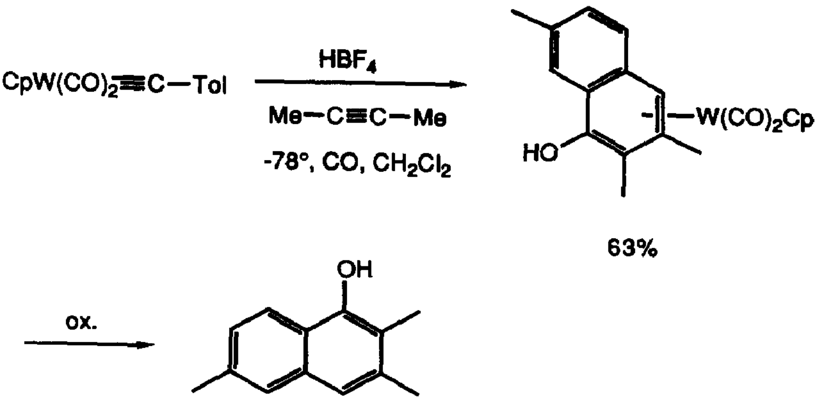
(382)

R = TMS, Ph



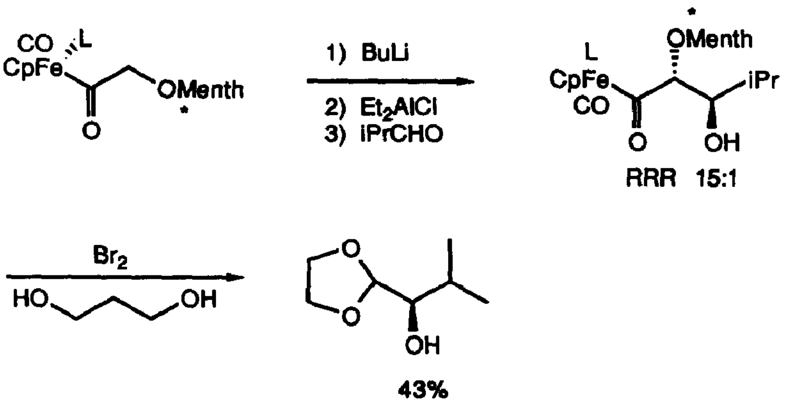
(383)

R^1 = CO$_2$Me, CONMe$_2$
R^2 = H, Me
R^3 = H, CO$_2$Me
R^4 = H, Me, CO$_2$Me

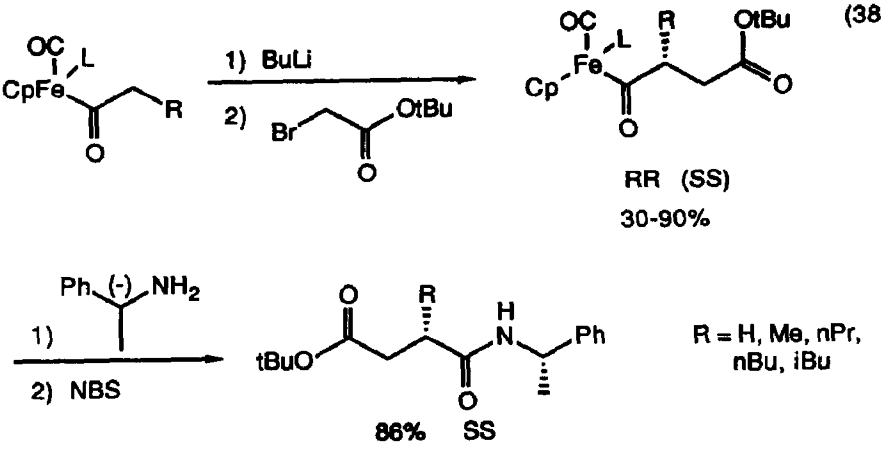

(384)

14. Alkylation of Metal Acyl Enolates

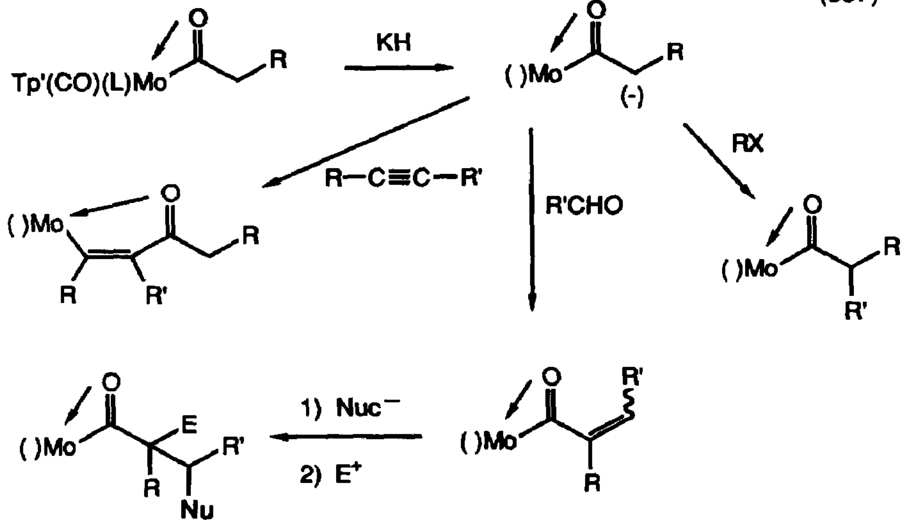
Chiral iron acyl enolates were alkylated with a high degree of stereoselectivity (equation 385) [452], (equation 386) [453]. Molybdenum acyl enolates were also easily α-alkylated (equation 387) [454].


(385)

(386)



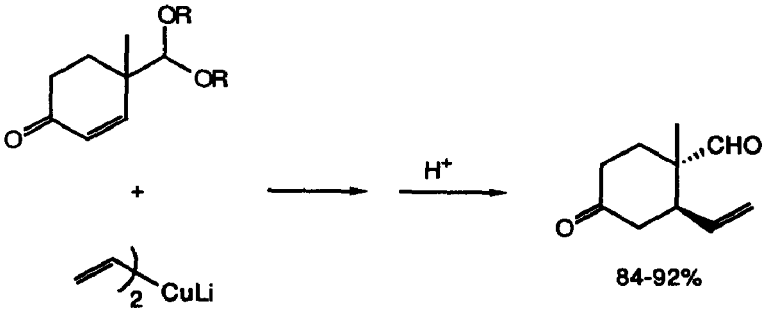
(387)



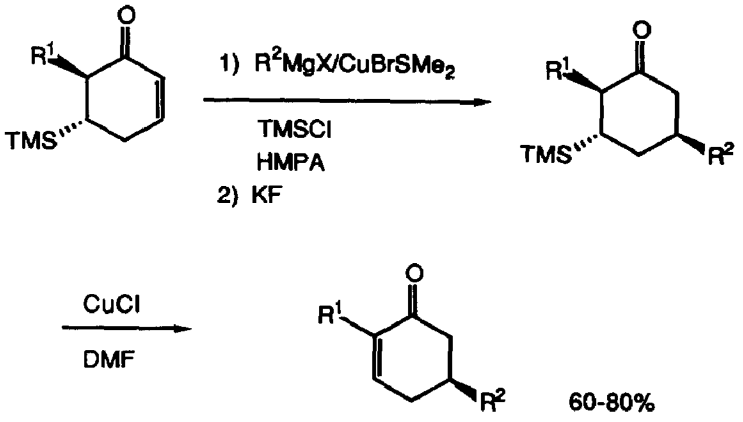
B. Conjugated Addition Reactions

Organocopper complexes continued to be the reagents of choice for conjugate addition reactions. A lecture dealing with organometallics in prostaglandin synthesis, with emphasis on copper conjugate addition reaction has been published [455]. The acceleration of conjugate addition of organocopper reagents by trimethylsilyl chloride was claimed to involve the initial coordination of the silane to the enone [456]. Conjugate additions of cuprates to cyclohexenones (equation 388) [457], (equation 389) [458], to acroleins (equation 390) [459], and β-nitroenones (equation 391) [460] were efficient processes. Zinc homoenolates added 1,4 to cyclohexenone in the presence of CuBr•SMe$_2$ (equation 392) [461]. Trimethylsilylmethyl cuprates added to β-bromocyclohexanones (equation 393) [462]. Vinyl cuprate added to a δ-carboethoxy enone to ultimately cyclize (equation 394) [463]. Conjugated amides bearing chiral amino groups underwent conjugate alkylation by cuprates with very high enantiomeric excess (equation 395([464].

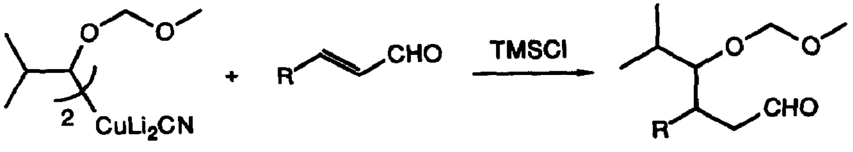
(388)

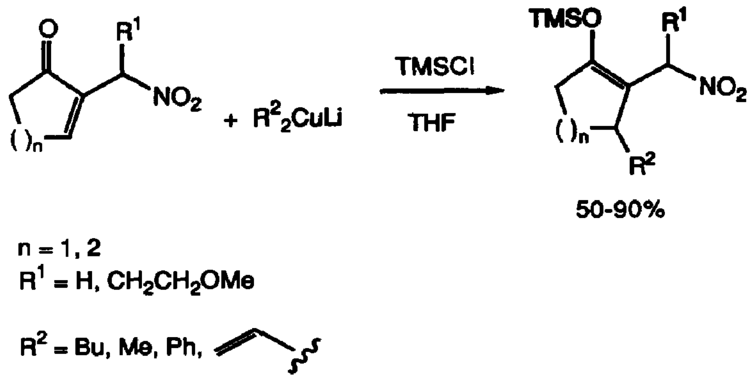


(389)



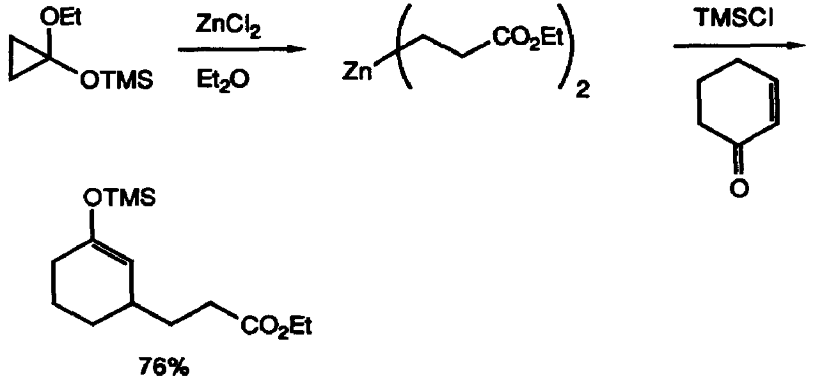
(390)



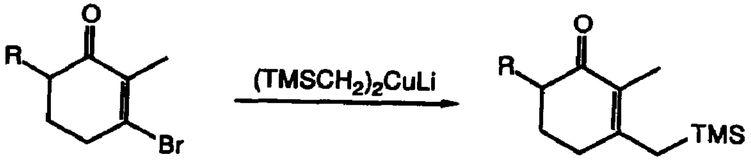


(391)

n = 1, 2
R^1 = H, CH_2CH_2OMe
R^2 = Bu, Me, Ph,

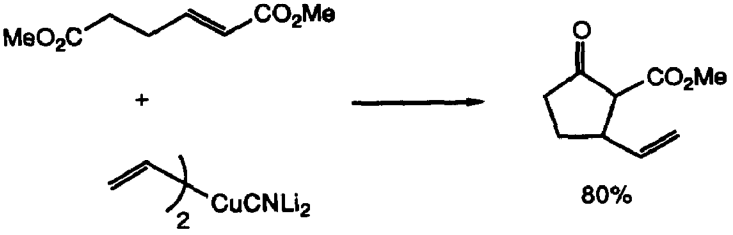


(392)

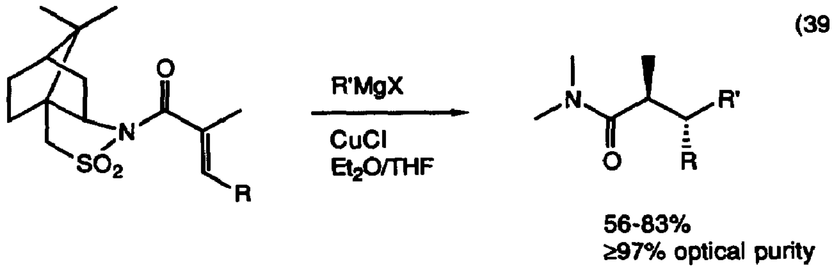


(393)

(394)



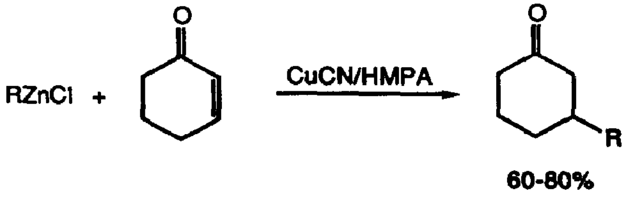
(395)

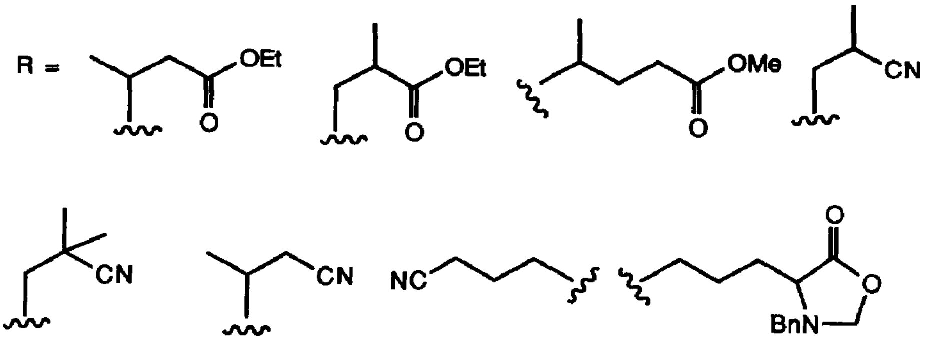


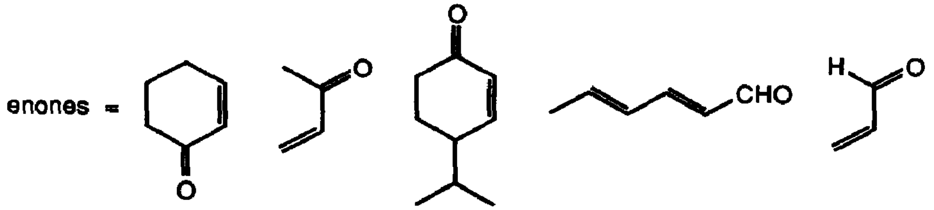
R = Me, Et, Bu, TBDMSCOCH$_2$
R' = Bu, Et, Me

Functionalized organocuprates, made from the corresponding organozinc reagents, were efficient reactants in conjugate addition reactions (equation 396) [465], (equation 397) [466], (equation 398) [467], (equation 399) [468]. α,β-Unsaturated chiral sulfoxides (equation 400) [469] and ketenimines (equation 401) [470] also underwent conjugate alkylation by organocopper reagents.

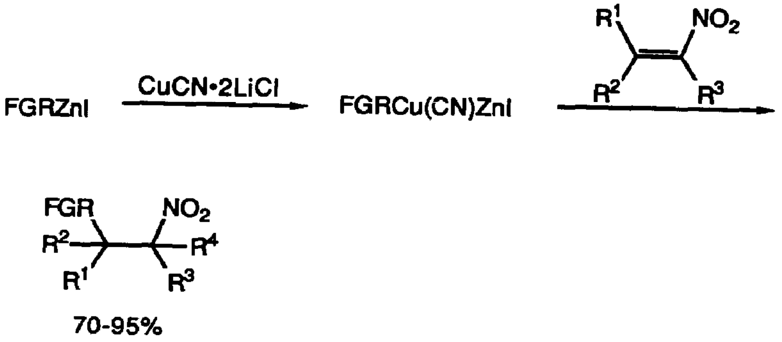
(396)

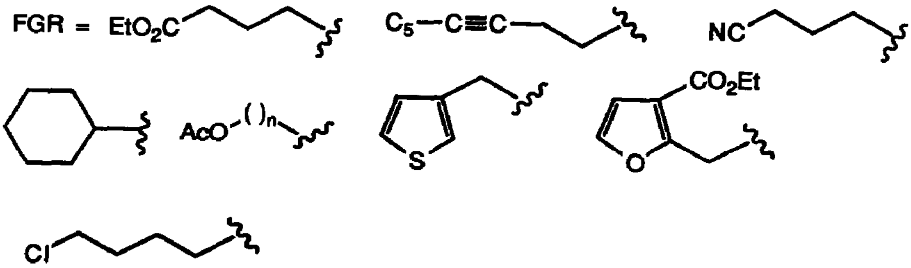



R =
OEt
O
OEt
O
OMe
O
CN
CN
CN
NC
O
O
BnN

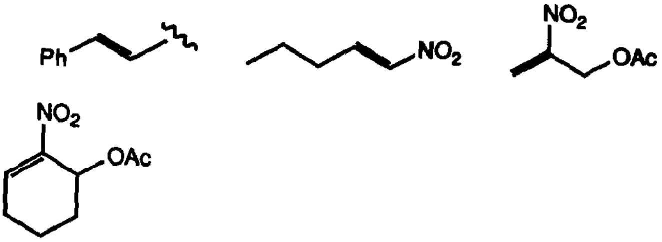

enones =
O
O
O
CHO
H
O

(397)

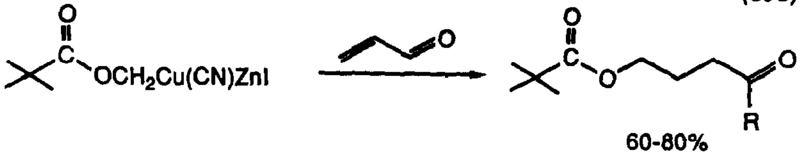

FGRZnI
CuCN•2LiCl
FGRCu(CN)ZnI
R1
NO2
R2
R3
FGR
NO2
R2
R4
R1
R3
70-95%


FGR =
EtO2C
C5—C≡C
NC
AcO
( )n
S
CO2Et
O
Cl

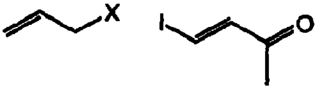
substrate =



(398)

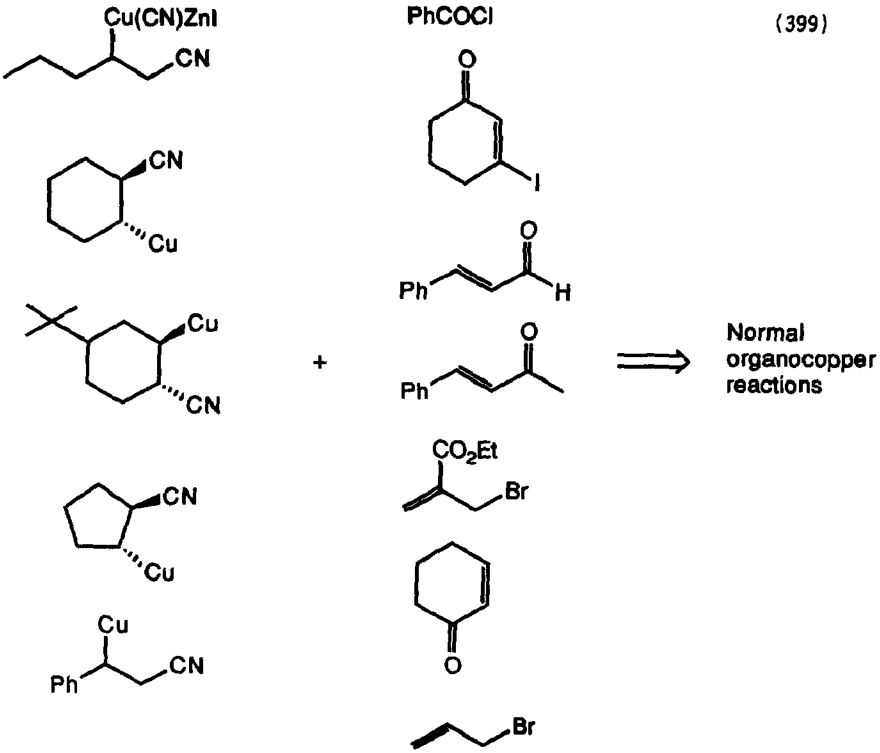


ICH$_2$Cu also works ‖ RCOCl, R$_3$SnCl,

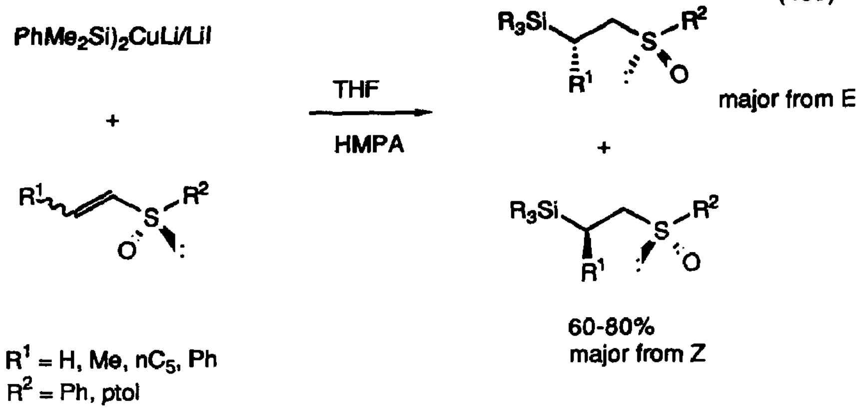


also react as is typical with RCu

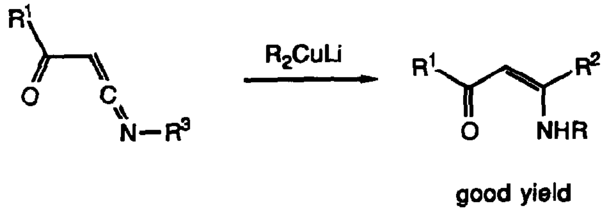
(399)



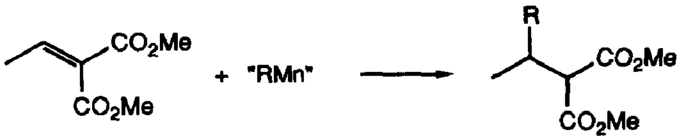
(400)



(401)

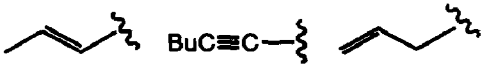


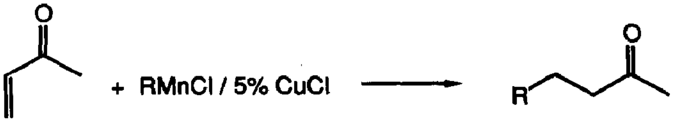
Manganese alkylidene malonates underwent conjugate addition reaction with organomanganese compounds (equation 402) [471], as did simple conjugated enones (equation 403) [472]. Transition metals were used to fine tune the Michael addition reaction of β-dicarbonyl compounds to enones [473]. Ruthenium hydrides catalyzed the Michael addition of cyanoesters to enones (equation 404) [474]. Nickel chloride/zinc catalyzed the Michael addition of cyano esters to enones (equation 404) [474]. Nickel chloride/zinc catalyzed the Michael alkylation of enones by halides (equation 405) [475]. With chiral ligands asymmetric induction was observed (equation 406) [476].



(402)

(full paper) R = Bu, Me, iPr, tBu, Ph,



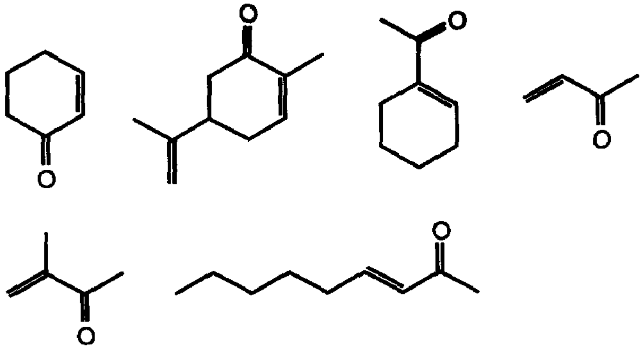


(403)

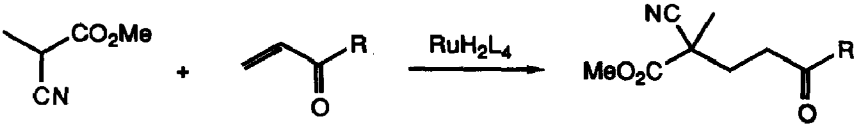
good yields

R = Bu, Ph, iPr, tBu,

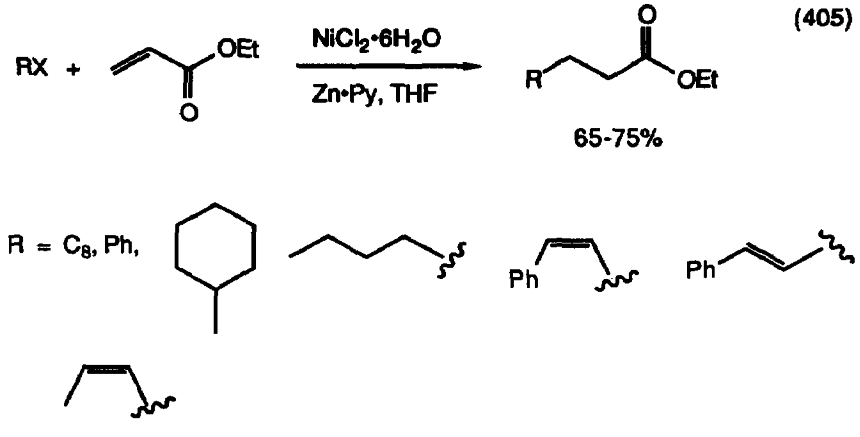
enone =



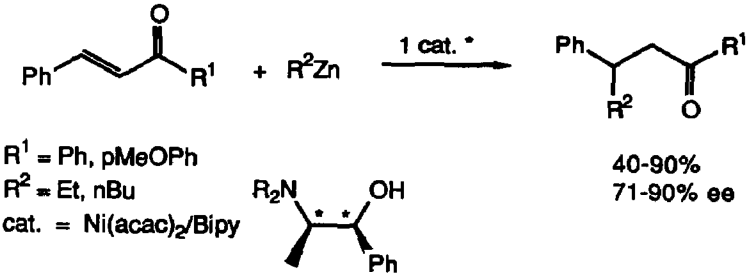
(404)



(405)

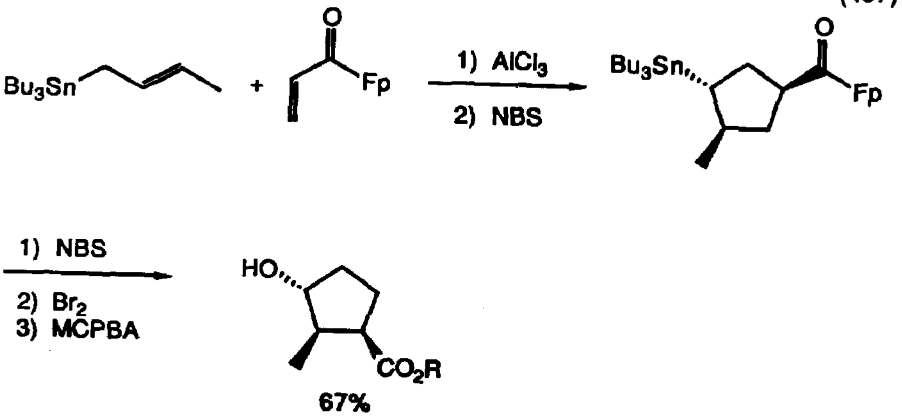


(406)

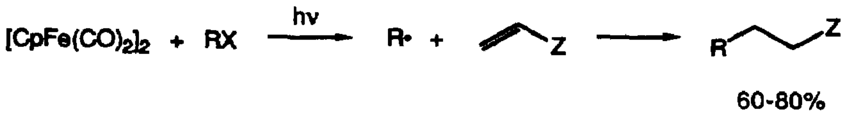


Other miscellaneous conjugate additions are described in (equation 407) [477], (equation 408) [478], (equation 409) [479], (equation 410) [480], (equation 411) [481], (equation 412) [482].

(407)

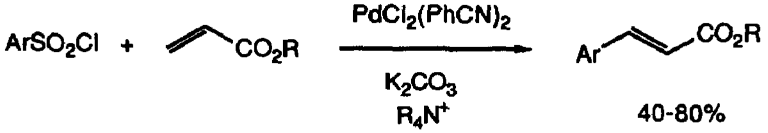


(408)



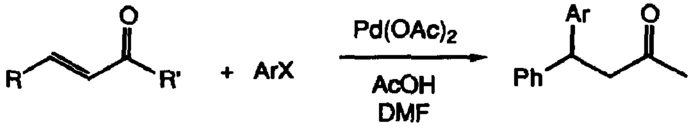
R = nC$_6$; Z = CN, CO$_2$Me, Ph$_2$, (CN)(OEt)

(409)

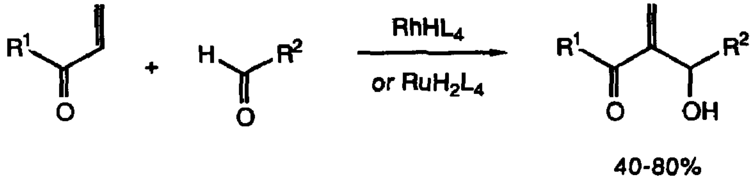


Ar = 1-Naphth, 2-Naphth, Ph, pMePh, pClPh,
R = nBu, Et

(410)

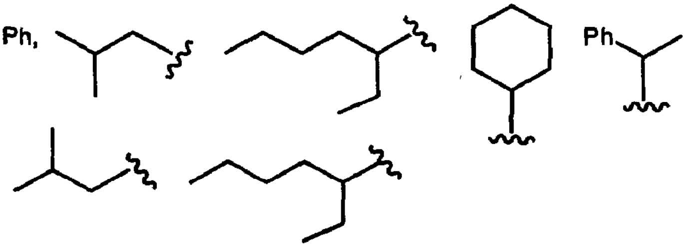


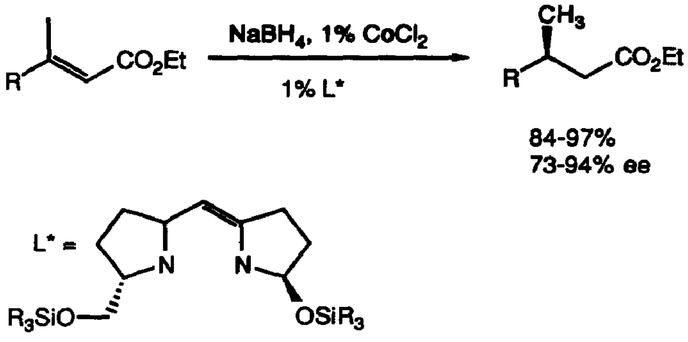
(411)



R^1 = Me, Et, nC$_5$, Ph,

R^2 = Et, nPr, Ph,





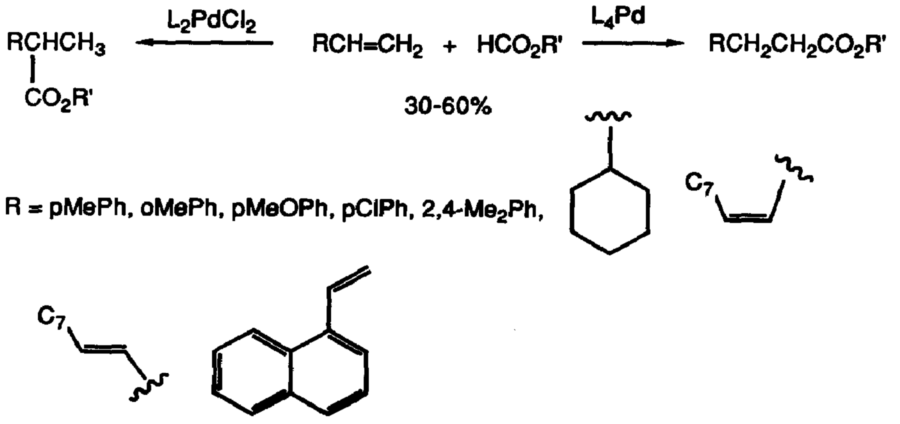
(412)

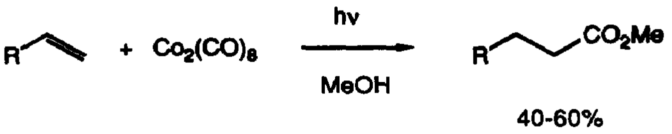
C Acylation Reactions

1. Carbonylation of Alkenes and Arenes

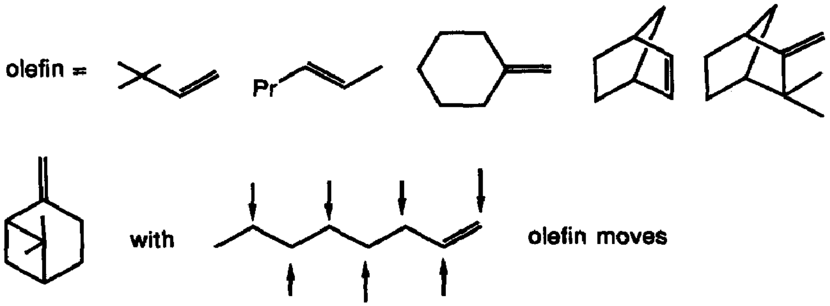
Catalytic synthesis of organic compounds by the carbonylation of unsaturated hydrocarbons and alcohols was the subject of a review (256 references) [483]. Palladium complexes catalyzed the addition of alkylformates to olefins to give esters (equation 413) [484], as did $Ru_3(CO)_{12}$ [485]. Olefins were hydrocarboxylated by cobalt carbonyl under photochemical conditions (equation 414) [486], by palladium chloride under electrochemical conditions [487], by $Co_2(CO)_8$ (pyridine) to produce higher aliphatic esters [488], by palladium phosphine complexes [489] and by sulfonated resin supported cationic palladium catalysts [490]. Styrene was oxidatively carbonylated to cinnamic acid by Pd/Cu/alkali earth systems [491].

(413)

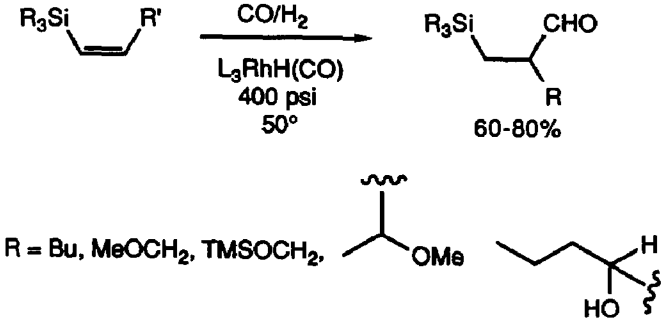




(414)

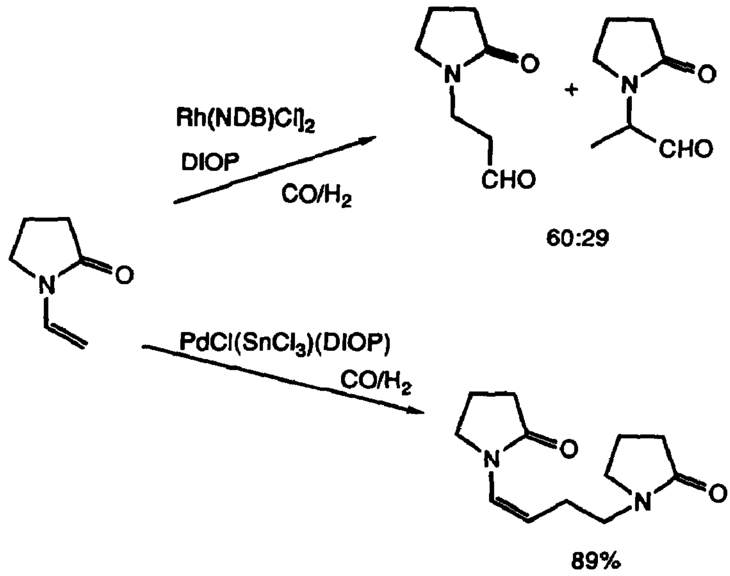


Vinyl silanes were hydroformylated using rhodium(I) catalysts (equation 415) [492]. Hydroformylation of N-vinylpyrrolidinone using rhodium catalysts gave aldehydes, while platinum catalysts led to dimerization rather than hydroformylation (equation 416) [493]. Hydroformylation of chromium complexed styrene went with high branched selectivity (equation 417) [494]. Aroma chemicals via hydroformylation of terpenes and other olefins was studied [495].

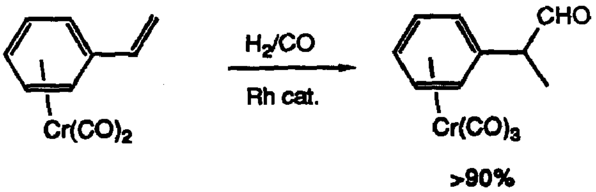


(415)

(416)

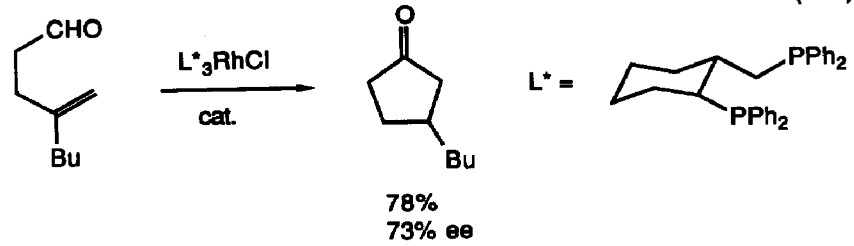


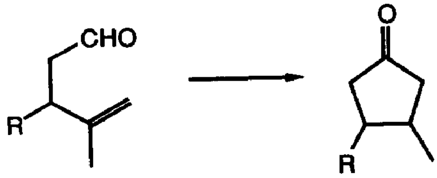
(417)



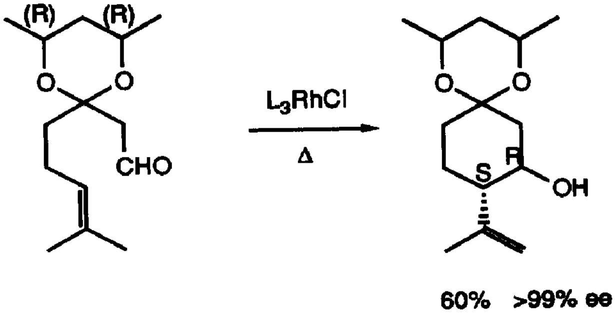
Chiral rhodium catalysts effect the intramolecular addition of aldehydes across olefins (equation 418) [496]. Chiral acetals close to the centers also resulted in high asymmetric induction (equation 419) [497-500].

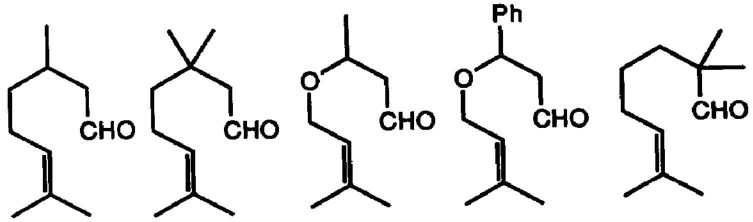
(418)



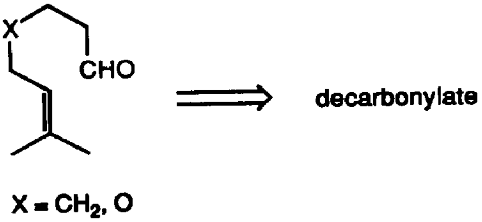


(419)



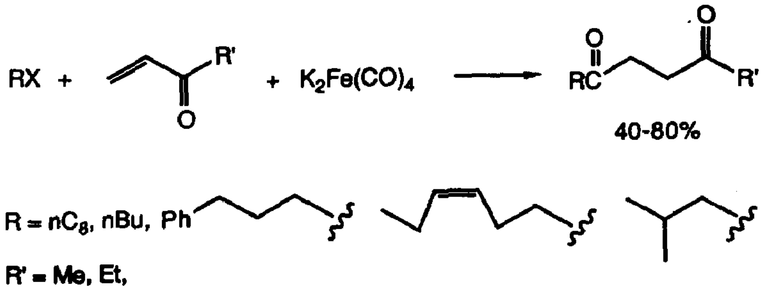


also close

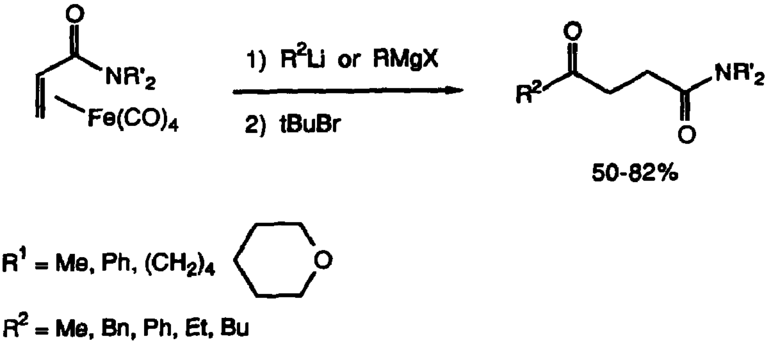


X = CH_2, O

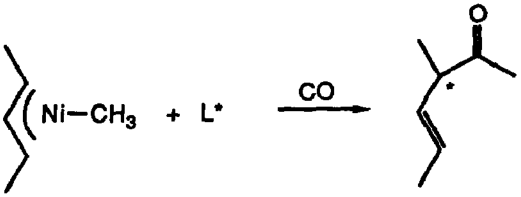
Iron carbonyl anions acylated conjugated enones with alkyl halides (equation 420) [501]. Iron carbonyl complexes of enones gave the same products when treated with organolithium reagents (equation 421) [502]. π-Allylnickel complexes were acylated by treatment with chiral ligands and carbon monoxide (equation 422) [503]. Palladium(II) salts promoted the 1,4-alkoxylation/acylation of dienes (equation 423) [504]. Palladium also catalyzed the carbonylation shown in equation 424 [505].



(420)

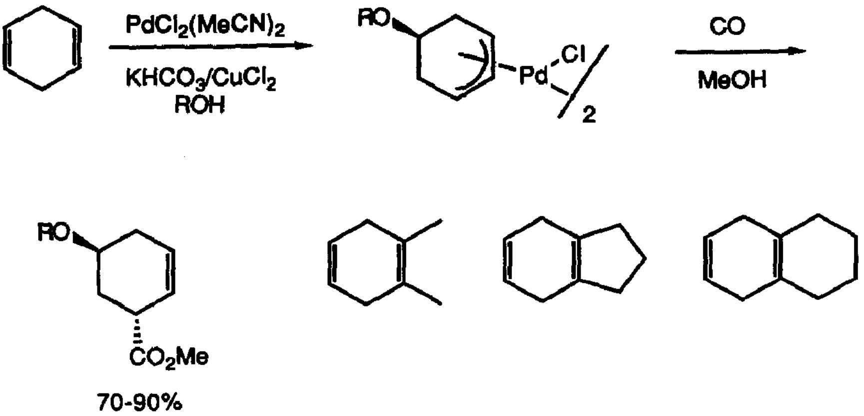


(421)

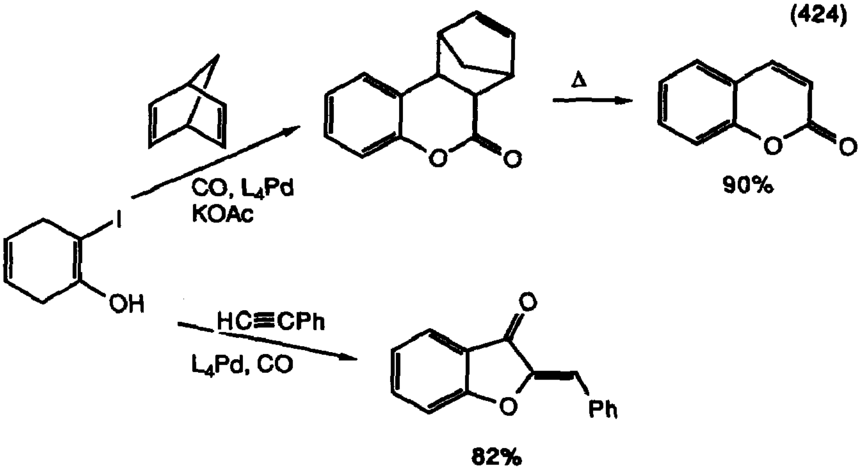


(422)

(423)



(424)



Palladium acetate in the presence of $K_2S_2O_8$, CF_3COOH, and 30 atm CO catalyzed (2 turns) the conversion of cyclohexane to a 1:2 mixture of cyclohexanone carboxylic and benzoic acid [506]. Ruthenium(0) complexes catalyzed the catalytic photochemical carbonylation of benzene [507]. Manganese(III) acetate formylated naphthalenes using malonic acid as the carbon source [508].

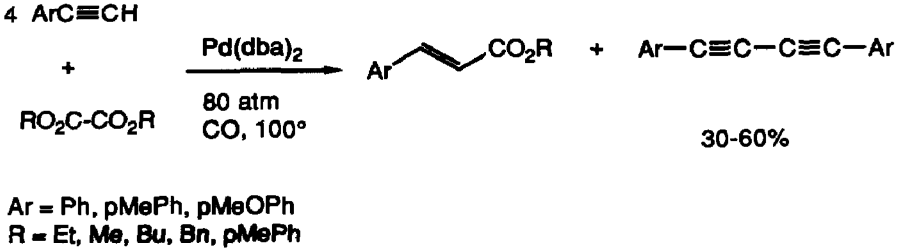
2. Carbonylation of Alkynes, Including the Pauson-Khand Reaction

The activation of alkynes with ruthenium complexes (equation 425) was reviewed (31 references) [509]. Palladium(0) complexes catalyzed the carboxylation of terminal alkynes by oxalates (equation 426) [510] [511]. Palladium acetate/triphenyl phosphine/perfluoroacetic acid catalyzed the hydrocarboxylation of acetylene to acrolein and acrylic acid [512]. Rhodium carbonyl complexes catalyzed the silyl formylation of alkynes (equation 427) [513].

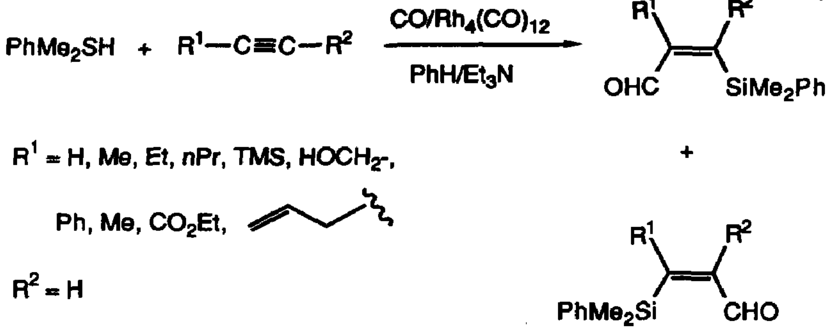
$$RC\equiv CH + CO_2 + R_2NH \xrightarrow{Ru} RCH{=}CH\text{-}O\text{-}\underset{\underset{O}{\|}}{C}\text{-}NR^2_2$$

(425)

(426)

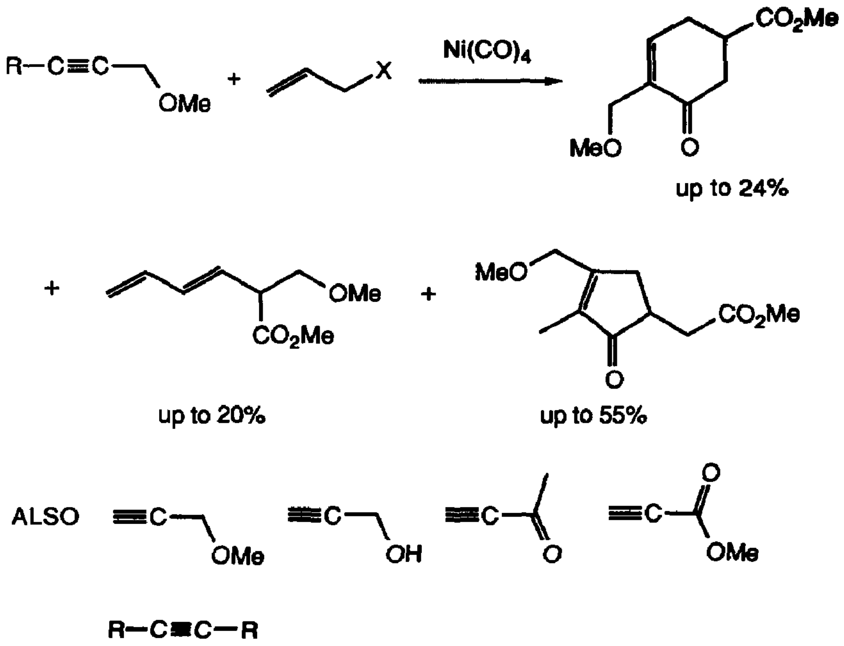


(427)

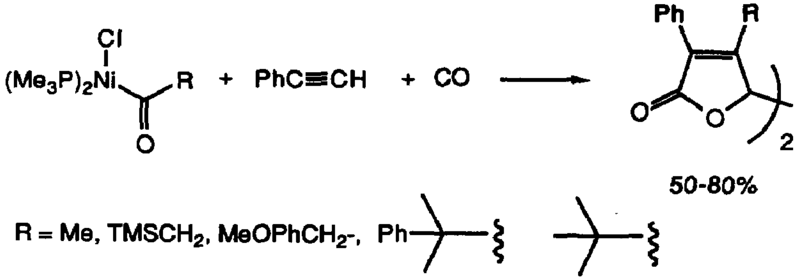


Nickel carbonyl catalyzed the cyclocarbonylation of propargyl ethers with allylic halides (equation 428) [514] and the cyclocarbonylation of terminal alkynes (equation 429) [515]. Cobalt carbonyl catalyzed a similar process (equation 430) [516] [517], (equation 431) [518]. Palladium on carbon catalyzed the carbonylative cyclodimerization of diynes (equation 432) [519].

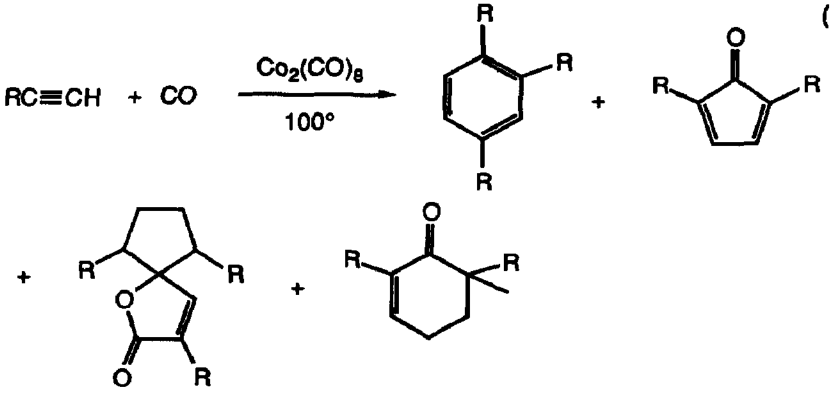
(428)

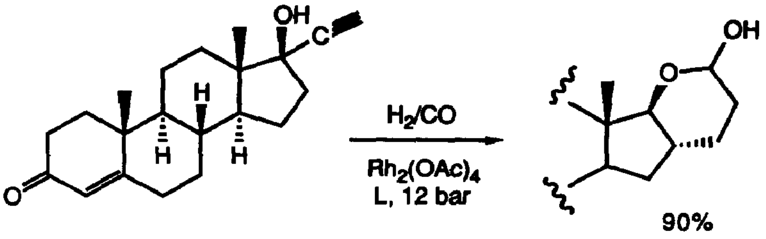

R—C≡C—
OMe
+
X
Ni(CO)4
CO2Me
MeO
O
up to 24%
+
OMe
CO2Me
+
MeO
CO2Me
O
up to 20%
up to 55%
ALSO
≡C—
OMe
≡C—
OH
≡C—
O
≡C—
O
OMe
R—C≡C—R

(429)

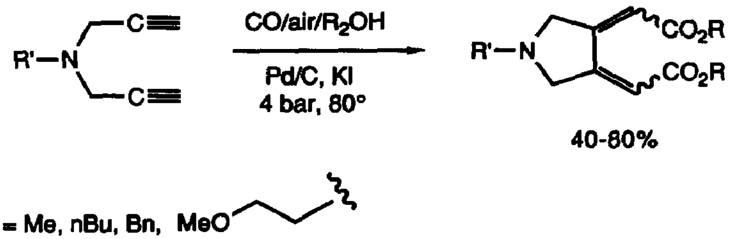

Cl
(Me3P)2Ni
R
O
+ PhC≡CH
+ CO
Ph
R
O
O
2
50-80%
R = Me, TMSCH2, MeOPhCH2-,
Ph

(430)


RC≡CH
+ CO
Co2(CO)8
100°
R
R
R
+
O
R
R
+
R
R
O
O
R
+
O
R
R



(431)



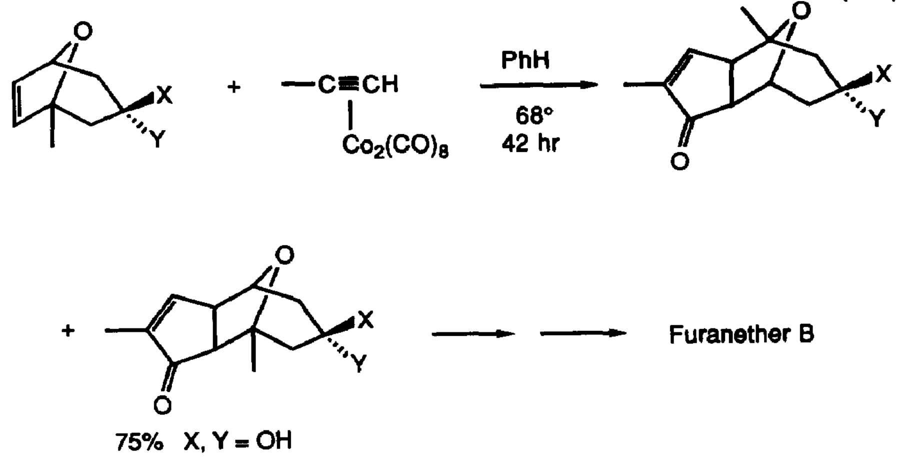
(432)

R = Me, nBu, Bn, MeO

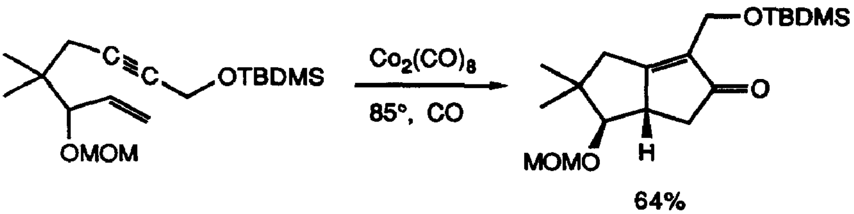
R' = HCO, MeCO, PhCO, $PhCH_2CO$, pTs, nBu, Bn, $MeOCOCH_2$

Cyclopentenone formation from alkyne-cobalt complexes was reviewed (46 references) [520]. "The Pauson-Khand cobalt cyclization reaction in a synthetic approach to corcolin" [521], "Synthesis of guaranolidians pseudo guaranolide natural product precursors based on the octacarbonyl dicobalt cyclization process" [522], and "The synthesis of plant growth regulators via alkynehexacarbonyl dicobalt complexes" [523] were all titles of dissertations. Inter- (equation 433) [524] and intramolecular Pauson-Khand reactions (equation 434) [525], (equation 435) [526][527][528], (equation 436) [529] were used to synthesize polycyclic cyclopentenone systems. Running the reaction on the surface of chromatographic supports increased the yield (equation 437) [530][531].

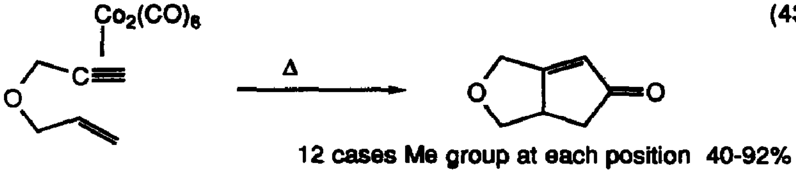
(433)



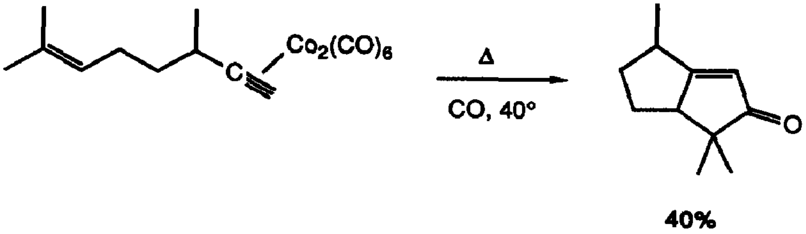
(434)



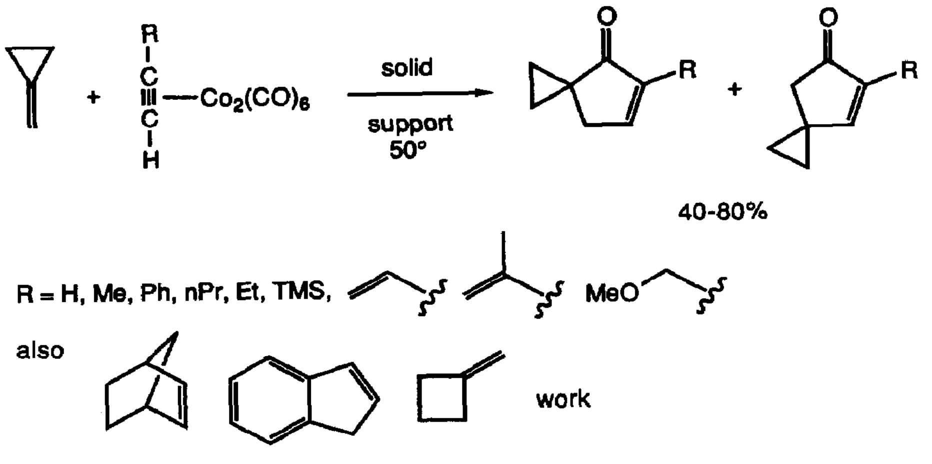
(435)



(436)



(437)

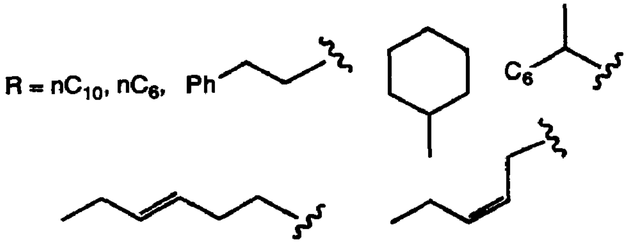


Support = SiO_2, Al_2O_3, $MgOSiO_2$, NaX Zeolite

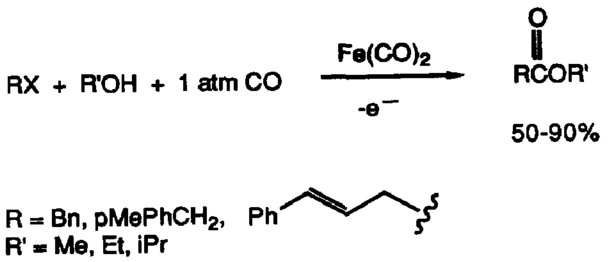
3. Carbonylation of Halides

Simple alkyl iodides (equation 438) [532], (equation 439) [533] and fluorinated halides (equation 440) [534] were catalytically converted to esters.

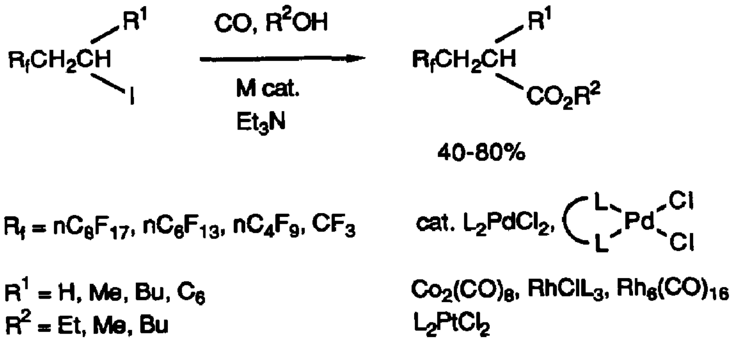
$$RI + CO + MeOH \xrightarrow[L_2PtCl_2\ \ 120°]{K_2CO_3} \underset{57\text{-}76\%}{RCO_2Me} \qquad (438)$$



(439)

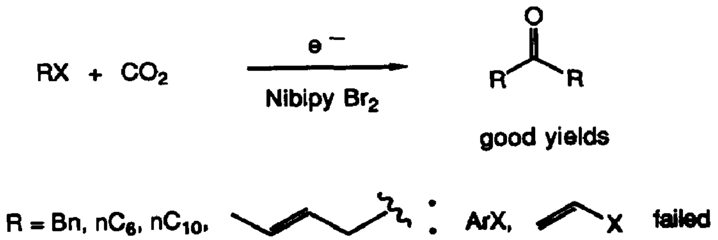


(440)



Halides were carbonylatively coupled (equation 441) [535] or formylated (equation 442) [536] under electrochemical conditions. Polymer-anchored palladium catalysts were used to carbonylate organic halides [537]. Benzyl chloride was carbonylated to phenylacetic acid using water soluble Ru(III)-EDTA complex catalysts [538]. Iron tetracarbonyl dianion acylated alkyl halides (equation 443) [539].

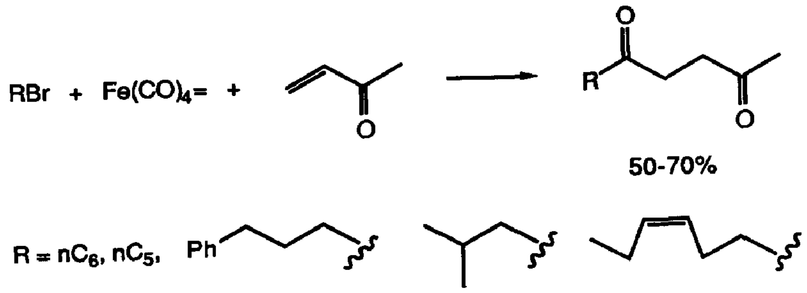
(441)



$$\mathrm{RX} + \mathrm{Fe(CO)_5} + \mathrm{HX} \xrightarrow{2e^-} \mathrm{RCHO} \qquad (442)$$

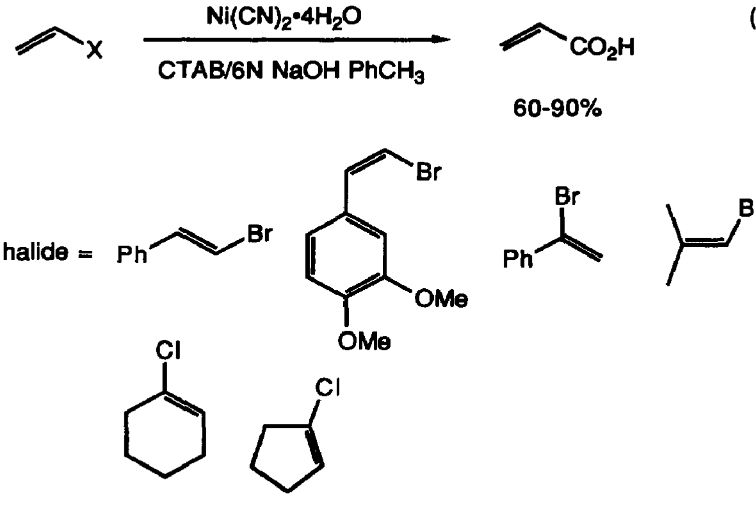
71% 88% conversion

$R = nC_5$



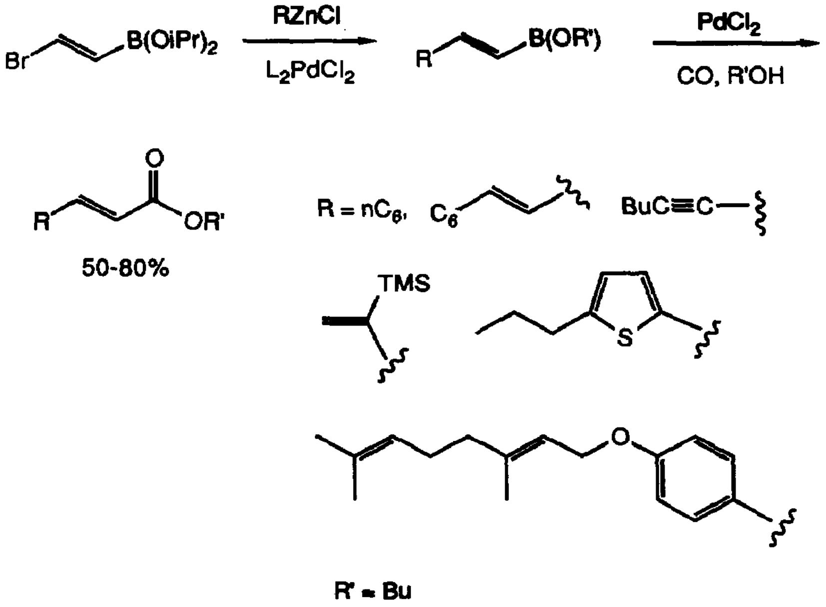
(443)

Vinyl halides were converted to acrylic acids using nickel(II) cyanide catalysts (equation 444) [540] or cobalt carbonyl catalysts [541] under phase transfer conditions. Palladium(II) catalyzed the carbonylation of vinyl boranes (equation 445) [542].

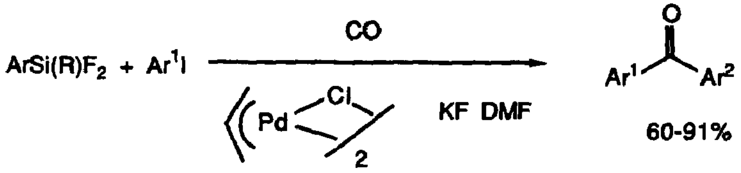


(444)

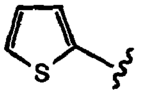
(445)



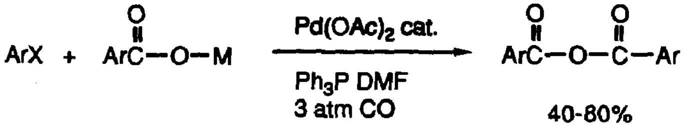
Aryl halides were carbonylated by phosphine palladium complexes [543] and cobalt(II) chloride [544]. Aryl chlorides were carbonylated by palladium(0) catalysts having *bis*-diisopropyl(phosphino)propane ligands, providing chelate assistance [545]. α-Chloronaphthalene and bromobenzene were carbonylated by dicobalt octacarbonyl/alkyl halide systems [546]. Under phase transfer conditions, palladium(0) complexes catalyzed the monocarbonylation of xylene dihalides [547], while palladium(II) complexes catalyzed the *bis*-carbonylation of *bis*-chloromethylarenes [548]. Aryl halides were carbonylated to aldehydes using palladium *bis*-(diisopropylphosphino)propane catalysts [549]. Aryl iodides were carbonylatively coupled to aryl fluorosilanes to give diaryl ketones (equation 446) [550]. Aryl halides were coupled to aryl carboxylic acid anhydrides by palladium acetate (equation 447) [551].



(446)

Ar^1 = Ph, pMePh, mMeOPh, pCF_3Ph, 

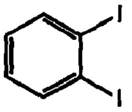
Ar^2 = pMeCOPh, pEtOPh, mCHOPh, 1-Naphth, pNiPh, $pMeO_2CPh$, 2-naphth



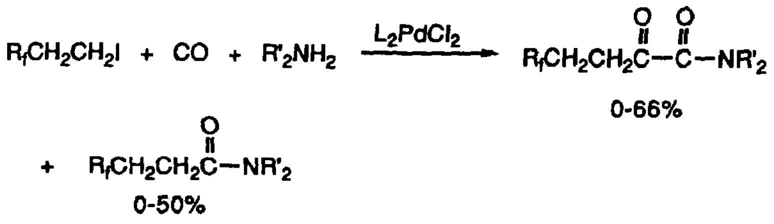
$$\mathrm{ArX} + \mathrm{Ar}\overset{\overset{\displaystyle O}{\|}}{C}\!-\!O\!-\!M \xrightarrow[\text{Ph}_3\text{P DMF} \atop \text{3 atm CO}]{\text{Pd(OAc)}_2\text{ cat.}} \mathrm{Ar}\overset{\overset{\displaystyle O}{\|}}{C}\!-\!O\!-\!\overset{\overset{\displaystyle O}{\|}}{C}\!-\!\mathrm{Ar}$$

40-80%

(447)

ArX = PhI, pMePh, pClPh, pO_2NPh, nC_{10} 

Double carbonylation has been reviewed [552]. Polystyrene bound palladium(0) complexes catalyzed the double carbonylation of halides to give α-keto esters or amides [553]. The mechanism of the palladium-catalyzed double carbonylation of aryl iodides has been stuided [554], as has the double carbonylation of iodoaryl alcohols or amines [555]. Fluorinated alkyl iodides were double carbonylated (equation 448) [556].



$$R_fCH_2CH_2I + CO + R'_2NH_2 \xrightarrow{L_2PdCl_2} R_fCH_2CH_2\overset{\overset{\displaystyle O}{\|}}{C}\!-\!\overset{\overset{\displaystyle O}{\|}}{C}\!-\!NR'_2$$

0-66%

$$+\ R_fCH_2CH_2\overset{\overset{\displaystyle O}{\|}}{C}\!-\!NR'_2$$

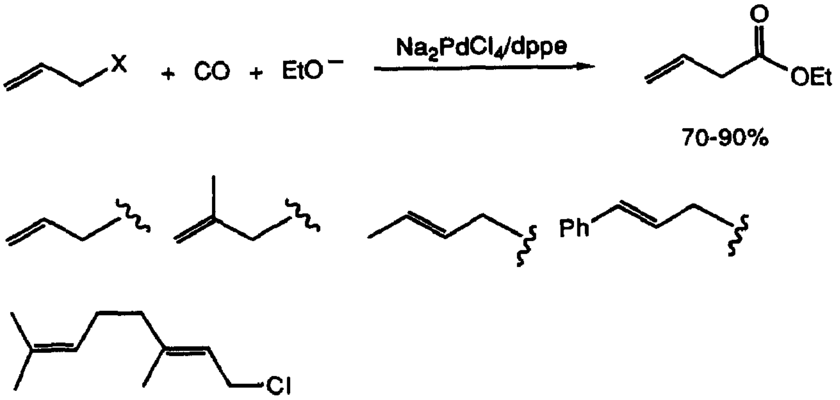
0-50%

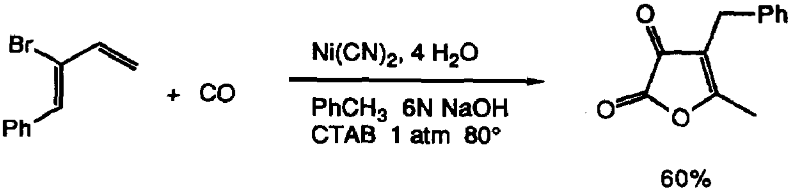
(448)

$R_f = C_8F_{17}, C_6F_{13}, C_4F_9, C_3F_7, C_2F_5CF_3$

Allyl halides were carbonylated to α,β-unsaturated esters by palladium catalysts (equation 449) [557]. 1-Phenyl-2-bromo-buta-1,3-diene was dicarbonylated by nickel catalysts (equation 450) [558]. Vinyl halides with pendent malonates cyclocarbonylated using palladium or nickel catalysts (equation 451) [559].

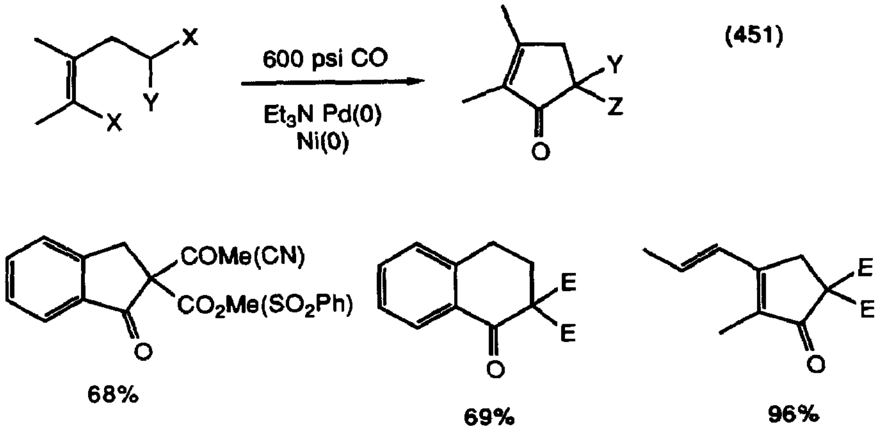
(449)





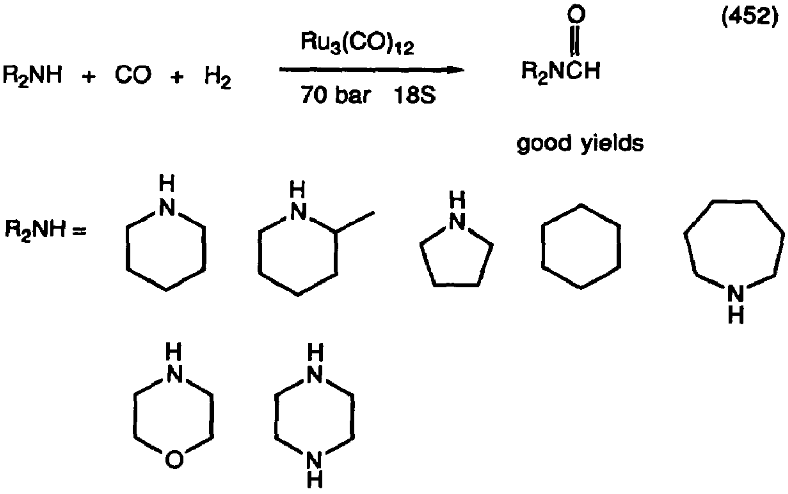
(450)

(451)

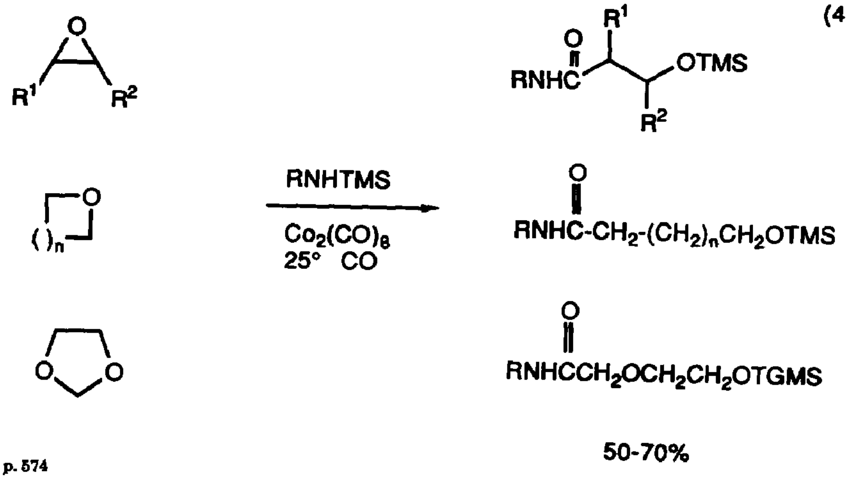


4. Carbonylation of Nitrogen Compounds.

Ruthenium complexes were used to carbonylate ammonia to urea [560]. The mechanism of the carbonylation of nitroaromatics to ureas has been reviewed (17 references) [561]. The effects of promoters, solvents and conditions on the oxidative carbonylation of aniline over palladium catalysts has been studied [562]. Ruthenium carbonyls catalyzed the N-formylation of secondary amines (equation 452) [563]. Cobalt carbonyl catalyzed the aminocarbonylation of epoxides by silylamines (equation 453) [564]. Amines were converted to α-keto amides by π-allyl palladium complexes (equation 454) [565]. Ortho amino aldehydes were also double carbonylated by palladium catalysts (equation 455) [566].

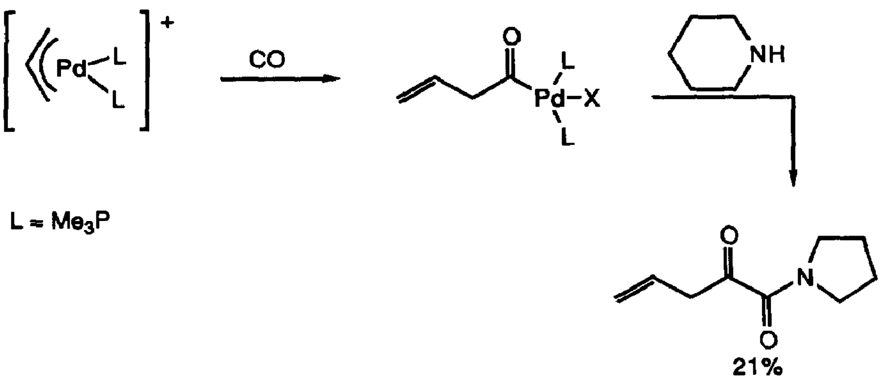


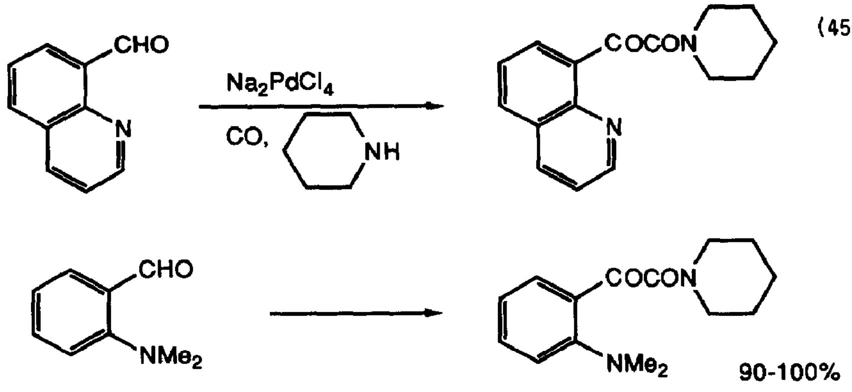
(452)



(453)

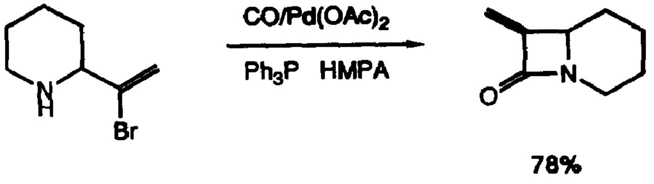
(454)





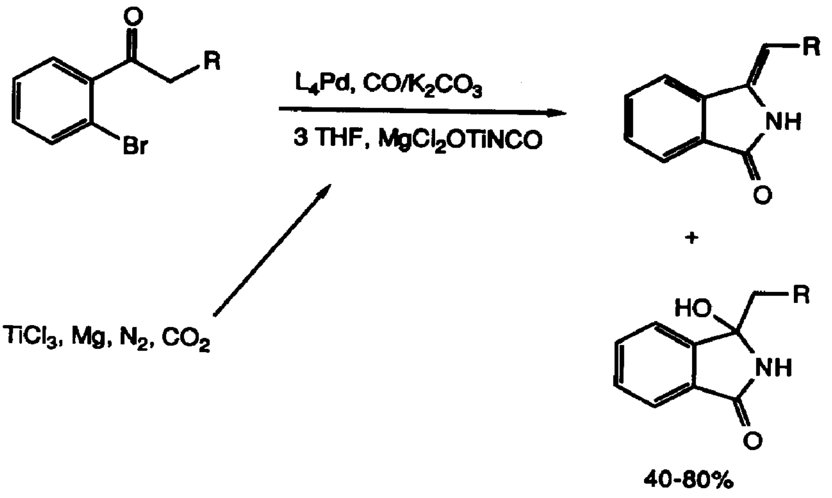
(455)

Lactams were made by palladium(0) catalyzed oxidative addition/carbonylation (equation 456) [567], (equation 457) [568], by o-metallation/carbonylation (equation 458) [569], by hydrometallation/carbonylation (equation 459) [570] and by carbonylative ring opening of aziridines (equation 460) [571] and azetidines (equation 461) [572].

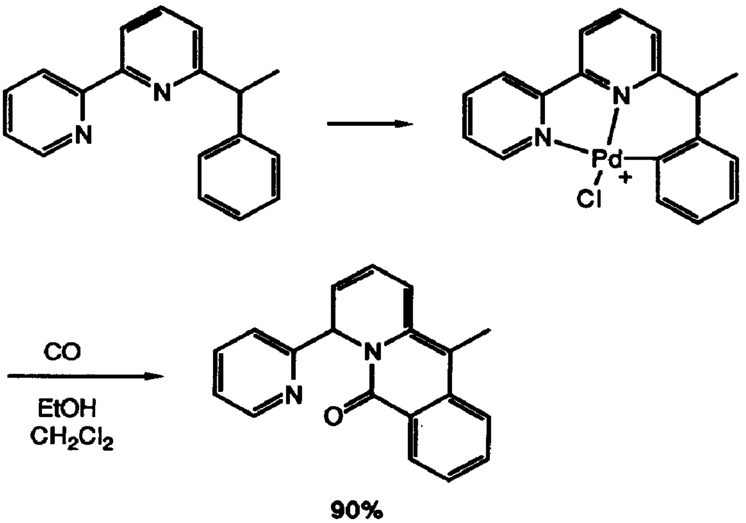


(456)

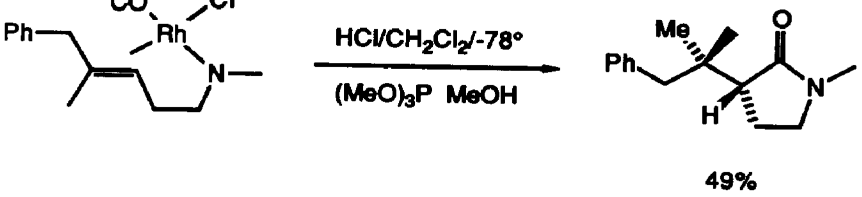
(457)


O
R
Br
L4Pd, CO/K2CO3
3 THF, MgCl2OTiNCO
TiCl3, Mg, N2, CO2
R
NH
O
+
HO
R
NH
O
40-80%

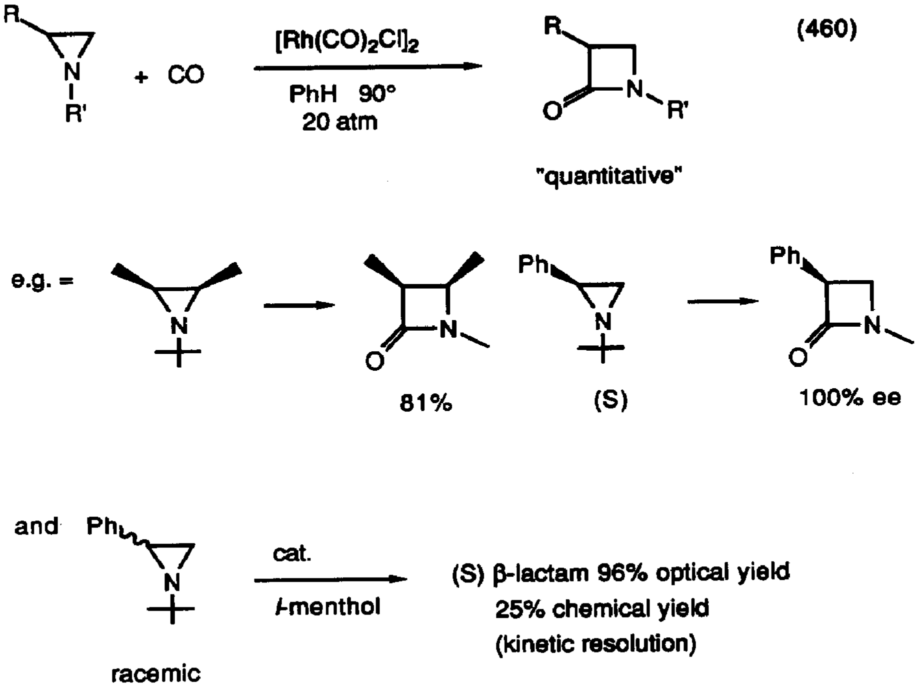
(458)


N
N
N
N
Pd
+
Cl
CO
EtOH
CH2Cl2
N
N
O
90%

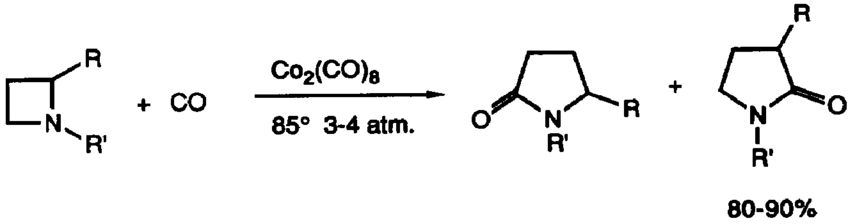
(459)


CO
Cl
Ph
Rh
N
HCl/CH2Cl2/-78°
(MeO)3P MeOH
Me
O
Ph
N
H
49%

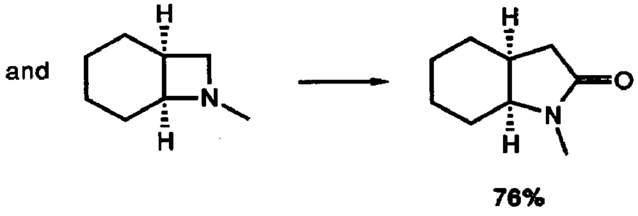
(460)



(461)



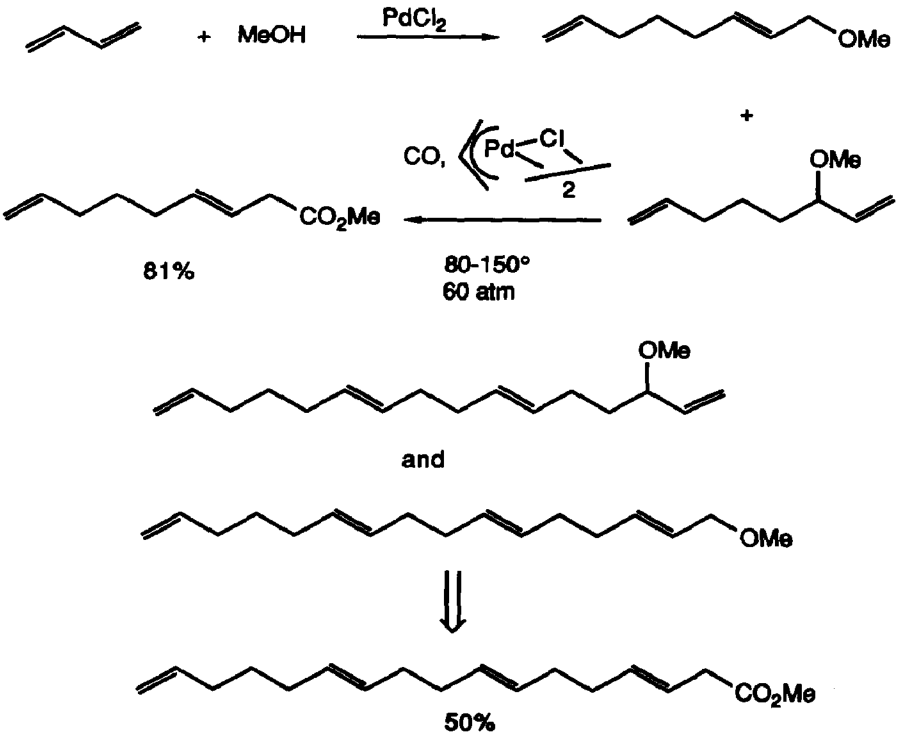
R = Ph, Me, CH_2Oh, CH_2OMe, CH_2O, COMe, CO_2Me
R' = Me, tBu



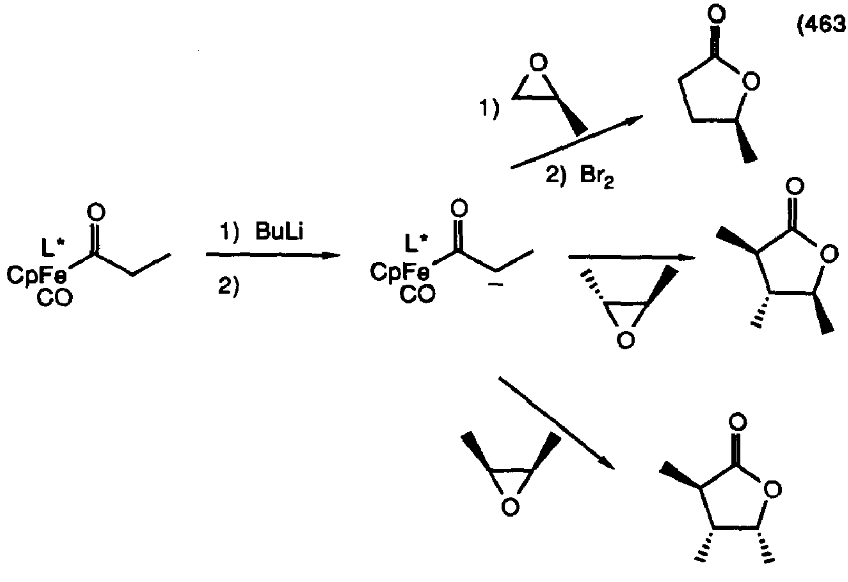
5. Carbonylation of Oxygen Compounds.

Alkoxyoctadienes were carbonylated to dienoic esters using π-allyl palladium catalysts (equation 462) [573][574]. Epoxides were converted to lactones via acyliron enolate chemistry (equation 463) [575]. Oxetanes and thietane were carbonylated to lactones and thiolactones by dicobalt octacarbonyl (equation 464) [576].

(462)

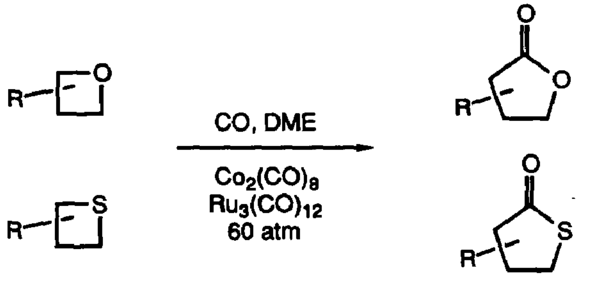


(463)



good yields
high diastereoselectivity

(464)



(inserts into least - substituted CX bond)

70-90%
R = H, 4-Me, 3-MeO

6. Miscellaneous Carbonylations.

A review (51 references) dealing with transition metal catalyzed carbonylation for synthesis of amino acids, 5-fluorouracils, and methacryl acid derivatives has appeared [577]. Organic synthesis with tricarbonyliron lactones has been reviewed (20 references) [578]. Aryl sulfonyl chlorides (equation 465)

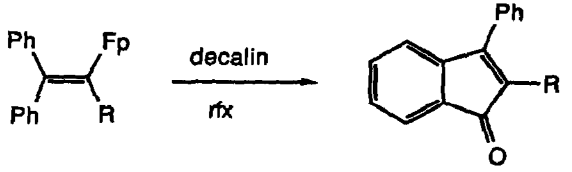
[579] and azo compounds (equation 466) [580] were converted to aryl carboxylic acids using palladium catalysts. Alkenyliron complexes formed indenones on thermolysis (equation 467) [581]. Labeled cyclopentadiene was made by reductive carbonylation of butadiene (equation 468) [582].

$$\mathrm{ArSO_2Cl} \xrightarrow{\mathrm{CO/PdL_4/Ti(OR)_4}} \mathrm{ArCO_2R} + \mathrm{ArSSAr} \qquad (465)$$

30-70% 14-40%

Ar = 1-Naphth

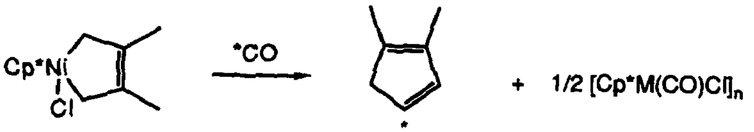
$$\mathrm{ArN{=}NSO_2Ar'} + \mathrm{CO} \xrightarrow{\mathrm{L_4Pd}} \mathrm{ArCO_2H} + \mathrm{Ar'CO_2H} + \mathrm{ArAr'} + \mathrm{ArCOAr'} \qquad (466)$$



(467)

78-86%

R = Ph, 1-Naphth



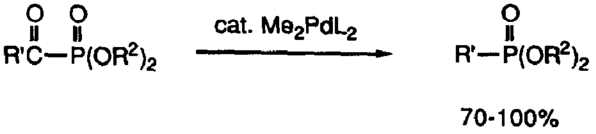
(468)

70-80%

7. Decarbonylation Reactions.

This was not a very popular endeavor, with a single example being reported this year (equation 469) [583].

(469)



$$\mathrm{R'}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}-\overset{\mathrm{O}}{\overset{\|}{\mathrm{P}}}(\mathrm{OR^2})_2 \xrightarrow{\text{cat. }\mathrm{Me_2PdL_2}} \mathrm{R'}-\overset{\mathrm{O}}{\overset{\|}{\mathrm{P}}}(\mathrm{OR^2})_2$$

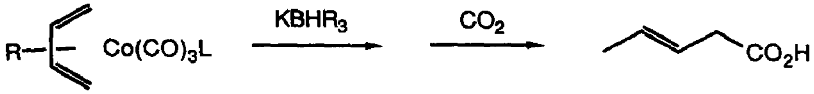
70-100%

R^1 = Ph, pMePh, pMeOPh, pClPh
R^2 = Me, Et

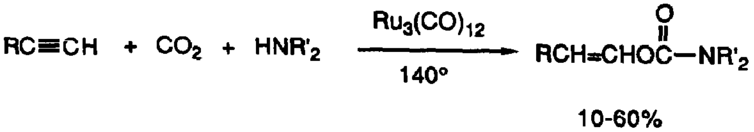
8. Reactions of Carbon Dioxide.

The catalytic carboxylation of olefins by carbon dioxide in the presence of palladium and rhodium complex catalysts was the subject of a report [584]. Dienes were carboxylated via reduction of cobalt carbonyl complexes (equation 470) [585]. Alkynes were hydrocarboxylated by carbon dioxide using $Ru_3(CO)_{12}$ (equation 471) [586] and reduced nickel catalysts (equation 472) [587], (equation 473) [588], (equation 474) [589].

(470)



(471)



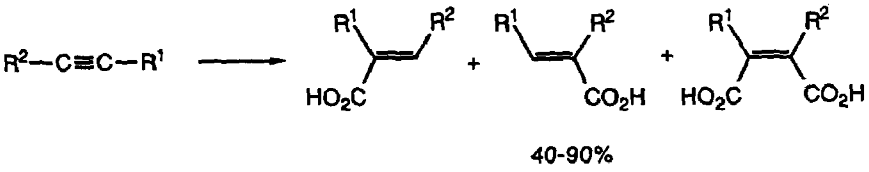
$$\mathrm{RC{\equiv}CH + CO_2 + HNR'_2} \xrightarrow[140^\circ]{\mathrm{Ru_3(CO)_{12}}} \mathrm{RCH{=}CHO}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{-NR'_2}$$

10-60%

R = nBu, Ph,
R_2NH = Et, Me, $(CH_2)_5$,

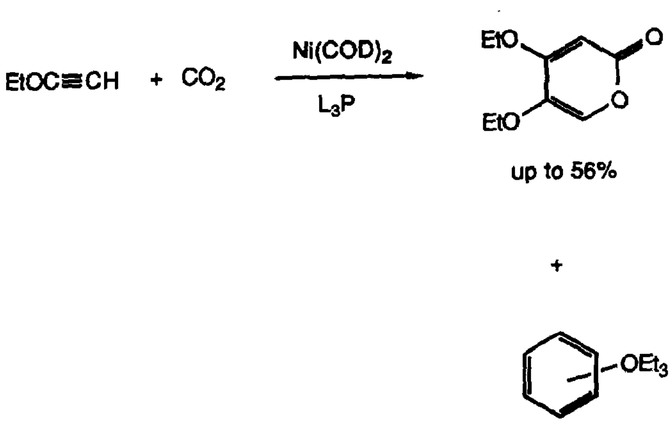
(472)

AND

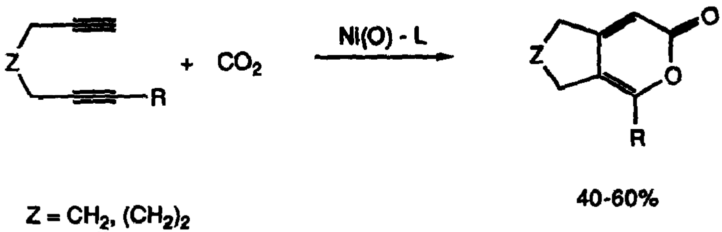


R^1 = nPr, Ph, TMS, Me
R^2 = nPr, TMS, CO_2Et, $CH_2CH_2OCOCH_3$

(473)

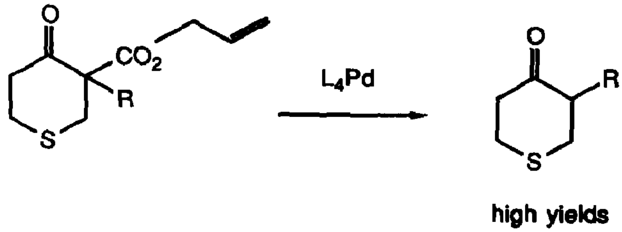


(474)

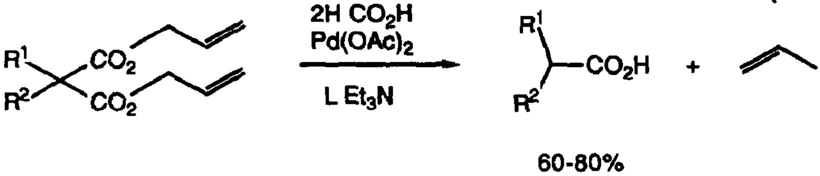


Z = CH_2, $(CH_2)_2$
R = H, iPr

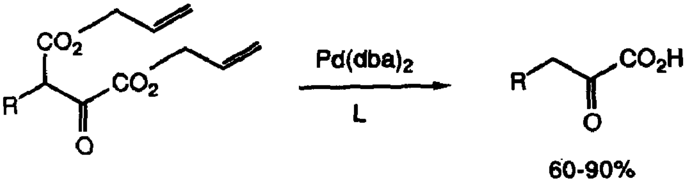
Carbon dioxide was extruded from allyl phenyl carbonates [590] and a variety of other allyl esters, by palladium(0) catalysts (equation 475) [591], (equation 476) [592], (equation 477) [593], (equation 478) [594].



(475)



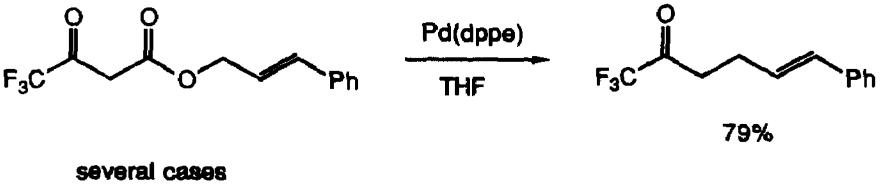
(476)



(477)

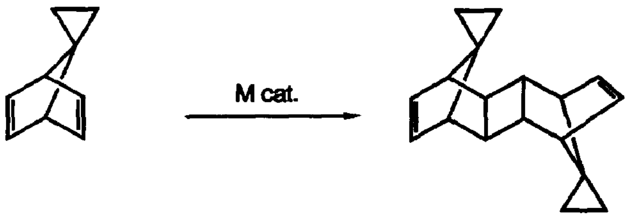
R = PhCH$_2$, pCF$_3$PhCH$_2$, oCF$_3$PhCH$_2$, oFPhCH$_2$, Ph, Me, ( )$_7$

(478)



D. Oligomerization including Cyclotrimerization of Alkynes and Metathesis Polymerization.

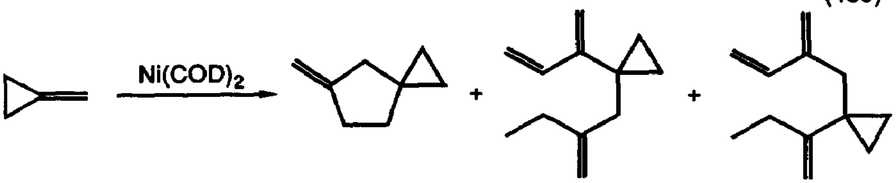
The complexes [η^6-arene Ni($SiCl_3$)$_2$] and [η^6-arene Co($SiCl_3$))] were efficient catalysts for the dimerization of ethene and propene [595]. The catalytic properties of free and alumina-supported triphenylphosphine nickel catalysts for dimerization of ethene were compared [596]. Palladium chloride dimerized ethene to linear butene []597], while nickel(0) complexes catalyzed the dimerization of styrene and stilbene [598]. Palladium(II) species on sepiolite catalyzed olefin dimerization [599]. Group(VIII) transition metals catalyzed the homodimerization of strained olefins (equation 479) [600], methylene cyclopropanes (equation 480) [601], and vinyl cyclopropanes [602]. Nickel(0) complexes catalyzed the cyclodimerization of butadiene (equation 481) [603].



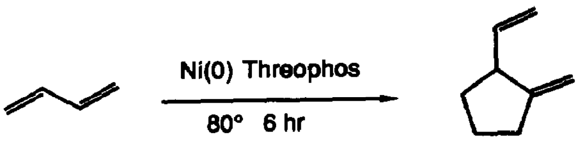
(479)

M = $Fe(CO)_5$, $Co(acac)_2$, $[Rh(CO)_2Cl]_2$, etc.

(480)



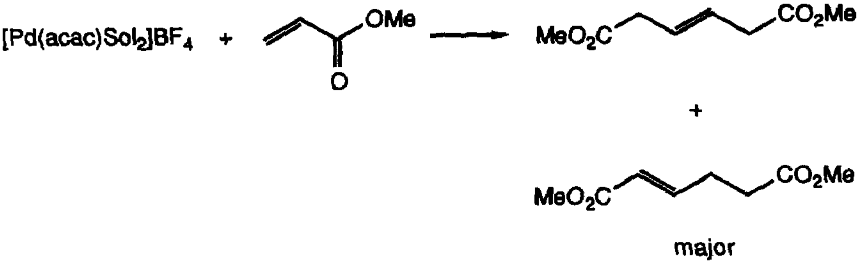
(481)



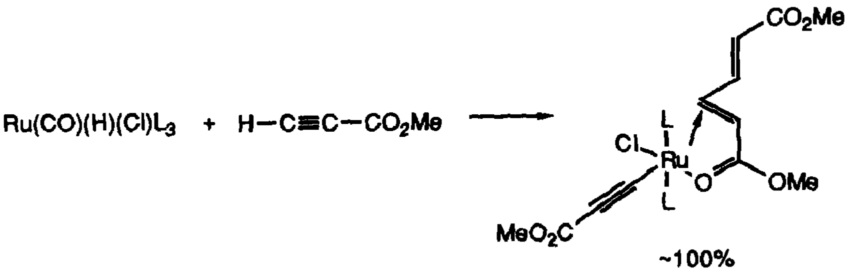
70% selectivity
90% conversion

Methyl acrylate was tail-to-tail dimerized by stoichiometric amounts of a cationic palladium(II) complex (equation 482) [605]. Zirconium(II) complexes dimerized and codimerized alkynes (equation 484) [606], as did cationic cobalt(I) complexes (equation 485) [607].

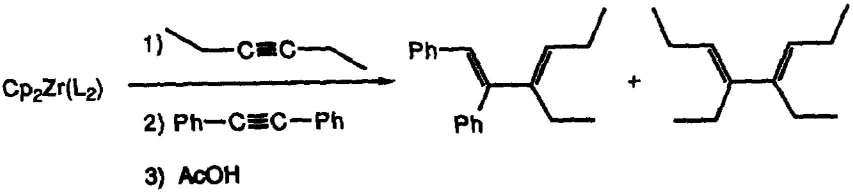
(482)



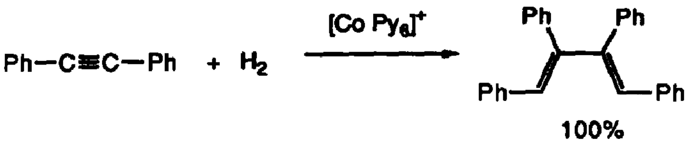
(483)



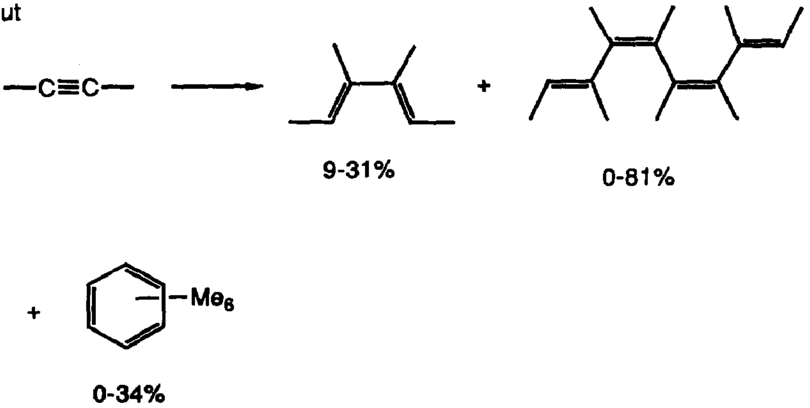
(484)



(485)

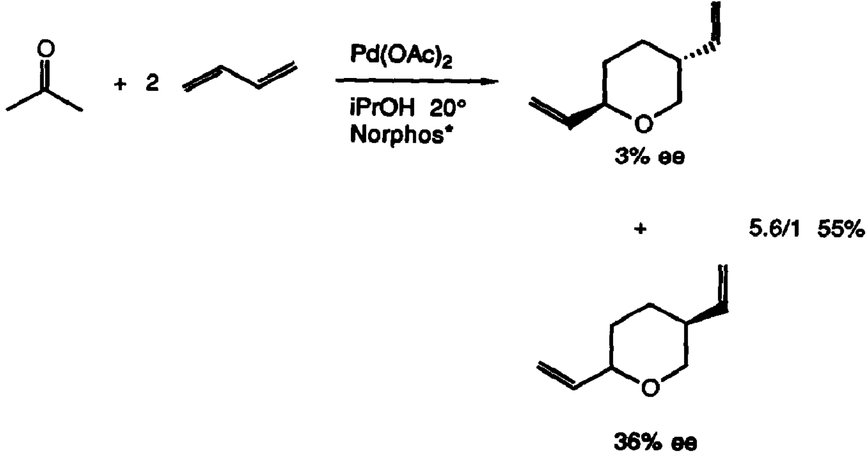


but

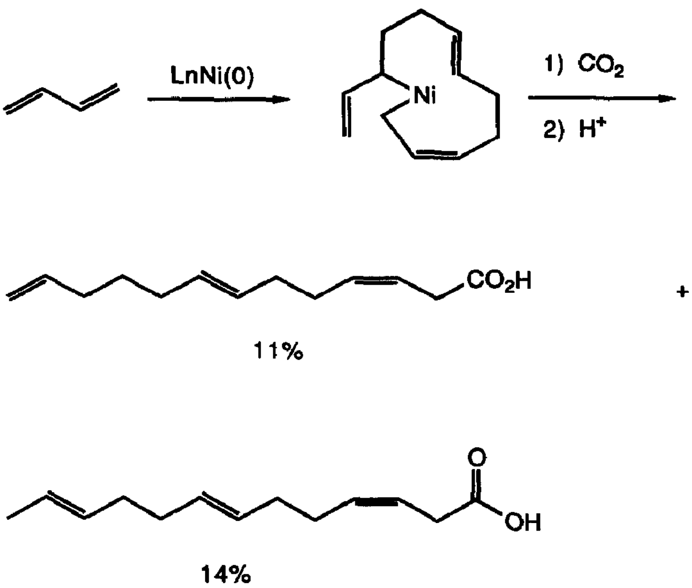


The stereochemistry of the palladium catalyzed telomerization of butadiene with diethylamine has been studied [608]. Palladium(II) acetate catalyzed the codimerization of acetone with butadiene (equation 486) [609]. Nickel(0) complexes cyclotrimerized butadiene, and then carboxylated the trimer with carbon dioxide (equation 487) [610].

(486)

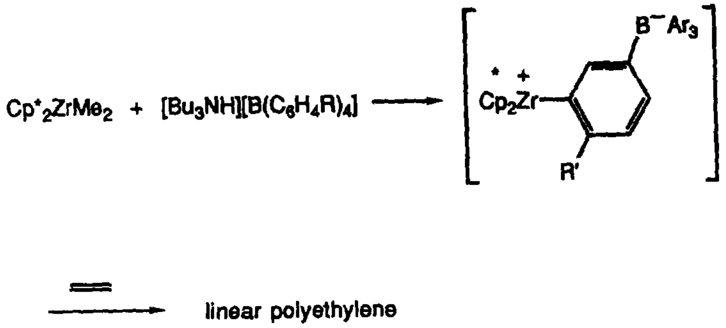


(487)

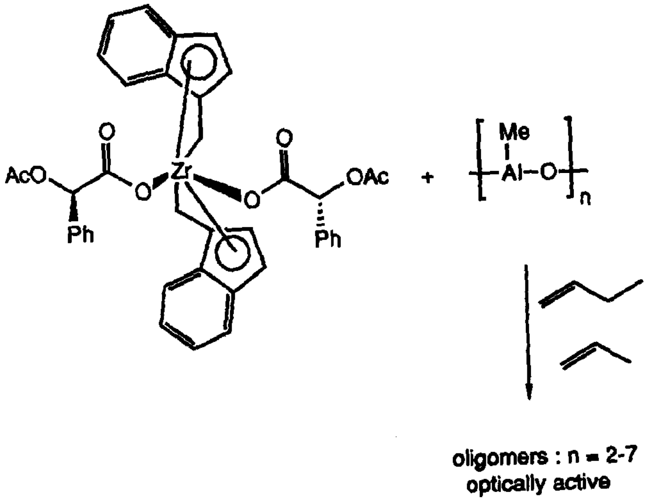


Cyclopentadienylzirconium borate complexes catalyzed the linear polymerization of ethylene (equation 488) [611]. Butenes were dimerized with >96% selectivity by η^3-allylnickel cyclooctadiene salts [612]. Ethylene was oligomerized by zirconium acetylacetonate supported on phosphorylated aluminum oxide [613]. Nickel hydrides were shown to be involved in nickel catalyzed ethene oligomerization [614]. Nickel pentane-2,5-dithionato complexes dimerized butenes efficiently [615]. Propene and 1-butene were asymmetrically oligomerized by a chiral zirconocene-aluminoxane catalyst (equation 489) [616]. The synthesis and characterization of chiral metallocene catalyst (equation 489) [616]. The synthesis and characterization of chiral metallocene cocatalysts for stereospecific α-olefin polymerization was reviewed (26 references) [617]. Alkyl aluminum/$YbCl_3$ catalysts cyclooligomerized alkenes in the presence of carbon monoxide (equation 490) [618]. Phenylallene [619] and bicyclopropylidene were oligomethylated by diazomethane in the presence of palladium acetate [620]. The total synthesis of polyprenoid natural products via palladium(0) catalyzed oligomerizations was reviewed (31 references) [621]. Nickel salts deposited on silica or alumina catalyzed olefin oligomerization [622].

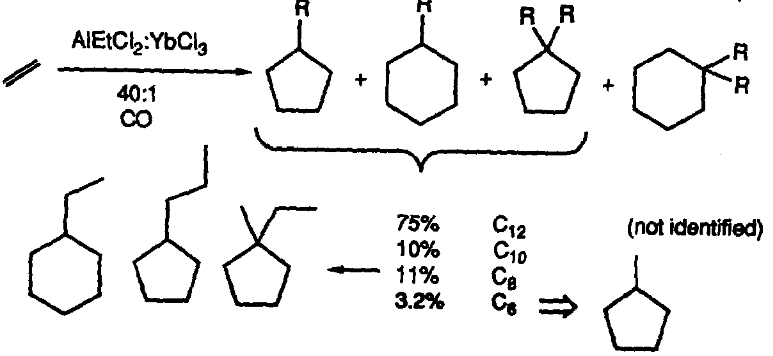
(488)


Cp*$_2$ZrMe$_2$ + [Bu$_3$NH][B(C$_6$H$_4$R)$_4$]
Cp$_2$Zr
* +
B$^-$Ar$_3$
R'
linear polyethylene

(489)

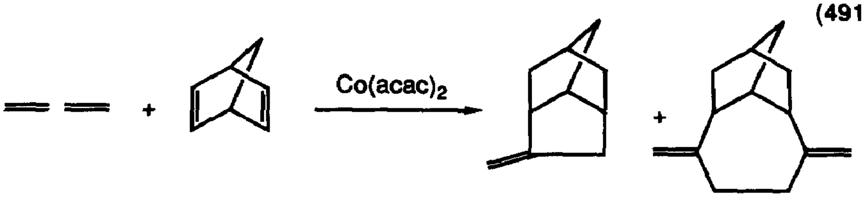

AcO
O
Zr
OAc
Ph
Me
Al
n
oligomers : n = 2-7
optically active

(490)

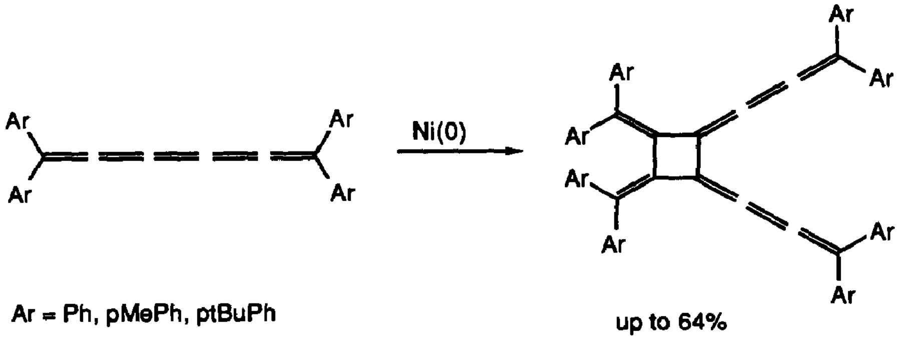

AlEtCl$_2$:YbCl$_3$
40:1
CO
R
R R
75% C$_{12}$
10% C$_{10}$
11% C$_8$
3.2% C$_6$
(not identified)

Procedures (conditions) for the optimum production of methylene-2-vinylcyclopentane by cyclodimerization of butadiene by nickel complexes have been developed [623]. Nickel acetylacetonate/alkyl aluminum catalysts codimerized propylene with 1-hexene [624]. The cooligomerization of allene with propyne was also catalyzed by nickel(0) complexes [625]. Allenes and norbornadiene were cooligomerized by cobalt catalysts (equation 491) [626]. Nickel(0) complexes dimerized cumulenes (equation 492) [627] and cyclotrimerized gem-dihaloolefins (equation 493) [628]. Aryl dihalides were coupled to distannanes (equation 494) [629] or dizinc reagents (equation 495) [630] to give oligomeric polyaromatic compounds.

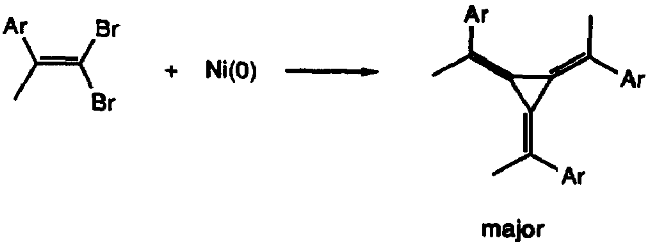
(491)



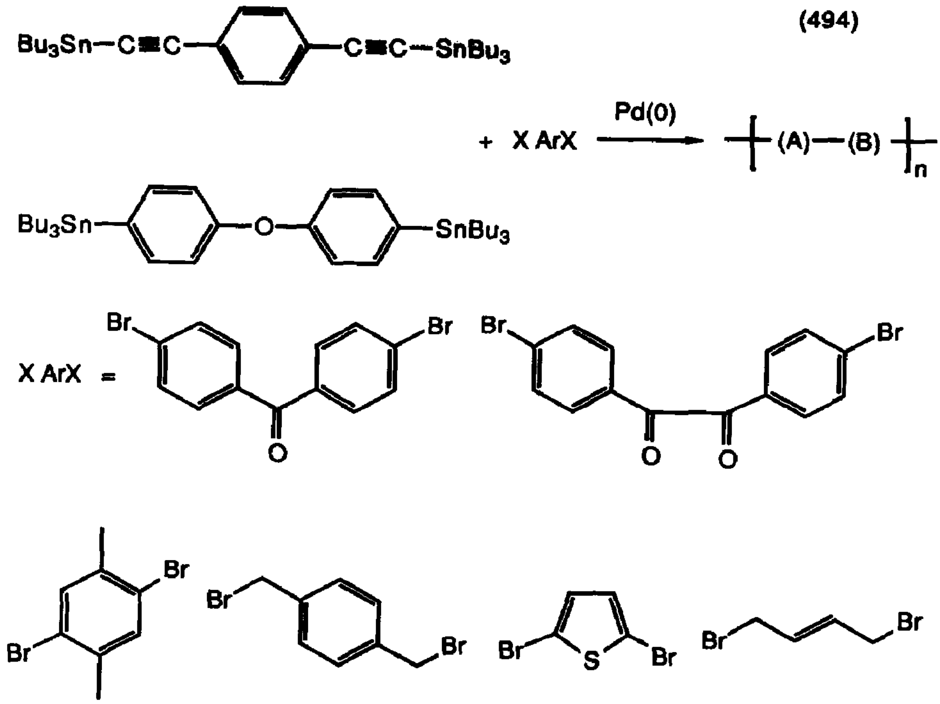
(492)



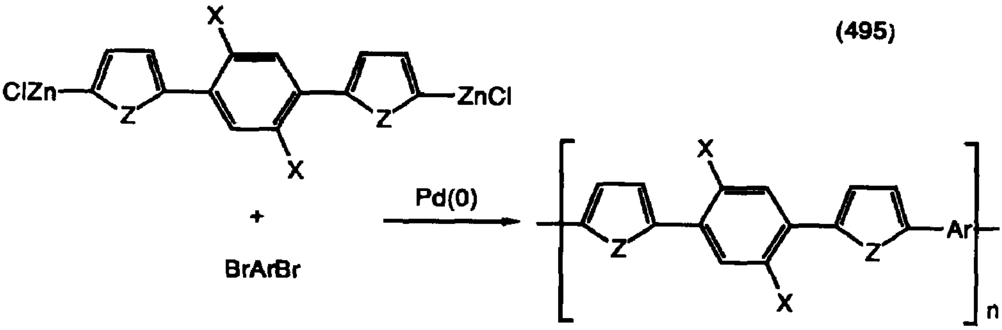
(493)



(494)



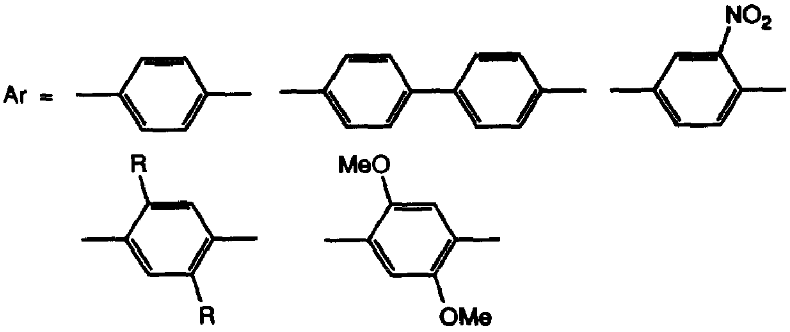
(495)



Z = S, O

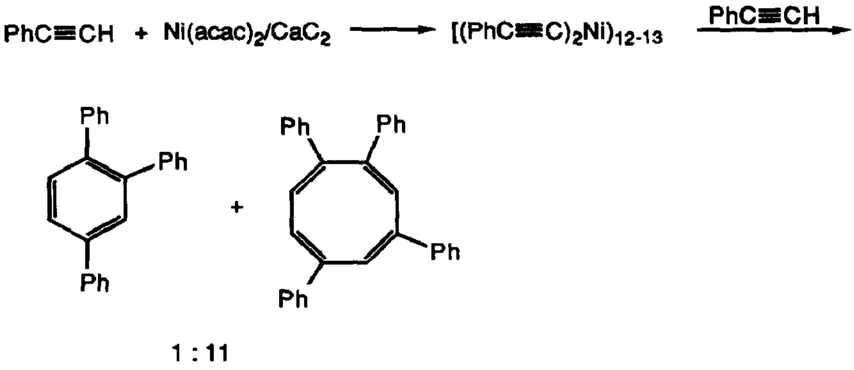
X = H, NO_2, Me, OMe

Y = H, Me, Bu

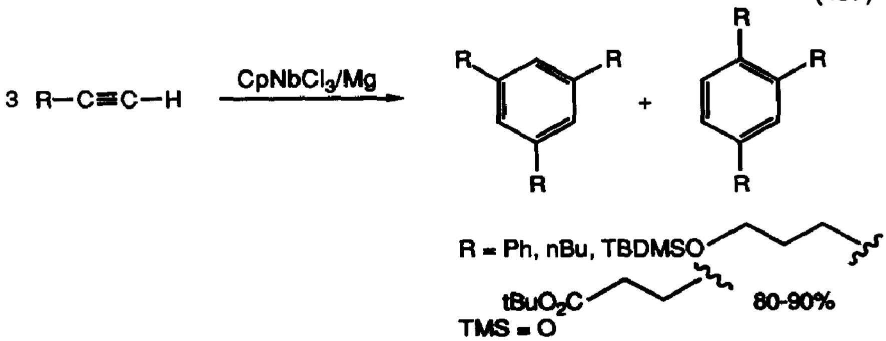


Phenylacetylene was cyclotrimerized by cobalt carbonyl clusters [631]. Alkyne alcohols were cyclotetramerized by nickel(0) catalysts [632]. Oligomeric nickel acetylides reacted with alkynes to give cyclic trimers and tetramers (equation 496) [633]. Reduced niobium complexes cyclotrimerized alkynes (equation 497) [634]. Rhodium(I) complexes cyclotrimerized alkynes (equation 498) [635].

(496)

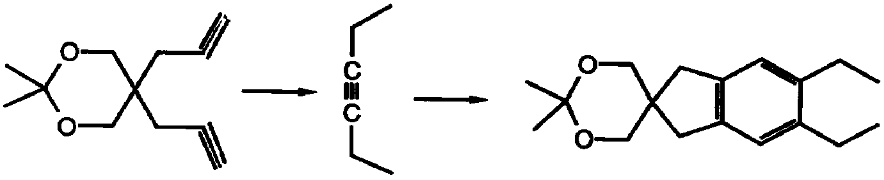


(497)



and

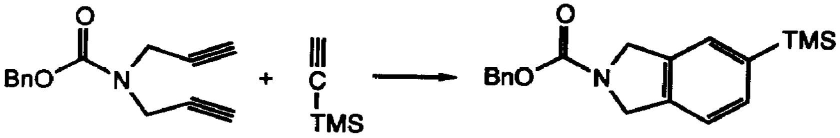
also



80%

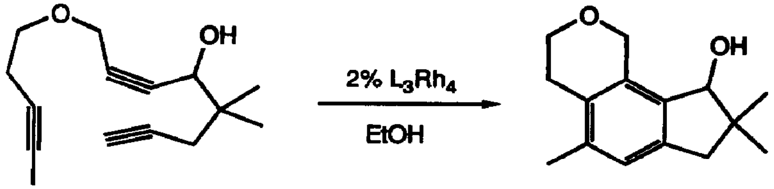
74%

and



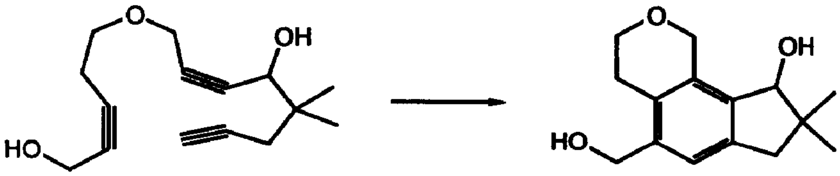
52%

(498)



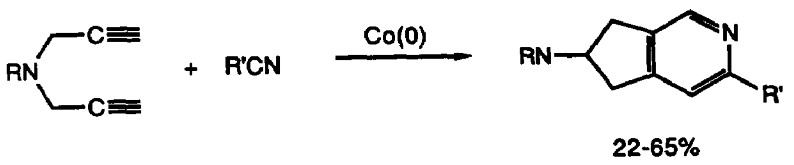
82%

and



86%

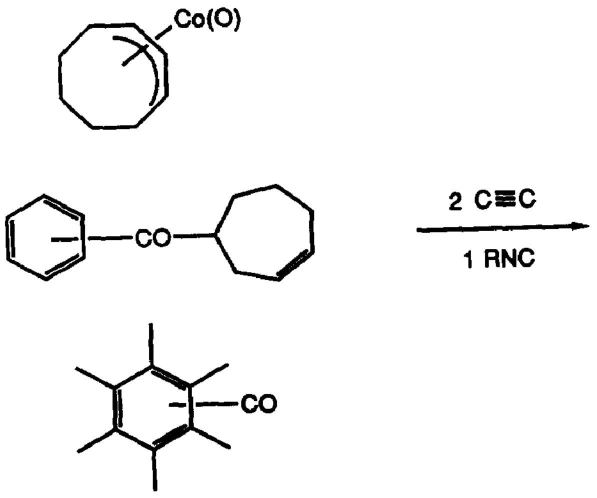
Cobalt-mediated syntheses of polyheterocycles have been reviewed (28 references) [636]. Dissertations entitled "Intermolecular [2+2+2] cycloaddition reactions of alkynes and alkenes mediated by cobalt" [637] and "Cobalt-mediated [2+2+2] cycloadditions of enamine double bonds to alkynes: activation of indole and applications to alkaloid synthesis [638] have appeared. Cobalt(0) complexes catalyzed the cocyclotrimerization of nitriles with (*bis*)-propargyl amines (equation 499) [639] and with simple alkynes to give pyridines (equation 500) [640], (equation 501) [641]. Cobalt(0) complexes also catalyzed the intramolecular cyclotrimerizations of diyne aldehydes (equation 502) [642]. Orthopalladated ferrocenylamines incorporated phenyl acetylene when heated (equation 503) [643].



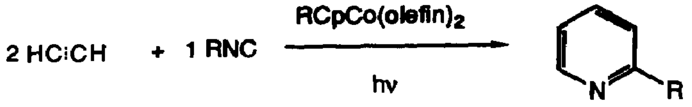
(499)

R = Me, iBu, Bu, Bn, CO_2Me, $AcEt_2CHO$, pTs

R' = Me, Ph, Bu

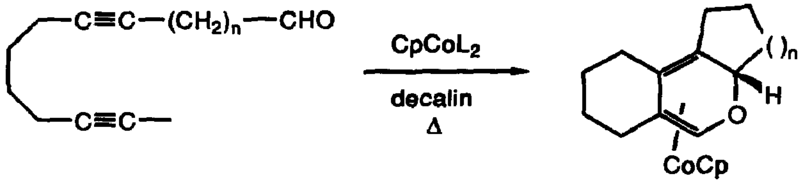


(500)



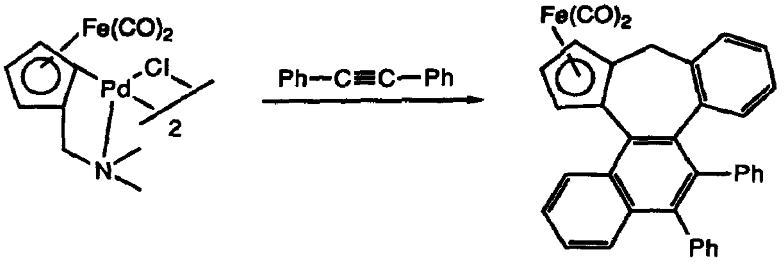
(501)

66,000 turnovers
up to 43% conversion



(502)

n = 1 8%
n = 2 45%

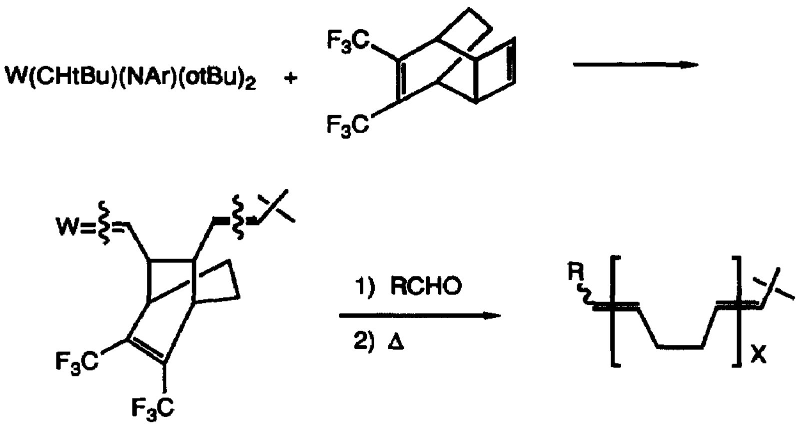


(503)

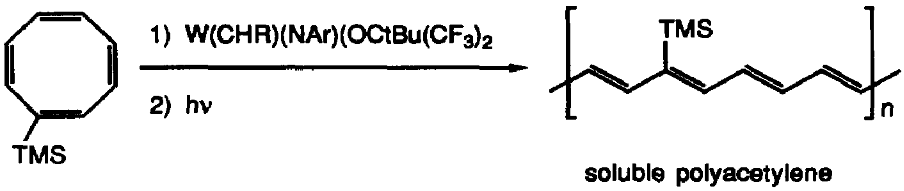
15%

Tungsten alkylidene complexes catalyzed the ring opening polymerization of cyclic polyenes (equation 504) [644], (equation 505) [645]. An initial metallocyclobutane thought to be involved was characterized by nmr spectroscopy [646]. Tantalum (equation 506) [647] and tungsten alkylidene complexes (equation 507) [648] gave living polymers of alkynes which could be capped by aldehydes or used to construct block copolymers.

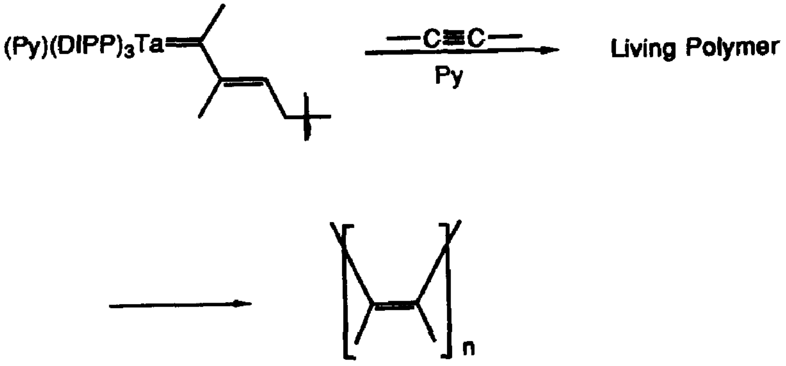
(504)


W(CHtBu)(NAr)(otBu)$_2$
+
F$_3$C
F$_3$C
W=
F$_3$C
F$_3$C
1) RCHO
2) Δ
R
X

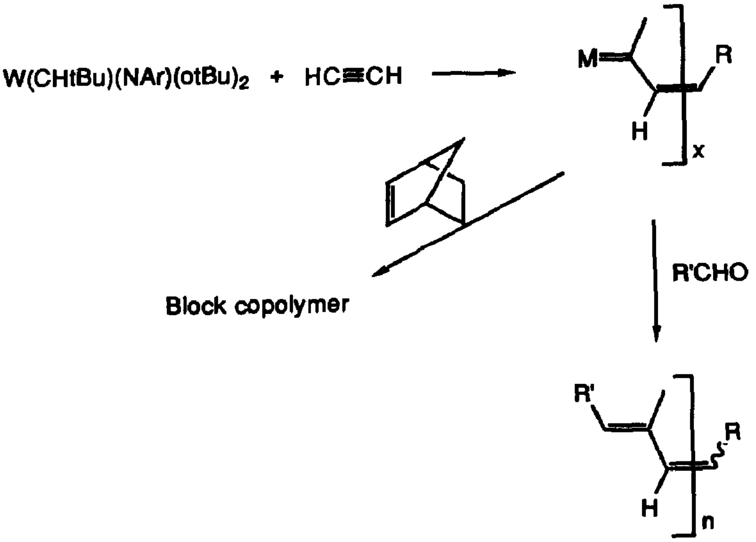
(505)


TMS
1) W(CHR)(NAr)(OCtBu(CF$_3$)$_2$
2) hν
TMS
n
soluble polyacetylene

(506)

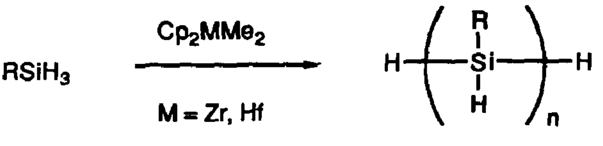

(Py)(DIPP)$_3$Ta
—C≡C—
Py
Living Polymer
n

(507)

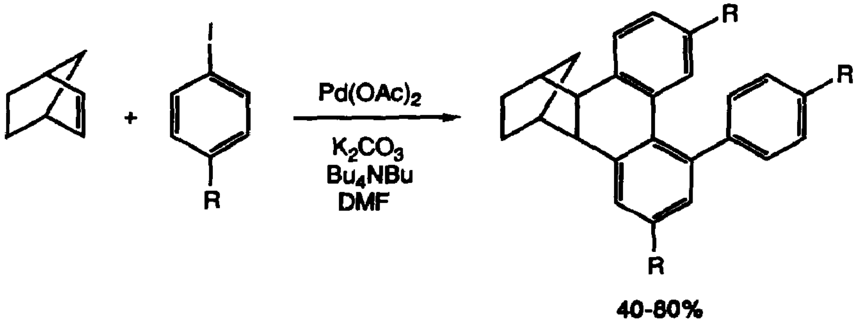


Monoalkylsilylhydrides were polymerized by zirconium and hafnium catalysts (equation 508) [649]. Palladium acetate catalyzed the reaction in equation 509 [650]. Iron carbonyls dimerized bridged bicyclic aldehydes (equation 510) [651].

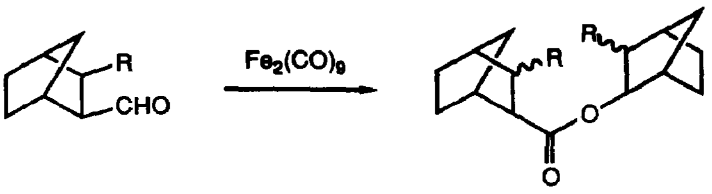
(508)



(509)



(510)



71%

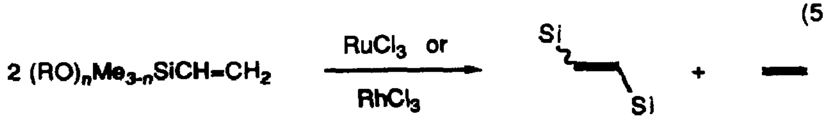
E. Rearrangements.

1. Metathesis

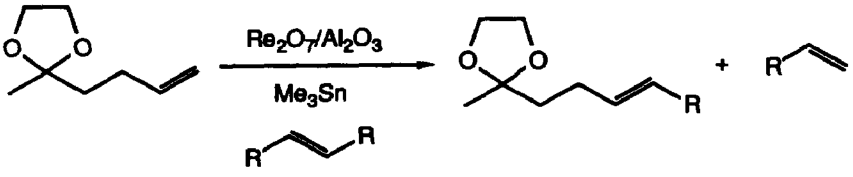
Olefin metathesis and related reactions were reviewed (69 references) [652] as was the mechanism of the olefin metathesis reaction (67 references) [653]. Photoinduced metathesis and other reactions of olefins by Group (VIB) metal carbonyls was reviewed (76 references) [654]. A simple preparation of the metathesis catalyst $Mo(NtBu)(CHtBu)[OCH(CF_3)_2]_2$ was developed [665]. 2-Pentene and 2-butene were metathesized by $[WBr_2(CO)_2(diene)]/AlCl_2Et$ catalysts [656]. Terminal alkenes were metathesized by $WCl_2(PhO)_4$ complexes [657]. 2-Methyl-2-alkenes were synthesized by cometathesis of α-olefins with isobutene [658]. The activity and selectivity of rhenium based catalysts for alkene metathesis was studied [659], as was the metathesis of alkenes over cesium nitrate-rhenia-alumina catalyst [660][661]. Propylene was metathesized over titanium tetrachloride/samarium or ytterbium particles [662]. The effect of support on the metathesis of propylene over anchored molybdenum and tungsten carbonyl catalyst was studied [663][664]. Treatment of silica gel with $(tBuO)_3W{\equiv}C\text{-}tBu$, gave a solid supported carbene complex which was an efficient metathesis catalyst for 1-octene [665].

Vinyl silanes were metathesized by $RuCl_3$ or $RhCl_3$ and Lewis acids (equation 511) [666][667]. Rhenium/alumina/tin catalysts metathesized olefins containing ketals or silylenol ethers (equation 512) [668]. Tungsten hexachloride/tetramethyl tin systems catalyzed the cometathesis of methyl oleate with α-olefins [669]. Pheromone components were synthesized by cometathesis reactions [670].

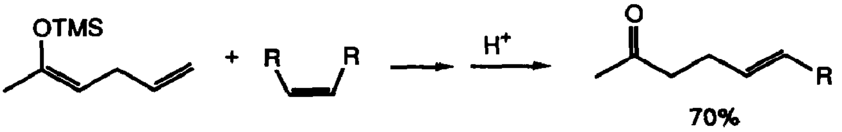
(511)



(512)



and



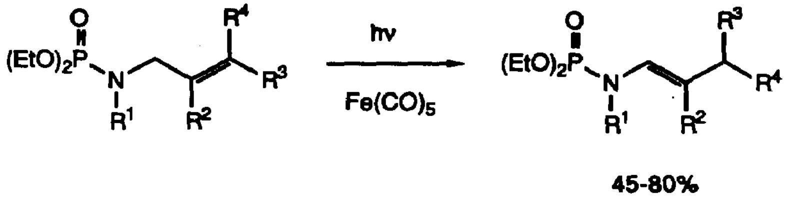
2. Olefin Isomerization

A variety of zirconium hydride isomerization catalysts were developed [671]. Titanocene dichloride/i-propyl Grignard systems isomerized 4-vinylcyclohexene [672]. Supported rhenium metathesis catalysts were studied for their activity in 1-butene isomerization [673].

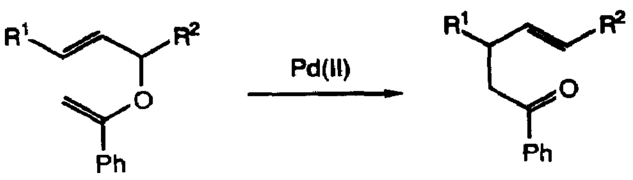
3. Rearrangement of Allylic and Propargylic Systems

N-Allylphosphonamides rearranged to eneamine systems when photolyzed with iron carbonyl (equation 513) [674]. Palladium(II) catalyzed oxy Cope rearrangements (equation 514) [675]. Allyl epoxides isomerized α,β-unsaturated carbonyl compounds when heated with rhodium(I) hydrides (equation 515) [676]. Propargyl alcohols rearranged to α,β-unsaturated aldehydes when treated with $RuCl_2L_3$ [677] or IrH_5 (L_2) [678] (equation 516). Alkynones rearranged to dienones under similar conditions (equation 517) [679].

(513)

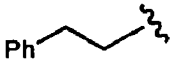


R^1 = H, Me, iPr, Bn, nC_9
R^2, R^3, R^4 = H, Me

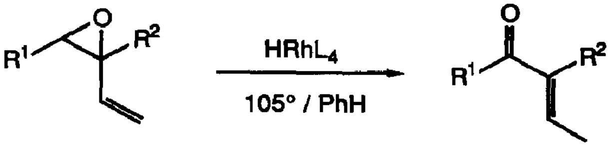


(514)

18-80%

R^1 = Me, nPr, H, 

R^2 = H, Me



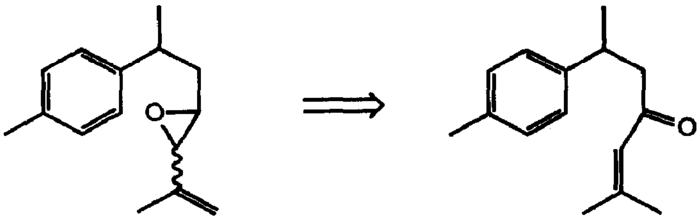
(515)

35-1%

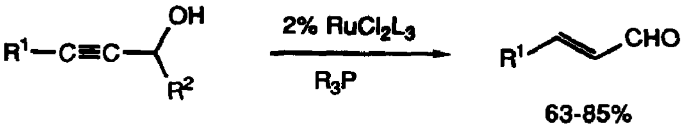
R^1 = H, nC_5, nC_8, Et

R^2 = Me, Et, Pr, H, nC_5

and



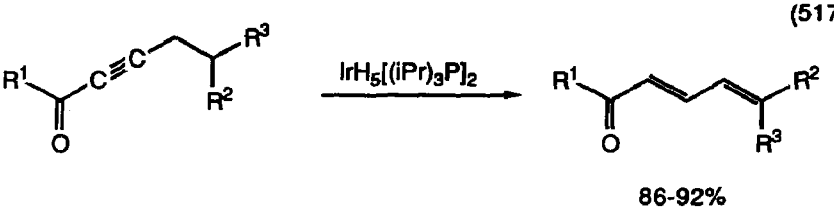
86%



(516)

$R^1 = C_6, C_5, C_7, C_4, Ph$
$R^2 = H, Me, Et, Ph$

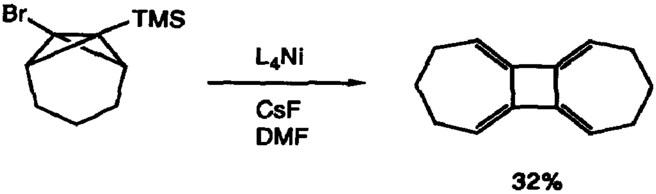
(517)



$R^1 = Me, Ph, Et$
$R^2 = C_5, Me, Et, Pr$
$R^3 = H, Me$

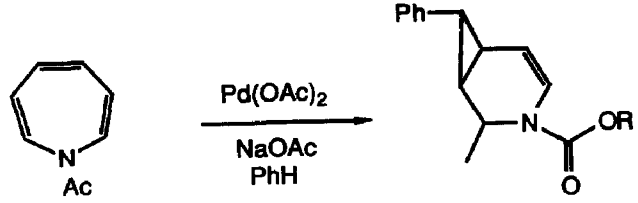
4. Skeletal Rearrangements

Skeletal transformations of saturated hydrocarbons on Group(VIII) metals was reviewed (57 references) [680]. Other skeletal rearrangements are shown in equation 518 [681]; equation 519 [682]; equation 520 [683]; equation 521 [684] and equation 522 [685].



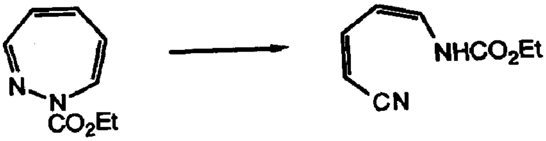
(518)

(519)

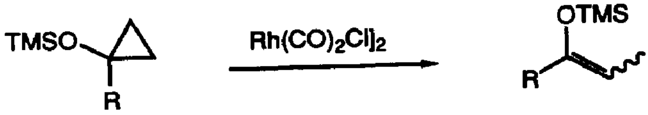


20%

and



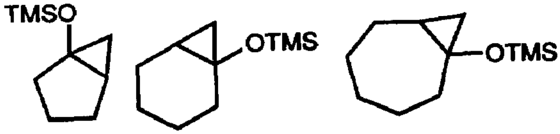
(520)



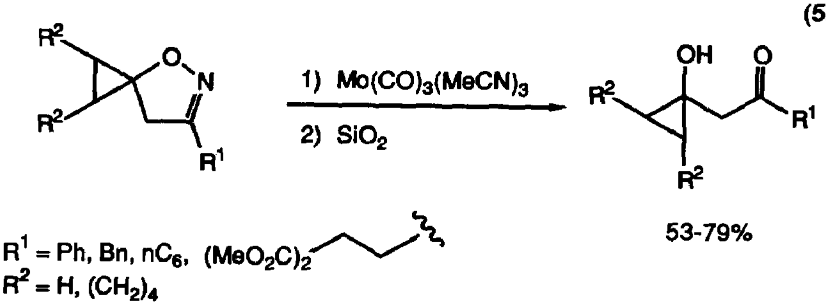
60-90%

R = Ph, H

also



(521)

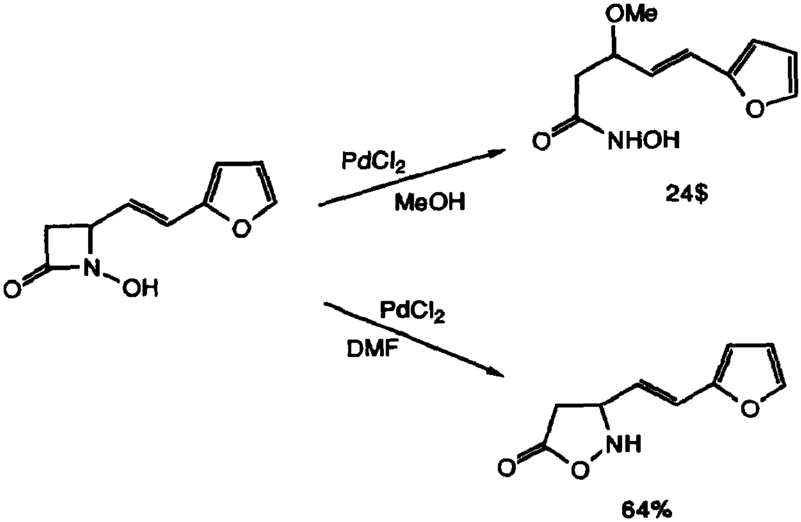


1) Mo(CO)$_3$(MeCN)$_3$
2) SiO$_2$
R^2
R^1
OH
O
N
53-79%
R^1 = Ph, Bn, nC$_6$, (MeO$_2$C)$_2$
R^2 = H, (CH$_2$)$_4$

also

Ph
O
N

(522)



PdCl$_2$
MeOH
OMe
NHOH
24$
OH
PdCl$_2$
DMF
NH
64%

5. Miscellaneous Rearrangements

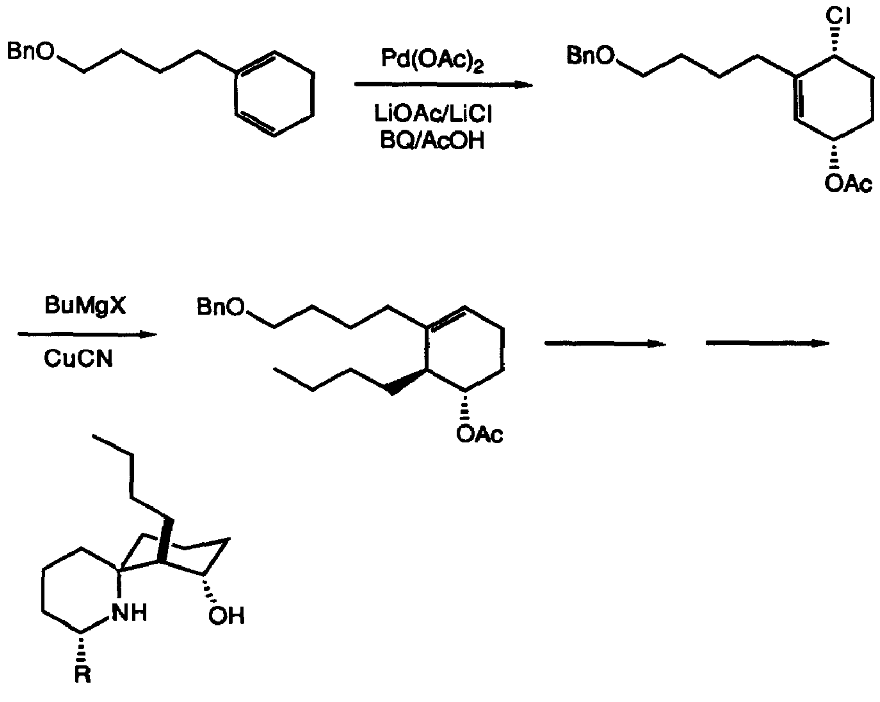
There were none to note this year.

III. Functional Group Preparations

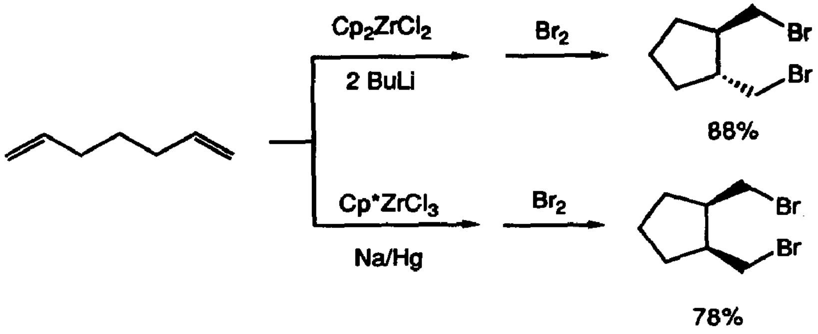
A. Halides

Metal-mediated additions to conjugated dienes has been reviewed (91 references) [686]. Palladium-catalyzed chloroacetoxylation of butadiene provided the starting material for the synthesis of [5E,9E]-5,9-hexa as dienoic acid [687], and the same process with a functionalized cyclohexadiene was used in an alkaloid synthesis (equation 523) [688]. Dihalides were prepared by halogenation of zirconacycles (equation 524) [689]. Secondary and tertiary chlorides were converted to bromides in good yield without elimination by treatment with HBr gas in the presence of catalytic amounts of iron(III) chloride [690]. Perfluoroalkyl iodides added to olefins in the presence of $L_3Ir(H)CO$ [691]. Copper(I) (equation 525) [692] and ruthenium(II) (equation 526) [693], (equation 527) [694] catalyzed the addition of chlorofluorocarbons to olefins.

(523)



(524)



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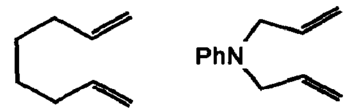

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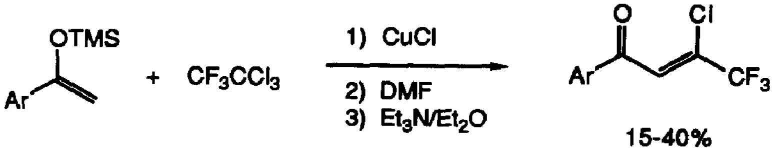
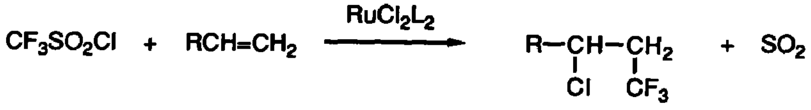


(526)

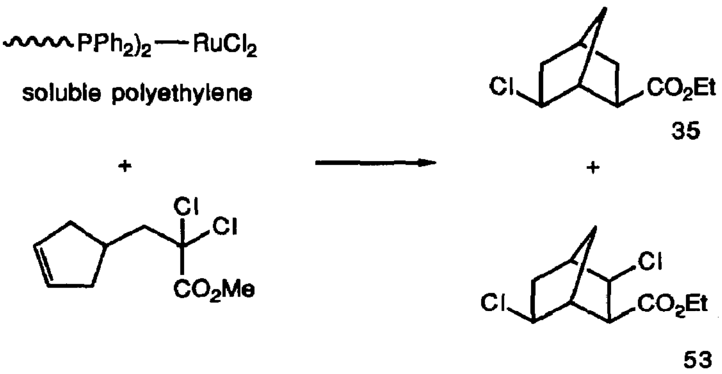


$$\mathrm{CF_3SO_2Cl + RCH{=}CH_2 \xrightarrow{RuCl_2L_2} R{-}\underset{\displaystyle Cl}{CH}{-}\underset{\displaystyle CF_3}{CH_2} + SO_2}$$

R = Ph, pClPh, mNO$_2$Ph, pMePh
nC$_6$, nC$_8$, nC$_{10}$, Et$_2$OC, PhCO$_2$

50-80%

(527)



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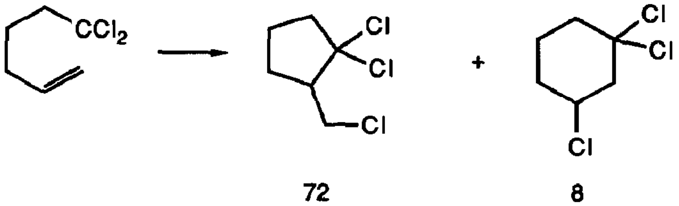
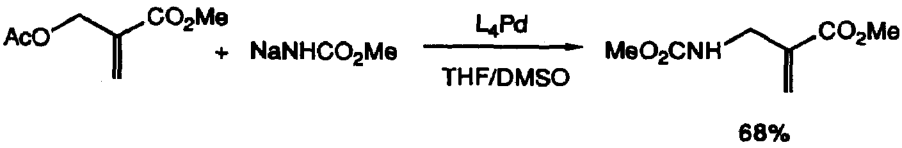
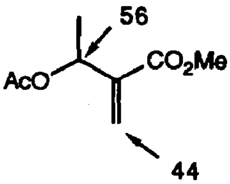


B. Amides, Nitriles

Palladium(0) complexes catalyzed the amidation of allyl acetates (equation 528) [695]. Lithium amides attacked chromium-complexed arenes (equation 529) [696]. Copper chloride catalyzed the alcoholysis of isocyanates to carbamates (equation 530) [697]. 4-Vinyl azetidinones were ring opened to amides by palladium(II) catalyzed alkylation (equation 531) [698]. Copper(I) chlorides catalyzed the amidation of aryl halides by aryl amides (equation 532) [699].

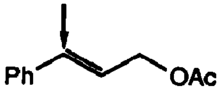
(528)

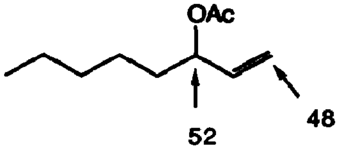




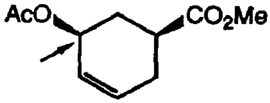
45%

72%

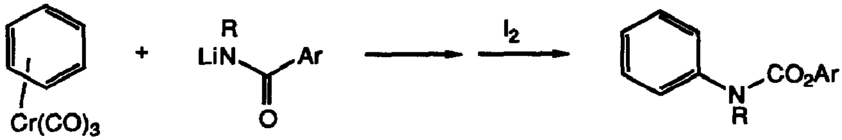




63%



(529)

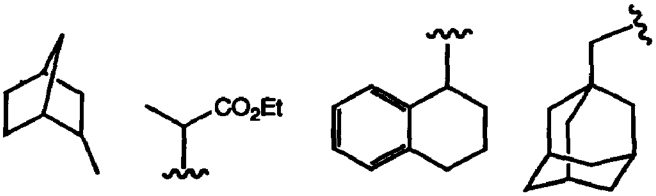


60-90%

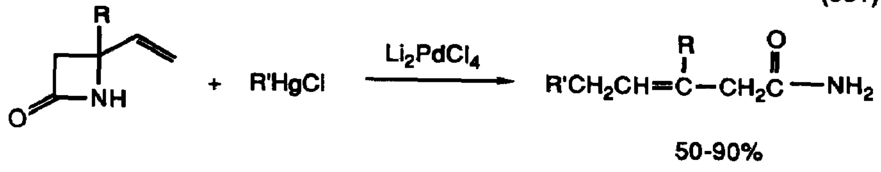
Ar = Ph, pMePh, pMeOPh, mNO$_2$Ph, β-naphth

(530)

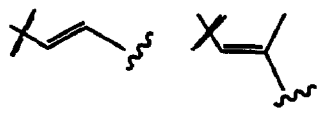
R = PhCH$_2$, tBu,



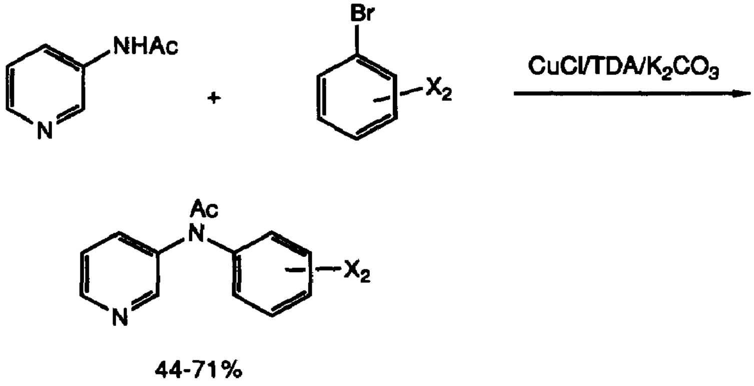
(531)



R = H, Me
R' = Ph, pMeOPh, mNO$_2$Ph,

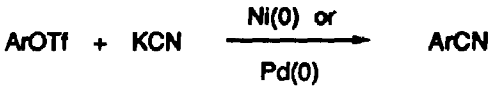


(532)



X_2 = H; 4F; 4CF$_3$O; 2CF$_3$; 3CF$_3$; 4CF$_3$; 4Cl; 2F; 3F; 2Cl; 3Cl; 3,4Cl$_2$; 2,3,4,5,6F$_5$

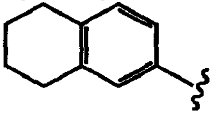
Aryl triflates were converted to aryl cyanides using nickel(0)/palladium(0) catlaysts (equation 533) [700][701]. Acetals were converted to cyanohydrin methyl ethers by trimethylsilyl cyanide and nickel, cobalt, palladium [702] and rhodium [703] catalysts (equation 534). Optically active cyanohydrin esters were made by addition of cyanide to chiral rhenium aldehyde complexes (equation 535) [704]. Cobalt complexed propargyl alcohols were converted to nitriles (equation 536) [705]. Benzyl amines were oxidized to nitriles over copper(I) chloride catalysts (equation 537) [706]. Nickel(0) catalysts promoted the hydrocyanation of eneynes (equation 538) [707]. The effect of R_3SnZ on the nickel(0) catalyzed addition of HCN to 1-cyano-2-butene were studied [708].

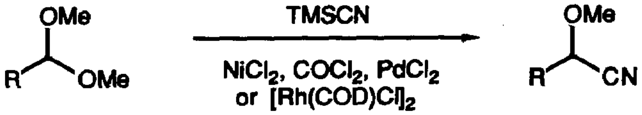


$$ArOTf + KCN \xrightarrow[Pd(0)]{Ni(0)\ or} ArCN \qquad (533)$$

60-90%

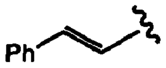
Ar = Ph, pMeOPh, pClPh, pMePh, oMePh, $pEtO_2CPh$, pBrPh, pAcOPh, pAcNHPh

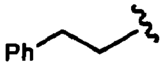




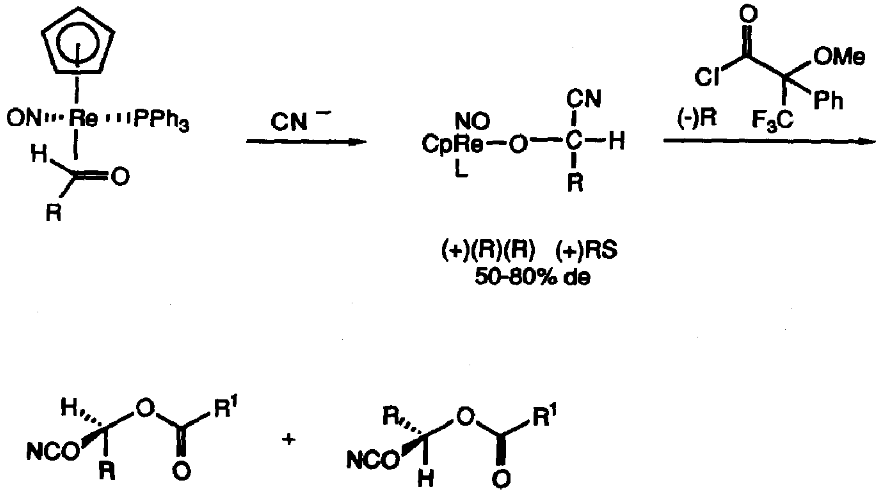
(534)

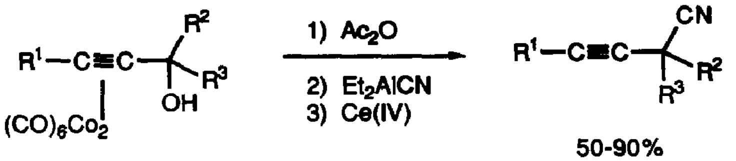
R = Ph, pMeOPh, C_8,





(535)





50-90%

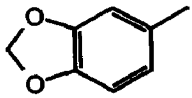
(536)

$R^1 = H, Me, C_5, Ph$
$R^2 = H, Me, Et, OEt$
$R^3 = H, Me, OEt$

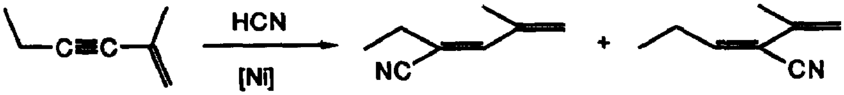
$$ArCH_2NH_2 \xrightarrow[\text{Mol. Sieve}]{CuCl/O_2/Py} ArCN \qquad (537)$$

96-99%

Ar = Ph; pMeOPh; 3,4MeO$_2$Ph; 1-Naphth; nPr; nC$_9$; nC$_4$;



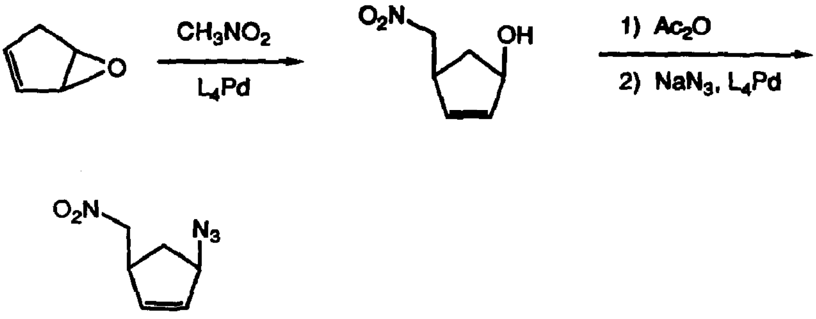
(538)



90 : 10 50%

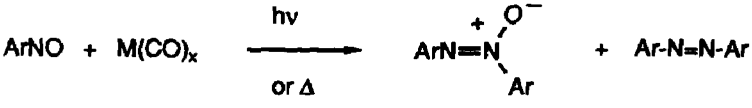
Palladium(0) catalyzed the replacement of allylacetate with azide (equation 539) [709]. Metal carbonyls oxidized nitroso compounds to azoxy compounds and azo compounds (equation 540) [710].

(539)



61%

(540)



$$\mathrm{ArNO} + \mathrm{M(CO)_x} \xrightarrow[\text{or } \Delta]{h\nu} \mathrm{ArN{=}\overset{+}{N}(O^-)Ar} + \mathrm{Ar{-}N{=}N{-}Ar}$$

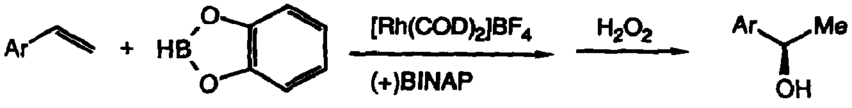
up to 90%

M = $Fe(CO)_5$, $Cr(CO)_6$

C Amines Alcohols

Chiral rhodium complexes catalyzed the asymmetric hydroboration of olefins (equation 541) [711], (equation 542) [712], (equation 543) [713]. Palladium(0) complexes catalyzed the hydroboration of dienes (equation 544) [714].

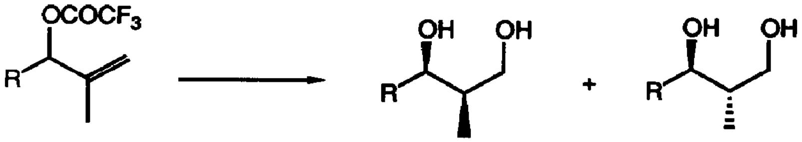
(541)



70-90%
~90% ee

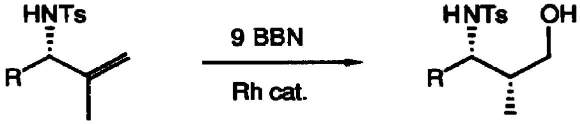
Ar = Ph, pMePh, pClPh, mClPh, pMeOPh, oMeOPh

(542)



high yields

	syn	anti
R = Ph	1	1.5
iPr	3.6	1
Bn	14.2	1
iPrCH$_2$	9.5	1
	6.9	1
	5.8	1

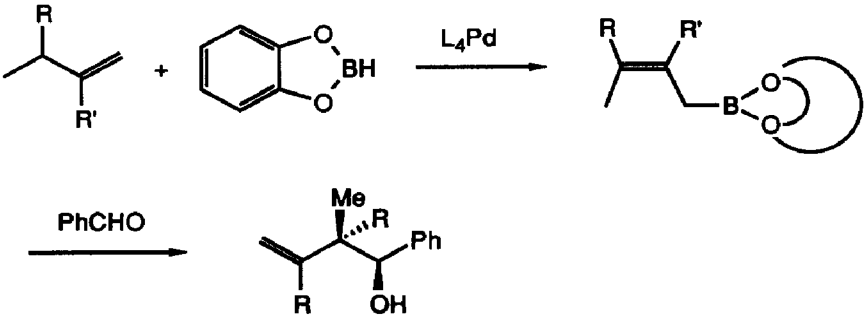


(543)

major

R = Bn, iPrCH$_2$, iPr, Ph, BnOCH$_2$-

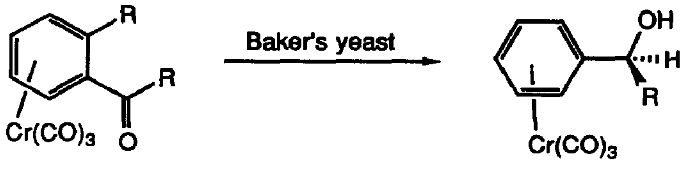
(544)



77-89%

Chromium complexed aryl ketones were stereospecifically reduced with Bakers yeast (equation 545) [715]. Asymmetric hydrogenation of β-keto carboxylic esters - a practical purely chemical access to β-hydroxy esters in high enantiomeric purity was the topic of a dissertation [716]. Epoxides were selectively reduced by hydrozirconation [717]. Zirconocene dihydride reduced α,β-unsaturated carbonyls to allylic alcohols [718].

(545)

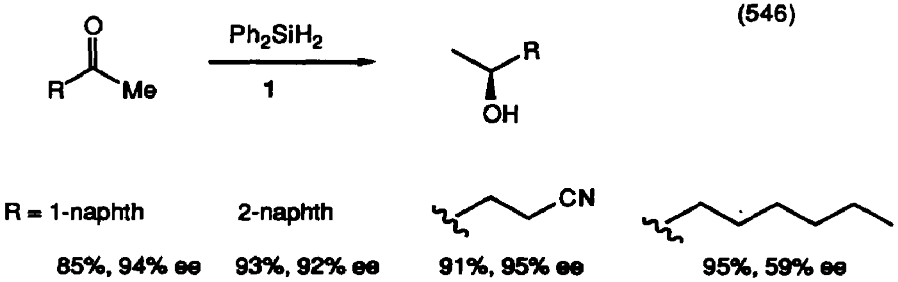


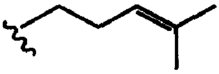
100% yield
≥99% ee

R = H, OMe; R' = Me

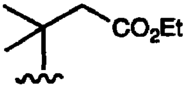
Chiral rhodium catalysts reduced ketones with silyl hydrides (equation 546) [719]. Olefins were oxidized to alcohols by oxygen in the presence of cobalt(I) salts (equation 547) [720][721]. The same complex catalyzed the addition of aldehydes to activated olefins (equation 548) [722]. Titanium isoproxides/tartrates were used to catalyze the kinetic resolution of thienyl and furyl benzylic alcohols (equation 549) [723][724].

(546)

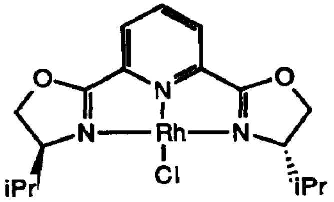




94%, 70% ee

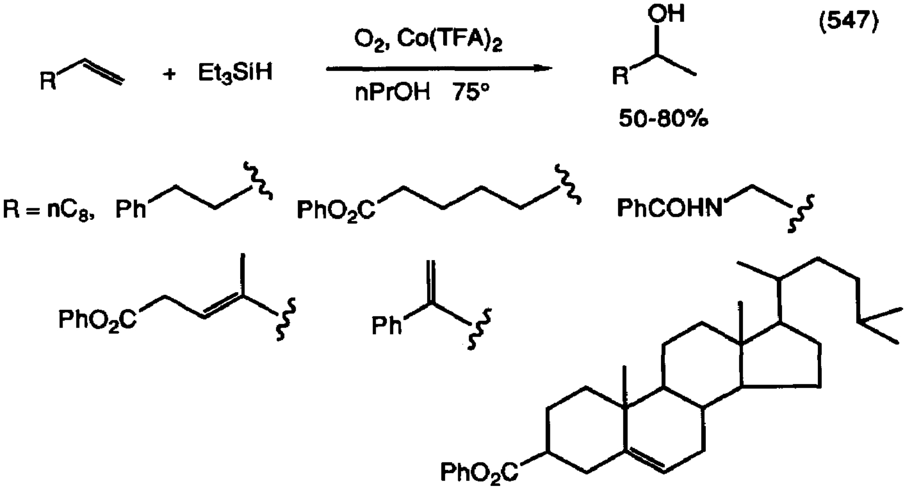


60%, 20% ee

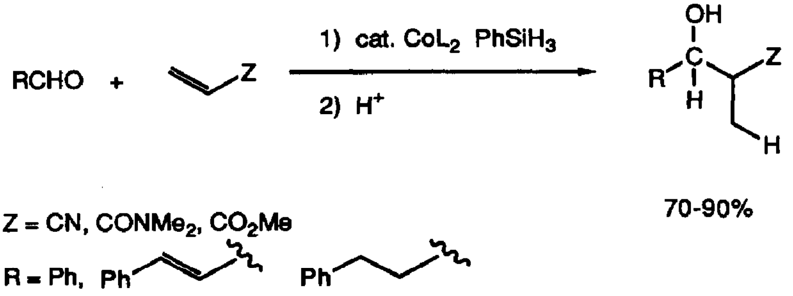


1

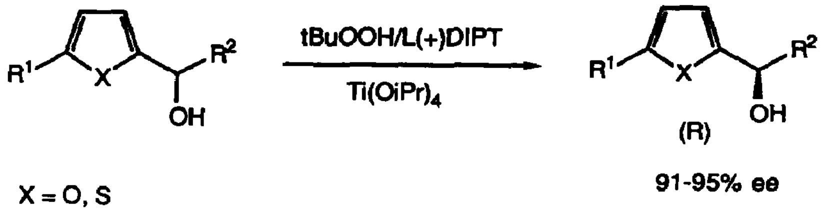
(547)



(548)

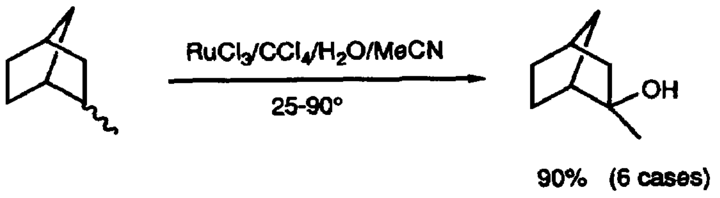


(549)



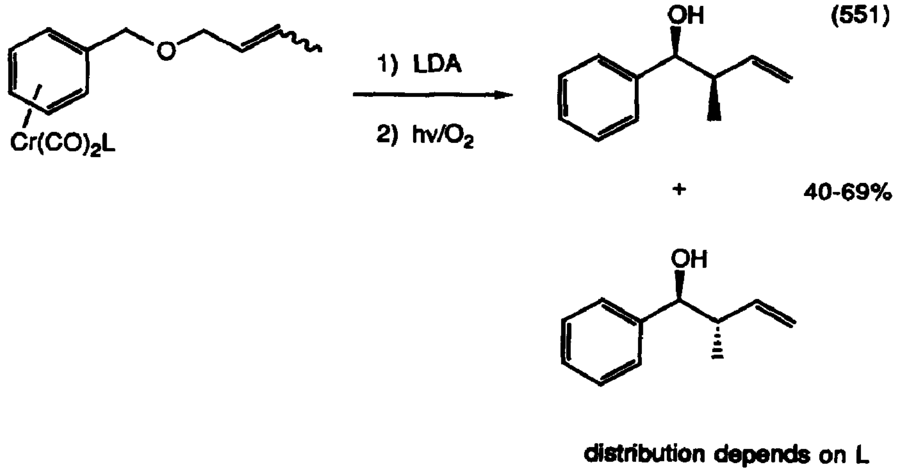
Polymer-immobilized Mn porphyrins catalyzed the oxidation of cholesterol and anthracene [725]. Ruthenium chloride oxidized 3°-CH bonds (equation 550) [726].

(550)



LDA isomerized chromium complexed allyl benzyl ethers to benzyl alcohols (equation 551) [727]. Wilkinson's catalyst decarbonylated aldoses by one carbon (equation 5522) [728].

(551)



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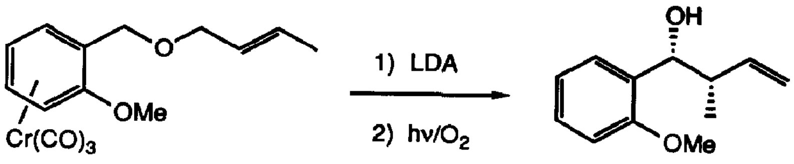
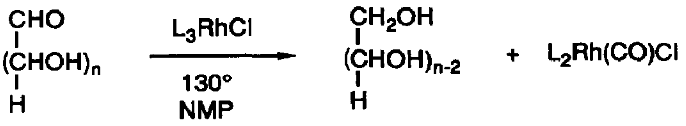


(+)R

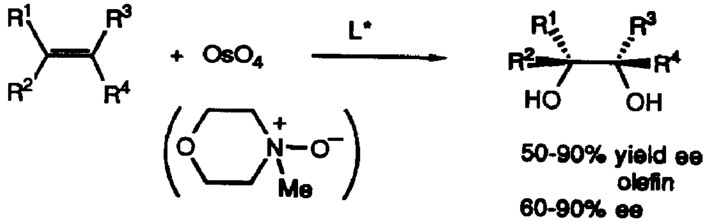
1R 2S 99% ee



$$\mathrm{CHO{-}(CHOH)_n{-}H} \xrightarrow[\text{130° NMP}]{\mathrm{L_3RhCl}} \mathrm{CH_2OH{-}(CHOH)_{n-2}{-}H} + \mathrm{L_2Rh(CO)Cl}$$

(552)

Asymmetric osmium tetroxide hydroxylation of olefins to vicinal diols was the subject of a dissertation [729]. This asymmetric *cis* hydroxylation has become quite efficient, and a number of chiral ligands, including (bis)-pyrrolidines [730][731], bis(dibenzyl)amines [732] and cinchona alkaloids [733][734] have been used (equation 553). Olefins were also *cis* hydroxylated by polymer-bound osmium tetroxide [735], and by hydrogen peroxide in the presence of tungstate catalysts (equation 554) [736].

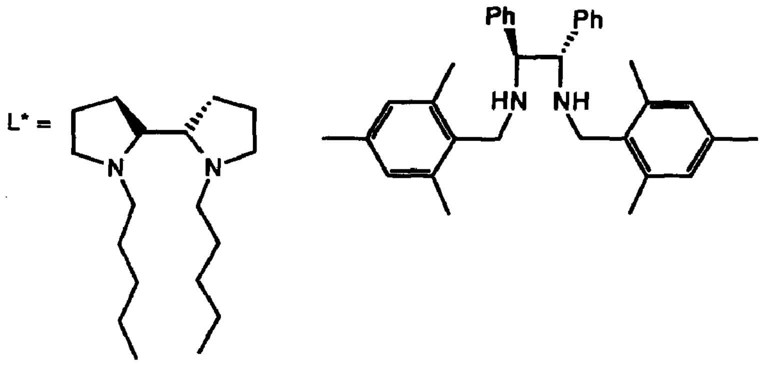


(553)

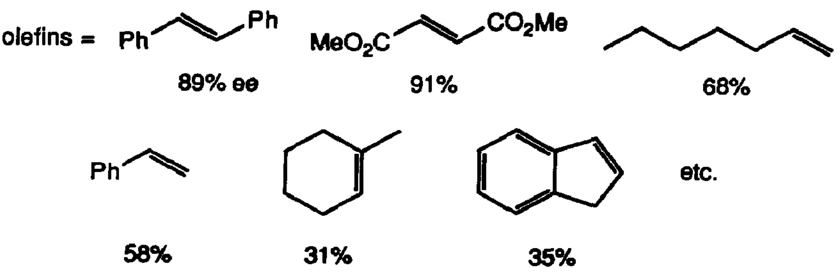
50-90% yield ee depends on olefin

60-90% ee

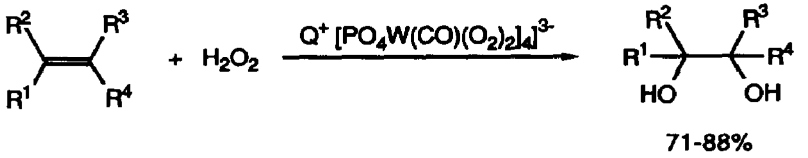
L* =



olefins =



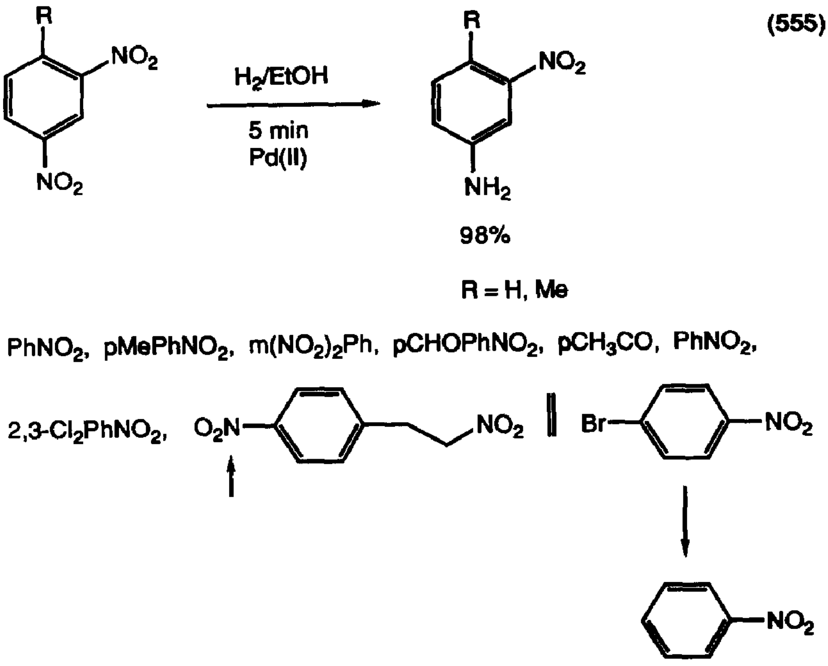
(554)



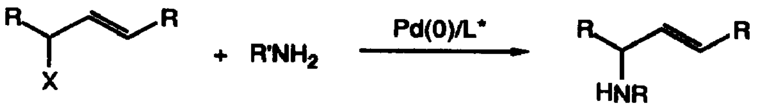
R^1 = Ph, H, nBu, Met, Et
R^2 = Me, H, $(CH_2)_3$, $(CH_2)_4$, $(CH_2)_5$
R^3 = Me, Et, H
R^4 = H, CH_2Cl_2, CH_3

2,4-Dinitroarenes were reduced to monoamino nitroarenes by palladium(II) catalysts on interlammellar montmorillonite (equation 555) [737]. Orthometallated palladium(II) complexes catalyzed the reduction of nitro alkanes and nitriles [738]. Ruthenium carbonyl catalyzed the reduction of o-nitrophenyl-allyl ether to o-aminophenol [739]. Nickel boride reduced aromatic nitro groups in the presence of olefin, aldehyde, ketone, nitrile, amide, carboxylic and acid carboxylic ester formation [740].

(555)



Soluble polyethylene-bound palladium(0) complexes catalyzed the allylic amination of allyl acetates [741]. Palladium(0) complexes with chiral ferrocenyl phosphine ligands catalyzed the allylic amination of allyl acetates with a high degree of asymmetric inductions (equation 556) [742]. Palladium(0) complexes also catalyzed the replacement of allylic acetates (equation 557) [743] by azide, and the amination of allyl epxodies (equation 558) [744].

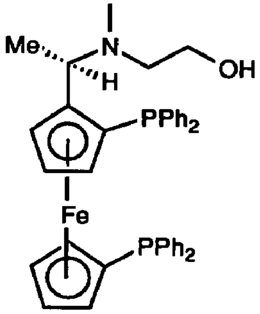


(556)

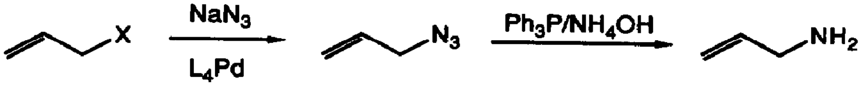
70-90% yield
up to 97% ee

R = Ph, Me, nPr, iPr
X = OCO_2Me, OCOMe, $OP(O)(OPh)_2$
R' = Bn, pTs, 3,4-$(MeO)_2Ph$

L* =

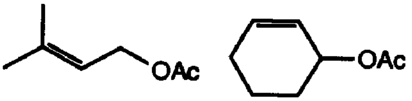


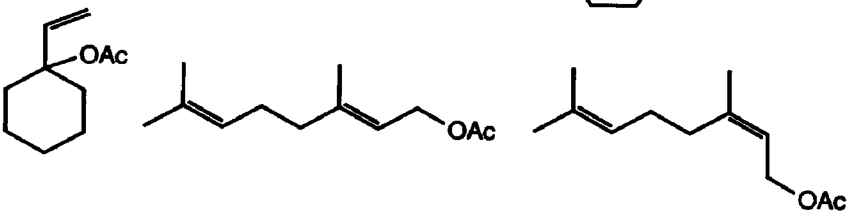
(557)

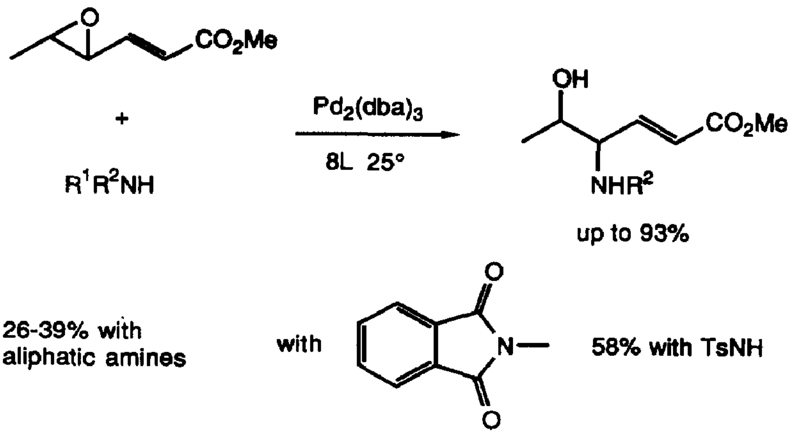


(full paper)

X = OAc, OCO_2Me, $OP(O)(OEt)_2$,

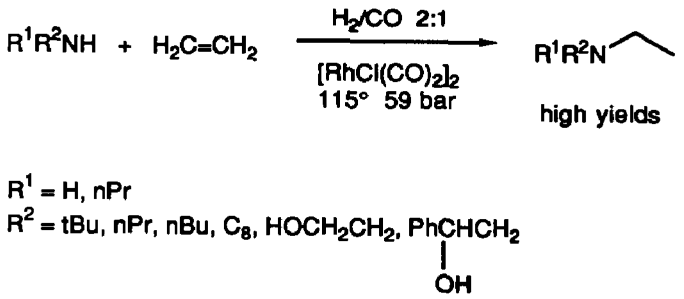




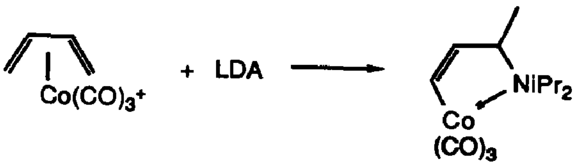


(558)

Functionalization of alkenes: "Catalytic amination of monoolefins" was the title of a review (117 references) [745]. Rhodium(I) complexes catalyzed the amination of ethylene (equation 559) [746]. Cationic diene cobalt complexes were aminated by LDA (equation 560) [747]. Zirconium imine complexes converted alkynes to allyl amines (equation 561) [748]. Cobalt carbonyl complexed propargyl amino were used as protecting groups in alkaloid synthesis (equation 562) [749].

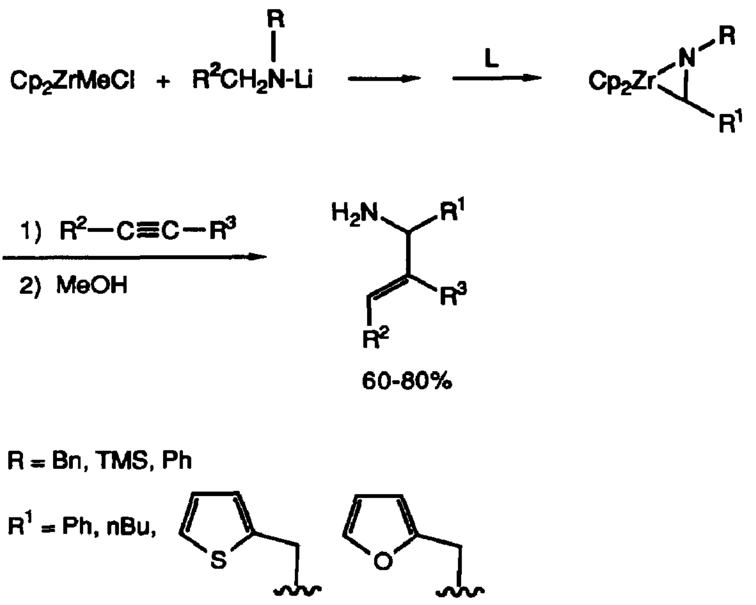


(559)

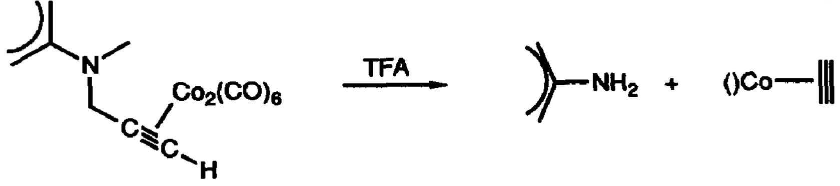


(560)

(561)



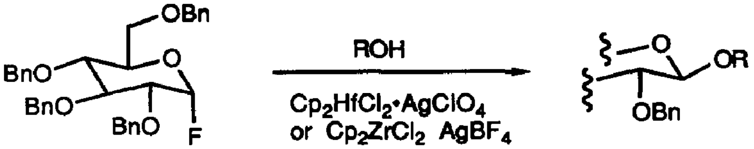
(562)



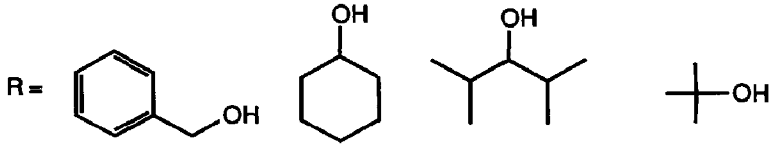
D. Ethers, Esters, Acids

Hafnium [750] and zirconium [751] complexes catalyzed the alkoxylation of fluorosugars (equation 563). Palladium(0) complexes catalyzed the allylation of sugar OH groups by allyl carbonates (equation 564) [752]. Palladium(II) complexes catalyzed the Michael addition of alcohols to conjugated enones (equation 565) [753]. Cobalt(II) chloride catalyzed the conversion of enol ethers to acetals (equation 566) [754]. Diarylethers were prepared by copper (equation 567) [755] and iron (equation 568) [756] assisted alkoxylation reactions. Rhodium(III) phosphine complexes catalyzed the conversion of ketones to ketals (equation 569) [757].

(563)

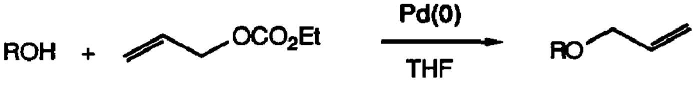


60-90%
α:β 85:15 to 100:6



sugars, 3β-cholestanol

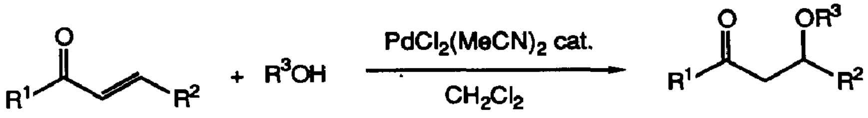
(564)



50-90%

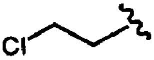
ROH = sugars, tartrates

(565)

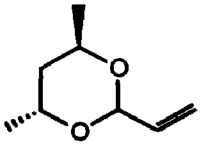


50-90%

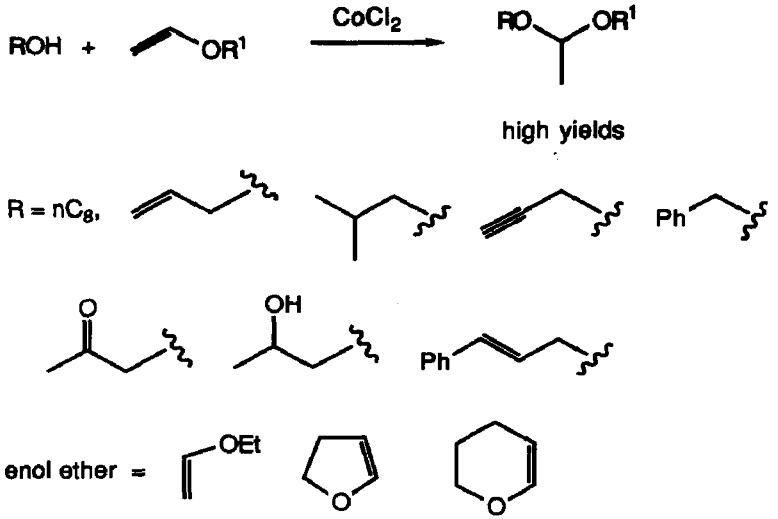
R^1 = Ph, Met, Et
R^2 = H, Me
R^3 = Me, Et, iPr, tBu, Bn, AcO$^-$,



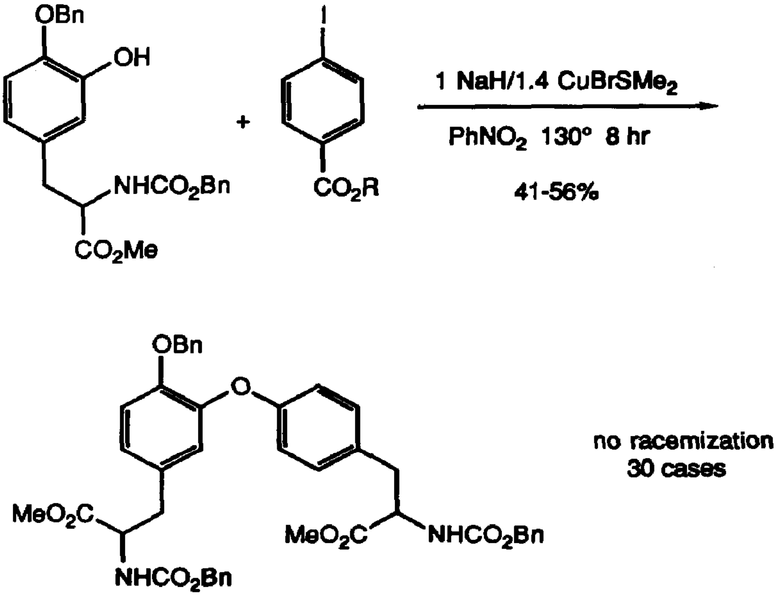
also



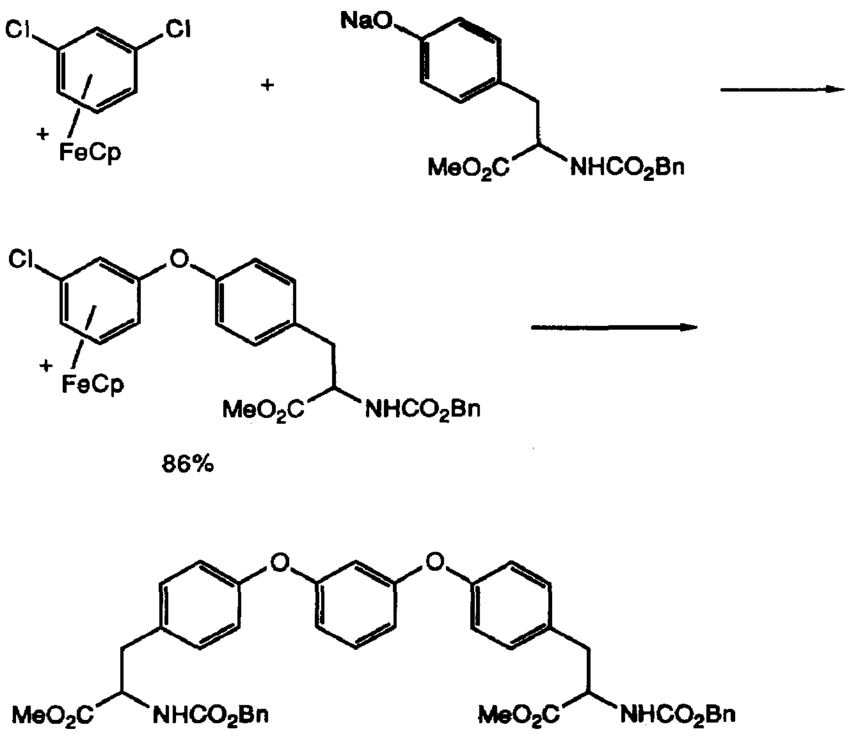
(566)


ROH
OR1
CoCl2
RO
OR1
high yields
R = nC8,
Ph
O
OH
Ph
enol ether =
OEt
O
O

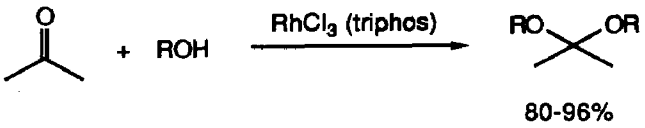
(567)

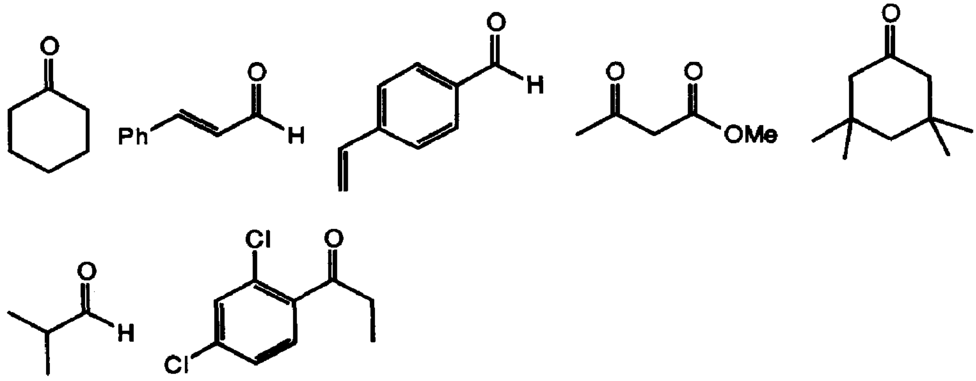

OBn
OH
NHCO2Bn
CO2Me
I
CO2R
1 NaH/1.4 CuBrSMe2
PhNO2 130° 8 hr
41-56%
OBn
O
MeO2C
NHCO2Bn
MeO2C
NHCO2Bn
no racemization
30 cases

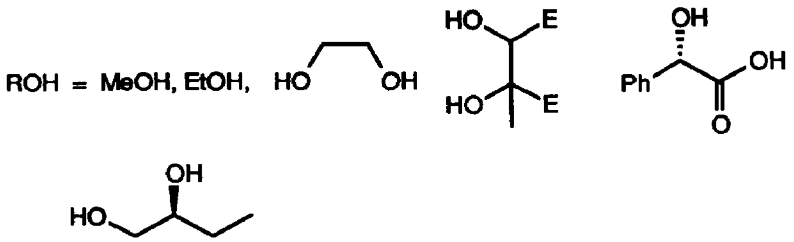
(568)


Cl
Cl
+
FeCp
+
NaO
MeO$_2$C
NHCO$_2$Bn
Cl
O
+
FeCp
MeO$_2$C
NHCO$_2$Bn
86%
O
O
MeO$_2$C
NHCO$_2$Bn
MeO$_2$C
NHCO$_2$Bn

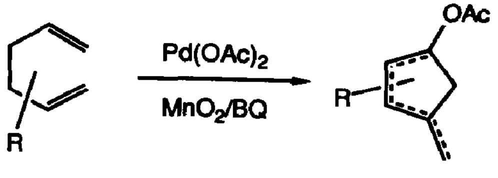
(569)


O
+
ROH
RhCl$_3$ (triphos)
RO
OR
80-96%


O
O
Ph
H
O
H
O
O
OMe
O
O
H
Cl
O
Cl

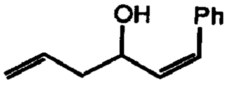


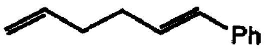
Palladium(II) acetate catalyzed the acetoxy cyclization of dienes (equation 570) [758] and the allylic acetoxylation of olefins [759] Cyclic ketones were oxidized to keto acids by molybdenum/vanadium catalysts (equation 571) [760]. Alkenes were oxidized to keto esters by ruthenium tetroxide (equation 572) [761], manganese(III) acetate α-acetoxylated enones (equation 573) [762] and ruthenium(II) complexes catalyzed the conversion of nitriles to nitrile hydroperoxides (equation 574) [763]. Exploratory studies of transition-metal catalyzed intramolecular radical cyclizations of unsaturated α,α-dichloroesters and acids was the topic of a dissertation [764].

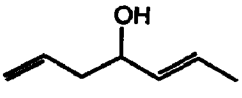


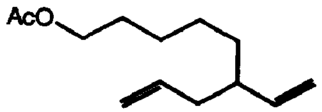
(570)

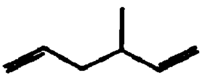
good yields

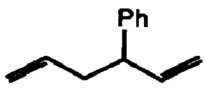


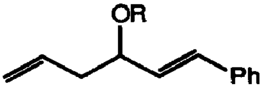


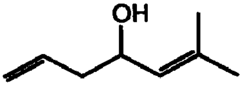


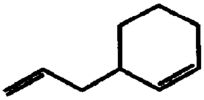


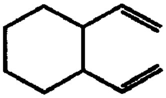




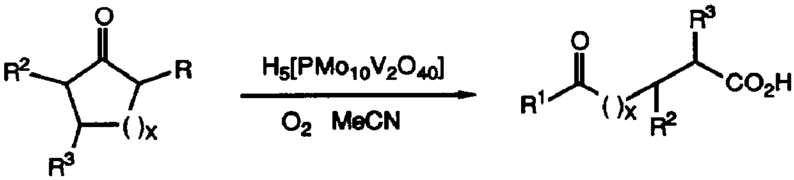






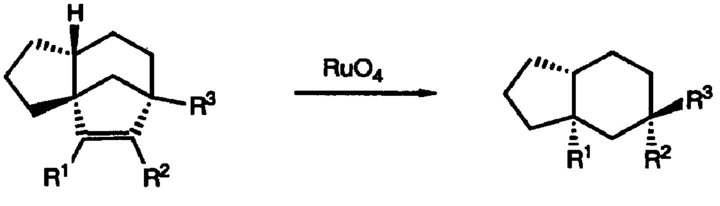


(571)



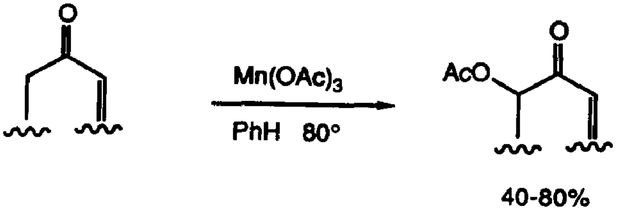
X = 1, 2
R^1 = Me, Ph
R^2 = Me, H
R^3 = H, Me

(572)

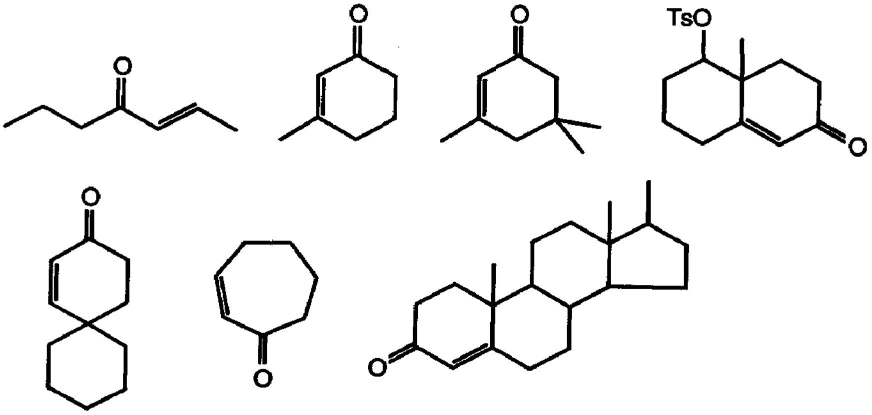


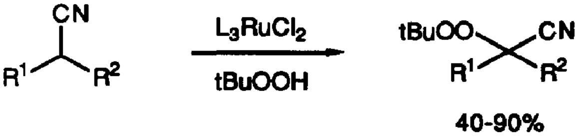
50-80%

R^1 = $COCO_2Me$, CO_2H, CO_2Me
R^2 = $COCO_2Me$, O
R^3 = $CH(OMe)_2$

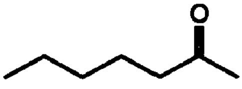


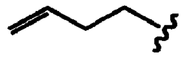
(573)





(574)

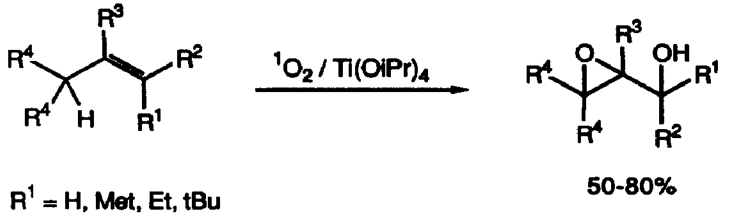
R^1 = Ph, pClPh, 

R^2 = Me, Ph, EtO, CHC_2, OTMS, 

E. Heterocycles

Transition metal catalyzed epoxidations have been reviewed (364 references) [765]. Titanium isopropoxide catalyzed the epoxidation of olefins by singlet oxygen (equation 575) [767]. Molybdenum complexes catalyzed the epoxidation of olefins by t-butylhydroperoxide (equation 576) [768]. Vinyl silanes were epoxidized by MCPBA, then ring opened (equation 577) [769].

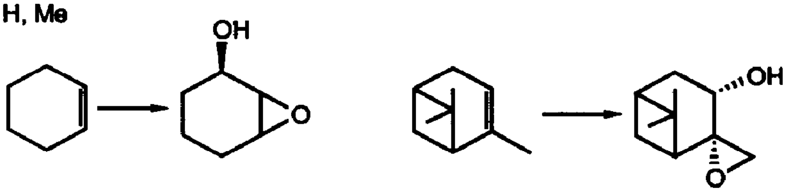
Ruthenium complexes catalyzed the epoxidation of steroids (equation 578) [770]. The mechanism of the Sharpless epoxidation was studied, and it was concluded that the sole intermediacy of monomeric titanium tartrate complexes was unlikely [771]. Allylic alcohols were epoxidized under Sharpless conditions then reduced using palladium(0) complexes (equation 579) [772]. A variety of tartrate ligands were prepared and studied for efficacy in the Sharpless epoxidation (equation 580) [773].



(575)

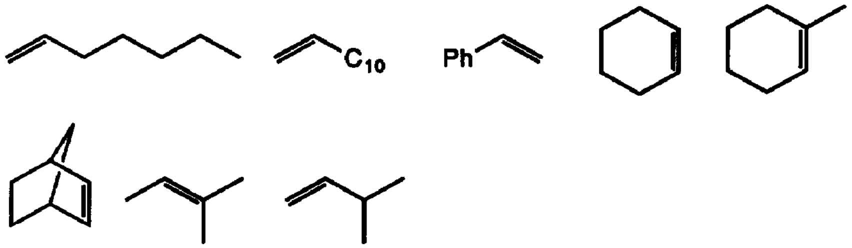
R^1 = H, Met, Et, tBu
R^2 = Me, iPr, H
R^3 = H, tBu, Me, Ph, CO_2Et
R^4 = H, Me

also



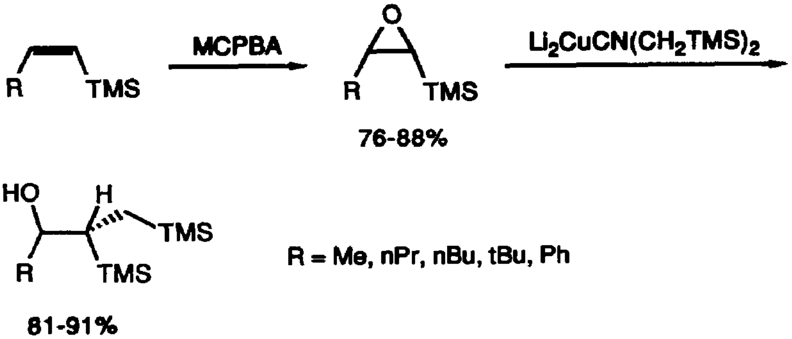
(576)

$$\text{tBuOOH} + \text{olefin} \xrightarrow{\text{MoO}_2\text{Cl}_2\text{[3-(diethoxyphosphinyl)camphor]}} \text{epoxide}$$

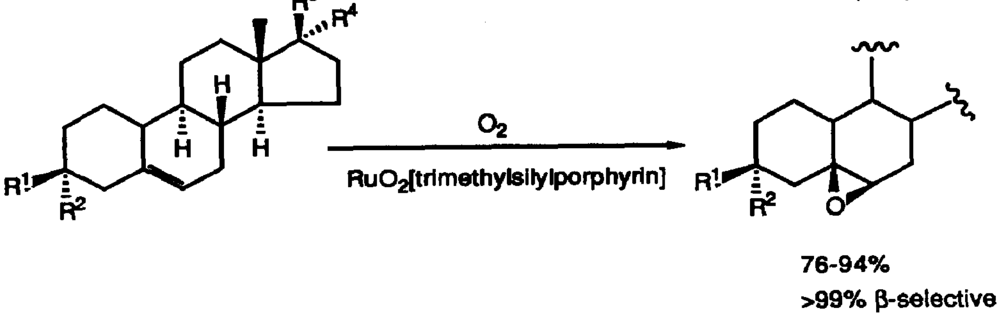


Polybutadiene ⟹ complete epoxidation 3h 25°

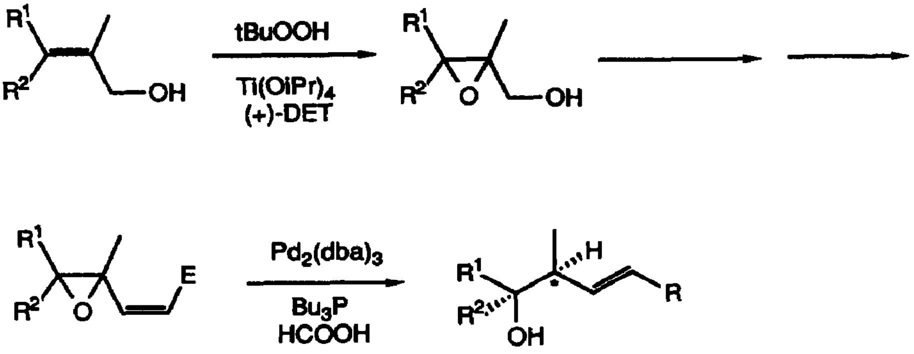
(577)



(578)



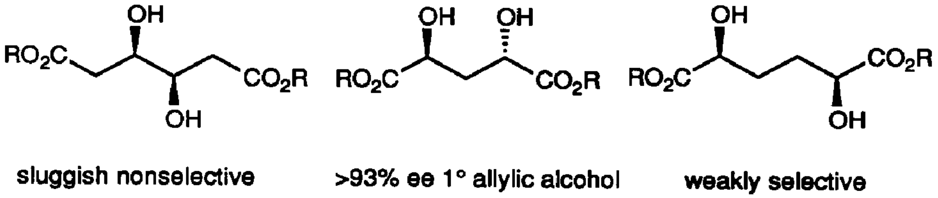
(579)



E olefin inverts, Z retains

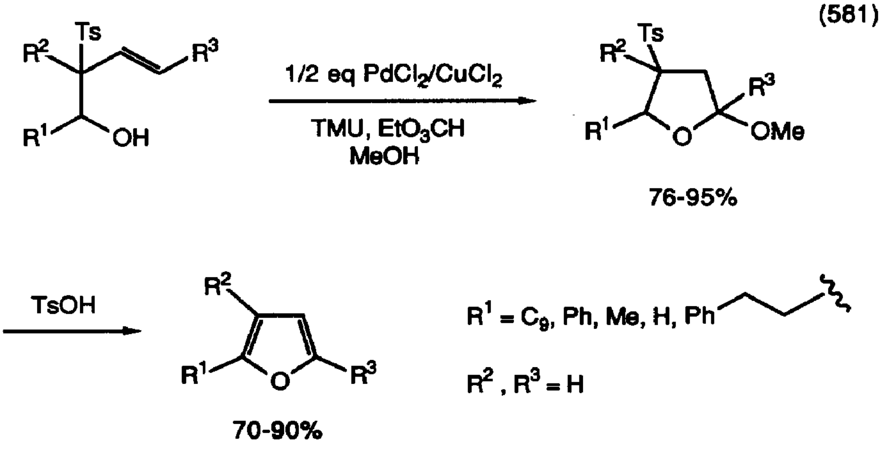
For Sharpless epoxidation

(580)

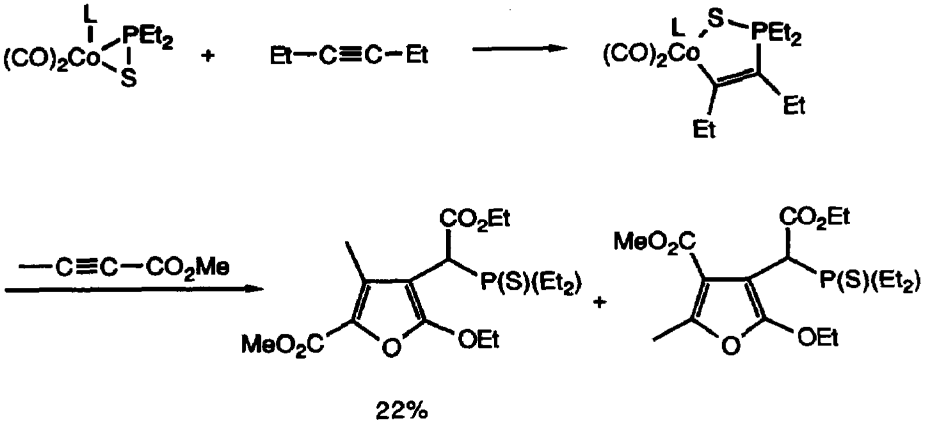


Stereoselective syntheses of functionalized tetrahydrofurans via intramolecular oxymetallations of allenes and syntheses of methyl nonactate homologs were the topics of a dissertation [774]. Furans were prepared by palladium-catalyzed intramolecular hydroxylation of olefins (equation 581) [775], by cobalt-catalyzed alkyne insertion (equation 582) [776], cobalt catalyzed endoperoxide decomposition (equation 583) [777] and by rhodium(I) assisted decarbonylations of keto sugars (equation 584) [778]. Tetrahydrofurans were prepared by palladium-catalyzed reactions of enol ethers with allyl alcohols (equation 585) [779], palladium catalyzed alkoxycarbonylation of olefins (equation 586) [780][781] and cobalt(II) catalyzed reaction of olefin with β-dicarbonyl compounds (equation 587) [782].

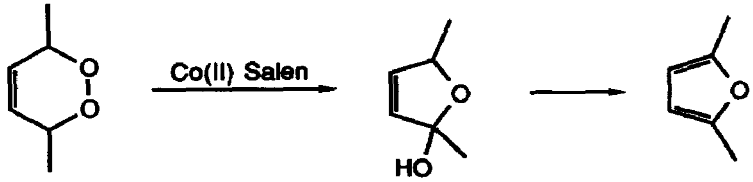
(581)



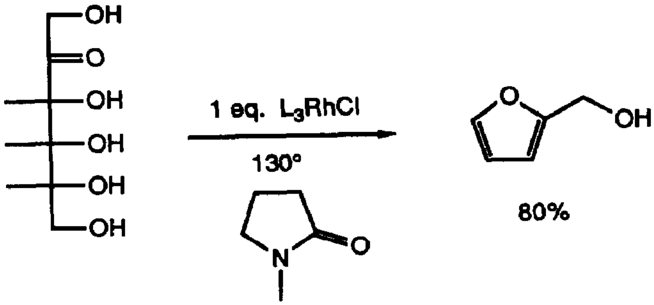
(582)



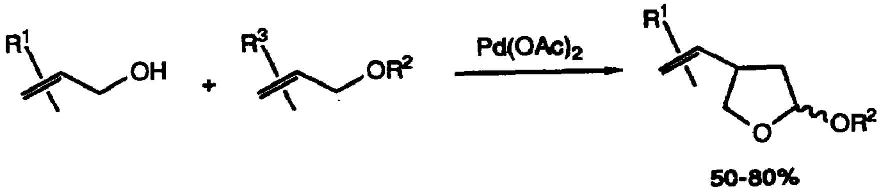
(583)



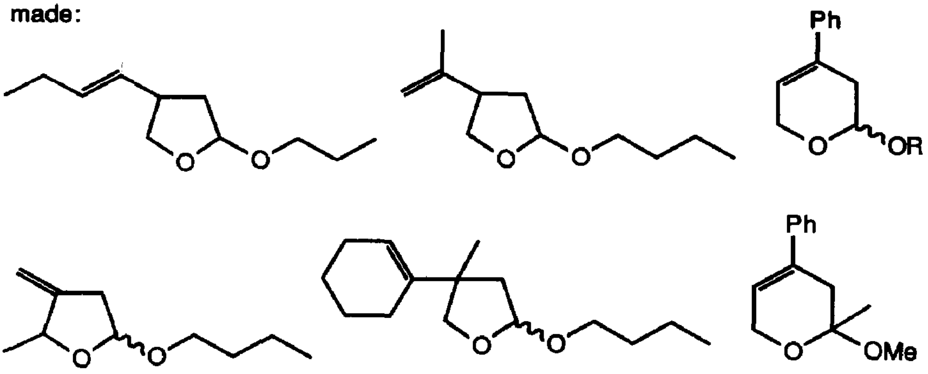
(584)



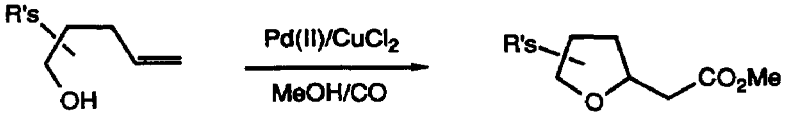
(585)



made:

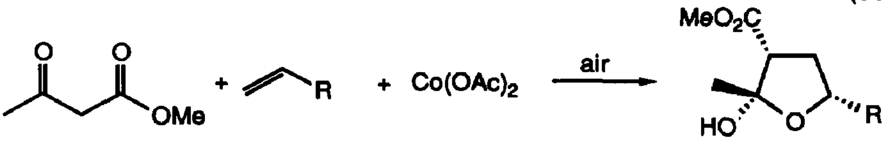


(586)



high yields

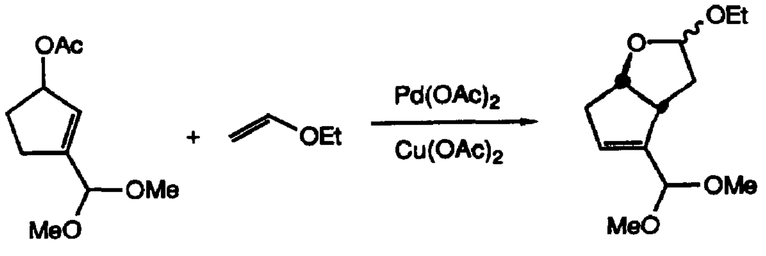
(587)



68-71%

R = Bu, C_8, C_9, C_{10}, C_{11}

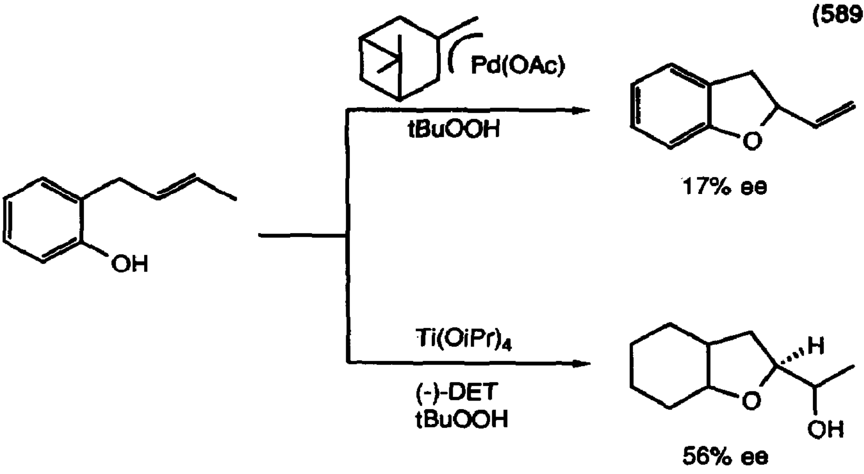
(588)[783]



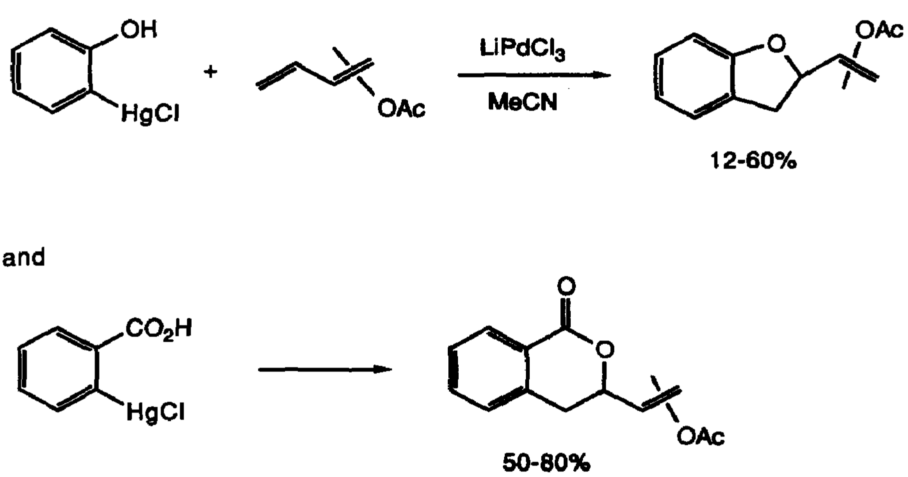
71%

Benzofurans were prepared by palladium(II) (equation 589) [784], (equation 590) [785] and copper catalyzed processes (equation 591) [786].

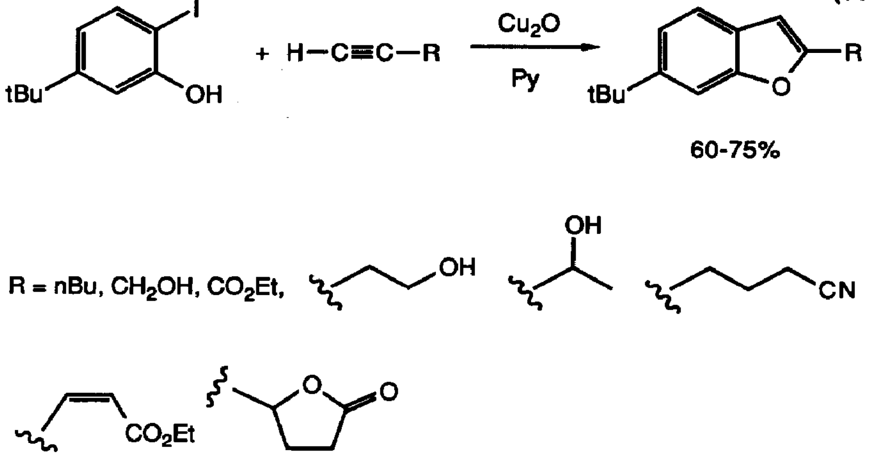
(589)



(590)

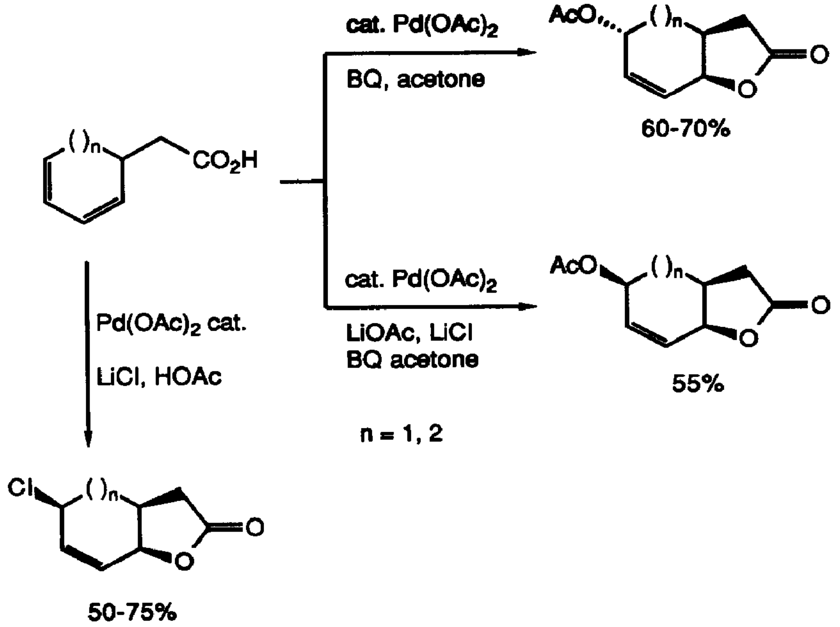


(591)

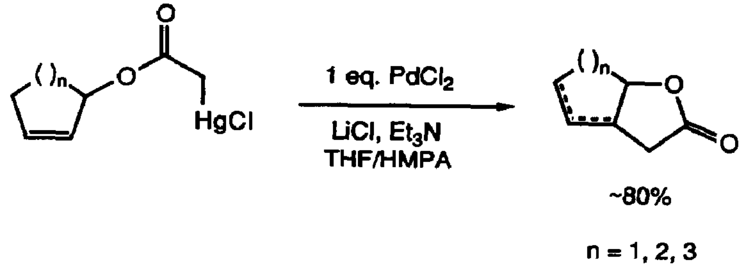


Lactones were prepared by palladium-catalyzed acetoxy-carboxylation of dienes (equation 592) [787], by intramolecular alkylation of cyclic olefins (equation 593) [788], by ruthenium-catalyzed oxidation of α,ω-diols (equation 594) [789], by palladium catalyzed reaction o-iodoesters with alkynes (equation 595) [790] and from bridging lactone complexes of cobalt (equation 596) [791].

(592)

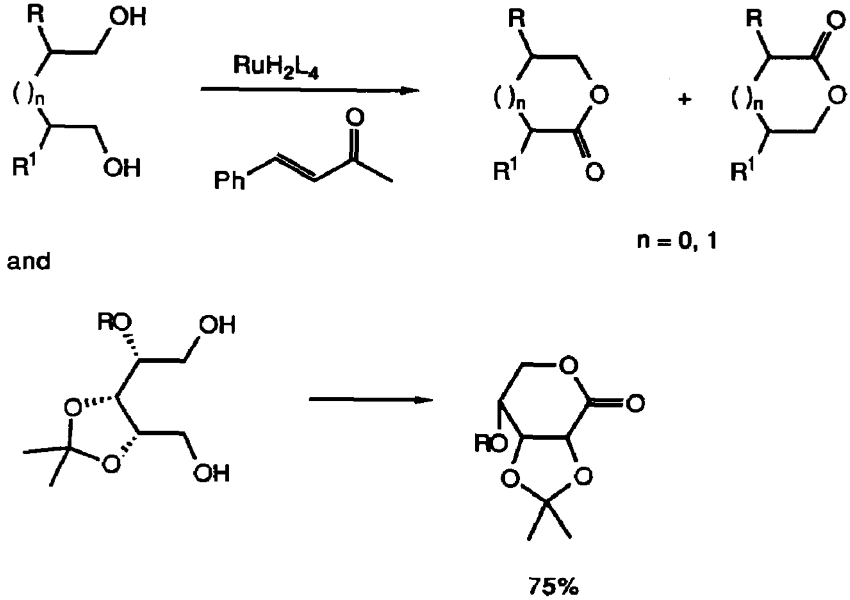


(593)



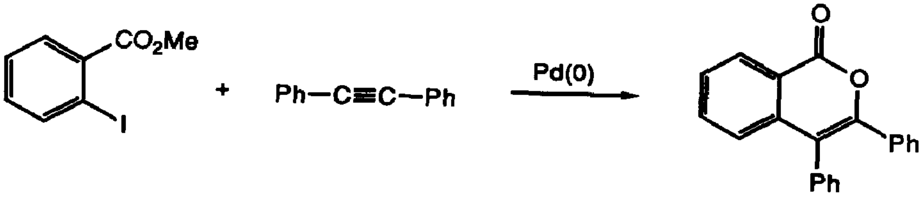
O
( )n
O
HgCl
1 eq. PdCl$_2$
LiCl, Et$_3$N
THF/HMPA
( )n
O
O
~80%
n = 1, 2, 3

(594)

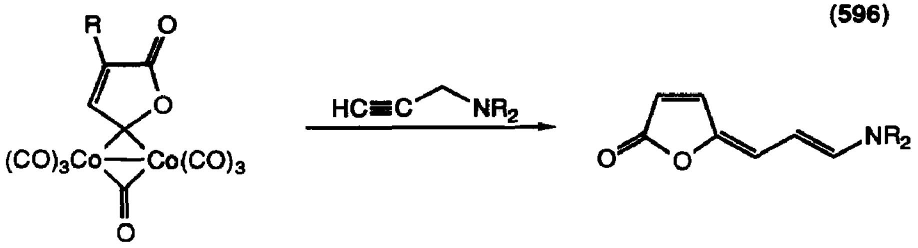


R
OH
( )n
R^1
OH
RuH$_2$L$_4$
O
Ph
R
( )n
O
R^1
O
+
R
O
( )n
O
R^1
n = 0, 1
and
RO
OH
O
O
OH
O
O
RO
O
O
75%

(595)

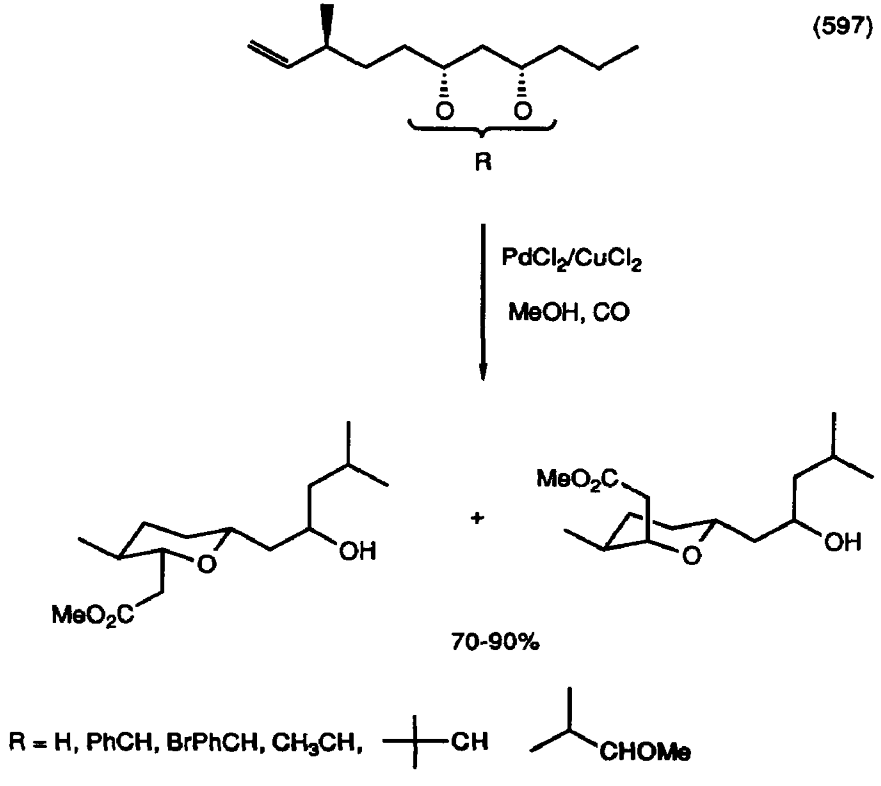


CO$_2$Me
I
+
Ph—C≡C—Ph
Pd(0)
O
O
Ph
Ph

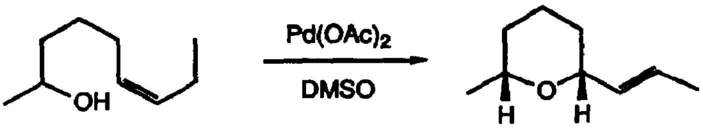


(596)

Pyrans were made by palladium(II) catalyzed alkoxylation of olefins (equation 597) [792], (equation 598) [793], by palladium(0) catalyzed reaction of magnesium alkoxide with allyl phenyl ethers (equation 599) [794][795] and by ruthenium(I) catalyzed hetero Diels-Alder reactions (equation 600) [796].

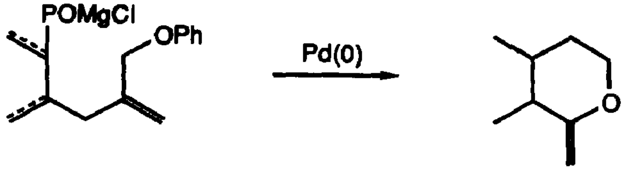


(597)



(598)

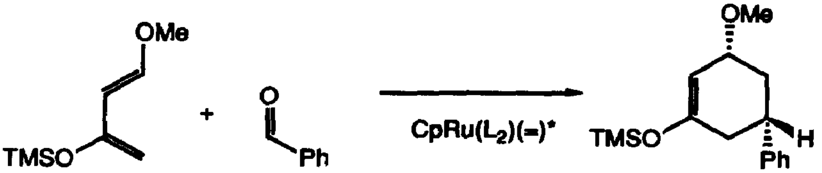
90%
no olefin isomerization



(599)

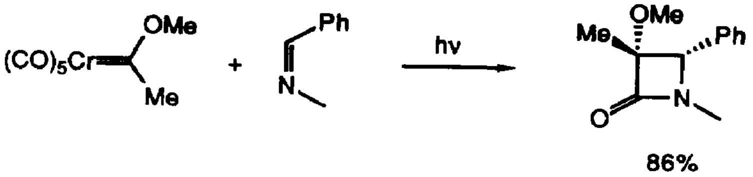
23-85%

(600)

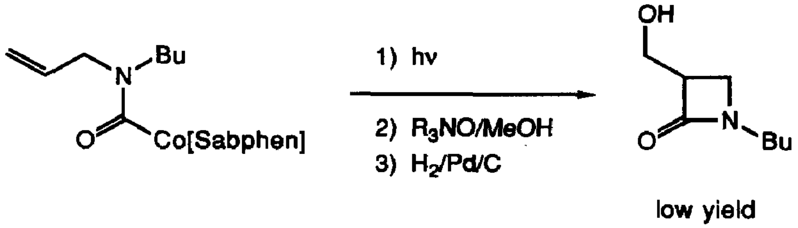


60-65%
30% ee

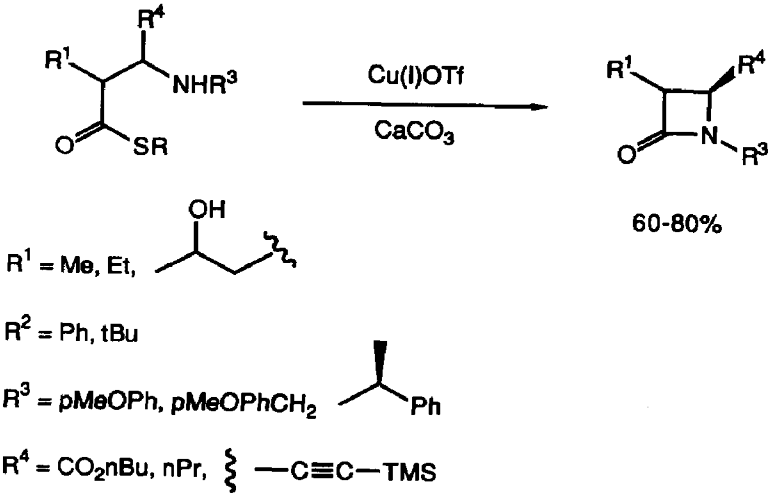
Dissertations entitled "Metal vinylidene complexes in β-lactam synthesis" [797], "The use of carbenoids in β-lactam synthesis" [798] and "Preparation and circular dichroism study of optically active iron-olefin complexes and approaches to the synthesis of penicillin using organoiron intermediates" [799] have appeared. β-Lactams were prepared by the photolytic reaction of imines with chromium carbene complexes (equation 601) [800], photolysis of amidocobalt complexes (equation 602) [801] and the copper triflate coupling of β-amino thioesters (equation 603) [802].



(601)



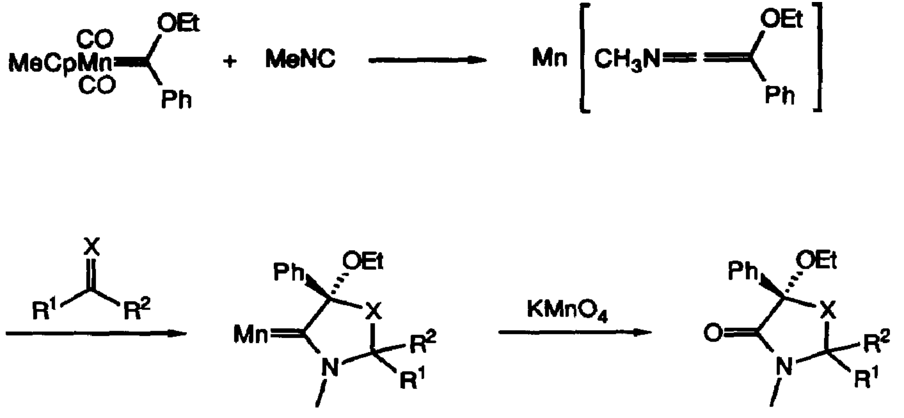
(602)



(603)

Pyrrolidinones were prepared by the reaction of ketenimine complexes of manganese with ketones (equation 604) [803], the ruthenium catalyzed chloroalkylation of N-allyltrichloroacetamides (equation 605) [804], the nickel(0) promoted reactions between isocyanates and olefins (equation 606) [805][806][807][808], the iron(III) chloride catalyzed amination of arenes (equation 607) [808] and by the palladium(II) catalyzed cyclization of alkynylbenzyl amides (equation 608) [809].

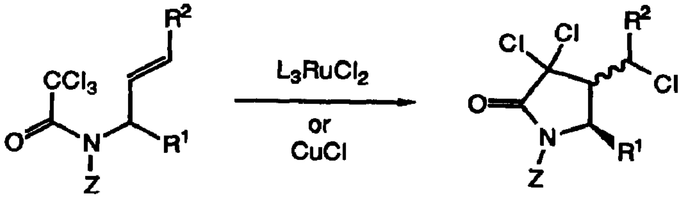
(604)



60-95%

X = O, S, NMe, NPh
R^1, R^2 = H, Me, =S, =O, =NPh, Ph

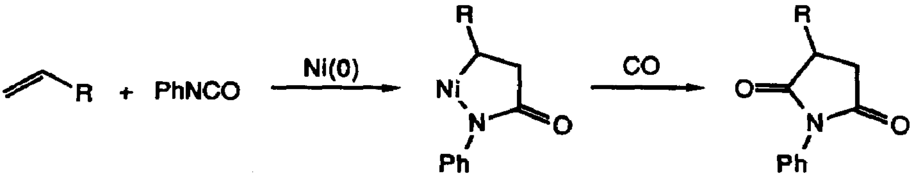
(605)

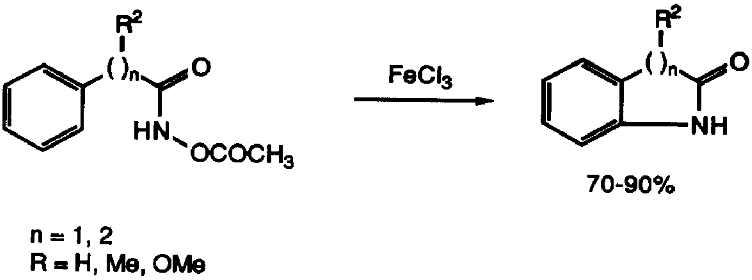


70-98%

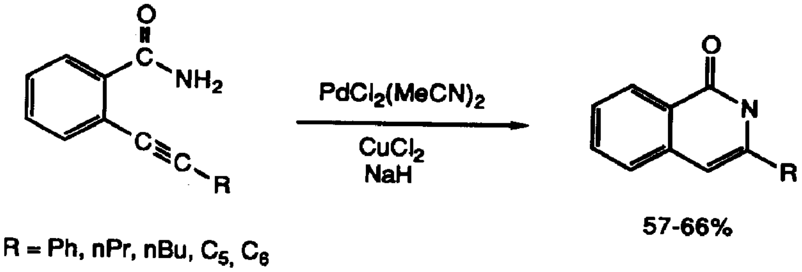
R^1 = Me
R^2 = H, nPr
Z = H, Bn, Ts, CBz, Ms, tBOC

(606)



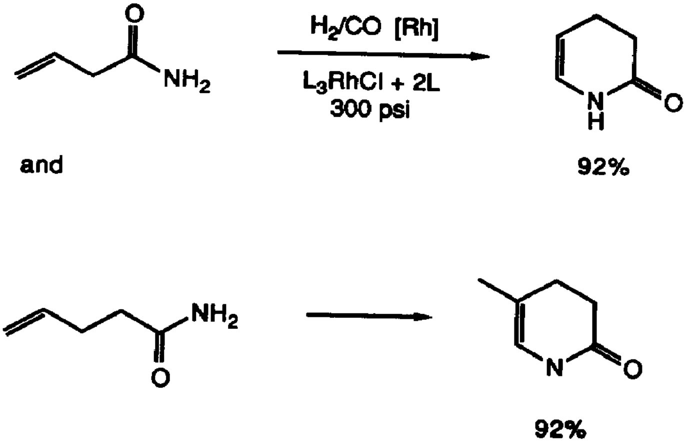


(607)



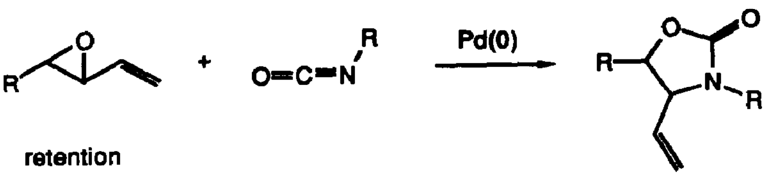
(608)

Pyridones were made by the rhodium catalyzed cyclization of unsaturated amides (equation 609) [810]. Oxazolidinones were prepared by the palladium(0) catalyzed reaction of epoxides with isocyanates (equation 610) [811][812].



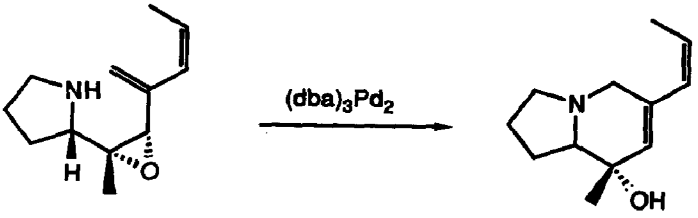
(609)

(610)

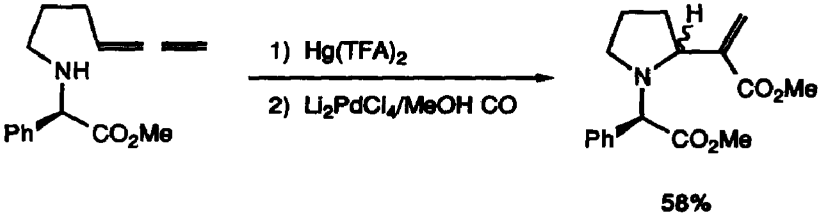


Indolizidines were prepared by palladium(0) catalyzed amination of alkyl epoxides (equation 611) [813]. Pyrrolidines were prepared by amination of allenes (equation 612) [814] while piperidines were formed from trimethylenemethane complexes and aziridines (equation 613) [815]. Ruthenium hydrides oxidatively cyclized ω-hydroxyamines to cyclic amines [816] Niobium trichloride converted azadienes and ketones to pyrroles (equation 614) [817]. $Cp_2Zr(CH_3)Cl$ also stitched together pyrroles from alkynes carbon monoxide and N-lithio-N-trimethylsilyl amine (equation 615) [818].

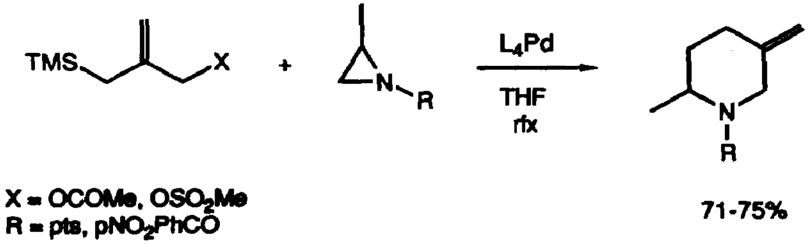
(611)



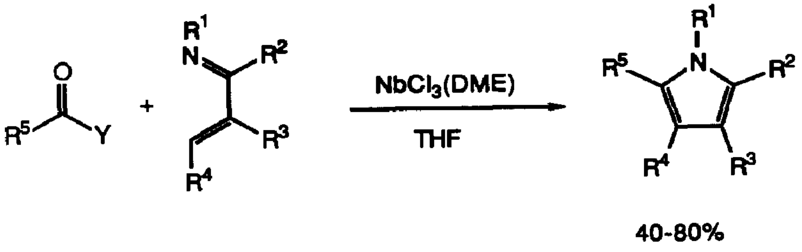
(612)

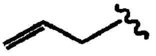
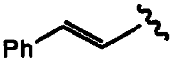
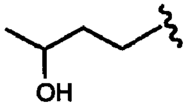


(613)

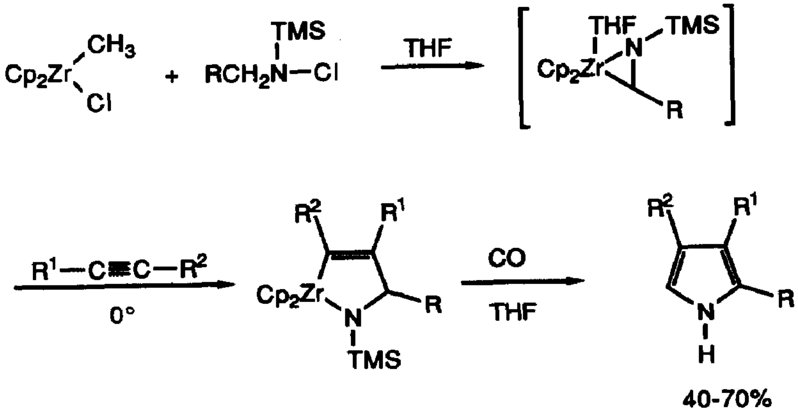


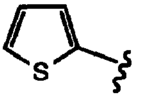
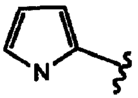
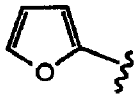
(614)

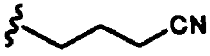


X = OEt, NMe$_2$, OMe
R^1 = Bn, nPr, 3,4-(MeO)$_2$Bu, 
R^2 = H, Me
R^3 = H, Me
R^4 = Ph, Me
R^5 = Me, H, CF$_3$, MeO$_2$CCH$_2$,  

(615)

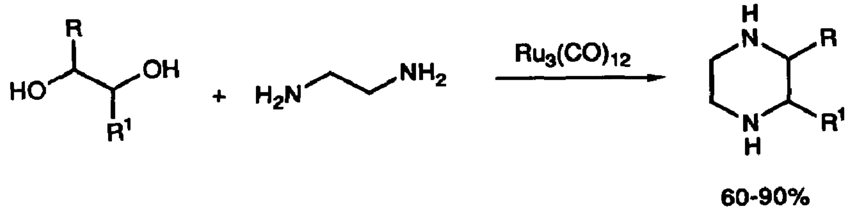


R = Ph, H, nC$_5$,   

R^1 = H, Ph, Me, nC$_8$
R^2 = Ph, H, Me, nPr, CH$_2$OTBS, 

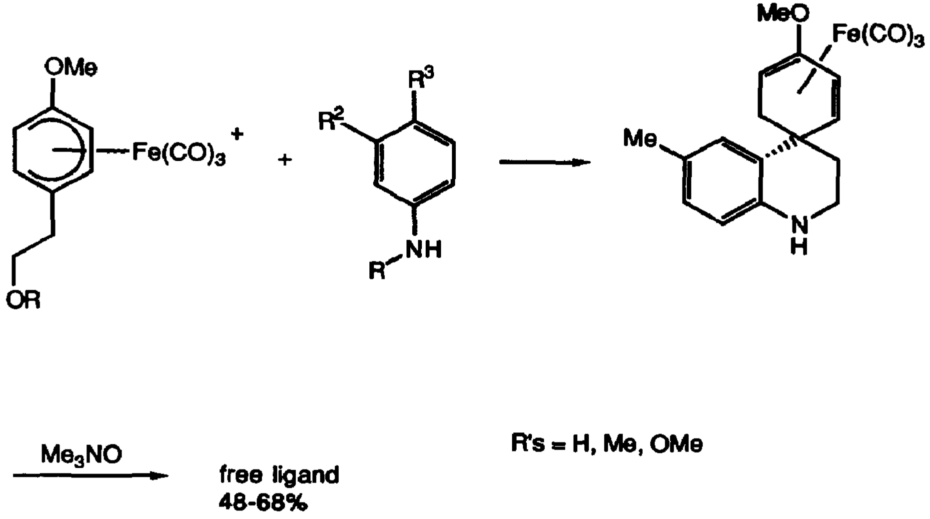
Piperzines were prepared by ruthenium catalyzed cyclocondensation of diols and diamines (equation 616) [819]. Spiro tetrahydroquinolines were prepared via cationic iron dienyl complex chemistry (equation 617) [820].

(616)



R's = Me, $(CH_2)_4$

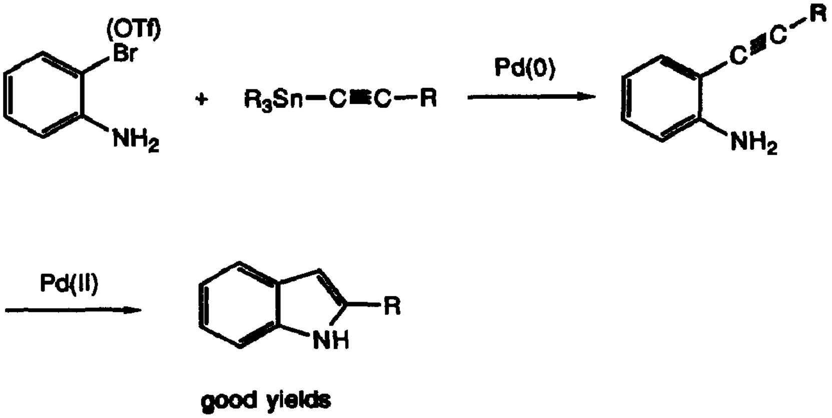
(617)



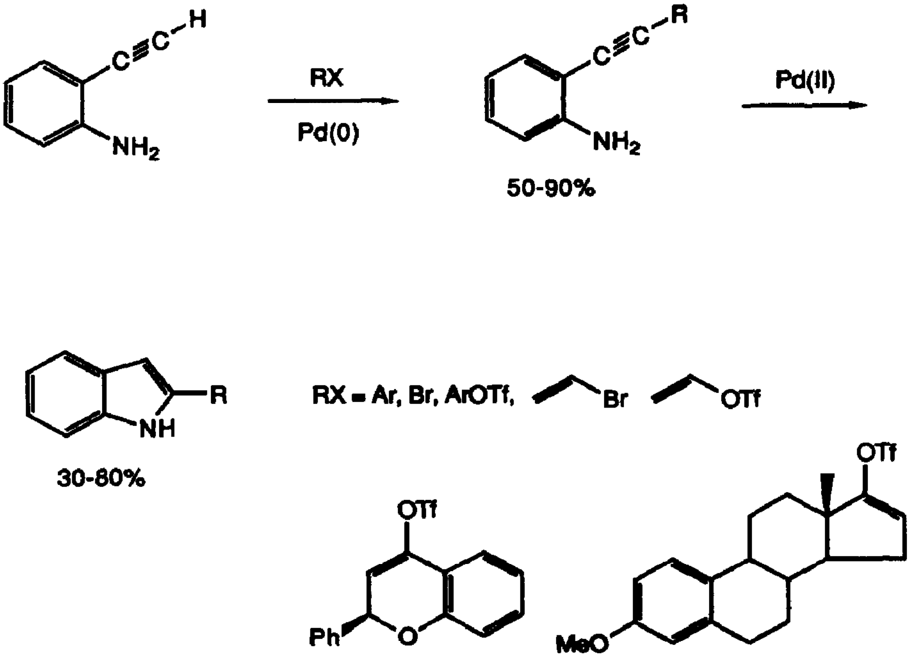
Reviews entitled "Synthesis and functionalization of indoles using transition metal reagents" (111 references) [812] and "Studies directed toward the ultimate synthesis for ergot alkaloids" (43 references) [822] have appeared. 2-Substituted indoles were make by the palladium(0) catalyzed coupling o-bromoanilines with alkynyl stannanes, followed by palladium(II) catalyzed cyclization (equation 618) [823] and closely related approaches (equation 619) [824], (equation 620) [825]. Tricyclic precursors to ergot alkaloids were synthesized using both palladium(0) and palladium(II) chemistry (equation 621)

[826]. N-Allyl-2-chloroanilines were cyclized to indoles using nickel(0) chemistry (equation 622) [827]. N-Acetyl-2-bromoanilines condensed with olefins in the presence of palladium(II) catalysts to give indoles (equation 623) [828]. Triphenyl amines were coupled to N-phenyl carbazoles by palladium(II) acetate (equation 624) [829]. Fused indoles were made by reaction of o-palladated quinolines with alkynes (equation 625) [830].

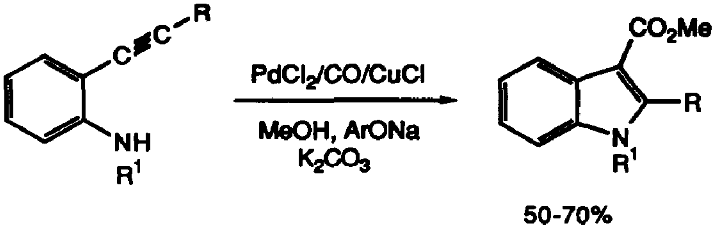
(618)



(619)

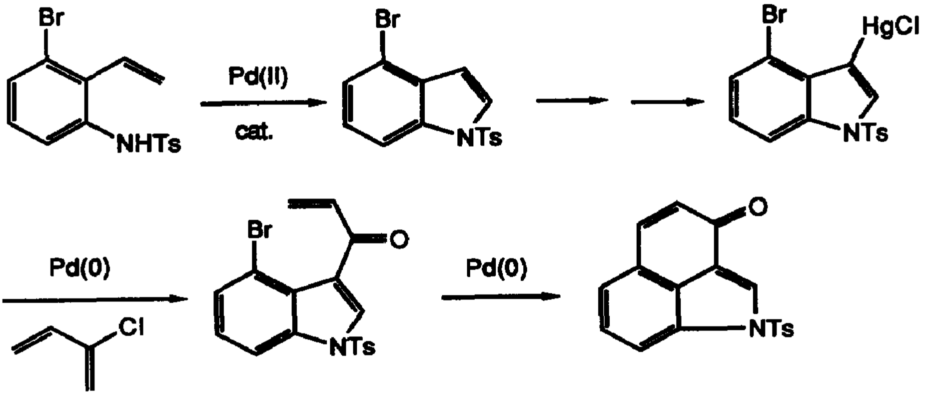


(620)

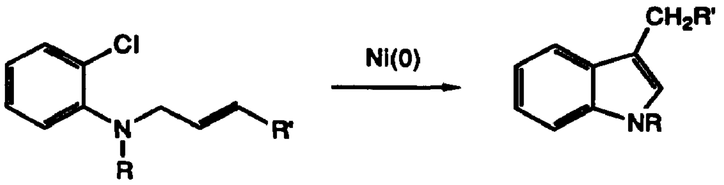


R = Bu, Ph; R^1 = H, SO_2Me

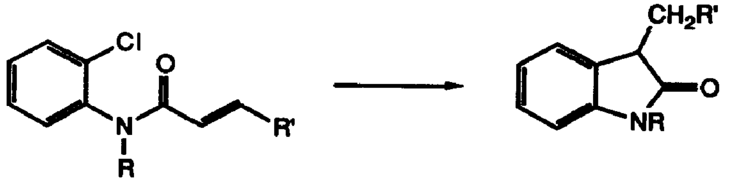
(621)



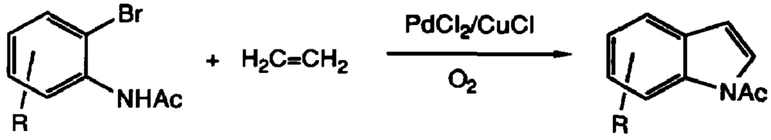
(622)



and

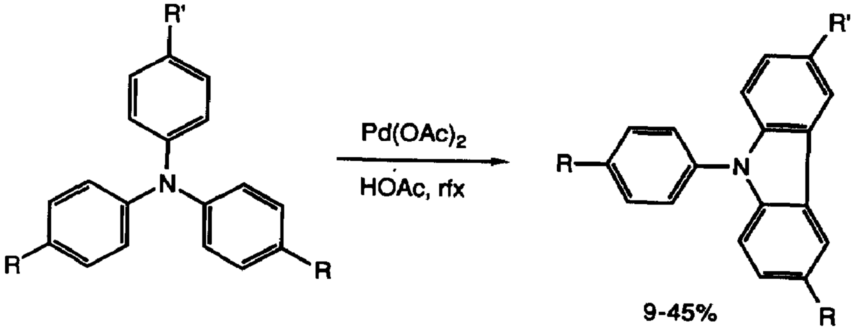


(623)



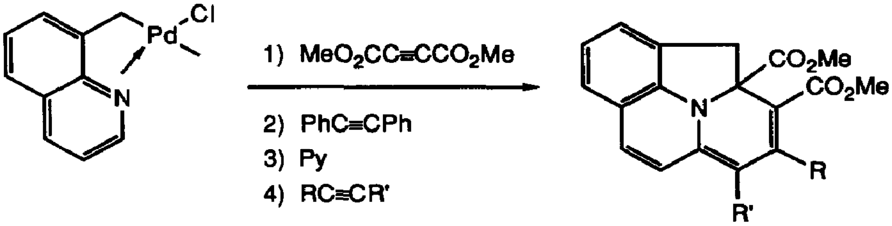
R = H, 4-Me, 4-CO_2Me, 5-OMe, 5-Me, 5-CO_2Me, 6-OMe, 6-Me, 6-Cl,
CO_2Me, 7-CO_2Me

(624)



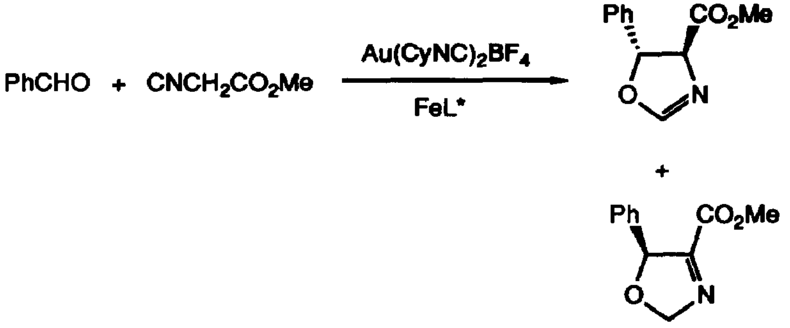
R = H, Me, CO_2Et, NO_2
R' = H, Me, CO_2Et, H

(625)



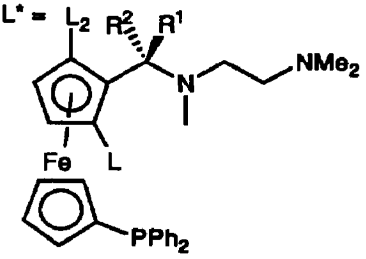
R = CO_2Me, CF_3
R' = Ph, CO_2Et, $pPhNO_2$, mCF_3Ph

The condensation of isocyanoesters (equation 626) [832], (equation 627) [833] or isocyano phosphonates (equation 628) [834][835] with carbonyl compounds using chiral ferrocenyl phosphin-amine ligands was useful for the synthesis of optically active oxazolines convertible to a number of important compounds. Tosylmethyl isonitrile condensed with chromium complexed benzaldehydes to give oxazolines (equation 629) [836].

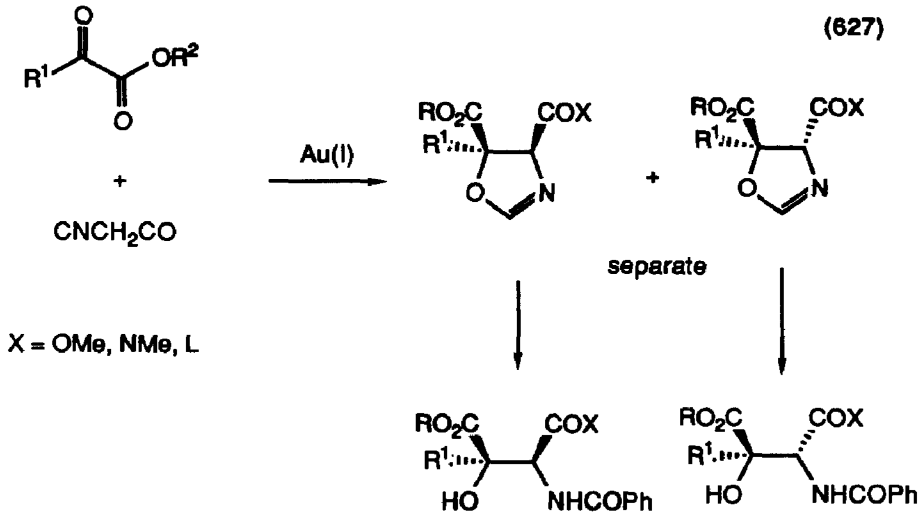


(626)

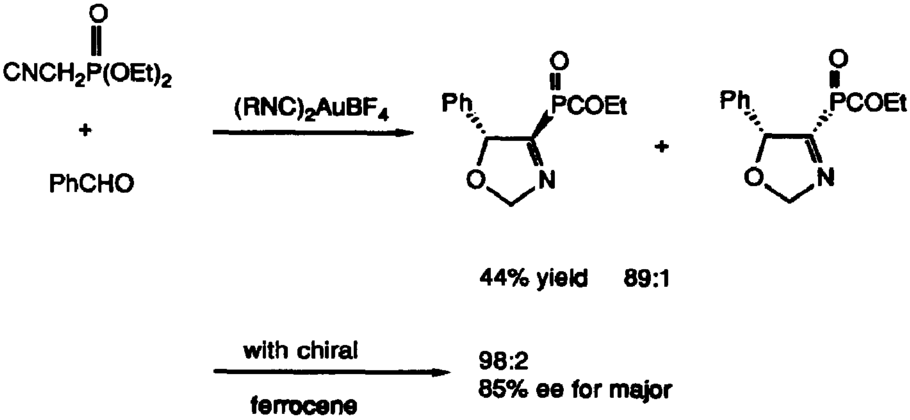
Optimum diastereoselectivity and enantioselectivity when L* has opposite planar and central chirality.



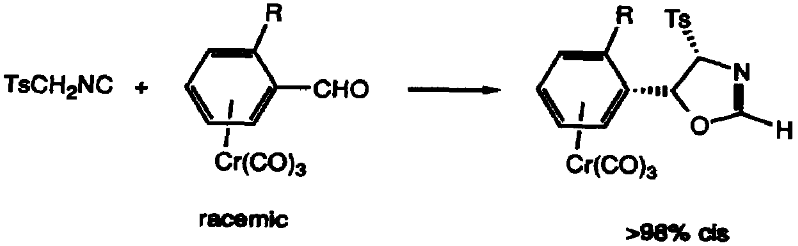
(627)


R^1
OR^2
+
CNCH$_2$CO
X = OMe, NMe, L
Au(I)
RO$_2$C
COX
R^1
O
N
+
separate
HO
NHCOPh

(628)

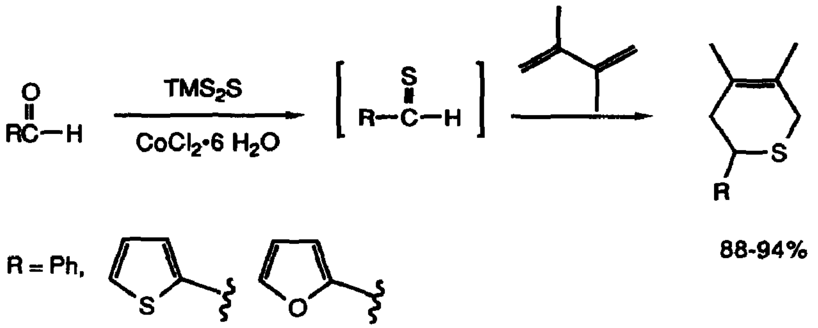

CNCH$_2$P(OEt)$_2$
+
PhCHO
(RNC)$_2$AuBF$_4$
Ph
PCOEt
O
N
+
44% yield 89:1
with chiral
ferrocene
98:2
85% ee for major

(629)

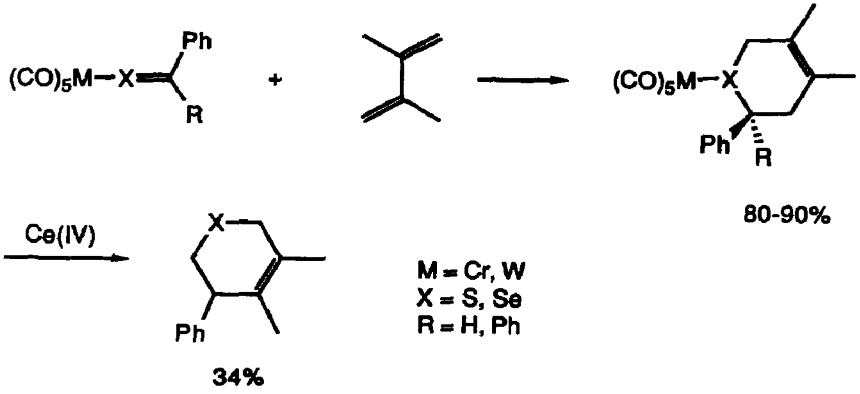

TsCH$_2$NC +
R
CHO
Cr(CO)$_3$
racemic
Ts
N
O
H
>98% cis

Sulfur heterocycles were prepared using cobalt catalysts (equation 630) [837] from metal carbonyl thioketone complexes (equation 631) [838], from SCl_2 cleavage of zirconacycles (equation 632) [839], from reactions of alkynes with iron-carbon disulfide complexes (equation 633) [840], and metal propargyl complexes (equation 634) [841].

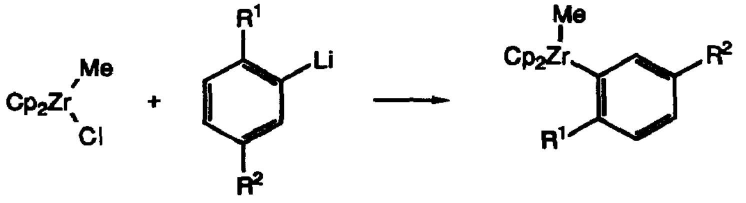
(630)

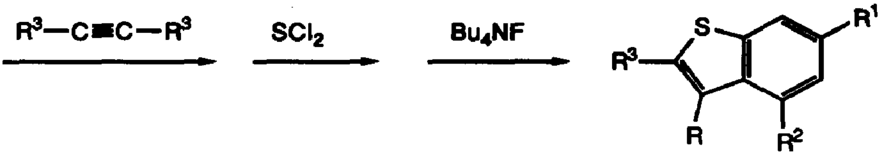


(631)



(632)

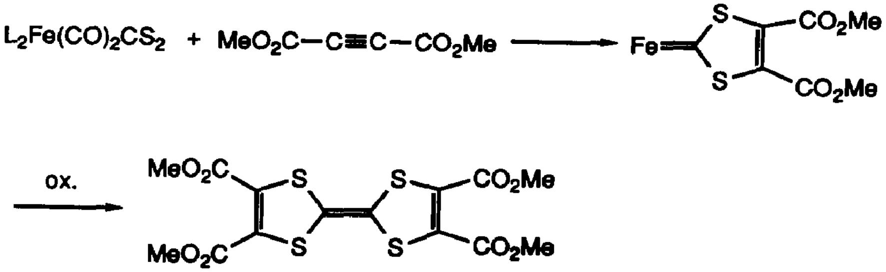




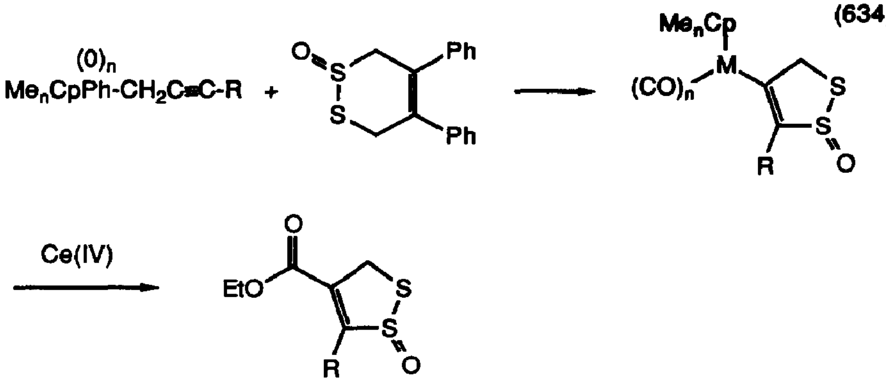
R^1 = Me, OMe
R^2 = H, Me, OMe
R^3 = Me, Et

60-80%

(633)



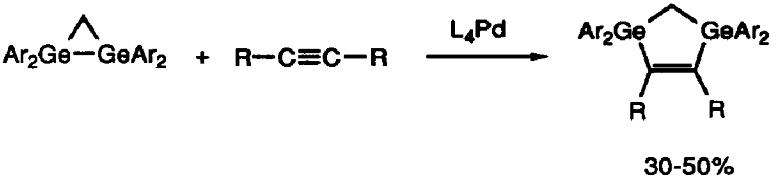
(634)



50-75%

Digermanium heterocycles were prepared from alkynes via palladium catalysis (equation 635) [842] and thienopyridines were prepared from carbamates and thiophene aldehydes [843].

"Ketenimine complexes from carbene complexes and isocyanides - versatile builidng blocks for carbocycles and nitrogen heterocycles" was the subject of a review (59 references) [844]. Low valent nickel-mediated reactions of Grignard reagents with an oxazoline, a thiazoline, isoxazoles, oxazoles, thiazoles and arylammonium salts was the topic of a dissertation [845]. Novel metalloheterocycles formed by [3+2], [2+2], and [2+3] cycloaddition reactions of cyclopentadienyl cobalt and rhodium complexes was the topic of a review (21 references) [846].

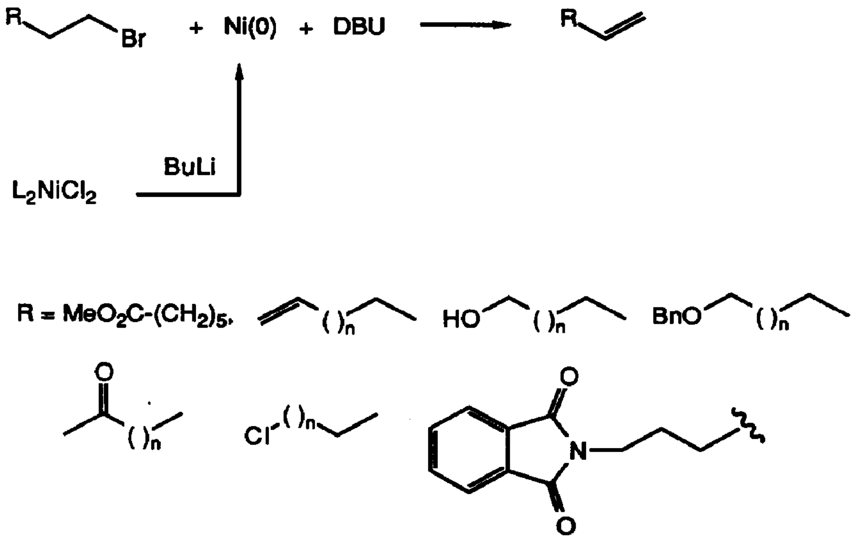


(635)

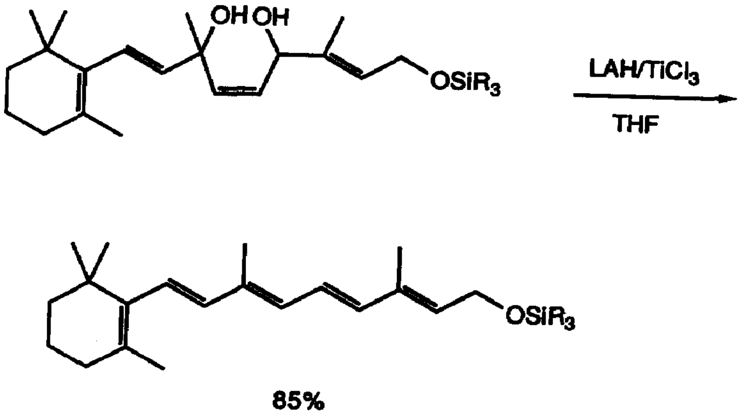
F. Alkenes, Alkanes

Nickel(0) complexes dihydrohalogenated terminal bromides to olefins (equation 636) [847]. Hexane was dehydrogenated to 1-hexene by PMe_3 and $Rh(Cl)(CO)(PMe_3)_2$ [848]. Polychloroalkanes were dehydrochlorinated by molybdenum hexacarbonyl [849].

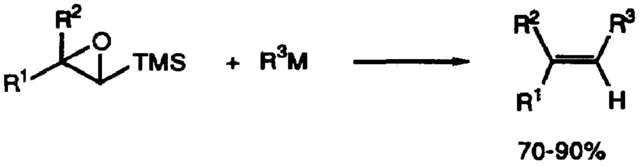
(636)



Synthesis of novel benzenoid molecules by low-valent titanium deoxygenation was reviewed [850]. 2-Butene-1,4-diols were deoxygenated by low valent titanium reagents (equation 637) [851][852]. Epoxysilanes were converted to olefins by organocopper species (equation 638) [853]. The nickel catalyzed reaction of cyclopropyl Grignard reagent with dithianes produced 1,3-dienes (equation 639) [854]. Palladium(0) catalyzed the rearrangement shown in equation 640) [855].

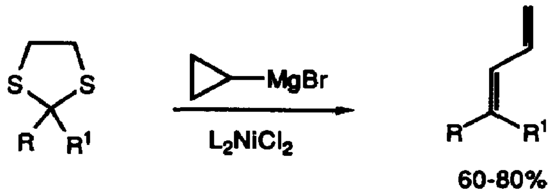


(637)



(638)

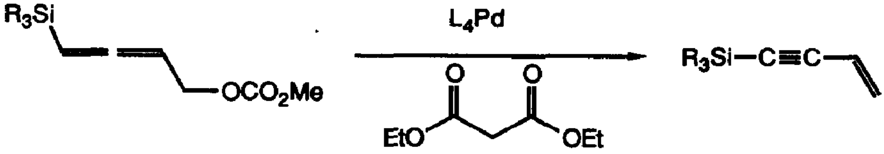
$R^1 = nC_5$, H, nBu
$R^2 = H, nC_5$, nBu, nC_6
$R^3M = Li_2Ph_2CuCN$, $Li_2(CH=CH_2)_2$, CN



(639)

R = Ph, 1-Naph, 2-Naph, oBrPh, pClPh
R^1 = H, Me, Ph

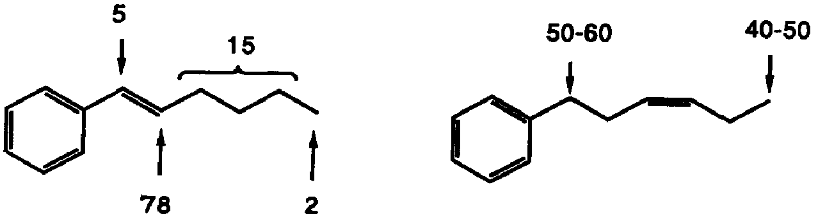
(640)

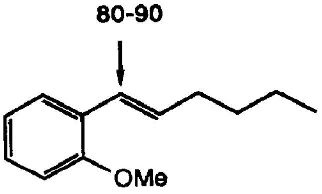


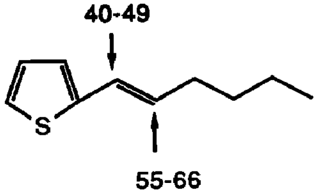
Organic halides were reduced to hydrocarbons by ethyl Grignard reagents in the presence of palladium(II) phosphine catalysts [856], while aryl halides were reduced to arenes by sodium borohydride in the presence of copper(I) chloride [857]. Diisobutyl aluminum hydride reduced aryl halides to arenes in the presence of titanium(IV) and Zr(IV) salts [858]. Thiols, sulfides and disulfides were reduced to hydrocarbons by nickel "complex reducing agents" [859].

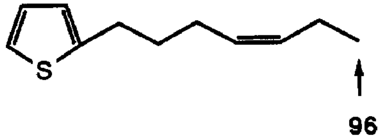
The influence of protecting groups and solvents in the hydrozirconation of oleyl ethers was studied [860]. Deuterozirconation of alkenes was studied (equation 641) [861] to determine where the Zr migrated. Conjugated enones were reduced to saturated enones by tin hydride/copper iodide (equation 642) [862] and by $[Ph_3PCuH]_6$ (equation 643) [863]. Alkynes were reduced to alkenes by silylhydrides in the presence of palladium catalysts (equation 644) [864], (equation 645) [865]. Samarium diiodide reduced alkynes to Z-alkenes in the presence of a proton source [866].

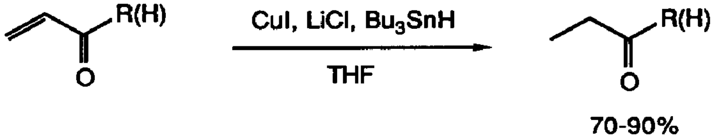
(641)



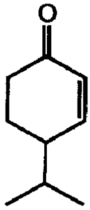

80-90
OMe

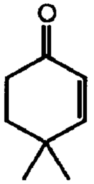

40-49
S
55-66

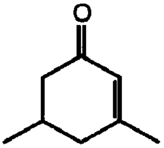

S
96

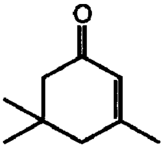

R(H)
O
CuI, LiCl, Bu_3SnH
THF
R(H)
O
70-90%

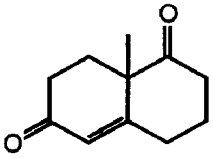
(642)

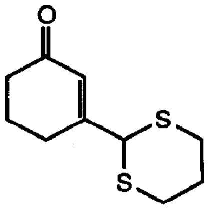

O

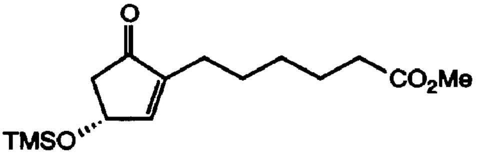

O

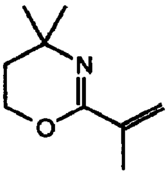

O

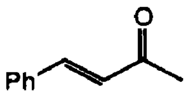

O

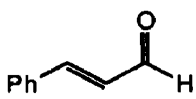

O
O

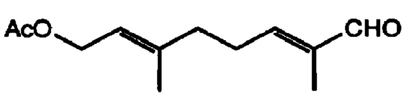

O
S
S


O
CO_2Me
TMSO

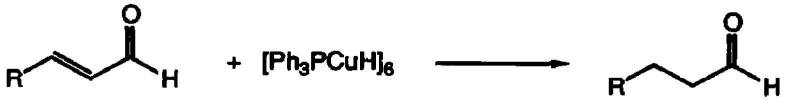

N
O

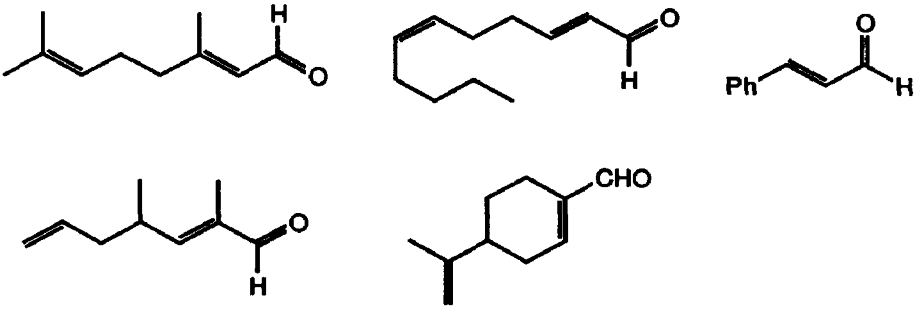

O
Ph

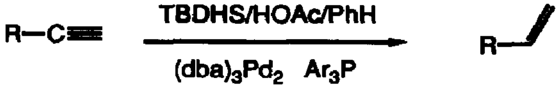

O
Ph
H


AcO
CHO

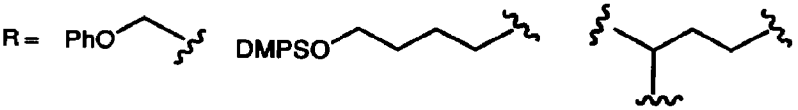
(643)



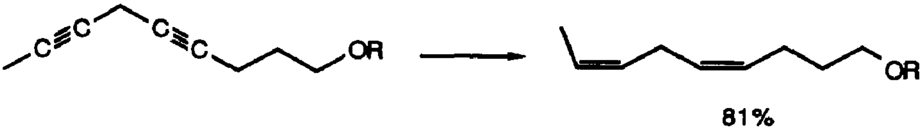


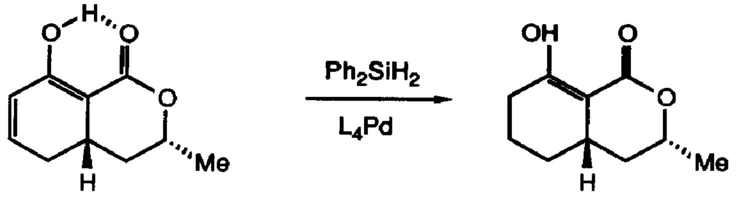


(644)



$Ph-C\equiv C-CO_2Et$ (cis)

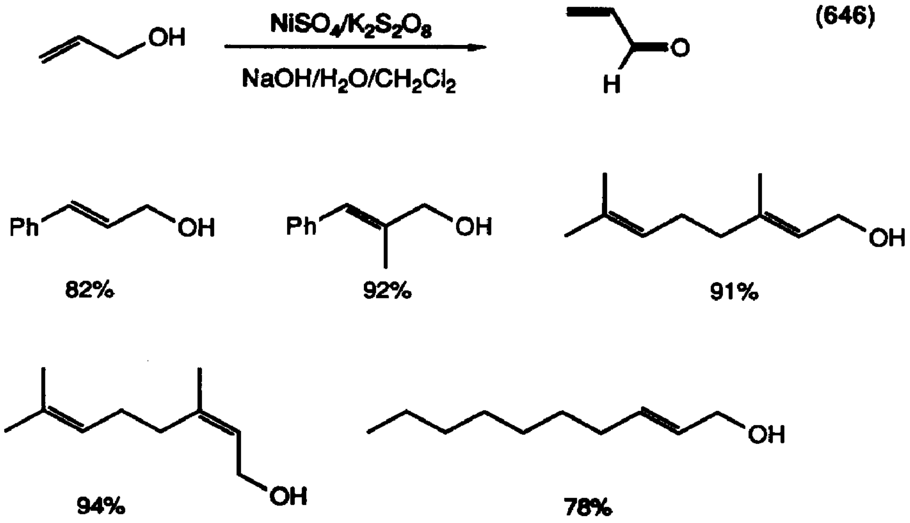




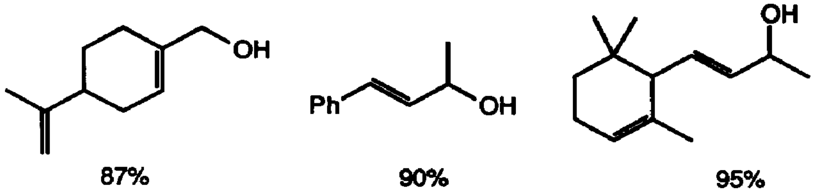
(645)

G. Ketones, Aldehydes

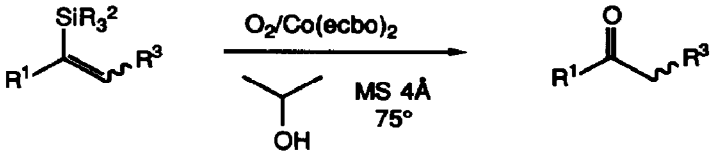
Oxidations using metal complexes (5 references) [867] and palladium(II) catalyzed reactions of olefins with oxygen nucleophiles (83 references) [868] have been reviewed. Cyclooctadiene was oxidized to primarily cyclooctene-4-one by hydrogen peroxide in the presence of palladium chloride [869]. Octene was oxidized to 2-octanone using $Pd(NO_2)_2(MeCN)_2$ as a catalyst [870]. Cyclooctane was oxidized to cyclooctanone and cyclooctanol by tBuOOH in the presence of $Mn_4O_2(O_2CCPh_3)_6(H_2O)_6$ [871]. Allyl alcohols were oxidized to unsaturated aldehydes by peroxydisulfate in the presence of nickel(III) sulfate (equation 646) [872]. Vinyl silanes (equation 647) [873] and silyl enol ethers (equation 648) [874] were oxidized to ketones.

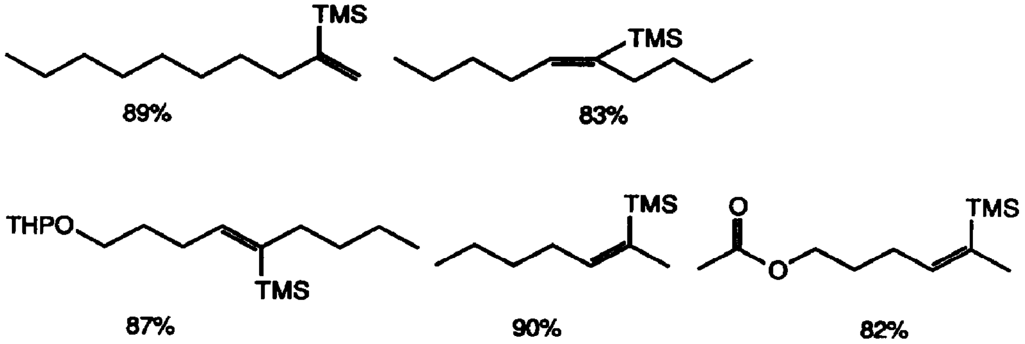


(646)

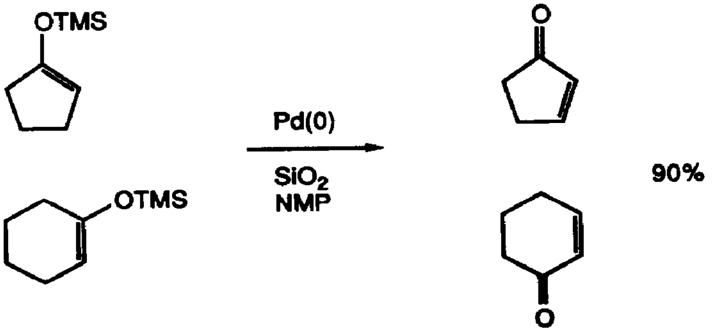

OH
87%
Ph
OH
90%
OH
95%

(647)


SiR$_3$^{2}
R^1
R^3
O$_2$/Co(ecbo)$_2$
MS 4Å
75°
OH
O
R^1
R^3


TMS
89%
TMS
83%
THPO
TMS
87%
TMS
90%
O
O
TMS
82%

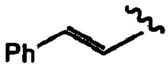
(648)


OTMS
OTMS
Pd(0)
SiO$_2$
NMP
O
O
90%

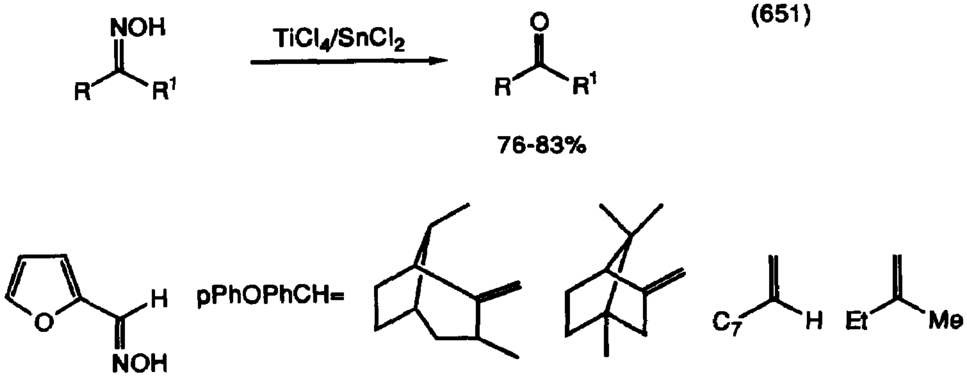
Acid chlorides were reduced to aldehydes by germanium hydrides in the presence of palladium(0) complexes (equation 649) [875]. Ketene complexes (equation 650) [876] and free ketenes (equation 651) [877] were made by reduction of dihalides. Oximes were hydrolyzed to ketones by titanium(IV) chloride/stannous chloride (equation 652) [878].

$$RCOCl + Bu_3GeH \xrightarrow[HMPA]{L_4Pd} \underset{80\text{-}90\%}{RCHO} \qquad (649)$$

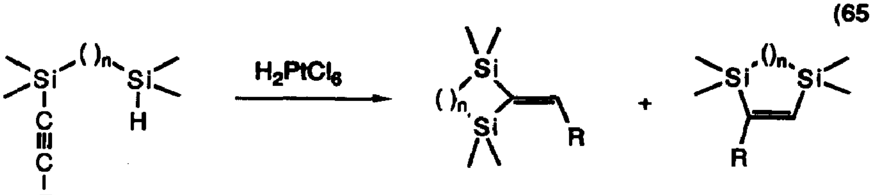
R = 2,4-Cl_2Ph, pNO_2Ph, Ph, 2-FPh,



$$Co_2(CO)_8 + CH_2Br_2 \xrightarrow{Zn} Co_2(CO)_7(CH_2{=}C{=}O) \qquad (650)$$



(652)



R = H,	n = 1	--	--	
	2	100	0	58%
	3	79	21	71%
R = Ph,	n = 2	100	0	42%
	3	100	0	60%

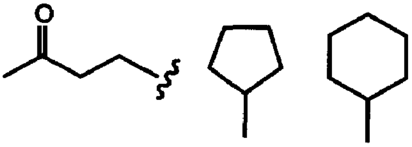
H. Organosilanes

Ketones were enantioselectively hydrosilylated by rhodium(I)/pyridinyl-oxazoline catalysts [879]. Chloroplatinic acid catalyzed the intramolecular hydrosilylation of alkynes (equation 652) [880]. Iridium hydrides catalyzed the o-silylation of alcohols (equation 653) [881]. Aroyl chlorides were converted to aryl silylchlorides by palladium catalysts (equation 654) [882]. Alkynes were silylstannylated using palladium(0) catalysts (equation 655) [883].

$$ROH + Et_3SiH \xrightarrow{IrH_2(THF)_2(PPh_3)_2]SbF_6} ROSiEt_3 \qquad (653)$$

2° > 1° > 3°

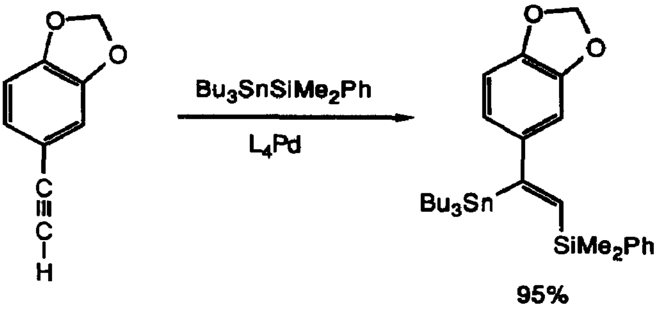
R = Me, Et, nPr, nBu, nC_5, nC_6, iPr, iBu, tBu, Ph, Cl_3CCH_2, $ClCH_2CH_2$-,



(654)

$$ArCOCl + (ClMe_2Si)_2 \xrightarrow{Pd\ cat.} ArSiMe_2Cl$$

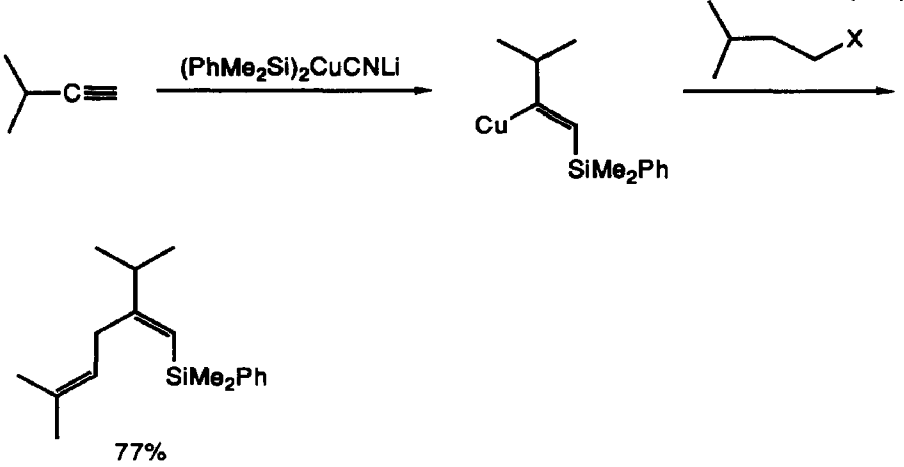
Ar = Ph, pMePh, pClPh, pClCOPh



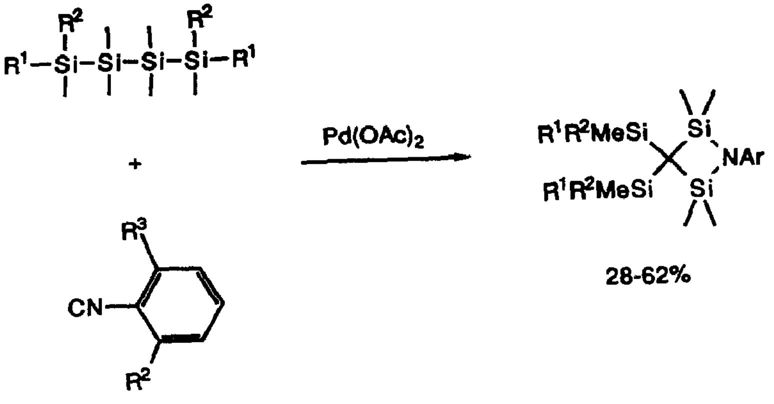
(655)

Alkynes were "carbosilylated" using copper silanes (equation 656) [884]. Silicon heterocycles were made from isonitriles with palladium catalysis (equation 657) [885]. Chromium complexed arenes were lithiated then silylated (equation 658) [885]. Cyclic ethers were silylatively ring opened using cobalt catalysts (equation 659) [886]. Diynes were cyclosilylated using nickel(0) catalysts (equation 660) [887].

(656)

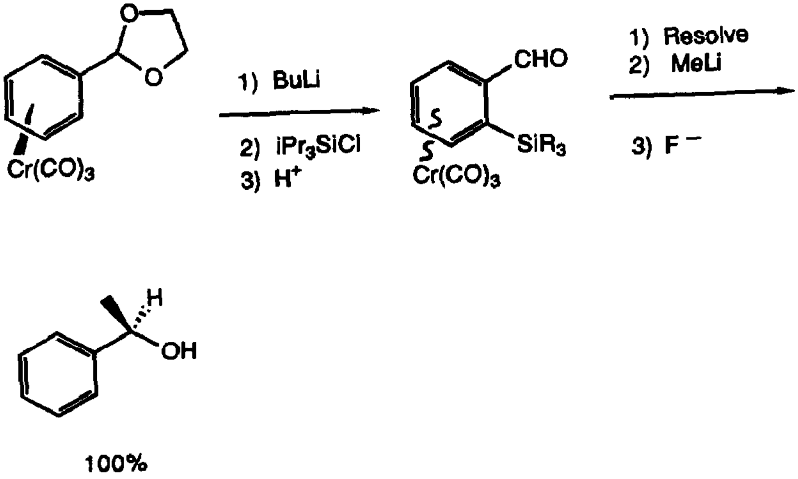


(657)

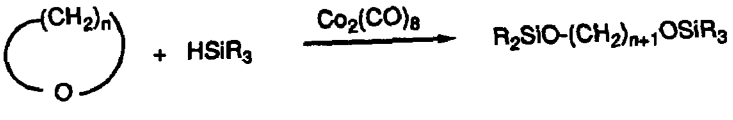


R^1R^2 = Me, Ph

(658)

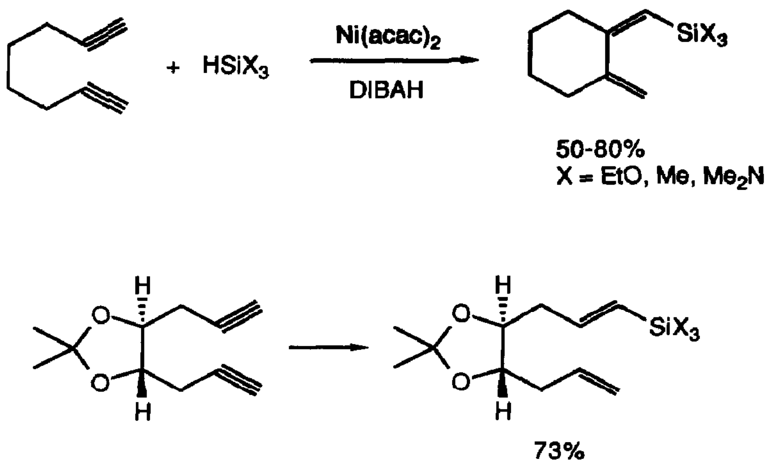


(659)

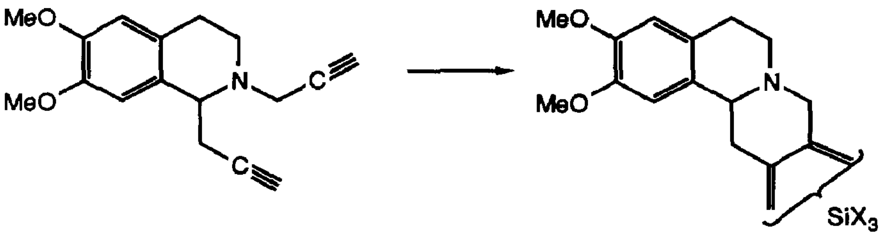


full paper, 30 examples, good yield

(660)



and

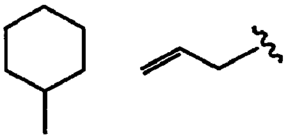


I. Miscellaneous

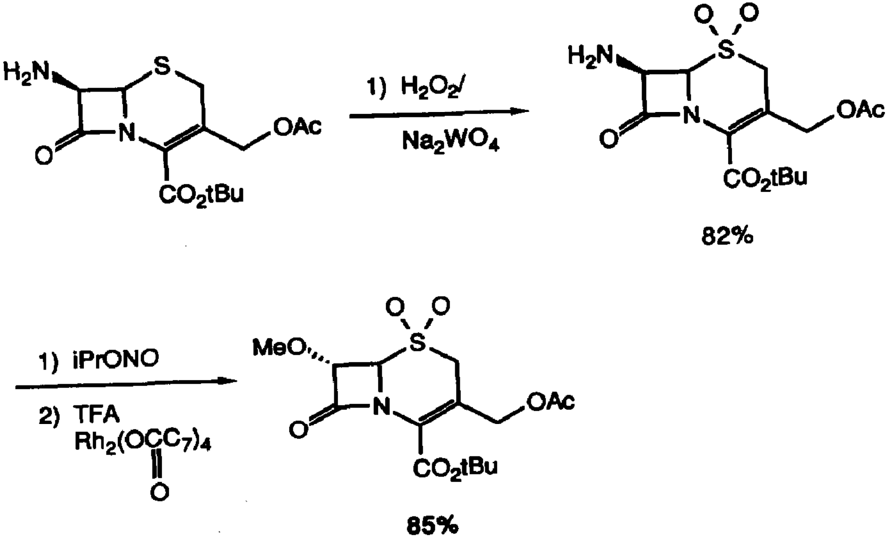
Bromides were converted to disulfides by MoS_4 complexes (equation 661) [888]. Cephams were oxidized to the sulfone by hydrogen peroxide with tungsten catalysts (equation 662) [889]. Chloromercurio nucleosides were thiolated using palladium catalysts (equation 663) [890]. Alkynyl sulfones were reduced to vinyl sulfones with copper catalysts (equation 664) [891].

$$RBr + [MS_4]^{-}R_2NH_2^{+} \longrightarrow \underset{70\text{-}90\%}{RSSR} \qquad (661)$$

R = Bn, pNO_2PhCH_2, $pCNPhCH_2$, $pClPhCH_2$, $mMeOPhCH_2$, nPr, Et, iPr,



(662)



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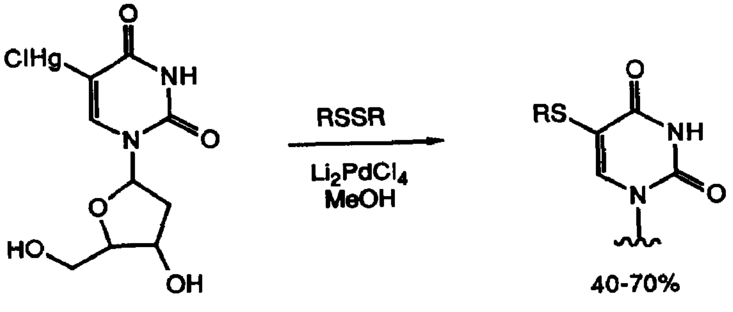
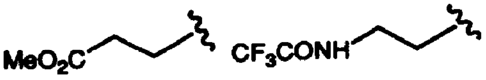
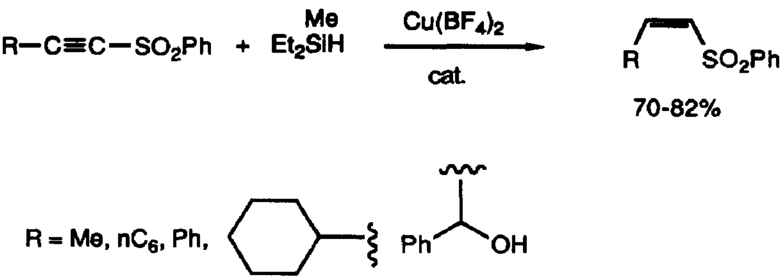


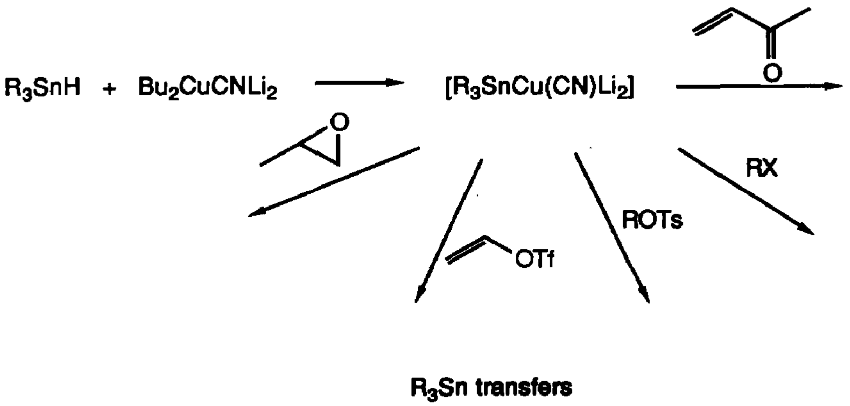
R = Bu, Ph, 

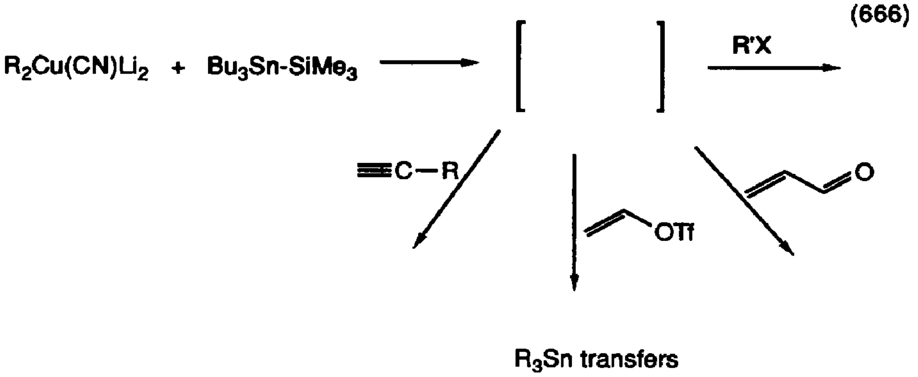
(664)



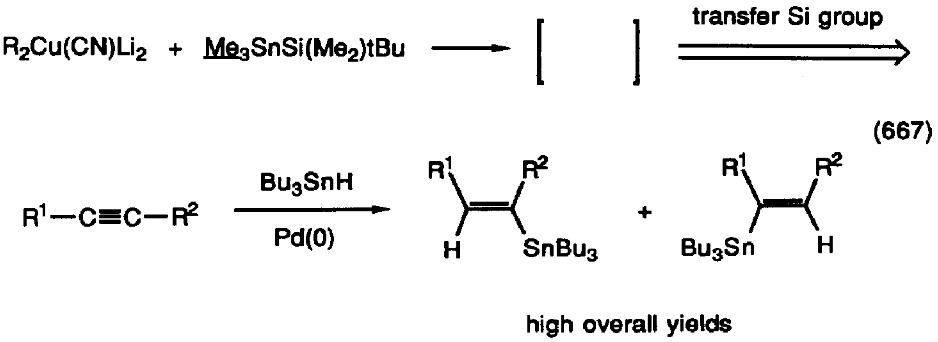
Polypeptides were cleaved from solid supports using L_2PdCl_2/Bu_3SnH [892]. Trialkylstannyl cuprates were prepared, and did normal cuprate chemistry, transferring the R_3Sn group to conjugated enones, halides, epoxides, tosylate vinyl triflates, and alkynes (equation 665) [893][894]. Copper reagents to transfer silyl groups were also developed (equation 666) [895]. Palladium(0) catalyzed the hydrostannylation of alkynes (equation 667) [896].

(665)

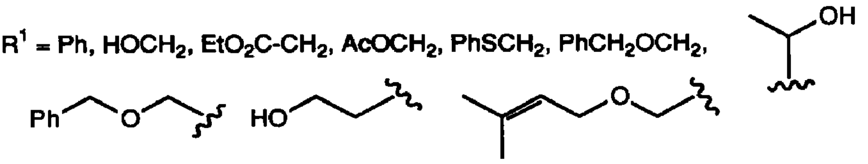




but

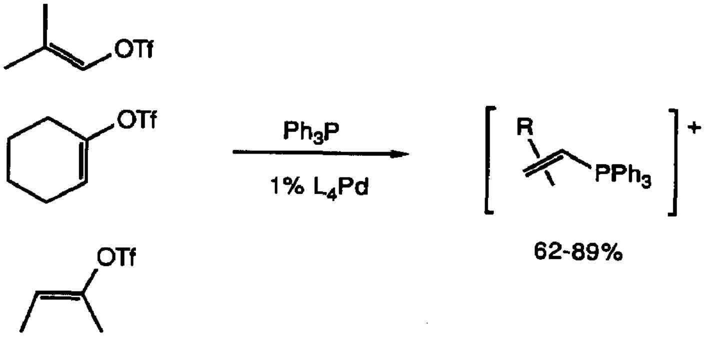


R^1 = Ph, $HOCH_2$, EtO_2C-CH_2, $AcOCH_2$, $PhSCH_2$, $PhCH_2OCH_2$,

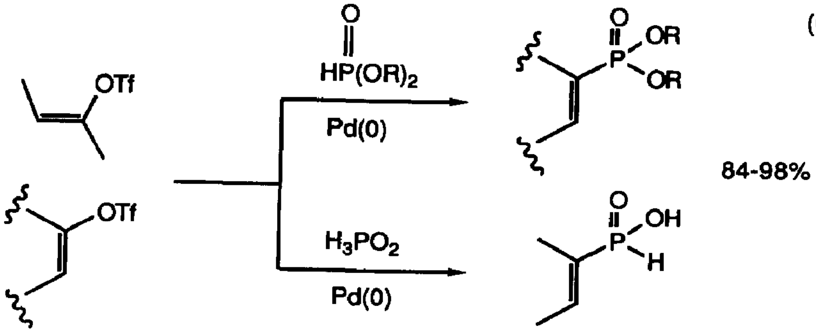


R^2 = H, Me, $AcOCH_2$, $PhSCH_2$, $PhCH_2OCH_2$

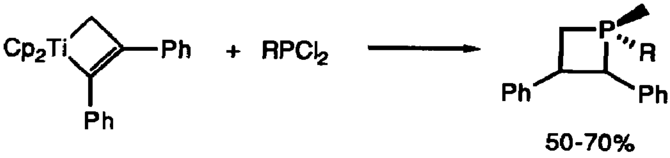
Vinyl triflates were converted to vinyl phosphonium salts using Pd(0) catalysts (equation 668) [897], and to vinyl phosphonates (equation 669) [898]. Other phosphorous compounds were made as in (equation 670) [899], (equation 671) [900], (equation 672) [901].



(668)



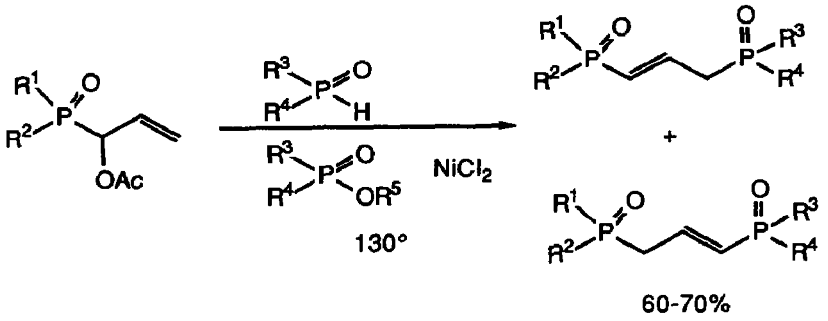
(669)



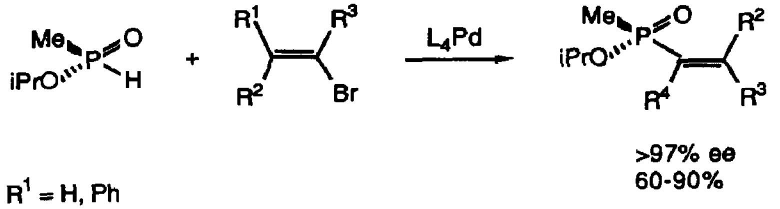
(670)

R = Ph, Et, tBu, EtO

(671)



(672)



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

IV. Reviews

The following reviews and dissertations have appeared.

"The nucleophilicity of metal complexes towards organic molecules", [902].

"Transition metal-tin chemistry", (583 references) [903].

"Enantioselective homogeneous catalysis involving transition metal allyl intermediates", (177 references) [904].

"Catalytic applications of transition metal complexes with sulfide ligands", (93 references) [905].

"Recent developments in organocopper chemistry", [906].

"Intramolecular stoichiometric (Li, Mg, Zn) and catalytic (Ni, Pd, Pt) metallo-ene reactions in organic synthesis", (42 references) [907].

"Boronic esters in stereo directed synthesis", (125 references) [908].

"Acyclic butadiene iron tricarbonyl complexes in organic synthesis", (109 references[909]

"Recent advances in the Reformatsky reaction", (197 references) [910]

"Ziegler-nickel catalysts for hydrogen transfer", [911].

"Metal complexes of small cycloalkynes and arynes", (166 references) [912].

"Transition metal mediated reactions of organic isocyanates", (162 references) [913].

"Catalytic applications of transition-metal complexes with sulfide ligands", (93 references) [914].

"Cyclopalladation reactions", (232 references) [915].

"Mimicking enzyme action: metal-catalyzed transformations of natural products", [916].

"Controlling metal-catalyzed organic syntheses. Part 6: The pragmatic concept of alternative and alternating order factors", [917].

"Organic syntheses, Part 3. Homogeneous catalysis", (0 references) [918].

"Homogeneous catalysis by transition-metal complexes", (348 references) [919].

"Application of organomanganese(II) in organic synthesis", (20 references) [920].

"Low-valent titanium reagents in organic synthesis. Part I", (60 references) [921].

"Uses of low-valent titanium reagents in organic synthesis", (196 references) [922].

"Transition metals in organic synthesis: annual survey covering the year 1987", (902 references) [923].

"Transition metal templates as catalysts for selective organic transformations" (~90 references) [924].

"Transition-metal-mediated reactions of organic isocyanates", (162 references) [925].

"Copper(I) chloride-catalyzed additions of organic polyhalides to olefins", (31 references) [926].

"Organic synthesis through the transmetalation reaction of organotin compounds. Regio- and stereoselective synthesis", (60 references) [927].

"Reactivity of titanocene methylidene with metal halides, alkene sulfides, and alkene oxides", (dissertation) [928].

"Organic syntheses using zirconium compounds. Recent developments", (67 references) [929].

"π-Allylnickel halides and their application in organic synthesis", (10 references) [930].

"Organometallic complexes as catalyst precursors: advantages and disadvantages", (14 references) [931].

"Catalysts and catalytic processes in the synthesis of drugs", (40 references) [932].

"Metallocenes as reaction intermediates", (75 references) [933].

"Metal complexes with small cycloalkynes and arynes", (166 references) [934].

"Chemistry of the transition metals: application to organic synthesis", (2 references), [935].

"Homogeneous catalysis of organic reactions by complexes of metal ions", (182 references) [936].

"Organomolybdenum complexes in organic synthesis: control of relative and absolute stereochemistry", (dissertation) [937].

"Stereoselective synthesis via iron acyl complexes", (dissertation) [938].

"Cobalt-mediated radical reactions in synthesis", (dissertation) [939].

"New applications of organotin reagents in organic synthesis", (31 references) [940].

"Organic syntheses with organoiron complexes", (dissertation) [941].

"Application of organopalladium complexes in organic synthesis", (41 references) [942].

"Cobalt complexes in organic synthesis", (27 references) [943].

"Recent progress in asymmetric synthesis via organocopper compounds" (>33 references) [944].

"Application of organocopper reagents in olefin preparation", (25 references) [945].

"Recent advances in catalytic asymmetric reactions promoted by transition metal complexes", (148 references) [946].

"Synthetic applications of enantioselective, organotransition-metal-mediated reactions", (107 references) [947].

"Enantioselective synthesis of organic compounds with optically active transition metal catalysts in substoichiometric quantities", (344 references) [948].

"Asymmetric catalysis in organic synthesis with industrial perspectives", (88 references) [949].

"Asymmetric synthesis catalyzed by chiral ferrocenylphosphine-metal complexes", [950].

"Enantiodifferentiating transformation of prochiral polyols utilizing menthone as the chiral template", (29 references) [951].

"Asymmetric homogeneous catalysis", (24 references) [952].

"Chiral auxiliaries", (14 references) [953].

"Asymmetric-induced reaction using organocopper reagents", (32 references) [954].

"Synthetic applications of enantioselective organotransition-metal-mediated reactions", (107 references) [955].

"Asymmetric catalysis. IL. Optically active binaphthyl derivatives - synthesis and use in transition-metal catalysts", [956].

"To activate, irradiate!", (19 references) [957].

"Organoiron routes towards compactin analogs", (dissertation) [958].

"Investigations of the synthesis and reactivity of chiral-at-iron benzylidene complexes", (dissertation) [959].

"Studies on the reactivity of titanocene alkylidenes and titanocene alkyl chlorides", (dissertation) [960].

"The nature of transition-metal-carbon σ-bonds. 11. Redox stability in σ-organo-transition-metal chemistry", (>140 references) [962].

"Transition metals in living systems - for the development of highly selective and efficient reactions", (9 references) [962].

"A study of the synthetic and mechanistic aspects of manganese-mediated carbon-carbon bond formation", (dissertation) [963].

"Molybdenum-mediated carbon-carbon bond formation", (dissertation) [964].

"Studies in the Ziegler nickel effect: metal-catalyzed hydrogen transfers", (dissertation) [965].

"Transfer hydrogenation reactions catalyzed by transition metal complexes", (163 references) [966].

"Catalytic reactions of sterically unhindered hydridobis(phosphine)platinum complexes", (>27 references) [967].

"Interaction of ketenes with organometallic compounds: ketene, ketenyl, and ketenylidene complexes", (179 references) [968].

"Chemical behavior of ketene ligands on polymetallic systems", (7 references) [969].

"Activation of carbon-hydrogen bonds of inert hydrocarbons", (20 references) [970].

"Metal complex catalyzed reactions which proceed via carbon-hydrogen bond activation", (50 references) [971].

"Activation of carbon-hydrogen bonds by metal complexes and its application in organic synthesis", (137 references) [972].

"Carbon-hydrogen bond activation by peralkylhafnocene and peralkylscandocene derivatives", (dissertation) [973].

"Carbon-hydrogen, carbon-carbon, tungsten-carbon, and tungsten-tungsten bond activation in the chemistry of $W_2R_2(OR')_4$ complexes", (dissertation) [974].

"New methodology for the catalytic functionalization of hydrocarbons", (172 references) [975].

"Remote functionalization of C-H and C-C bonds by "naked" transition-metal ions (Cosi Fan Tutte)", (25 references) [976].

"The activation and functionalization of aromatic carbon-hydrogen bonds by rhodium isonitrile complexes", (dissertation) [977].

"Reactivity of heterodiene metal carbonyl complexes", (12 references) [978].

"Carbon-carbon bond forming reactions of organotransition metal enolate complexes", (9 references) [980].

"Development of metal homoenolate chemistry and its application to synthesis of fine chemicals", (12 references) [981].

"Cobaloximes as catalysts", (54 references) [982].

"Cobalt-mediated radical reactions in organic synthesis", (20 references) [983].

"Rhodium-catalyzed hydroboration", (8 references) [984].

"Complexes with transition metals as in situ cottonogenic derivatives", [985].

"C-1 chemistry by using hetero elements", (34 references) [986].

"Allyl esters as carboxy protecting groups in the synthesis of glycopeptides", [987].

"Heterocyclic sonochemistry", (90 references) [988].

"Metal cluster compounds - chemistry and importance homogeneous and heterogeneous catalysis", (166 references) [989].

"Dibenzylidene acetone complexes of transition metals", (162 references) [990].

"Silicon hydrides in organic synthesis", (16 references) [991].

"Ketenimine complexes as building blocks for N-heterocyclic and carbocyclic compounds", (25 references) [992].

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