# TRANSITION METALS IN ORGANIC SYNTHESIS
# ANNUAL SURVEY COVERING THE YEAR 1987

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## CONTENTS

## I. General Comments

This annual survey covers the literature for 1987 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 are.

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.

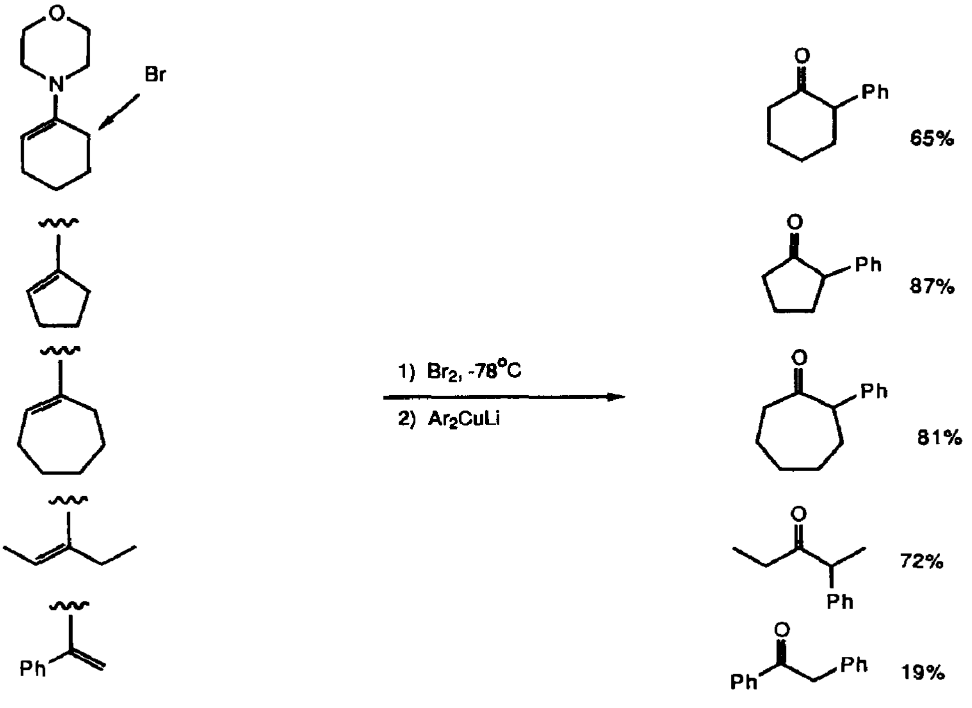
## II. Carbon-Carbon Bond-Forming Reactions

### A. Alkylations

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

Ketones were α-arylated by a process involving α-bromination of the corresponding enamine, followed by reaction with aryl cuprate reagents (equation 1) [1]. Vinyl selenides were alkylated by dialkylcuprates (equation 2) [2]. Lithium dimethylcuprate underwent reaction with secondary tosylates by an $S_N2$ type process, but with secondary bromides or iodides by a single electron transfer process (equation 3) [3]. The involvement of radical intermediates in the reaction of alkyl halides with cuprates was the topic of a dissertation [4]. A variety of organic halides were arylated efficiently by the reagent resulting from the reaction of two equivalents of phenylmagnesium bromide with one equivalent of copper(I) iodide (equation 4) [5]. Muscalure was synthesized using the reaction of oleyl tosylates with Grignard reagents in the presence of copper salts [6]. Alkylmanganese(II) complexes alkylated vinyl triflates and phosphates (equation 5) [7].

(1)



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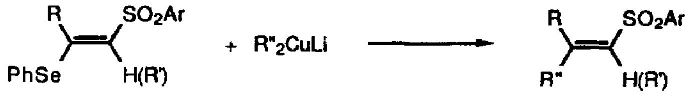
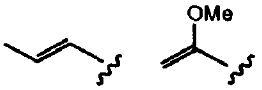


R = H, $nC_8$, TMS, nBu, Ph, $HOCH_2CH_2$-

R' = H, nBu, Me, Ph

R" = Me, nBu, tBu, Ph, secBu,



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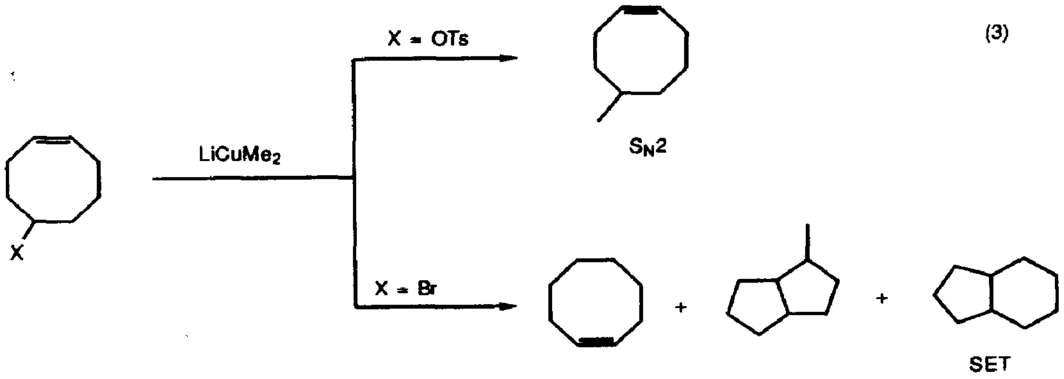

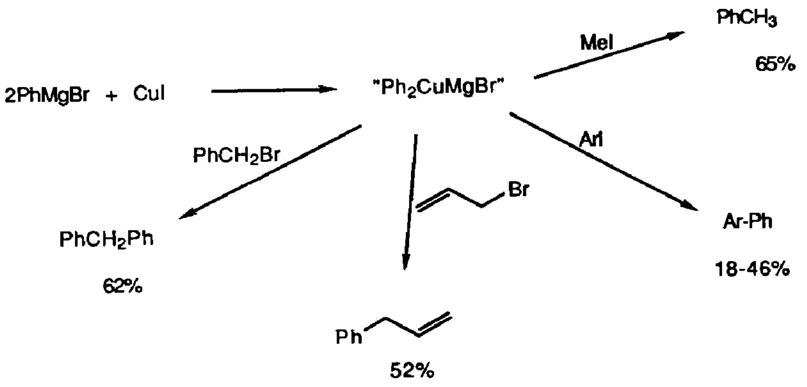

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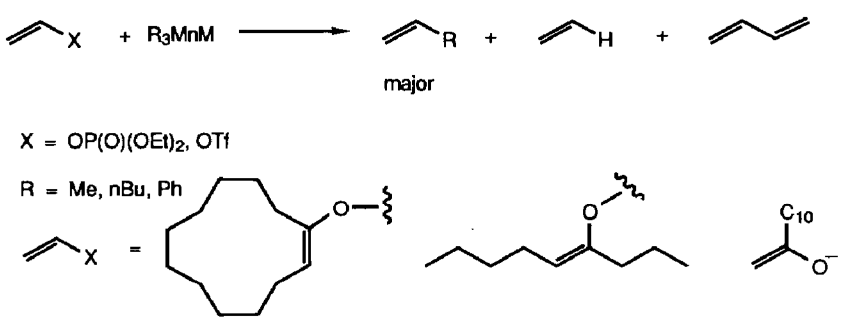
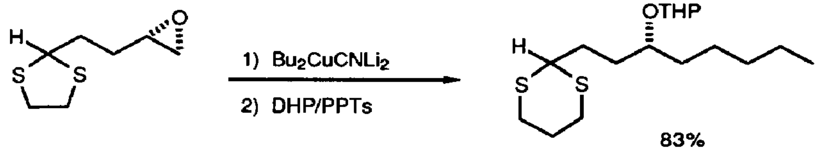


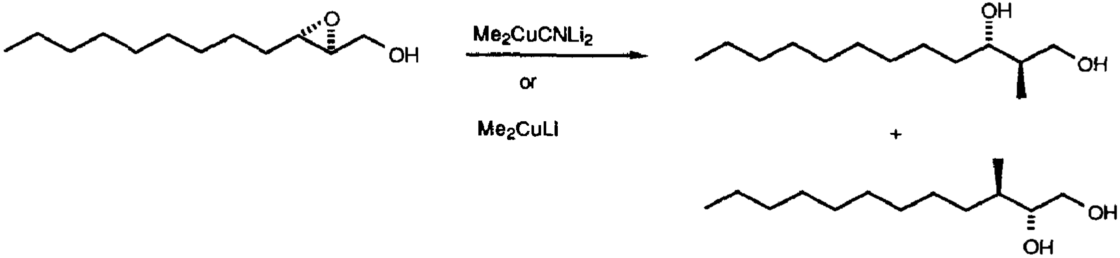
(5)

Long-chain γ-alkoxyaldehydes were prepared by alkylation of epoxides by higher order cyanocuprates (equation 6) [8]. The regiochemistry of the ring opening of epoxyalcohols depended on reaction conditions (equation 7) [9]. Chiral epoxides (from Sharpless epoxidation) underwent alkylation by organocuprates with high stereoselectivity (equation 8) [10], (equation 9) [11]. Reduction of CuI·PBu$_3$ by sodium naphthalenide produced a reactive copper species which formed organocopper reagents readily (equation 10) [12].

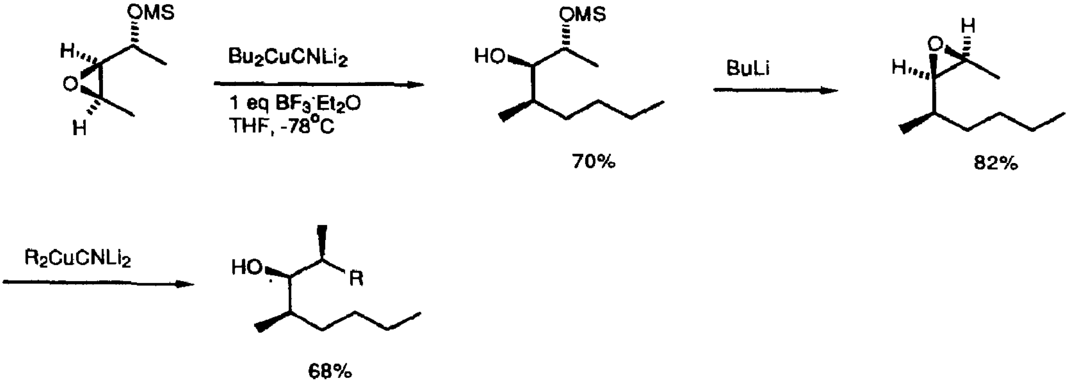


(6)

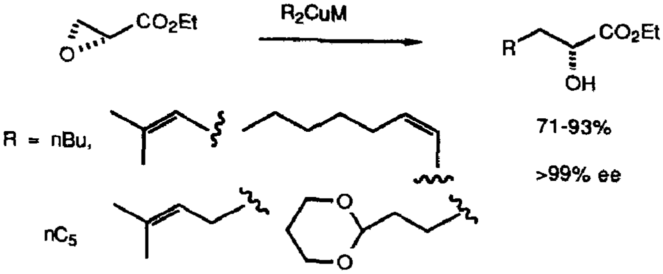
(7)


OH
Me₂CuCNLi₂
or
Me₂CuLi
OH
+
OH
OH

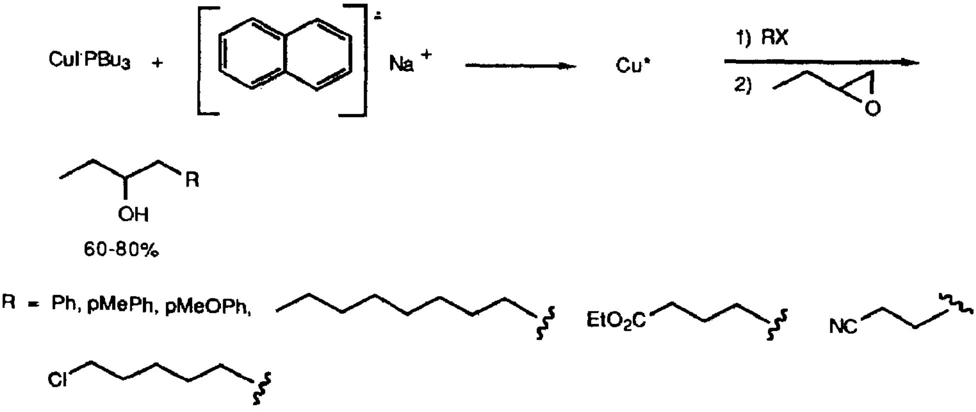
(8)


OMS
H
O
H
Bu₂CuCNLi₂
1 eq BF₃·Et₂O
THF, -78°C
OMS
HO
70%
BuLi
H
O
H
82%
R₂CuCNLi₂
HO
R
68%

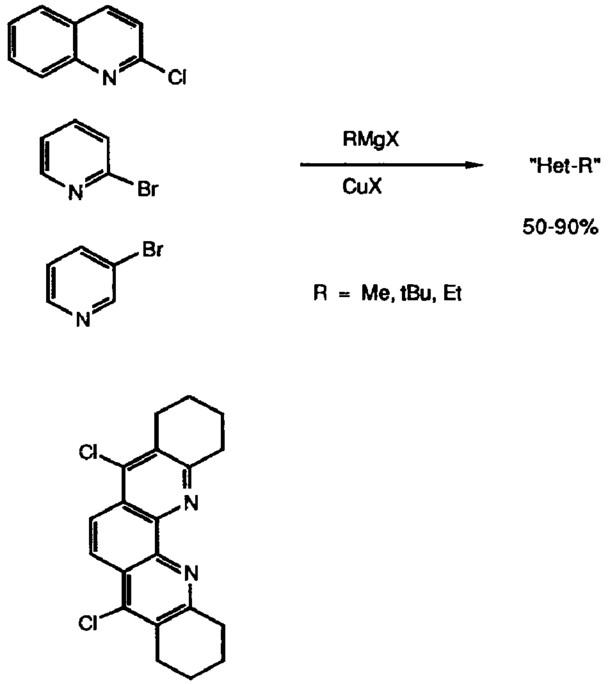
(9)


CO₂Et
O
R₂CuM
R
CO₂Et
OH
71-93%
>99% ee
R = nBu,
nC₅
O
O

(10)

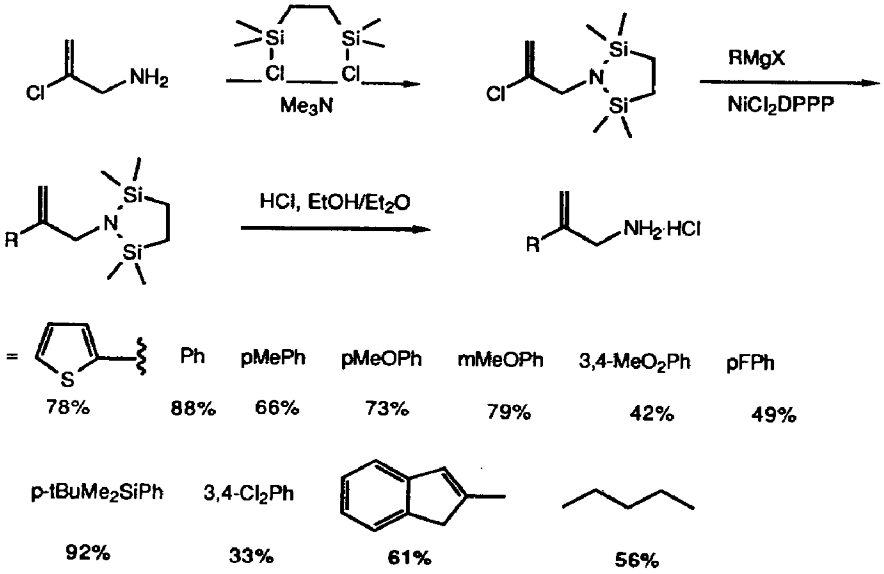

CuI·PBu₃ +
Na⁺
Cu*
1) RX
2)
O
R
OH
60-80%
R = Ph, pMePh, pMeOPh,
EtO₂C
NC
Cl

A variety of heteroaromatic halides were alkylated using copper-catalyzed Grignard reactions (equation 11) [13]. This topic has also been reviewed [14]. β-Chloroallylamines were alkylated using nickel-catalyzed Grignard chemistry (equation 12) [15]. A variety of transition metals, including nickel(II), iron(III),

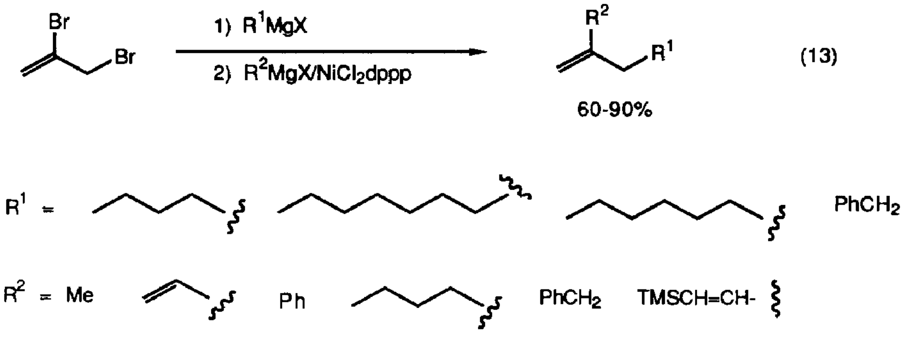


(11)

(12)

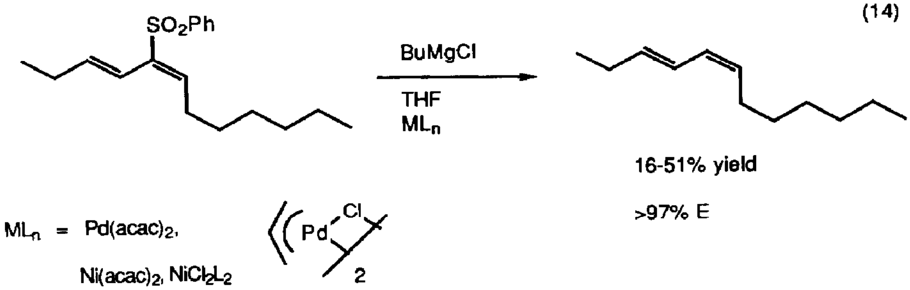


and cobalt (III) were used to catalyze the cross-coupling reactions of Grignard reagents with vinyl chloride [16]. 2-Bromo-allyl bromides were dialkylated via nickel catalyzed Grignard reactions (equation 13) [17]. Palladium and nickel complexes catalyzed the alkylation of vinyl sulfones by Grignard reagents (equation 14) [18]. Chiral nickel and palladium complexes were used to

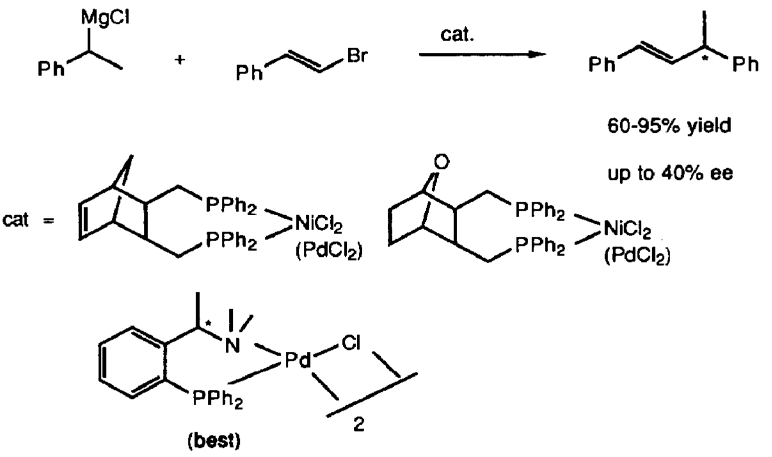


(13)

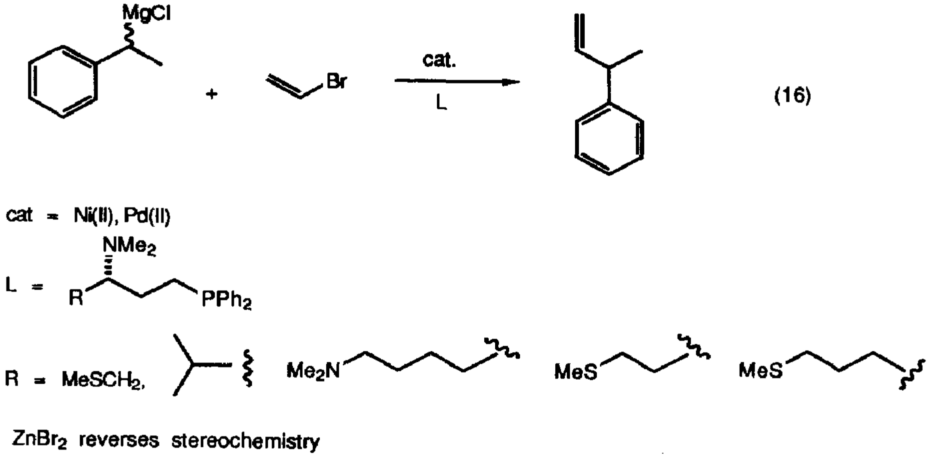
(14)



(15)



catalyzed the asymmetric alkylation of vinyl halides by α-phenethyl Grignard reagents (equation 15) [19], (equation 16) [20]. Copper and palladium salts were used to catalyze the reaction of Grignard reagents with 1,1-ω-trichloro-1-alkenes (equation 17) [21]. This process was used to synthesize propylure.



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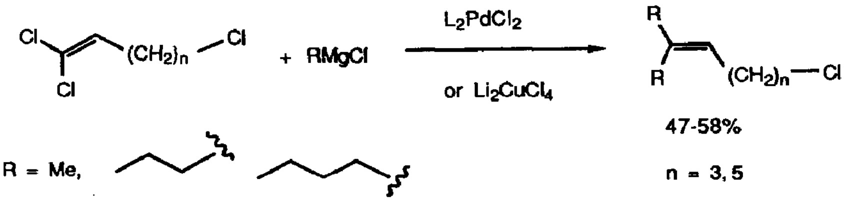
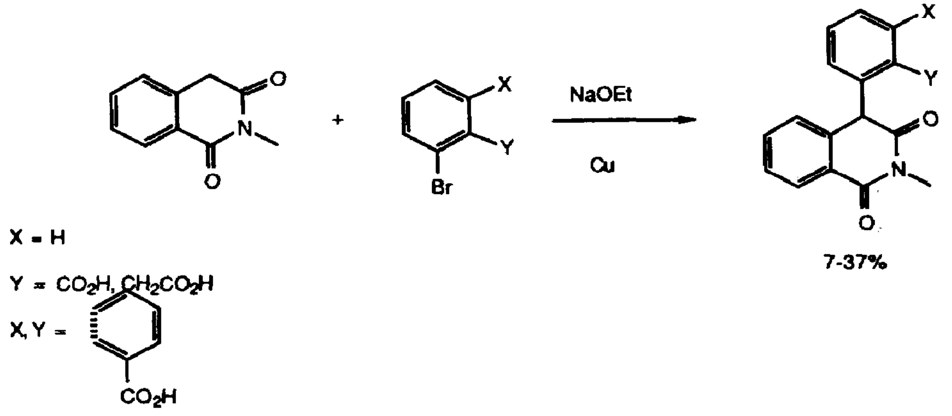


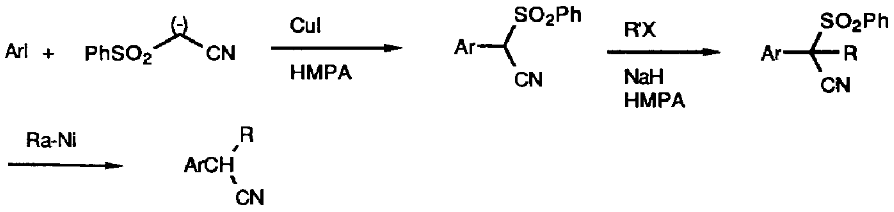
(17)

Copper salts were used to catalyze the coupling of stabilized carbanions with aryl halides (equation 18) [22], (equation 19) [23]. Amines and perfluoroalkyl iodides were coupled by nickel, palladium and platinum catalyst (equation 20) [24].



(18)

(19)



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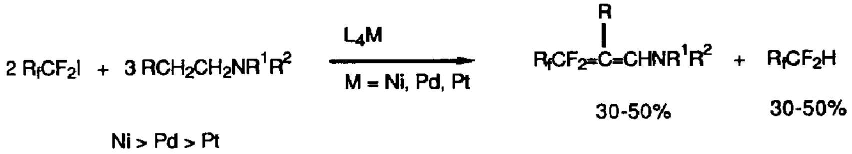
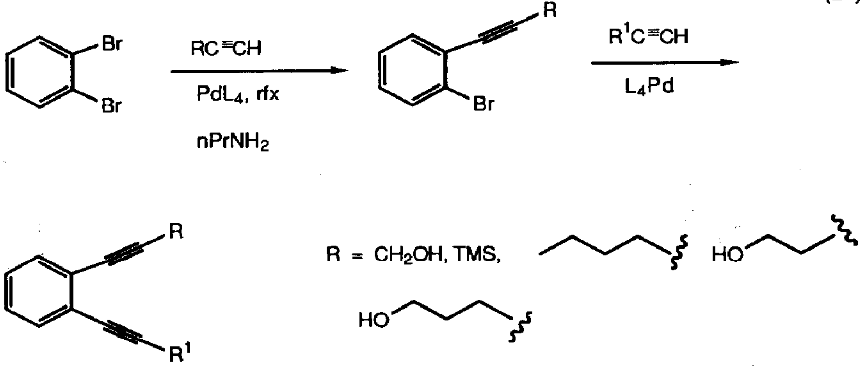


Amine = $Et_3N$, $nPr_3N$, $Et_2NMe$, $nPr_2NMe$

Terminal acetylenes were efficiently coupled to aryl halides (equation 21) [25], (equation 22) [26], (equation 23) [27], (equation 24) [28]; heteroaromatic halides (equations 25-27) [29], and vinyl halides (eqution 28) [30], (equation 29) [31], (equation 30) [32], (equation 31) [33] by mixed palladium/copper catalysts in the presence of amines.

(21)



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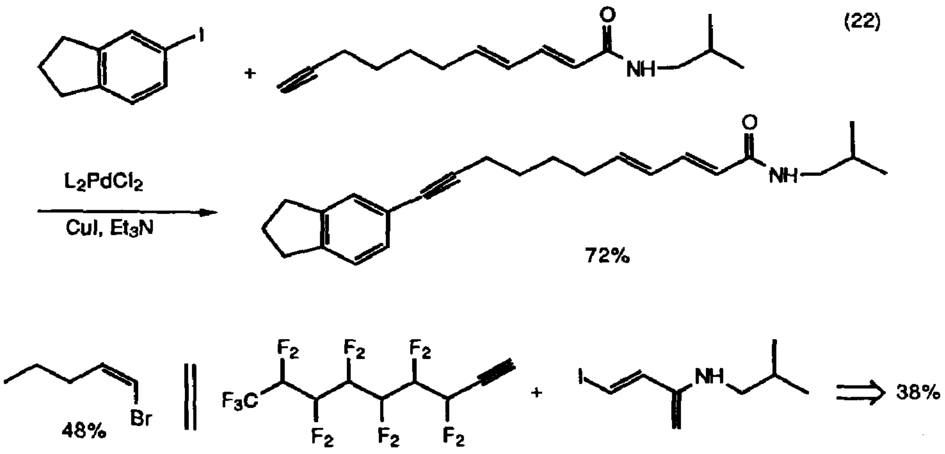

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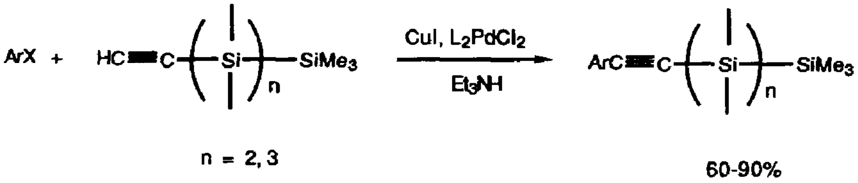
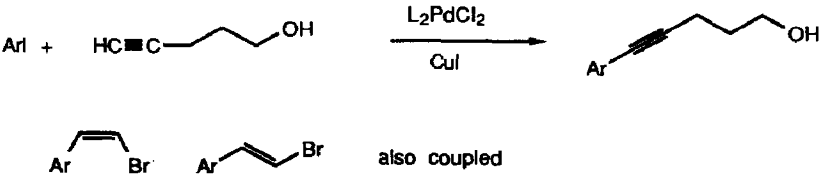
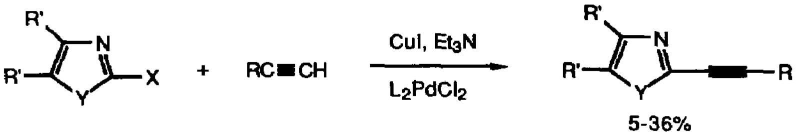


Ar = pMeOPh, Ph, pPh-Ph, pClPh, pAcOPh, pCNPh, mCNPh, pNO$_2$Ph, 2-Naphth

(24)



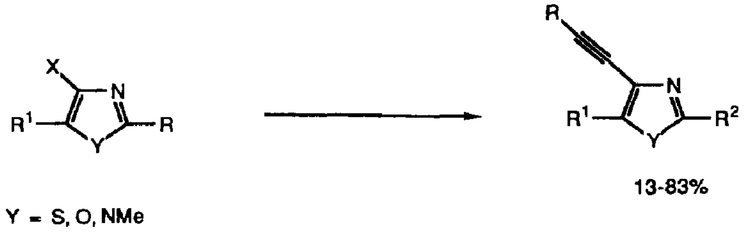


(25)

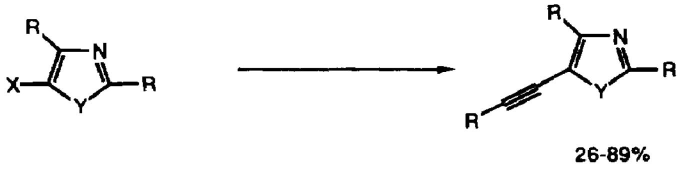
Y = S, NMe

X = Br, I

R' = H, Ph

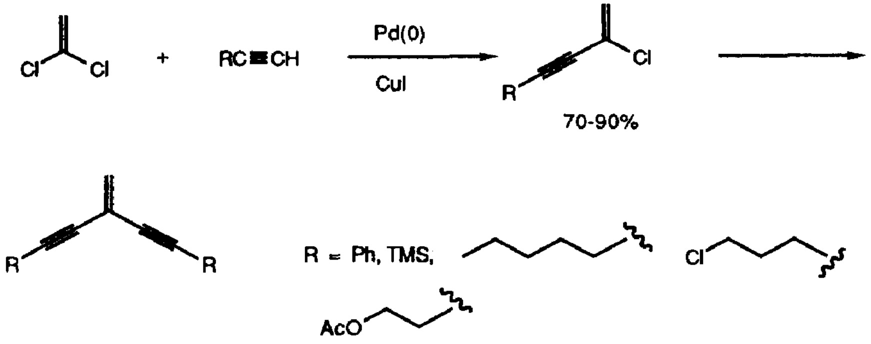

X
R¹
R
N
Y
Y = S, O, NMe
R
R¹
R²
13-83%

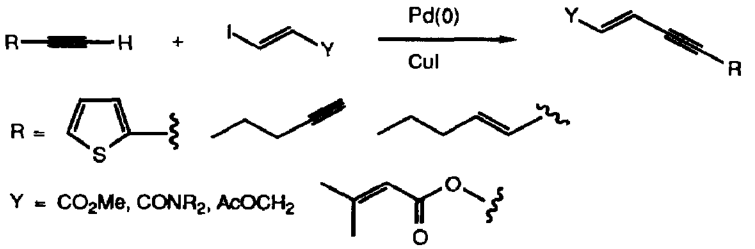
(26)


R
X
R
N
Y
R
R
R
26-89%

(27)

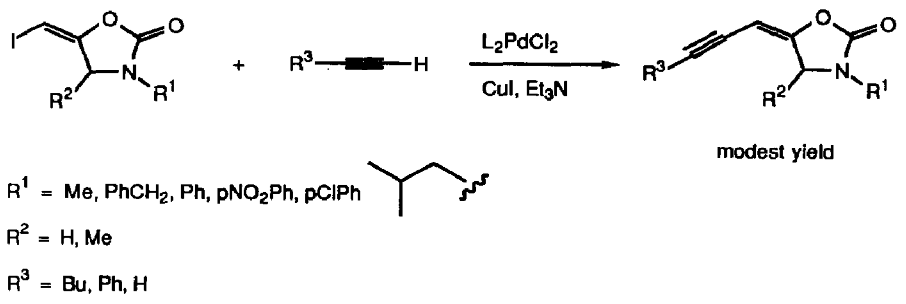
(28)


Cl
Cl
+
RC≡CH
Pd(0)
CuI
R
Cl
70-90%
R
R
R = Ph, TMS,
Cl
AcO

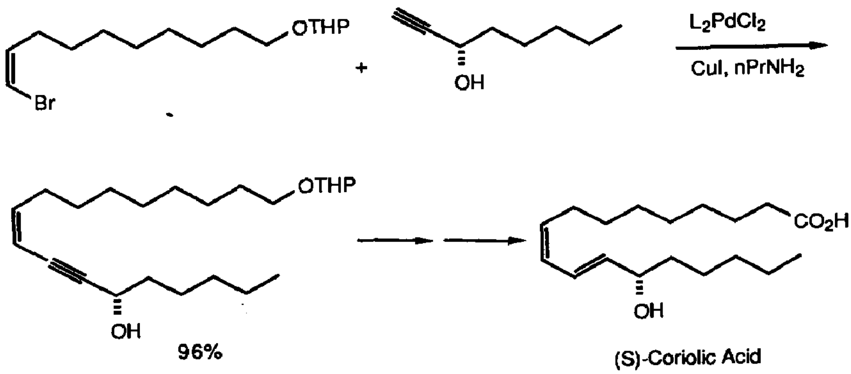

R
H
+
I
Y
Pd(0)
CuI
Y
R
R =
S
Y = CO₂Me, CONR₂, AcOCH₂
O
O

(29)

(30)

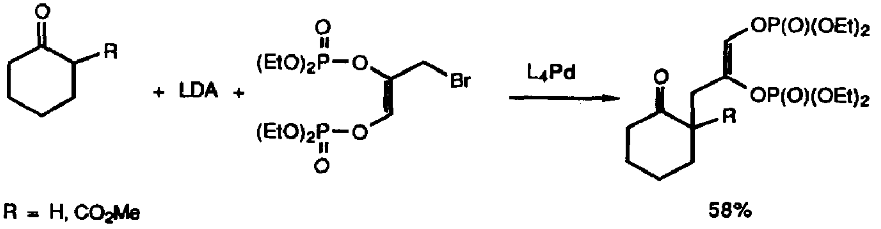


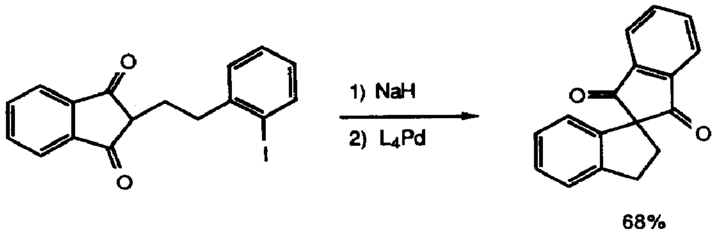
(31)



The most active area for research for the alkylation of halides currently involves oxidative addition/transmetallation/reductive elimination processes, catalyzed by palladium(0) or nickel(0) complexes, and involving transmetallation from main group organometallics. Thus, palladium(0) catalyzed the coupling of lithium enolates to allylic halides (equation 32) [34], sodium enolates to arylhalides (equation 33) [35], vinylalanes to aryl halides (equation 34) [36], and vinylalanes (equation 35), and zirconium species (equation 36) to vinyl halides [37].

(32)





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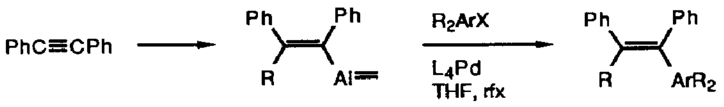
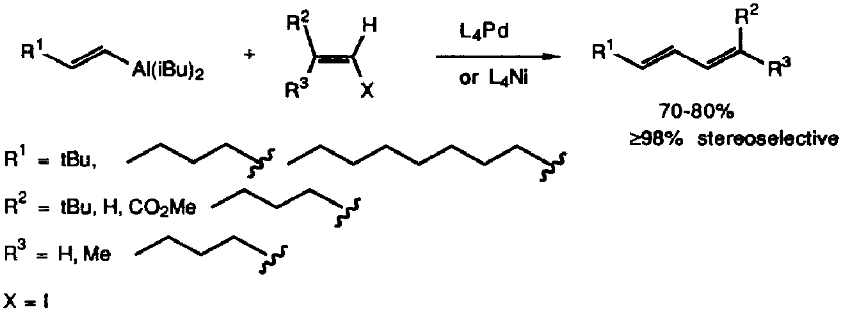


(34)

R = Et, H

$R_2$ = pMeO, pMe$_2$N, CH$_2$CH$_2$O, pMe



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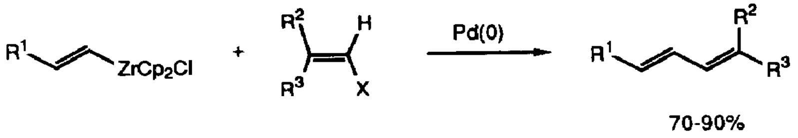


(36)

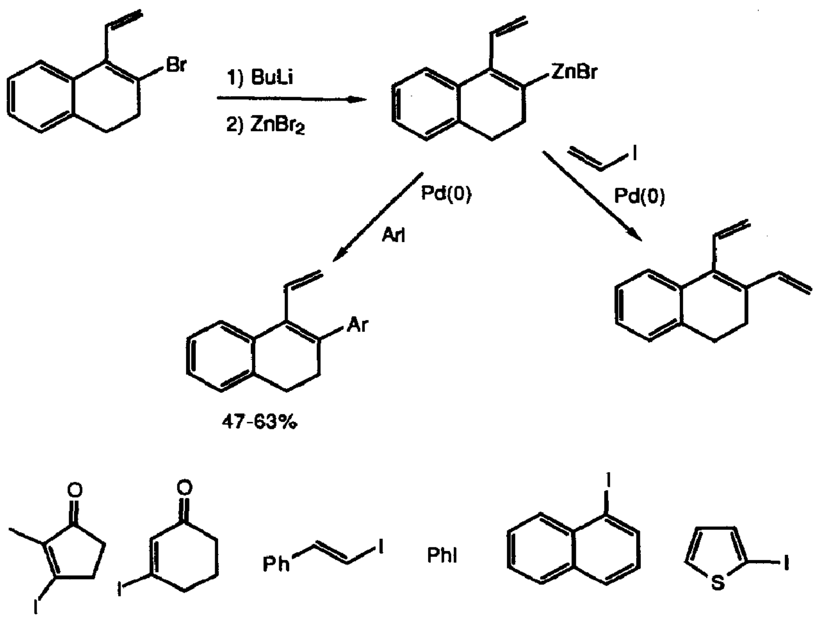
$R^1$ = nC$_5$, nC$_6$, nBu, THPOCH$_2$

$R^2$ = nBu, H, CO$_2$Me

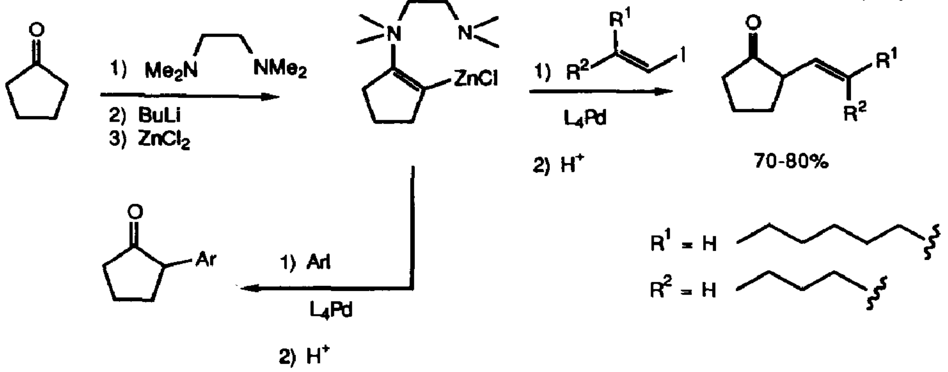
$R^3$ = H, Me

Transmetallation from zinc to palladium is one of the most extensively used processes for alkylation of halides. Thus vinyl halides (equation 37) [38], (equation 38) [39], (equation 39) [40], (equation 40) [41], (equation 41) [42], (equation 42) [43], (equation 43) [44, 45], (equation 44 and 45) [46], aryl halides (equation 46) [47], (equation 47) [48], (equation 48) [49], (equation 49 and 50)

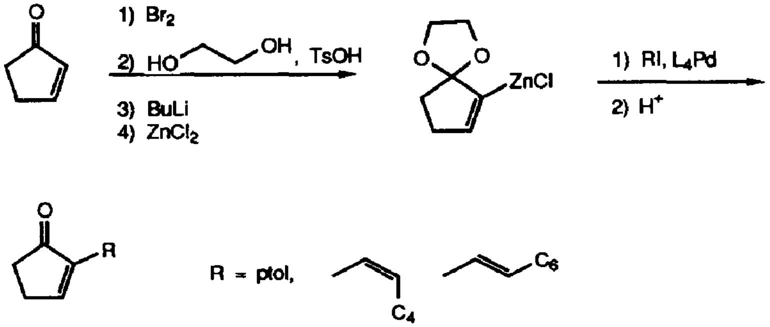
(37)


Br
1) BuLi
2) ZnBr$_2$
ZnBr
Pd(0)
ArI
Pd(0)
I
Ar
47-63%
O
O
I
I
Ph
I
PhI
I
S
I

(38a)

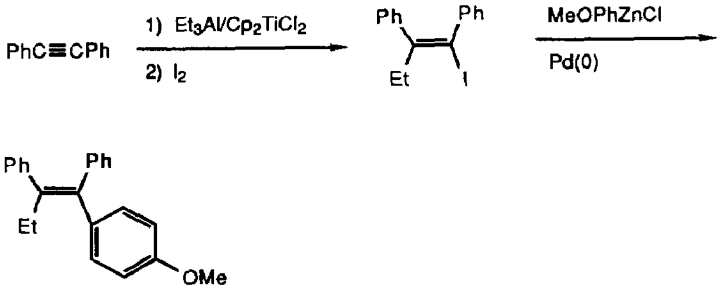

O
1) Me$_2$N NMe$_2$
2) BuLi
3) ZnCl$_2$
N
N
ZnCl
R$^1$
1) R$^2$
I
L$_4$Pd
2) H$^+$
O
R$^1$
R$^2$
70-80%
O
Ar
1) ArI
L$_4$Pd
2) H$^+$
R$^1$ = H
R$^2$ = H

(38b)

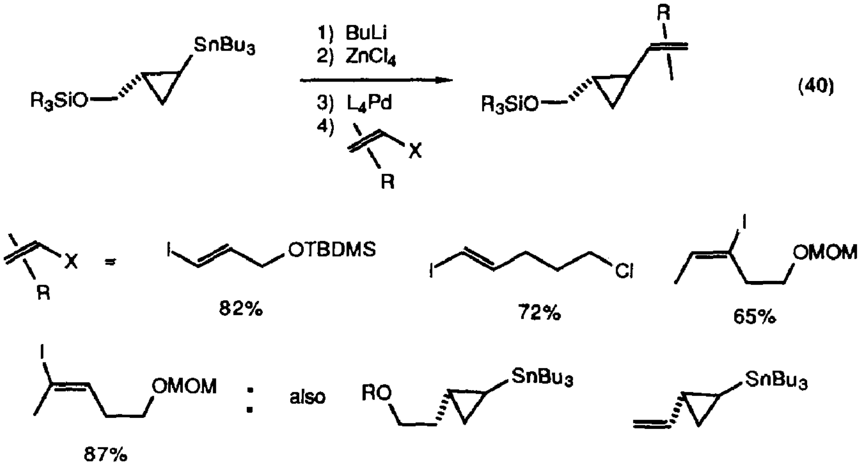

O
1) Br$_2$
2) HO OH, TsOH
3) BuLi
4) ZnCl$_2$
O O
ZnCl
1) RI, L$_4$Pd
2) H$^+$
O
R
R = ptol,
C$_4$
C$_6$

[50], aryl and vinyl triflates (equation 51) [51], (equation 52) [52], (equation 53) [53], α-bromonitriles (equation 54) [54] and perfluoroalkyliodides (equation 55) [55] could be alkylated by organozinc reagents.

(39)



(40)



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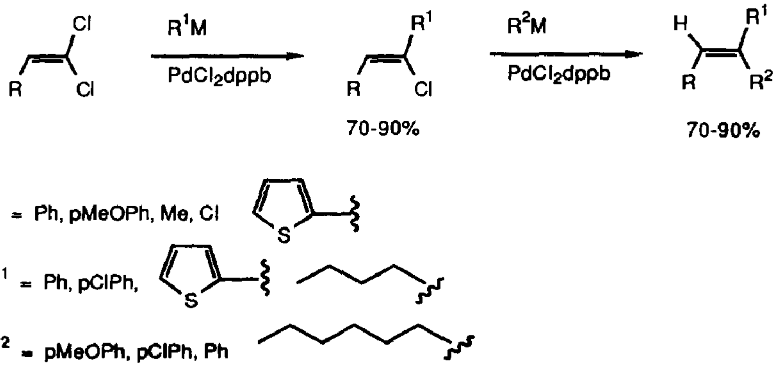
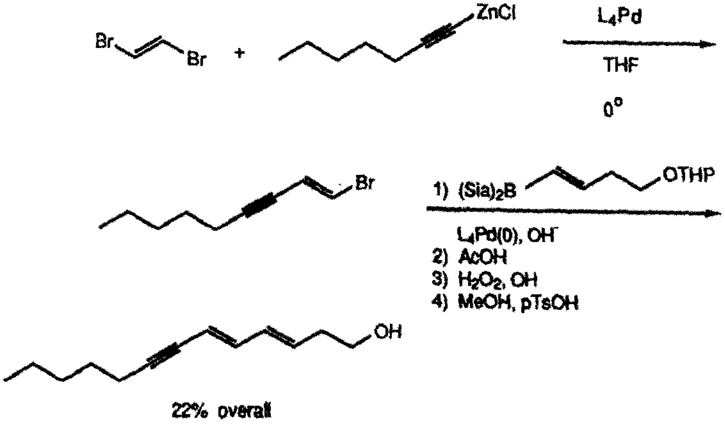
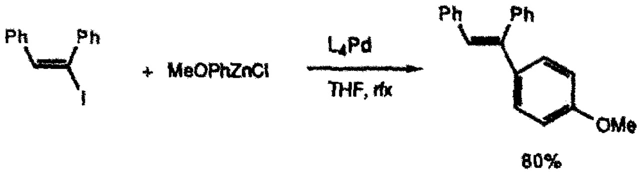


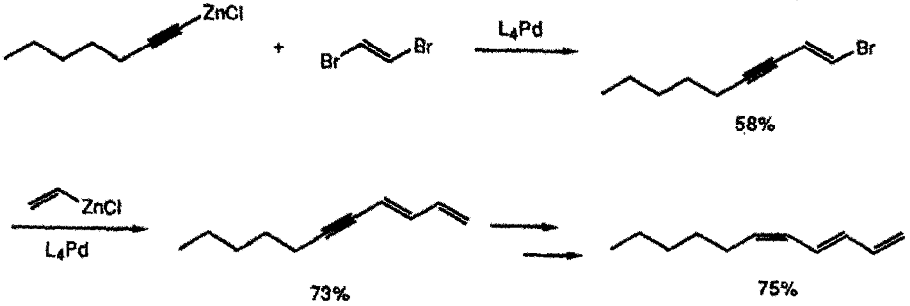
(42)


Br
Br
+
ZnCl
L₄Pd
THF
0°
Br
1) (Sia)₂B
OTHP
L₄Pd(0), OH⁻
2) AcOH
3) H₂O₂, OH
4) MeOH, pTsOH
OH
22% overall

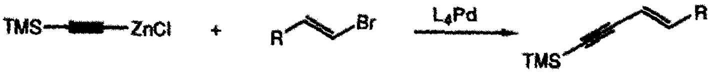
(43)


Ph
Ph
I
+ MeOPhZnCl
L₄Pd
THF, rfx
Ph
Ph
OMe
80%

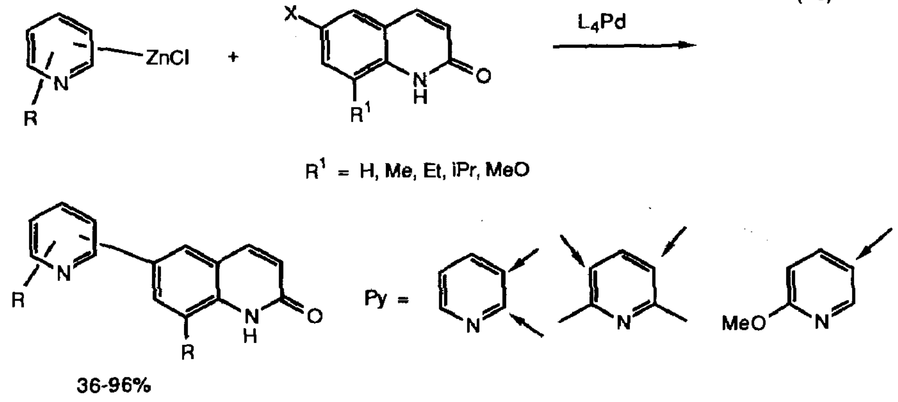
(44)


ZnCl
+
Br
Br
L₄Pd
Br
58%
ZnCl
L₄Pd
73%
75%

(45)

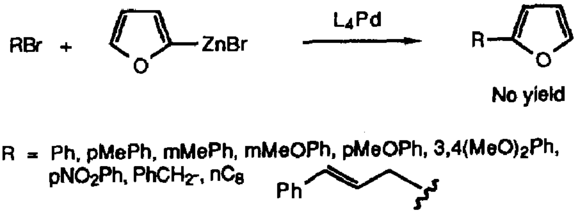

TMS
ZnCl
+
R
Br
L₄Pd
R
TMS

(46)



$R^1$ = H, Me, Et, iPr, MeO

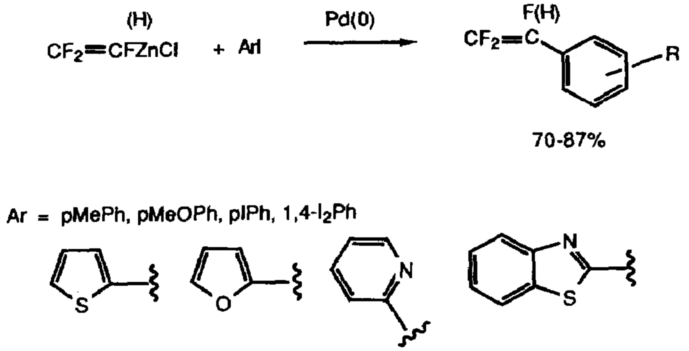
36-96%



(47)

No yield

R = Ph, pMePh, mMePh, mMeOPh, pMeOPh, 3,4(MeO)$_2$Ph, pNO$_2$Ph, PhCH$_2$-, nC$_8$

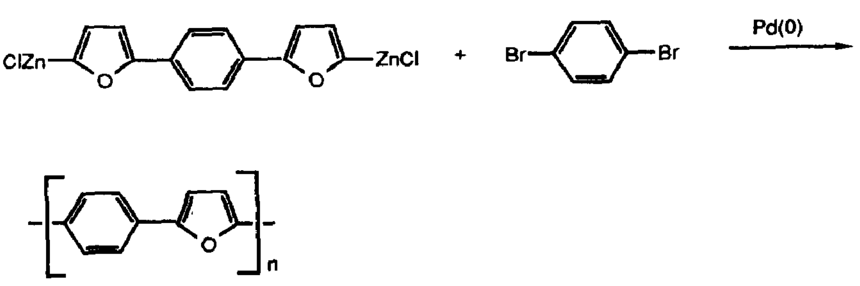


(48)

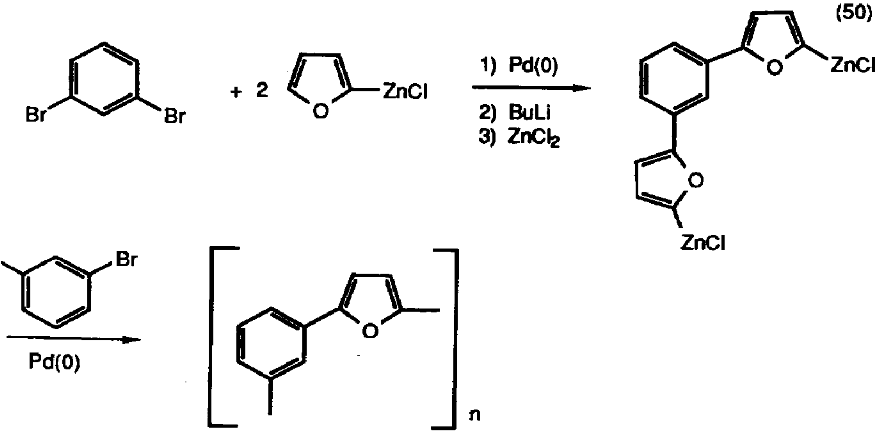
70-87%

Ar = pMePh, pMeOPh, pIPh, 1,4-I$_2$Ph

(49)



(50)



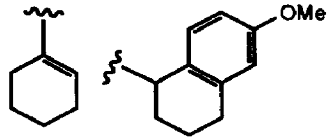
$$\mathrm{ArOSO_2R_f} + \mathrm{RZnCl} \xrightarrow[\mathrm{LiCl}]{\mathrm{L_4Pd}} \underset{60\text{-}70\%}{\mathrm{ArR}} \qquad (51)$$

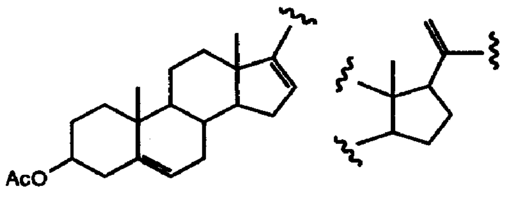
R = $\mathrm{PhCH_2}$, $\mathrm{EtO_2CCH_2CH_2}$ $\mathrm{nBu{-}C{\equiv}}$ $\mathrm{MeOH_2C{-}C{\equiv}}$ $\mathrm{PhC{\equiv}C{-}}$

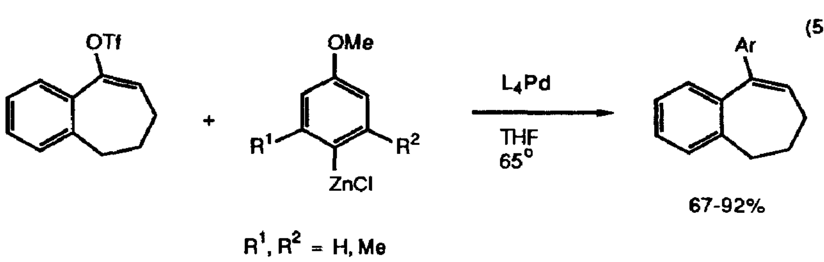
Ar = Ph, pClPh, mClPh, oClPh, $\mathrm{pNO_2Ph}$, mMeOPh

$$\mathrm{BrZnCH_2CO_2tBu} + \mathrm{ROTf} \xrightarrow{\mathrm{L_2Pd(0)}} \underset{40\text{-}60\%}{\mathrm{RCH_2CO_2tBu}} \qquad (52)$$

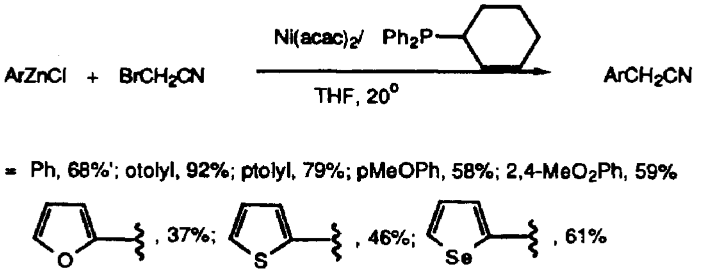
R = pClPh, pMeSPh, 2-Naphth



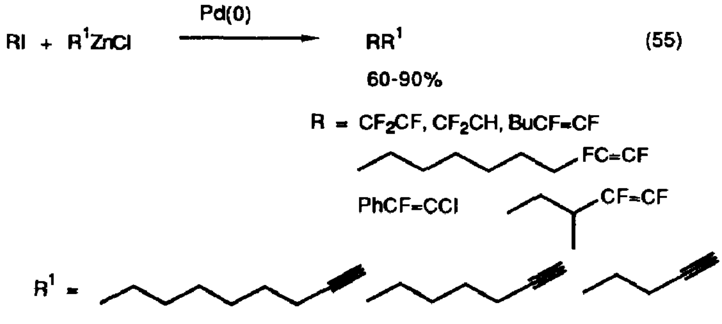




(53)

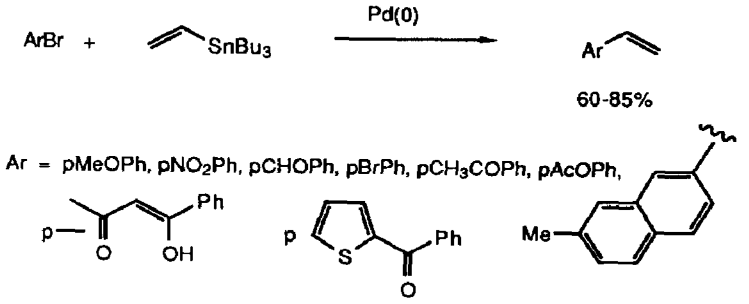


(54)



(55)

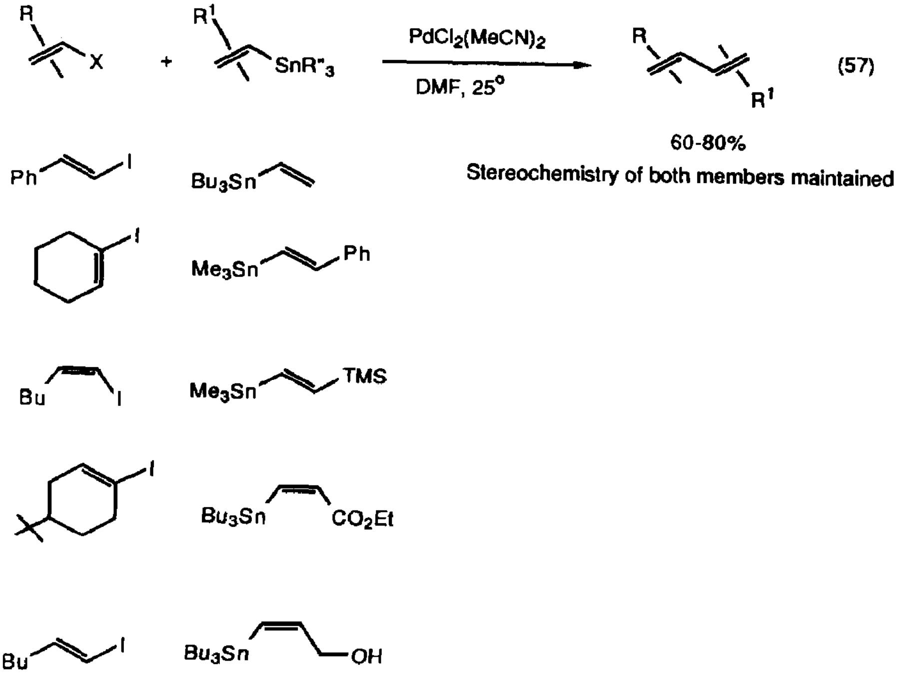
Alkyltin reagents are a close second to alkyl zinc reagents for the oxidative addition/ transmetallation/reductive elimination process discussed above. Thus, palladium(0) catalyzed the coupling of aryl halides with vinyl tins (equation 56) [56], vinyl halides with vinyl tins (equation 57) [57], vinyl halides with acetylenic tins (equation 58) [58], aryl halides with allyl tins (equation 59) [59], vinyl and allyl tins with perfluoroiodides (equation 60) [60], heteroaromatic halides with vinyl tins (equation 61) [61], heteroaromatic tins with heteroaromatic halides (equation 62) [62], heteroaromatic tins with aryl halides (equation 63) [63], α-alkoxyvinyl tins with aryl, vinyl, and allyl halides (equation 64) [64], (equation 65) [65], aryl trilfates with alkyl, vinyl, aryl, and acetylenic tins (equation 66) [66], vinyl triflates with vinyl tins (equation 67) [67], and cyclization of vinyl triflates with vinyl tins (equation 68) [68]. It is not clear that any other possible combination exists.



(56)

60-85%

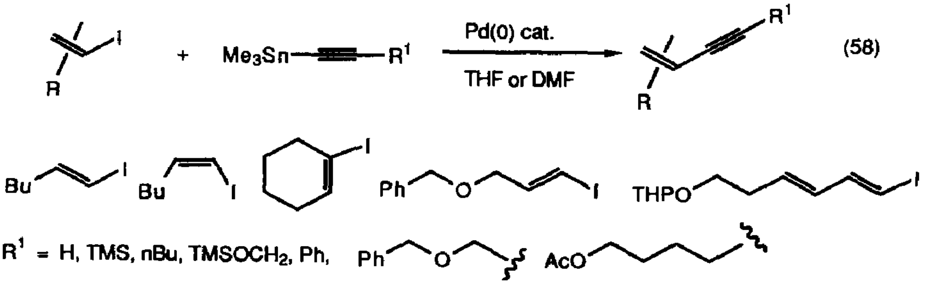
Ar = pMeOPh, pNO$_2$Ph, pCHOPh, pBrPh, pCH$_3$COPh, pAcOPh,



(57)

60-80%

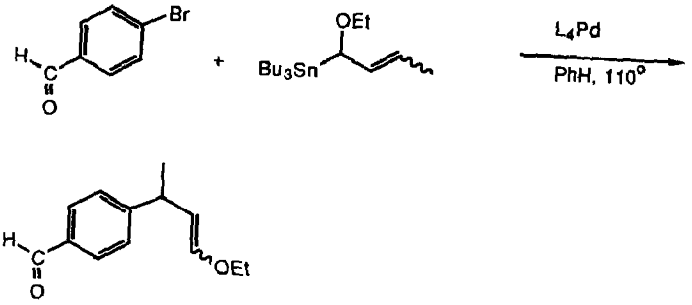
Stereochemistry of both members maintained



(58)

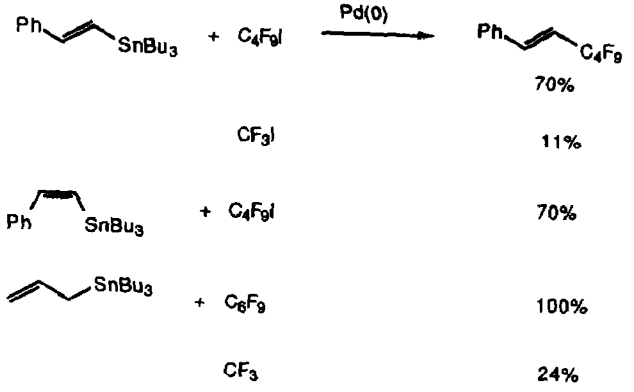
R$^1$ = H, TMS, nBu, TMSOCH$_2$, Ph,

(59)

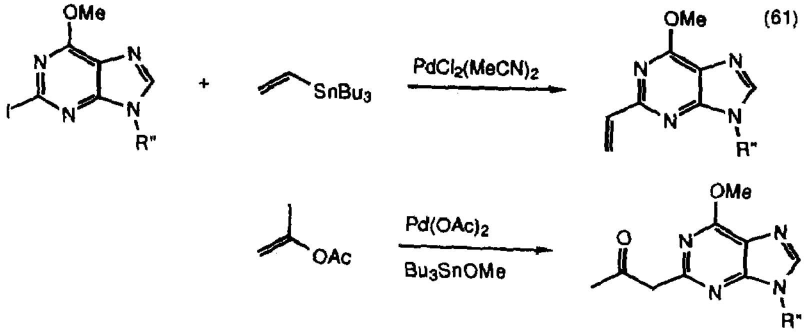


80-95%
Z/E = 25/75

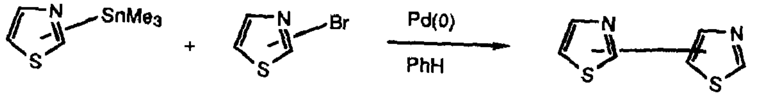
(60)



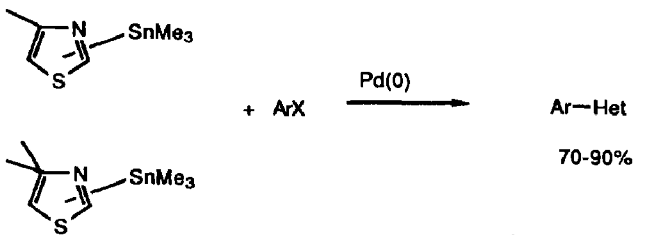
(61)



(62)

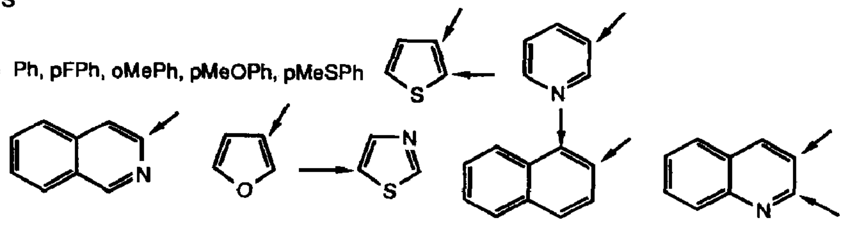


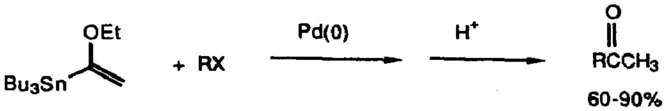
2,2'; 2,4'; 2,5'; 4,4'; 5,5'



(63)

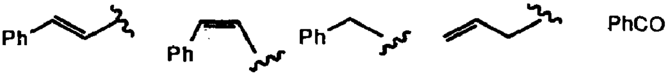
Ar = Ph, pFPh, oMePh, pMeOPh, pMeSPh





(64)

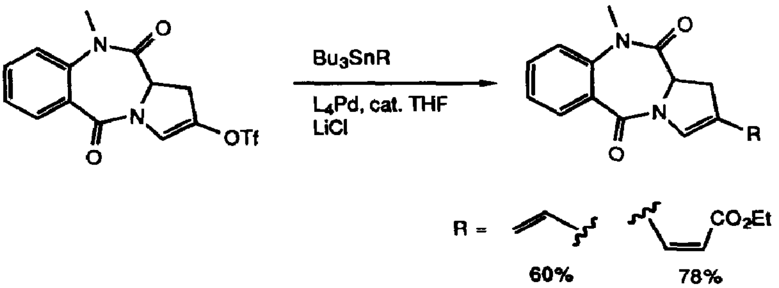
R = Ph, pMePh, mMePh, oMePh, pClPh, pMeOPh, pAcPh, pCNPh, $pNO_2Ph$



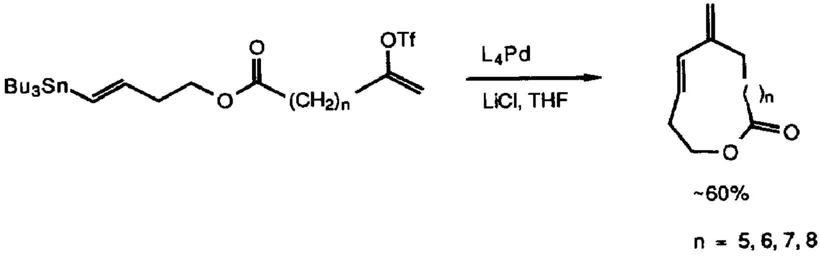
(65)

$$ArOTf + R^1SnR_3 \xrightarrow[LiCl]{Pd(0)} ArR^1 \qquad (66)$$

30 cases



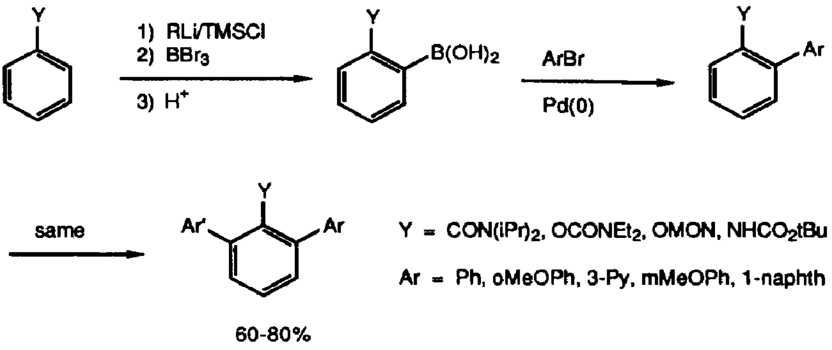
(67)



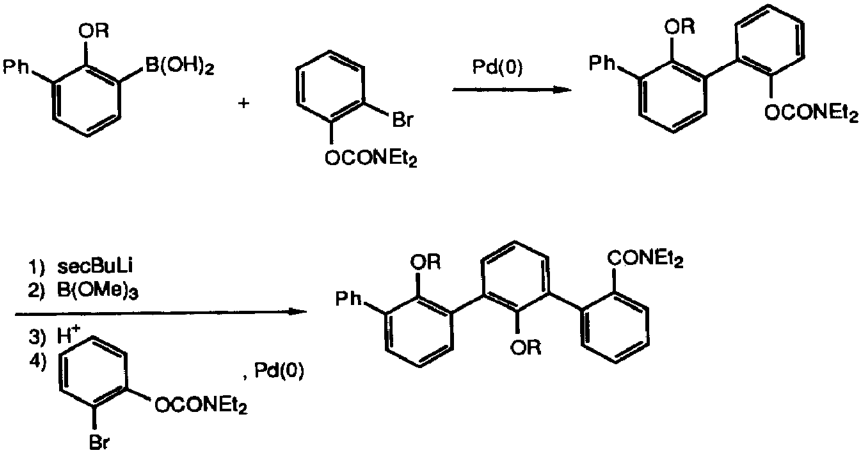
(68)

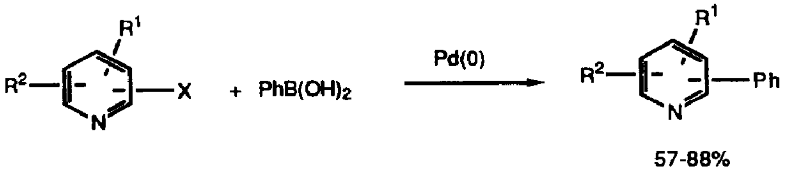
Transmetallation from boron to palladium has also been used extensively in organic synthesis. Thus coupling of aryl halides with aryl borates (equation 69) [69], (equation 70) [70], heteroaromatic halides with aryl borates (equation 71) [71], vinyl halides with aryl borates (equation 72) [72], (equation 73) [73], and vinyl borates with vinyl halides (equation 74) [74], (equation 75) [75], (equation 76) [76], including the most spectacular of all of these coupling reactions (equation 77) [77], have all been quite successful.

(69)



(70)





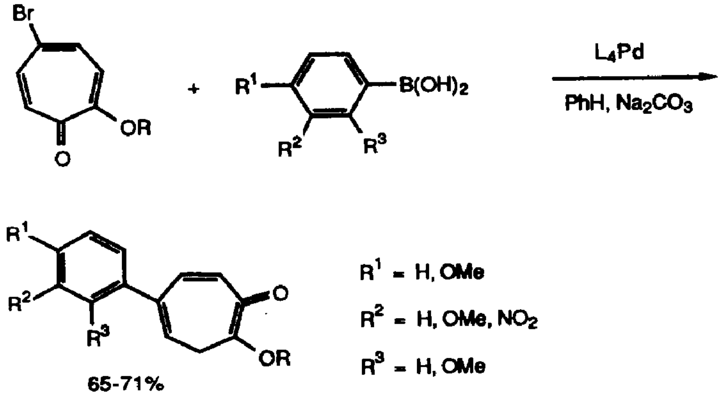
(71)

X = 2-Br, 5-Br

$R^1$ = 3-OH, 2-NH$_2$, 2-NO$_2$

$R^2$ = H, Me

(72)

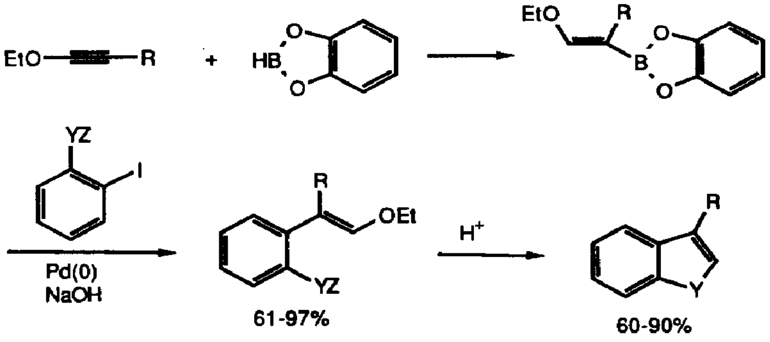


$R^1$ = H, OMe

$R^2$ = H, OMe, NO$_2$

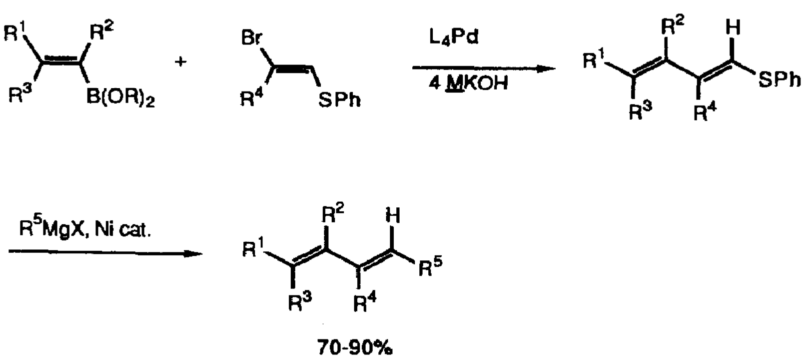
$R^3$ = H, OMe

(73)

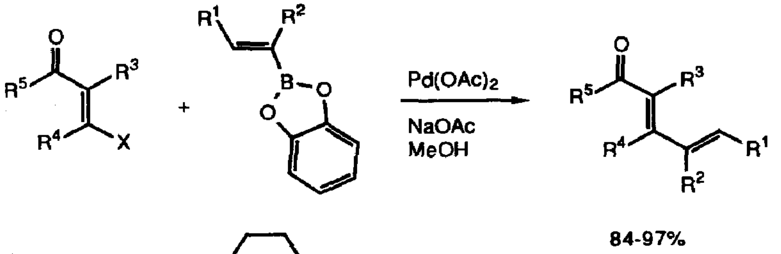


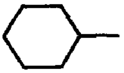
YZ = NH, NHAc, OMe

(74)



(75)



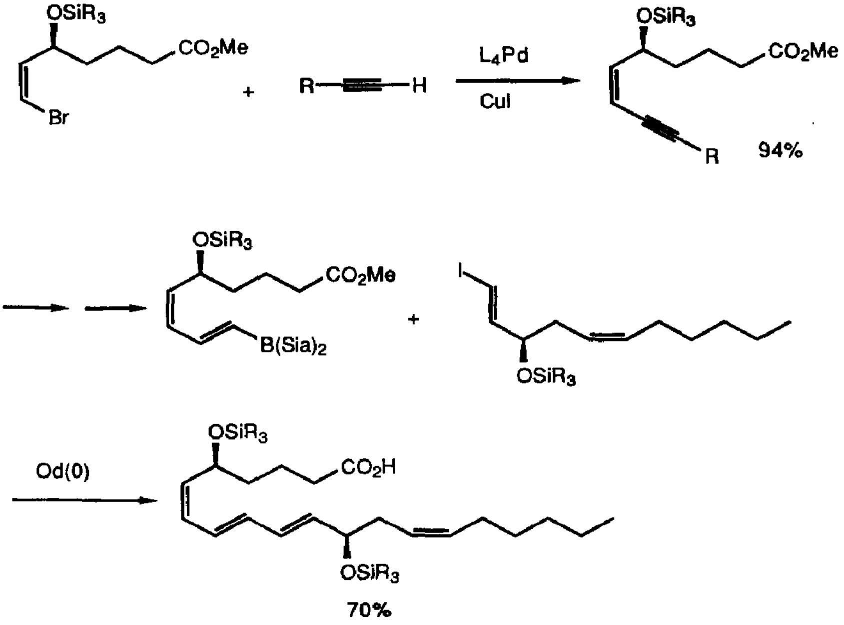
R$^1$ = tBu, Et, Ph, nBu 

R$^2$ = H

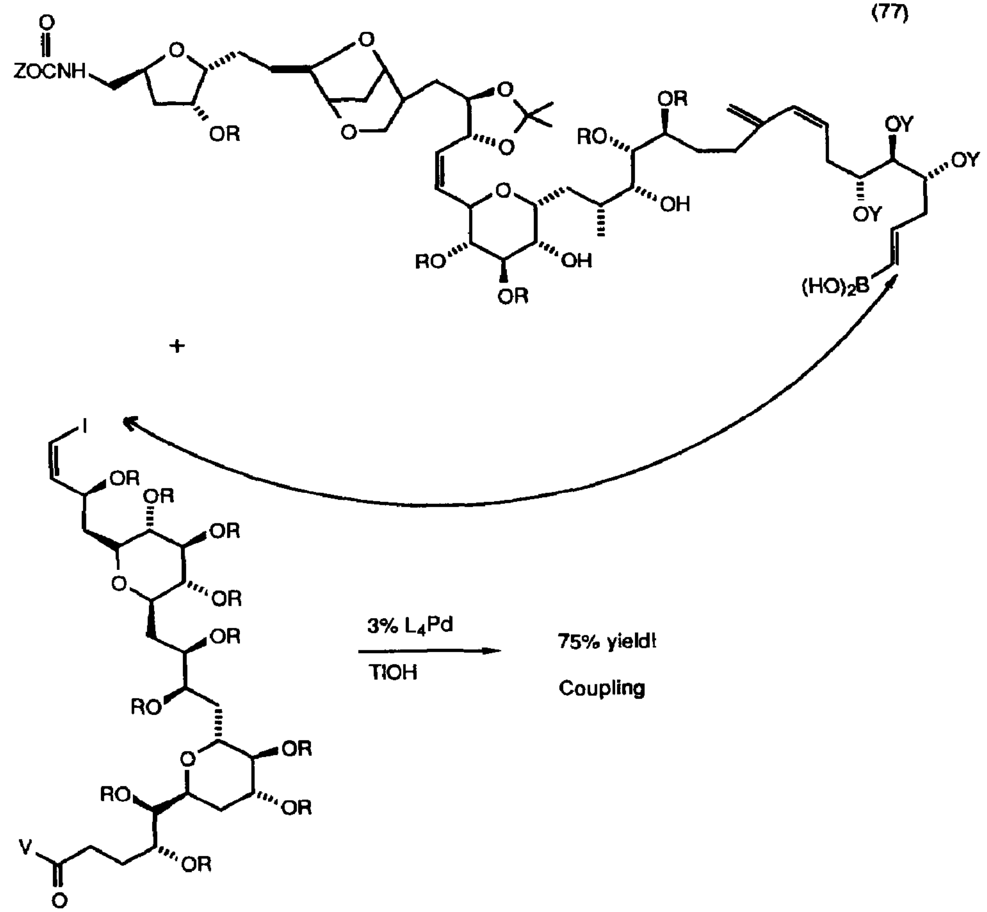
R$^3$ = Me, H

R$^4$ = (CH$_2$)$_3$, (CH$_2$)$_2$

(76)

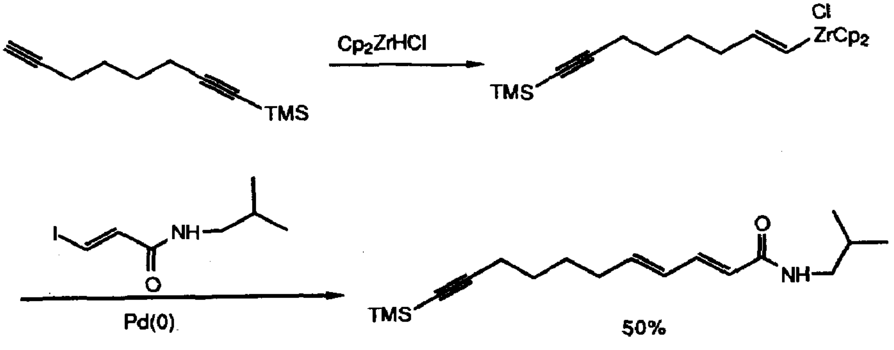


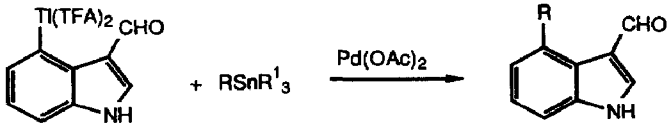
(77)



Vinyl zirconiums and vinyl iodides were coupled by palladium catalysts (equation 78) [78]. Palladium acetate catalyzed the alkylation of 4-indolylthallium compounds by alkyl tins (equation 79) [79]. Palladium catalyzed vinylic substitution of aryl halides on polymeric nitrogen supports was the topic of a dissertation [80].

(78)



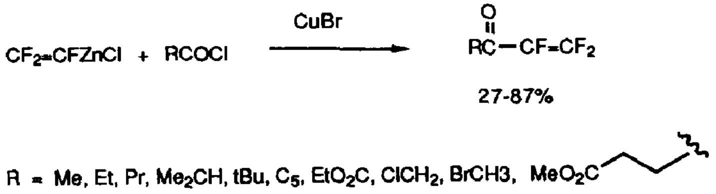


(79)

R = alkyl, Py, Ph

## 2. Alkylation of Acid Derivatives

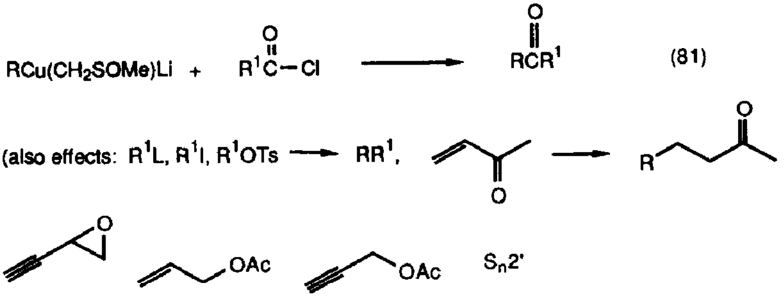
Perfluorozinc chlorides alkylated acid halides in the presence of CuBr (equation 80) [81]. Mixed cuprates with dimethylsulfoxide anion as the nontransferable group alkylated acid chlorides (equation 81) [82]. Thermally stable 2-pyridoxyalkylcuprates alkylated acid halides [83]. Copper(II) salts catalyzed the acylation terminal alkynes (equation 82) [84].



$$CF_2{=}CFZnCl + RCOCl \xrightarrow{CuBr} RC(=O){-}CF{=}CF_2 \quad (80)$$

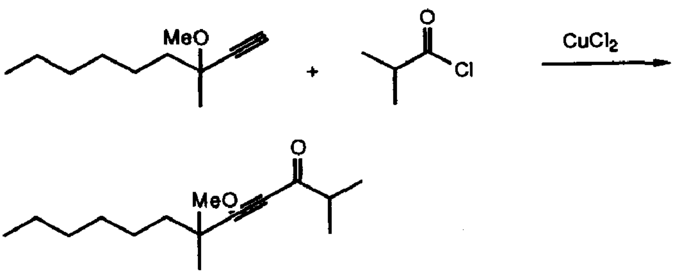
27-87%

R = Me, Et, Pr, $Me_2CH$, tBu, $C_5$, $EtO_2C$, $ClCH_2$, BrCH3, $MeO_2C$

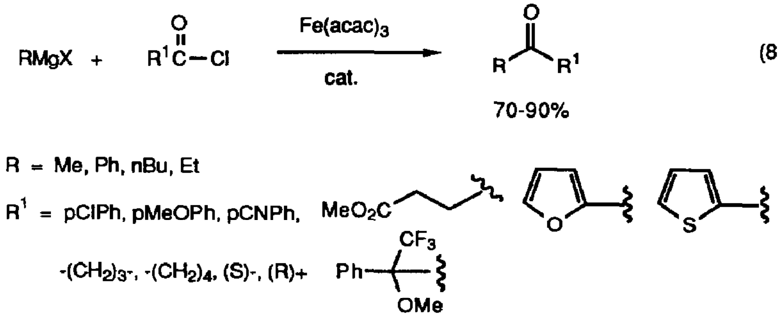


$$RCu(CH_2SOMe)Li + R^1C(=O){-}Cl \longrightarrow RC(=O)R^1 \quad (81)$$

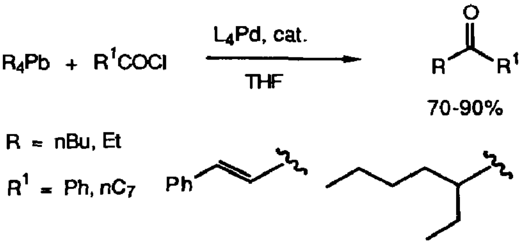
(82)



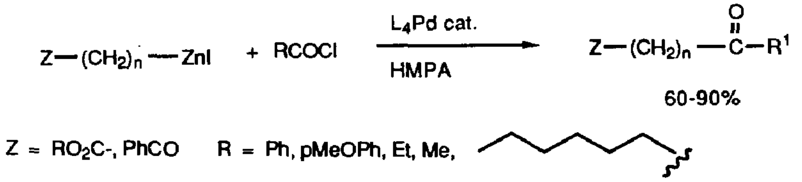
Iron(III) acetylacetonate catalyzed the reaction of Grignard reagents with acid halides to give ketones (equation 83) [85]. Palladium(0) complexes catalyzed the alkylation of acid halides by alkylated reagents (equation 84) [86], alkyl zinc reagents (equation 85) [87], and vinyl tin reagents (equation 86) [88]. Palladium(0) complexes also catalyzed the acylation of olefins by acid chlorides (equation 87) [89], (equation 88) [90], (equation 89) [91]. Palladium(0) and ruthenium(II) complexes catalyzed the acylation of epoxides by acid chlorides (equation 90) [92].



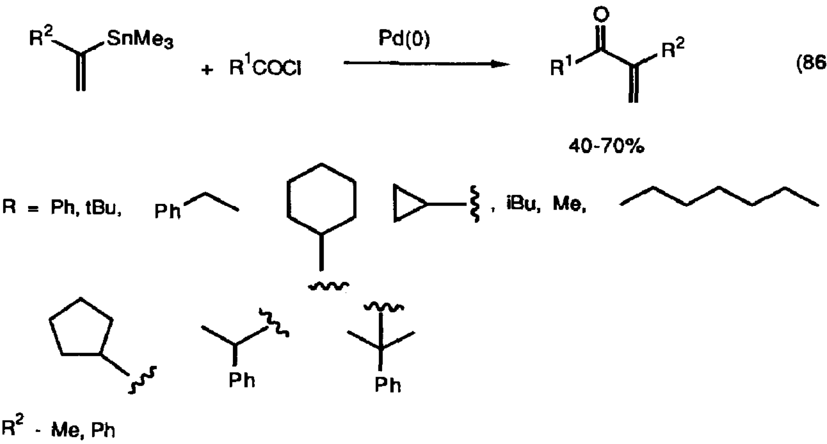
(83)



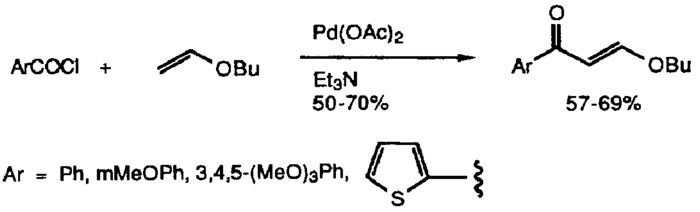
(84)



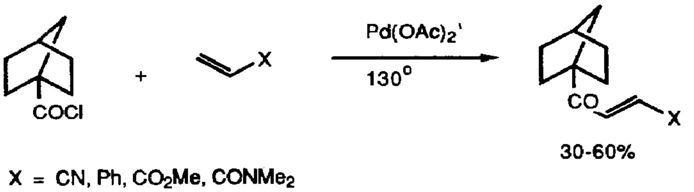
(85)



(86)

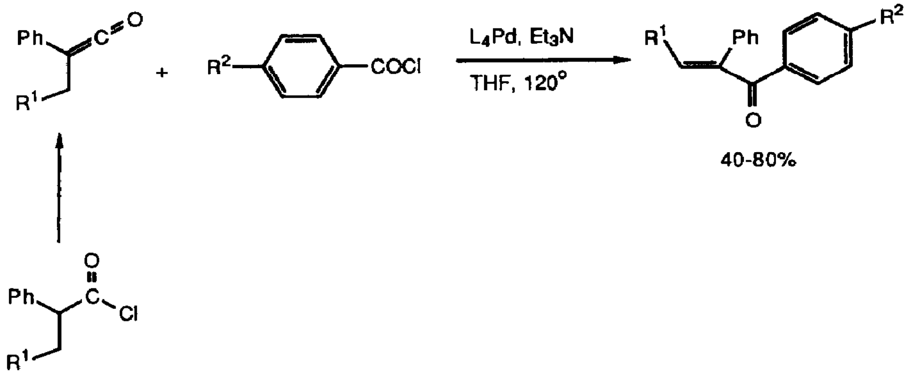


(87)

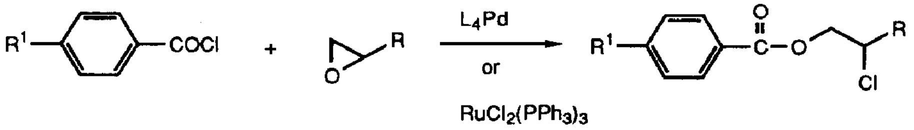


(88)

(89)



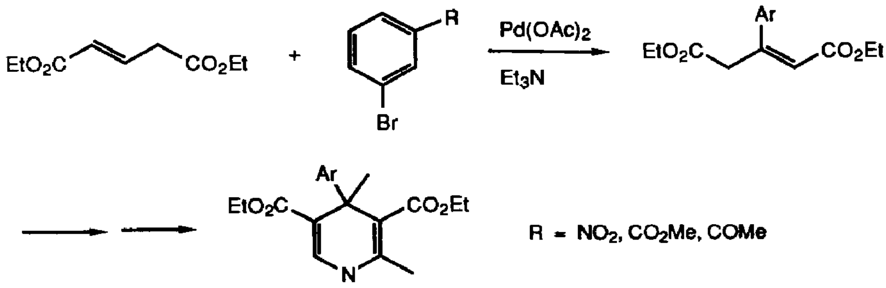
(90)



## 3. Alkylation of Olefins

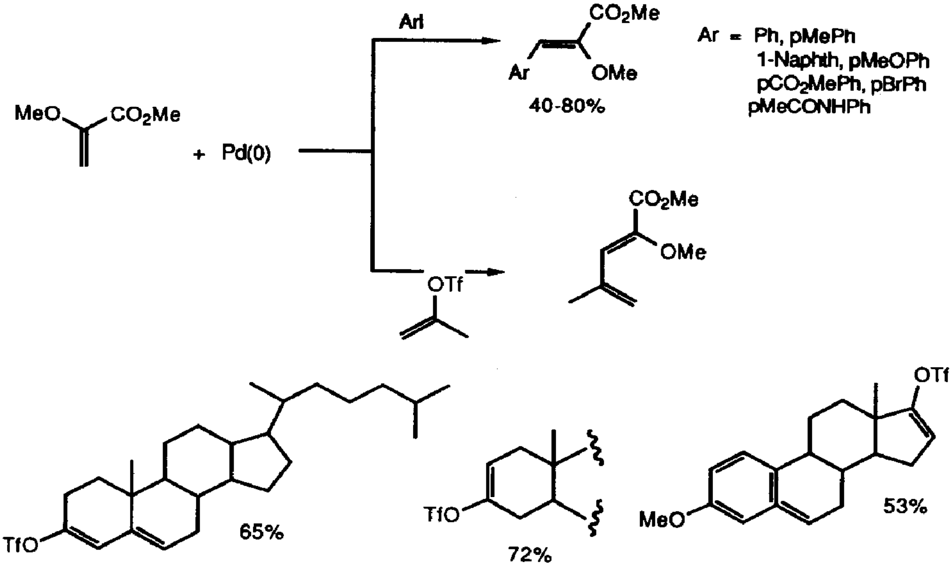
By far the most extensively used procedure for the alkylation of olefins is the Heck reaction-oxidative addition/olefin insertion/b-hydride elimination/reductive elimination processes-catalyzed by palladium(0) complexes. This is the topic of a brief review [93]. Functionalized olefins such as conjugated enones (equation 91) [94], (equation 92) [95], were arylated. The same process was used for the construction of bridged annullated [2,2] paracyclophanes (equation 93) [96], and indoles (equation 94) [97]. Enol ethers bearing ester groups also were efficiently arylated (equation 95) [98]. The regiochemistry of enol ether alkylation was carefully studied (equation 96) [99]. Vinyl silanes also were arylated by arylpalladium complexes (equation 97) [100].

(91)



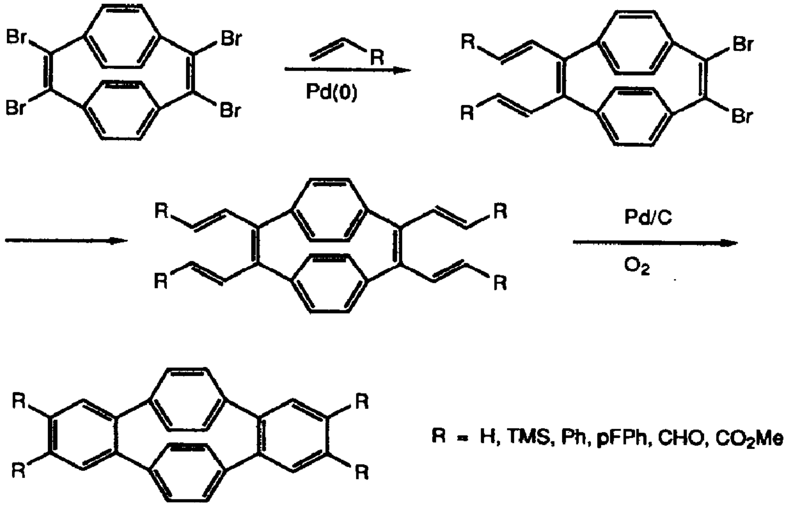
R = $NO_2$, $CO_2Me$, COMe

(92)



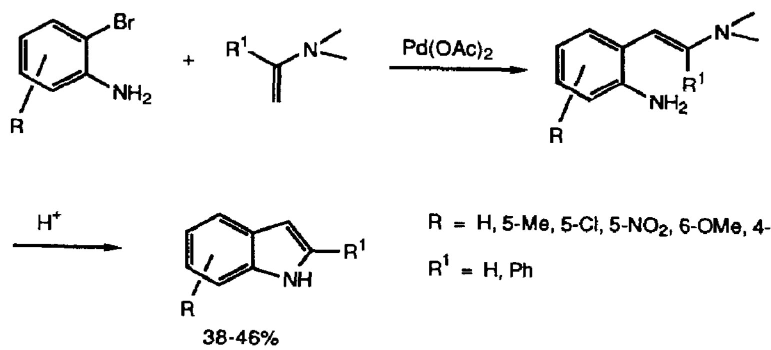
Ar = Ph, pMePh
1-Naphth, pMeOPh
$pCO_2MePh$, pBrPh
pMeCONHPh

(93)



R = H, TMS, Ph, pFPh, CHO, $CO_2Me$

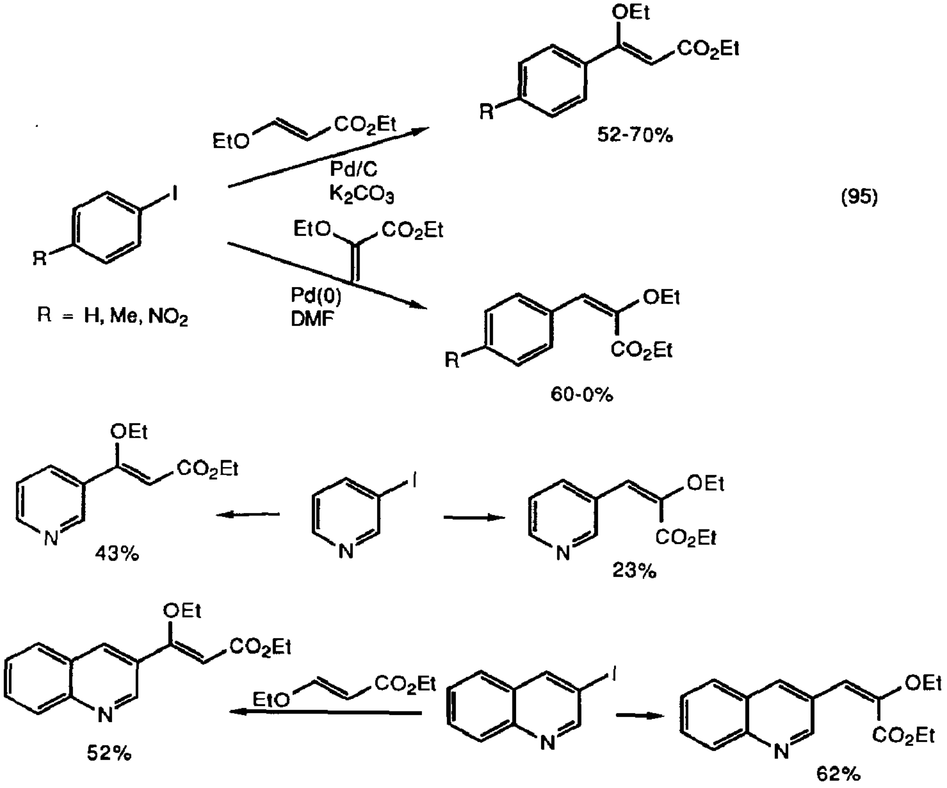
(94)

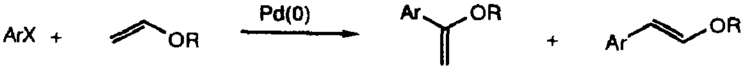


R = H, 5-Me, 5-Cl, 5-NO$_2$, 6-OMe, 4-CO$_2$Me

R$^1$ = H, Ph

(95)





(96)

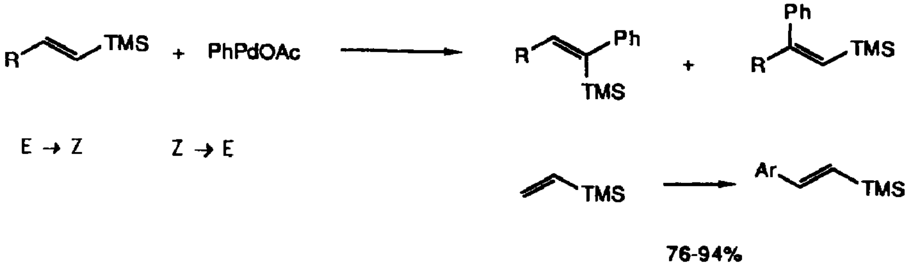
1) R = nBu: α or β -Me's favor α -arylation

2)  - exclusively α

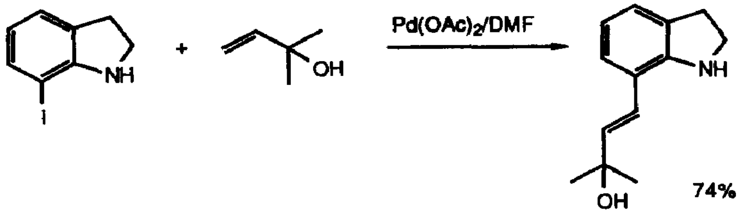
3) pMeOPh ⟶ α ; pNO$_2$Ph ⟶ β

4) Pd(OAc)$_2$ ⟶ β ; L$_4$Pd ⟶ α

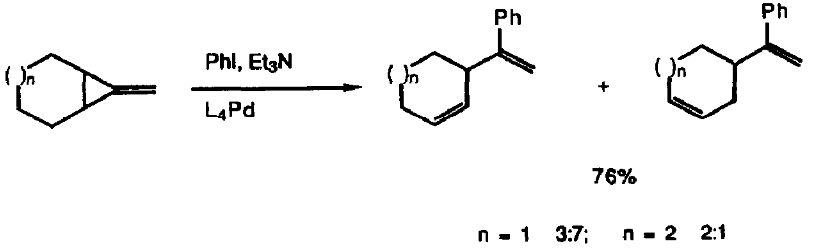
(97)



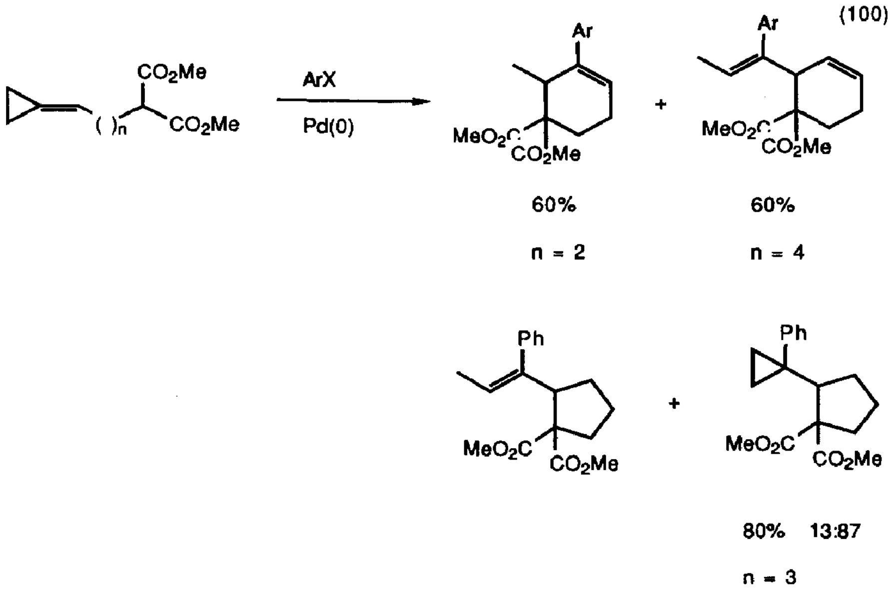
7-Iodoindolines were olefinated cleanly under Heck reaction conditions (equation 98) [101]. Methylene cyclopropanes were arylated with ring opening (equation 99) [102], (equation 100) [103]. Arylation of allylic alcohols produced ketones (equation 101) [104]. 2-Arylbutadienes were prepared by Heck arylation of $SO_2$- diene cycloadducts (equation 102) [105].



(98)



(99)



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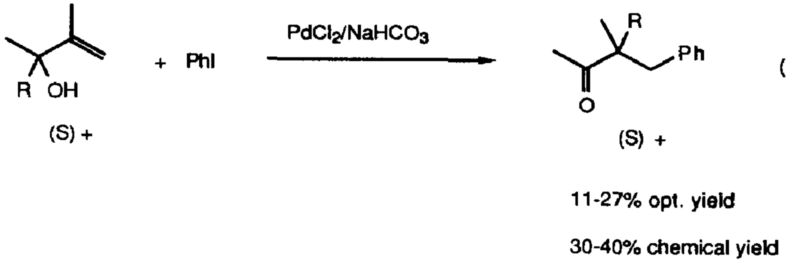
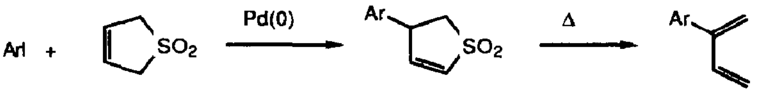


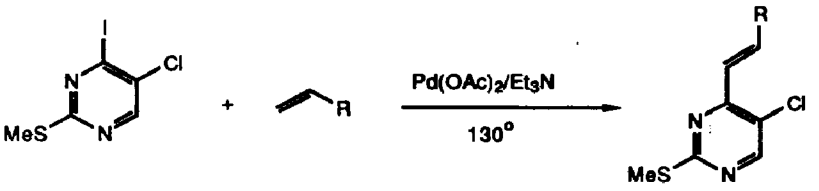
(101)



(102)

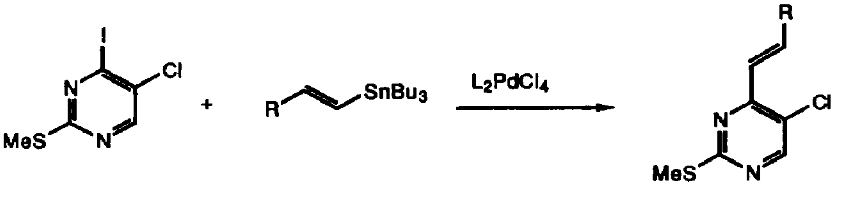
Ar = Ph, 70%; pMePh, 65%; pMeOPh, 66%; pAcOPh, 56%; 2,3,4$(MeO)_3$Ph, 45%
pBrPh, 59%; $pMeO_2CPh$, 52%

Heteroaromatic halides were olefinated under Heck conditions (equation 103) and alkylated by vinyl (equation 104) and alkyltins (equation 105) [106], as were heteroaromatic triflates (equation 106) [107]. Intramolecular versions were used to prepare spirocyclic (equation 107) [108], and heterocyclic compounds (equation 108) [109].



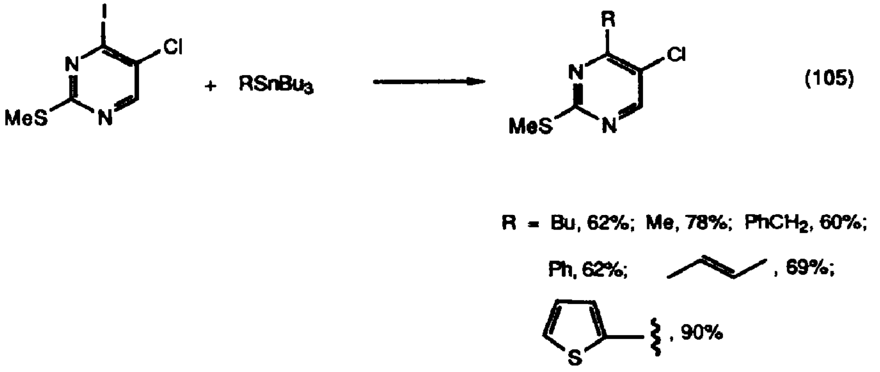
(103)

R = Ph, 80%; CO$_2$Me, 46%; nBu, <10%



(104)

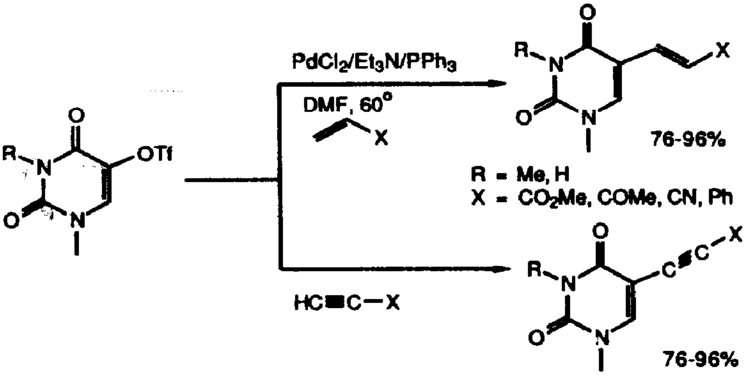
R = Ph, 90%; H, 71%; Me, 80%;
CH$_2$OTHP, 66%



(105)

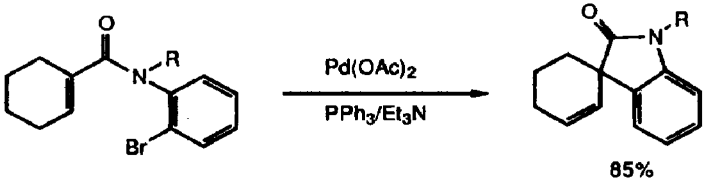
R = Bu, 62%; Me, 78%; PhCH$_2$, 60%;
Ph, 62%; , 69%;
, 90%

(106)



R = Me, H
X = CO$_2$Me, COMe, CN, Ph

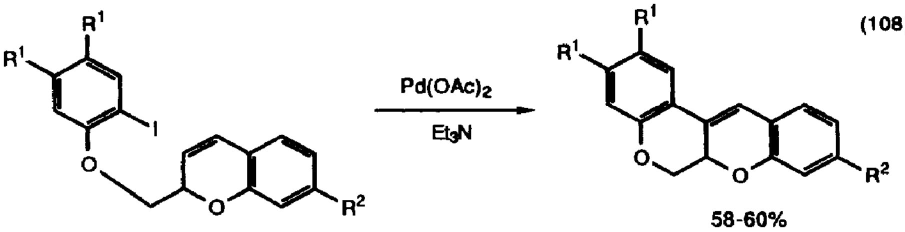
X = CH$_2$OH 72%; Ph, 83%; TMS, 59%



(107)

13 cases studied

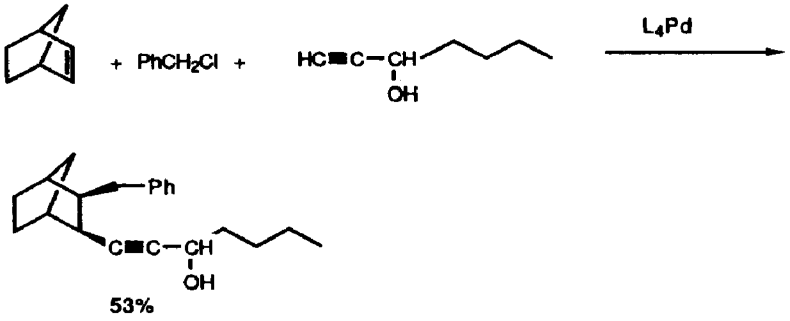
(108)

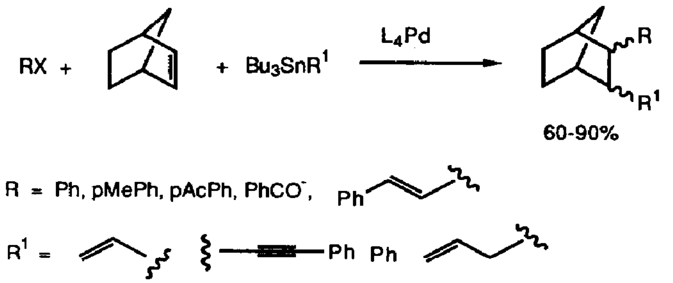


$R^1, R^2 = H, OMe$

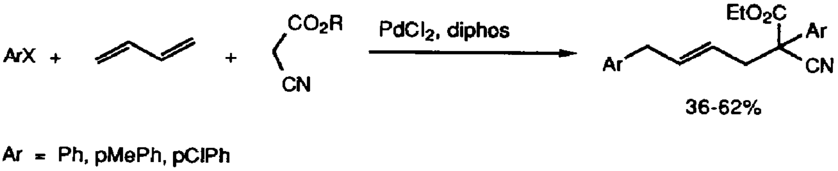
Olefin insertion has been combined with further trapping of the σ-alkyl palladium species to further functionalized norbornenes (equation 109) [110], (equation 110) [111], and butadiene (equation 111) [112], as well as allenes (equation 112) [113]. Silica and polystyrene supported palladium catalysts were used to arylate conjugated enones with iodobenzene [114]. Perfluoroalkyl iodides were added to alkenes using iron as the catalyst (equation 113) [115].

(109)

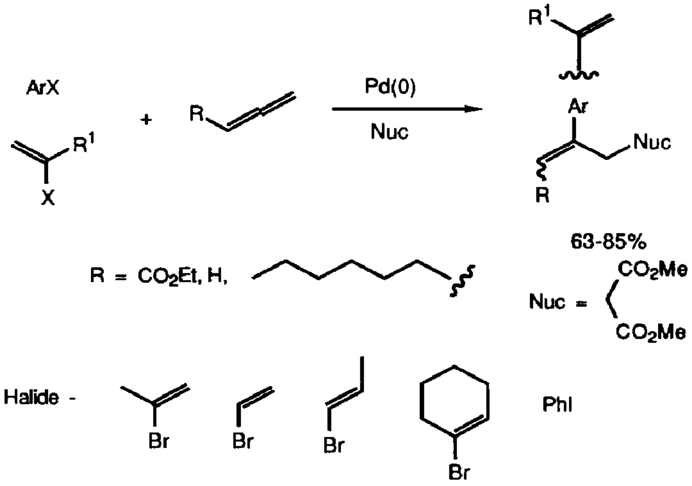




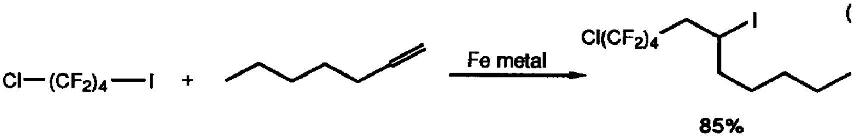
(110)



(111)

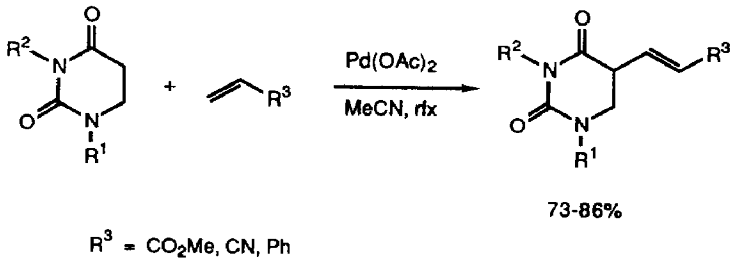


(112)



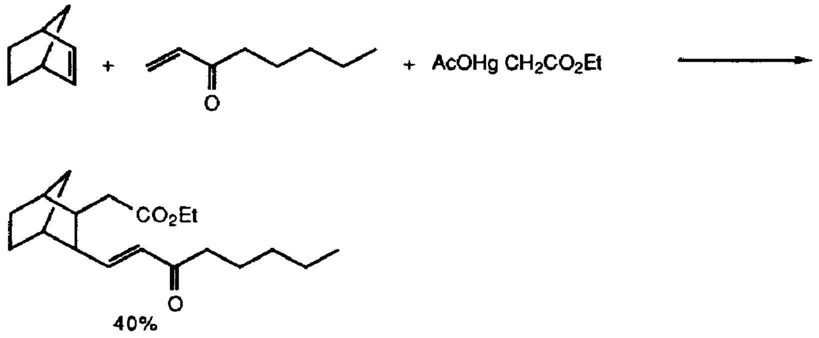
(113)

Direct metallation/insertion (equation 114) [116], and transmetallation-insertion from mercury (equation 115) [117], (equation 116) [118][119], (equation 117) [120], (equation 118) [121], from tin (equation 119) [122], from thallium (equation 120) [123], from zinc (equation 121) [124], and from manganese (equation 122) [125] have also been used to alkylate olefins.

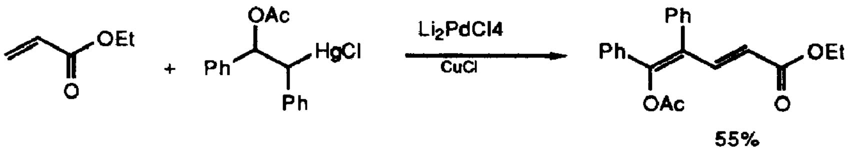


(114)

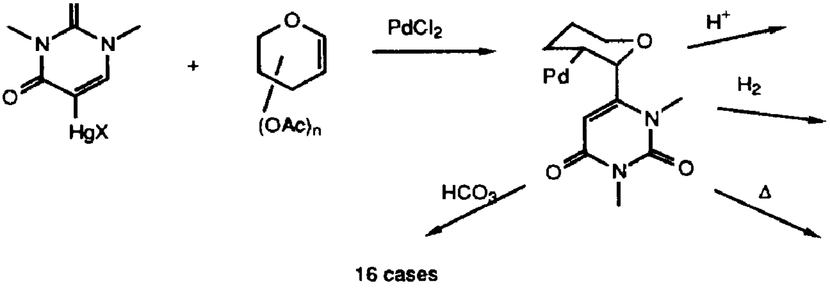
(115)



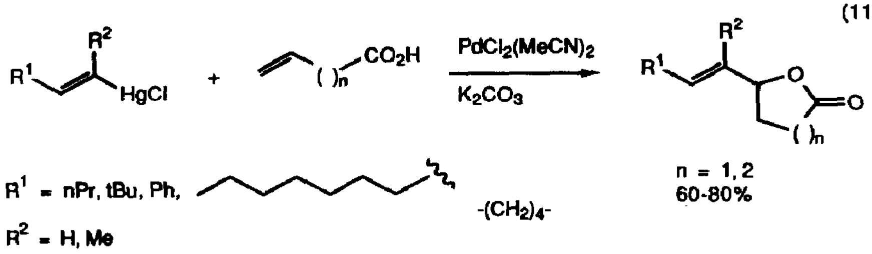
(116)



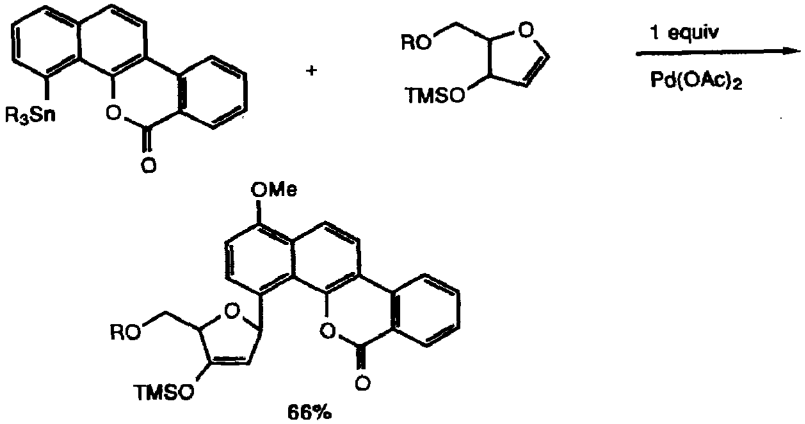
(117)



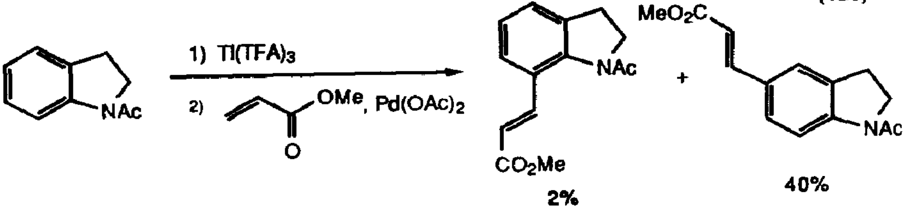
(118)

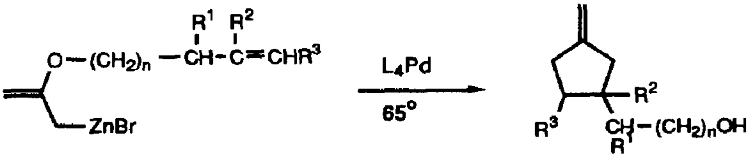


(119)

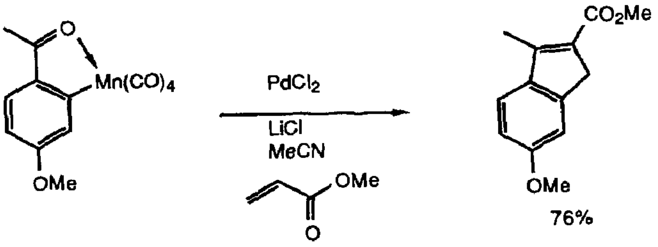


(120)



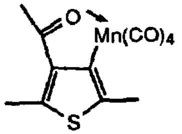


(121)



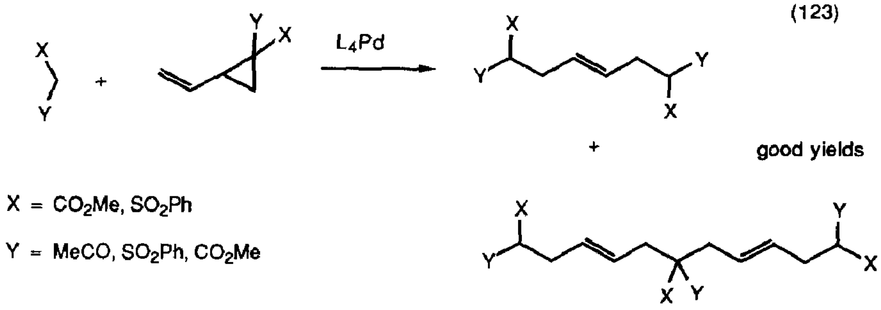
(122)

also

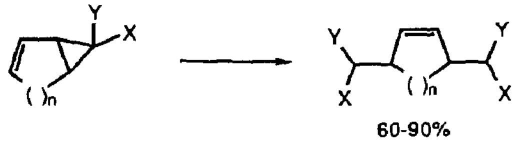


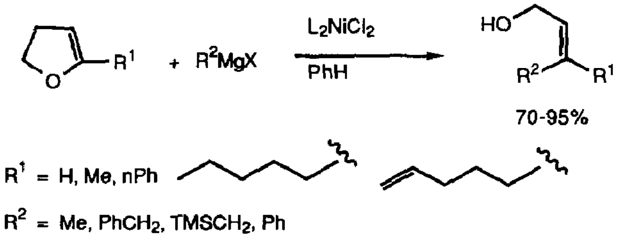
A dissertation dealt with the synthesis of (+)-Prelog-Djerassi lactone methylester via the palladium(0) catalyzed alkylation of homochiral malonates [126]. Vinylcyclopropanes were alkylated by stabilized carbanions under palladium(0) catalysis (equation 123) [127]. Cyclic enol ethers were ring-opened by nickel-catalyzed Grignard reactions (equation 124) [128].

(123)



also

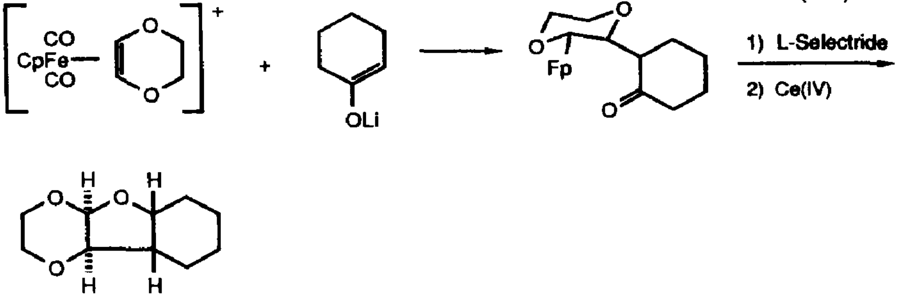


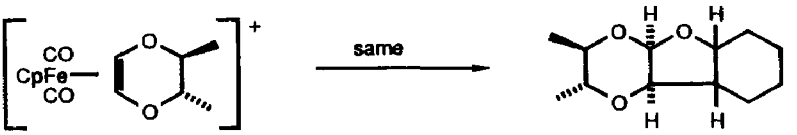


(124)

The reaction of enolates with cationic iron/enol ether complexes has been used to synthesize cyclic polyethers (equations 125 and 126) [129] and to functionalize olefins (equation 127) [130].

(125)

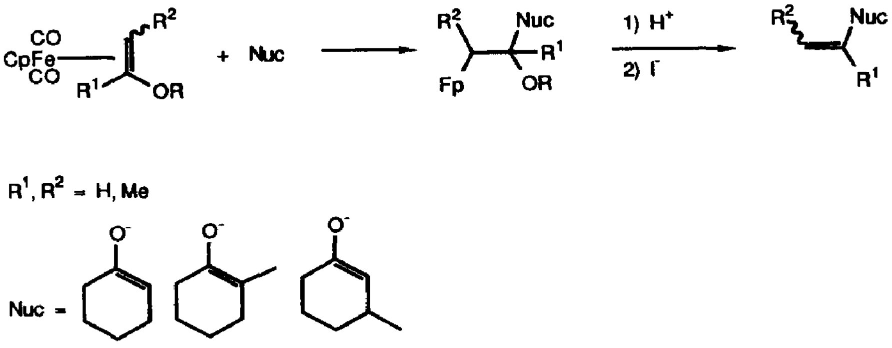




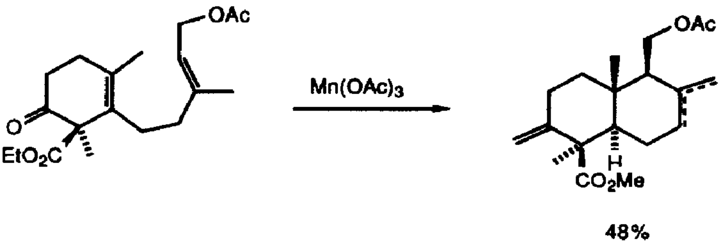
(126)

75% yield 14:1 de

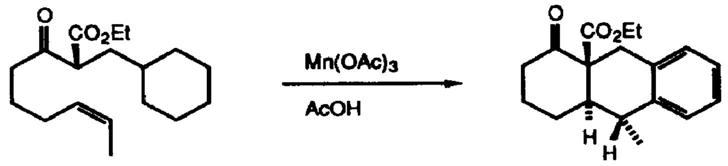
(127)



Manganese(III) acetate has been used to effect a large number of oxidative cyclizations of polyolefins with enolates (equation 128) [131], (equation 129) [132], (equation 130) [133], (equation 131) [134]. Intramolecular versions have also been developed (equation 132) [135], (equation 133) [136]. Cobalt salen complexes were used to promote the radical addition of organic halides to olefins (equation 134) [137].

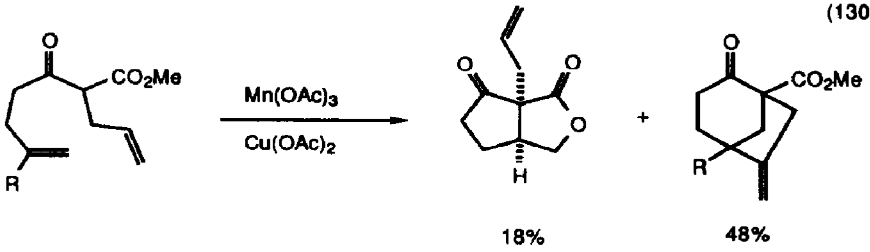


(128)

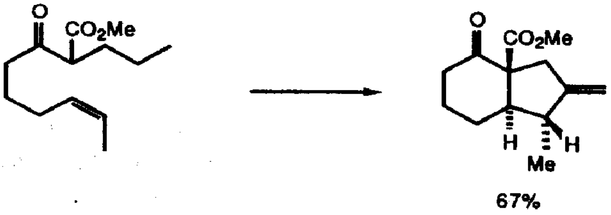


(129)

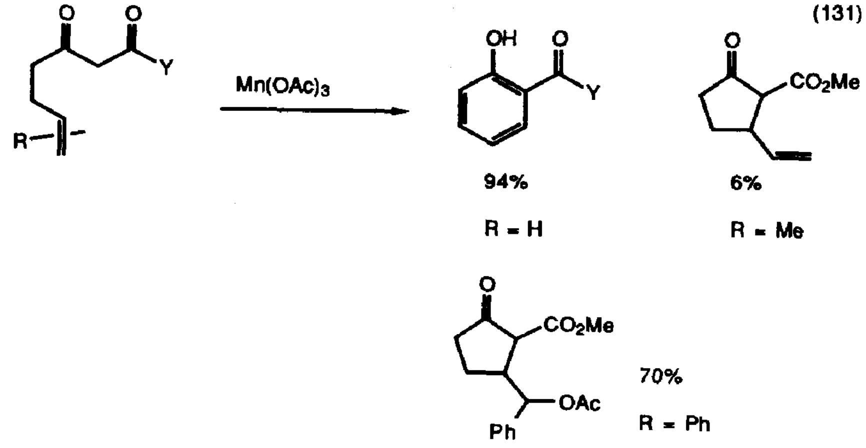
(130)



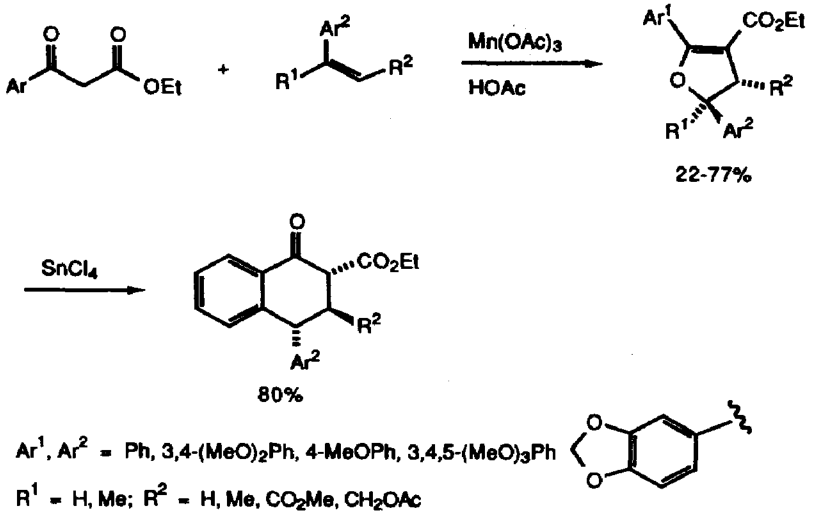
and



(131)

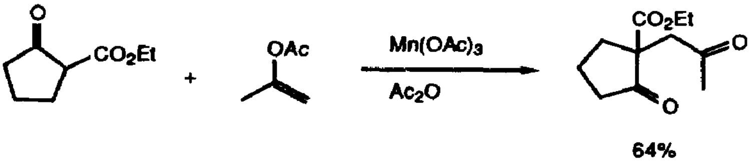


(132)



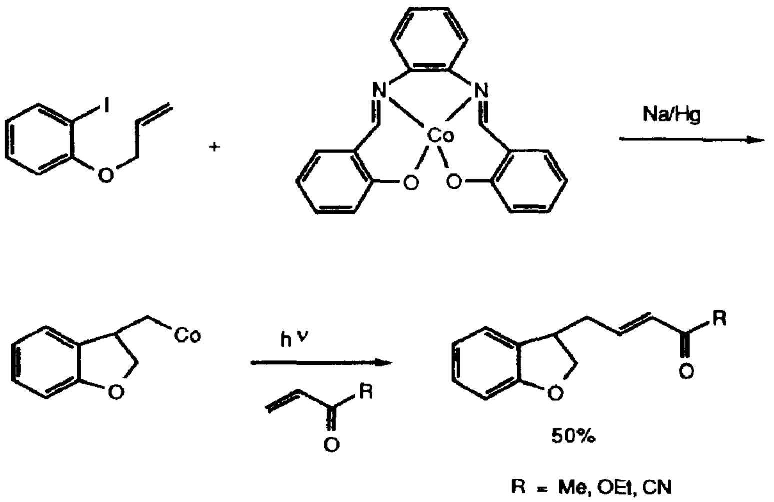
$Ar^1$, $Ar^2$ = Ph, 3,4-$(MeO)_2$Ph, 4-MeOPh, 3,4,5-$(MeO)_3$Ph

$R^1$ = H, Me; $R^2$ = H, Me, $CO_2Me$, $CH_2OAc$



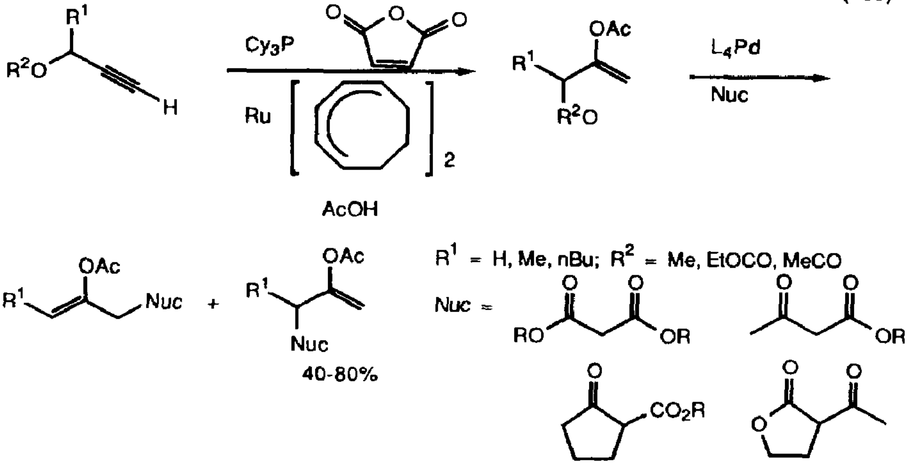
(133)

(134)

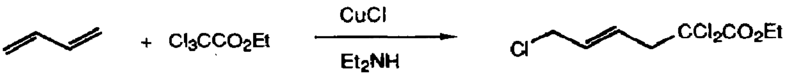


Propargyl ethers were acetoxylated using ruthenium(II) catalysts, then alkylated using palladium(II) catalysts (equation 135) [138]. Copper complexes catalyzed the 1,4-addition of chloroacetic acids to butadiene (equation 136) [139]. The full details on the synthesis, structure and alkylation of chiral vinylrhenium complexes and the mechanism of 1,3-asymmetric induction have been published [140].

(135)

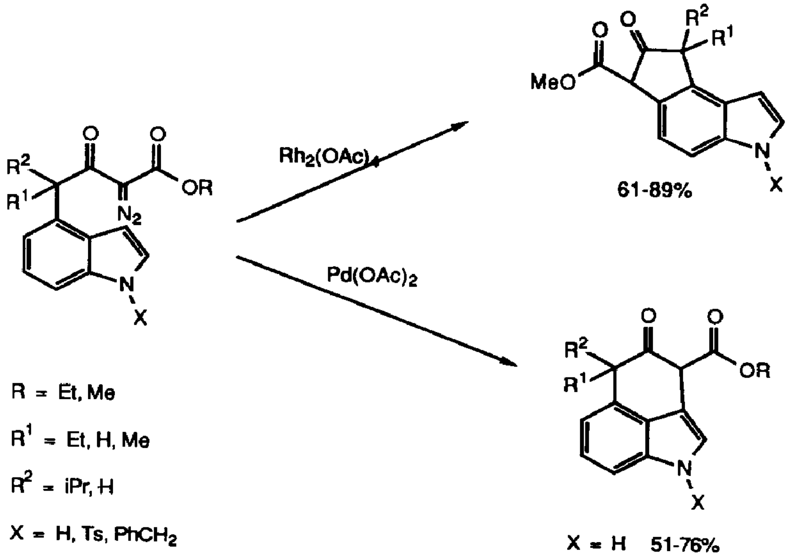


(136)



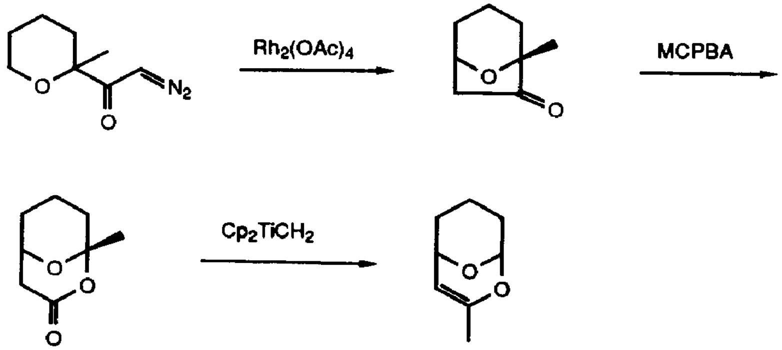
## 4. Decomposition of Diazoalkanes and Other Cyclopropanations.

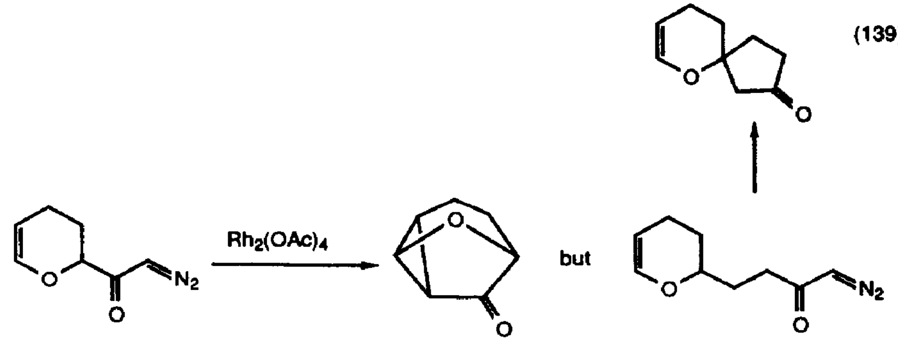
"Transition-metal catalyzed decomposition of aliphatic diazo compounds. New results and applications in organic synthesis" was the title of a review having 417 references [141]. Rhodium(II) acetate was used to catalyze a large number of C-H insertion processes producing various ring systems (equation 137) [142], (equation 138) [143], (equation 139) [144], (equation 140) [145], (equation 141) [146], (equation 142) [147], (equation 143) [148], (equation 144) [149], (equation 145) [150], (equation 146) [151]. With furans, α,β,γ,δ-unsaturated aldehydes were produced (equation 147) [152].



(137)

(138)

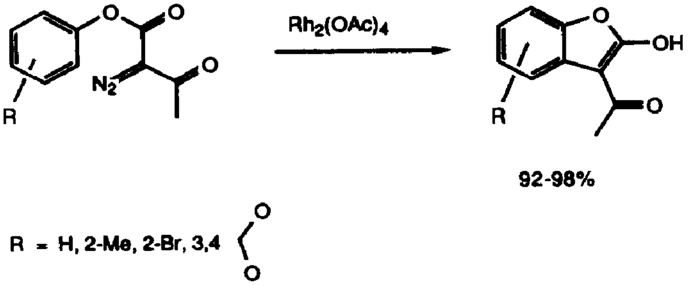




(139)

58%

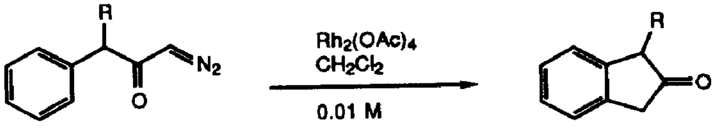
8 cases of various chain lengths to give various cyclic structures



(140)

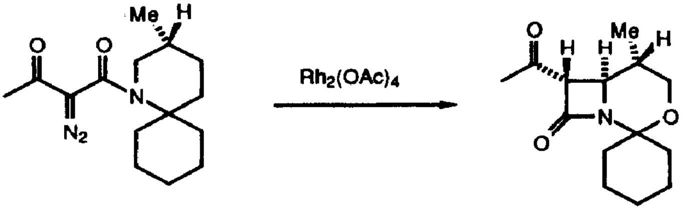
92-98%

R = H, 2-Me, 2-Br, 3,4



(141)

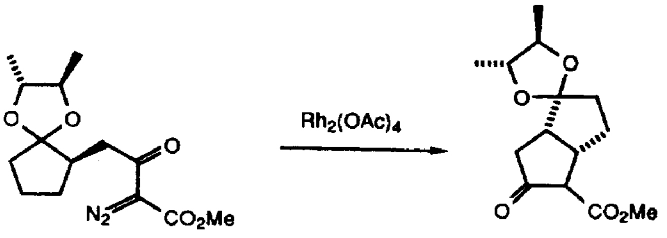
26-65%



(142)

63%

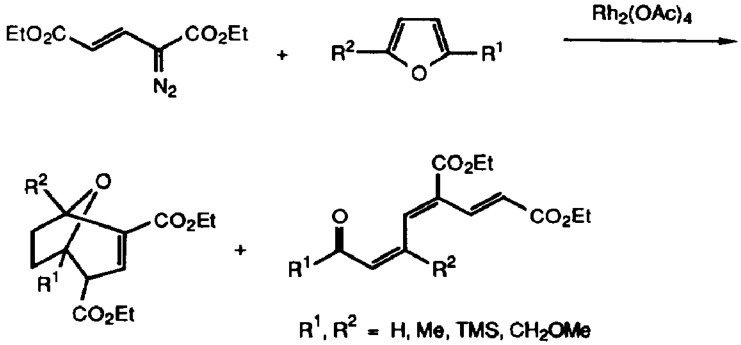
optically active but no ee



65%

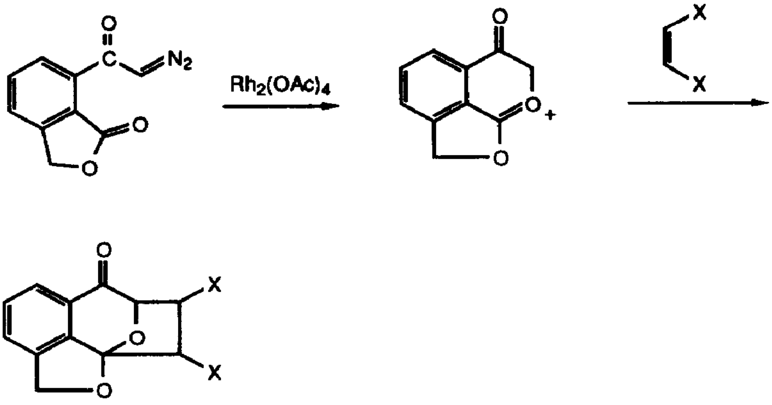
(143)

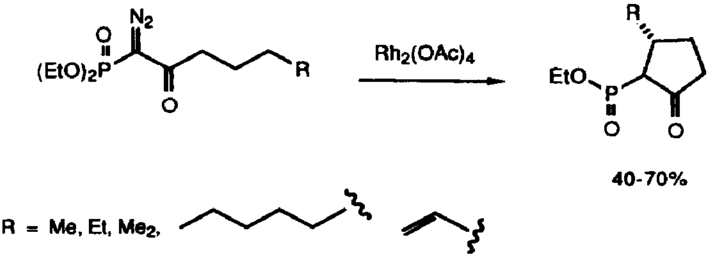
(144)



R$^1$, R$^2$ = H, Me, TMS, CH$_2$OMe

(145)

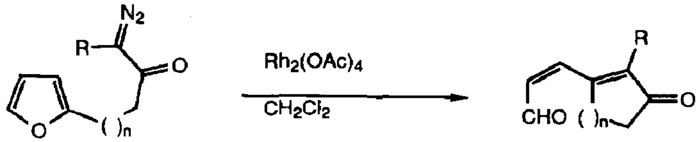




40-70%

R = Me, Et, Me$_2$,

(146)



(147)

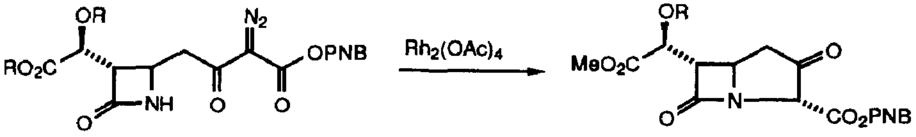
n = 1, 2

75-95%

R = H, Me, CO$_2$Et

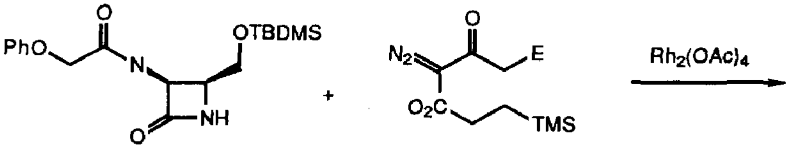
Rhodium acetate also catalyzed N-H (equation 148) [153], (equation 149) [154], O-H (equation 150) [155] and S-insertion reactions (equation 151) [156], (equation 152) [157], (equation 153) [158].

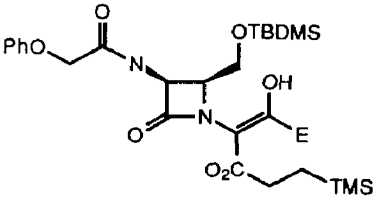
(148)



57%

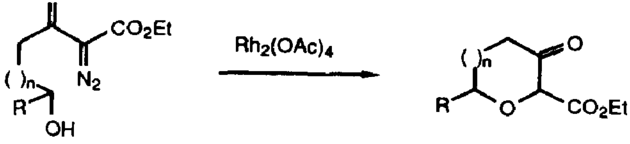
(149)





40-50%

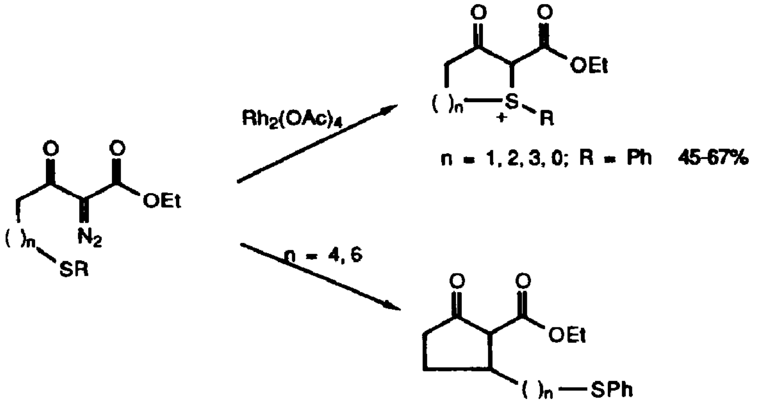
E = H, PhCH$_2$, CH(OAc)CH$_3$, CH(OTBDMS)Ph
=CHCH$_3$, Br, 3-py fail



(150)

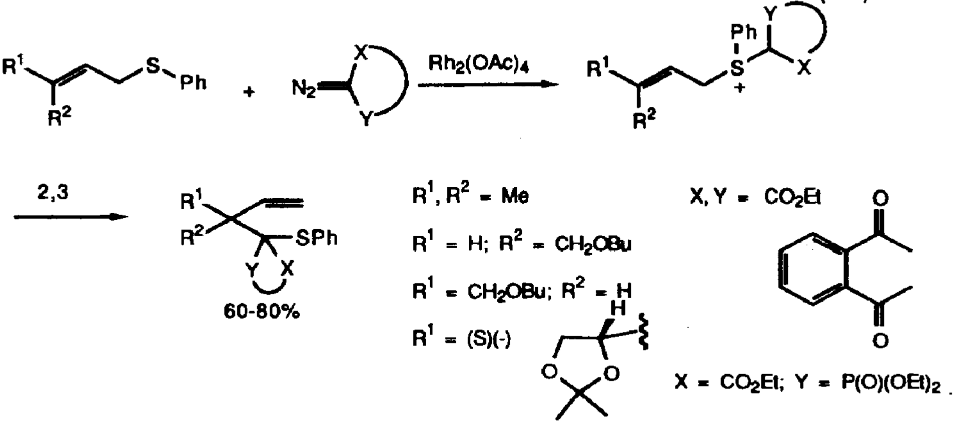
n = 2; 60%

n = 1; 80%

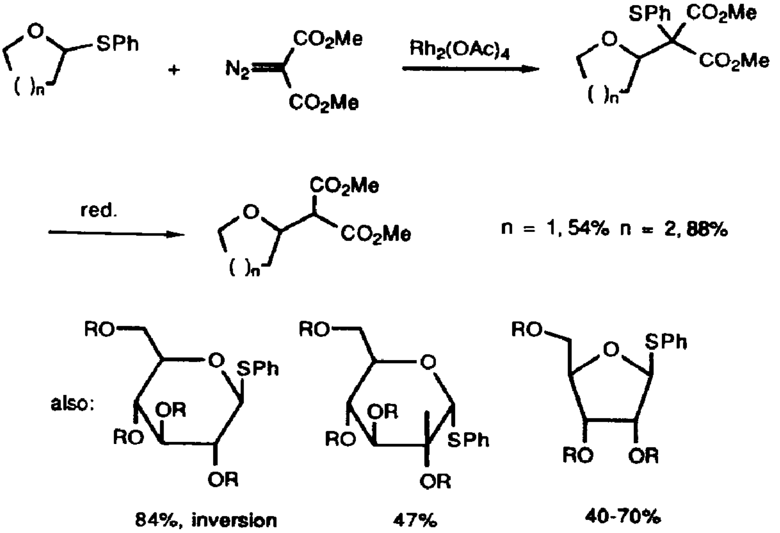

Rh$_2$(OAc)$_4$
OEt
N$_2$
( )$_n$
SR
O
( )$_n$
S
+
R
n = 1, 2, 3, 0; R = Ph 45-67%
n = 4, 6
( )$_n$ —SPh

(151)

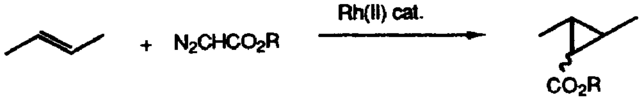
(152)


R$^1$
R$^2$
S
Ph
+
N$_2$
X
Y
Rh$_2$(OAc)$_4$
Ph
S
+
2,3
SPh
60-80%
R$^1$, R$^2$ = Me
R$^1$ = H; R$^2$ = CH$_2$OBu
R$^1$ = CH$_2$OBu; R$^2$ = H
R$^1$ = (S)(-)
H
O
X, Y = CO$_2$Et
X = CO$_2$Et; Y = P(O)(OEt)$_2$ .

(153)

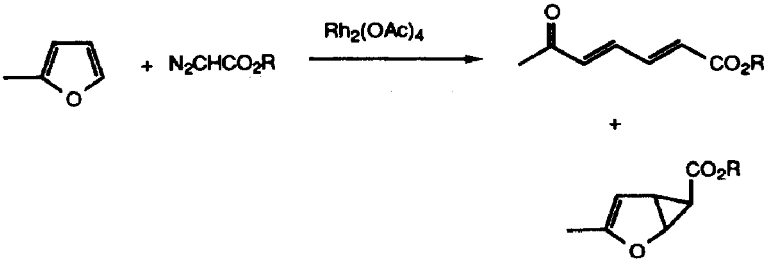

O
SPh
( )$_n$
+
N$_2$
CO$_2$Me
Rh$_2$(OAc)$_4$
red.
n = 1, 54% n = 2, 88%
also:
RO
OR
84%, inversion
47%
40-70%

Rhodium(II) acetate also catalyzed the efficient cyclopropanation of olefins by diazo compounds. The stereoselectivity of this cyclopropanation depended on the carboxylate (equation 154) [159]. 2-Methyl furan (equation 155) [160], cyclopropene carboxylates (equation 156) [161], cyclopentadiene (equation 157) [162] and cyclooctatetraene (equation 158) [163] were all cyclopropanated efficiently. Copper(I) chloride catalyzed the cyclopropanation of cyclooctene by diazomalonates (equation 159) [164]. Copper semicorrin catalysts also worked (equation 160) [165]. 2-Phenylcyclopropane carboxylic acids were synthesized with high optical purity by chiral copper(II) complex catalyzed cyclopropanation of styrene [166]. Vinyl and allylic halides were cyclopropanated by diazomethane in the presence of copper(I) chloride [167]. Palladium(II) acetate catalyzed the cyclopropanation of tricyclo[3.2.0.0$^{2,7}$]hept-3-ene by diazomethane (equation 161) [168].



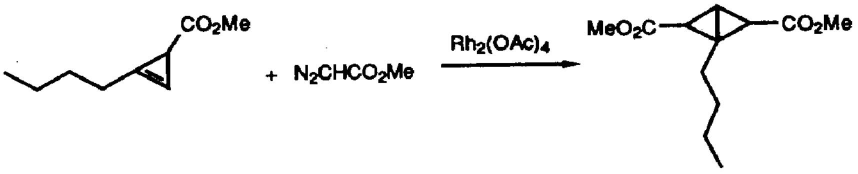
(154)

$Rh_2(NHCOCH_3)_4$ gave best trans/cis 10:1

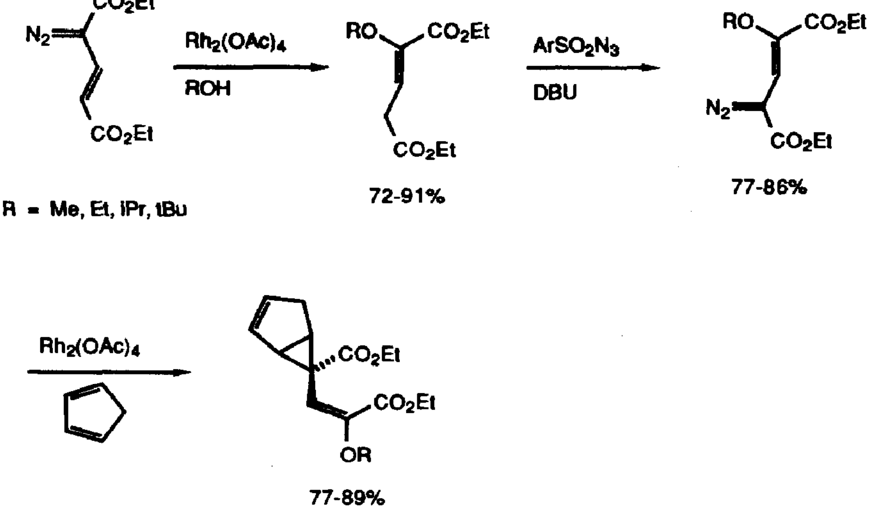


(155)

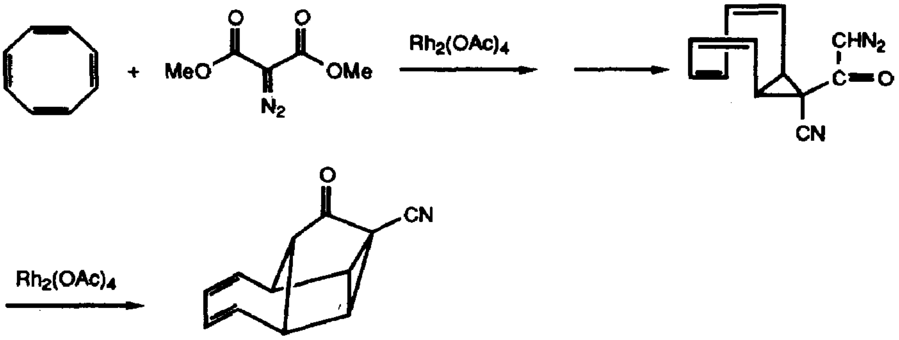
(156)



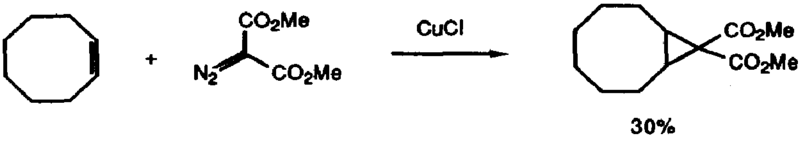
(157)

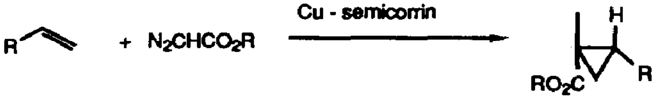


(158)

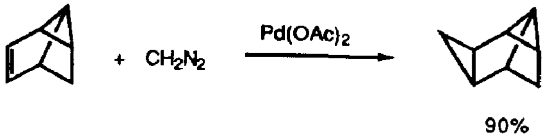


(159)





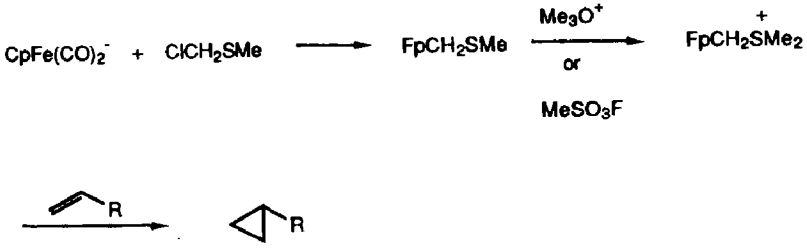
(160)



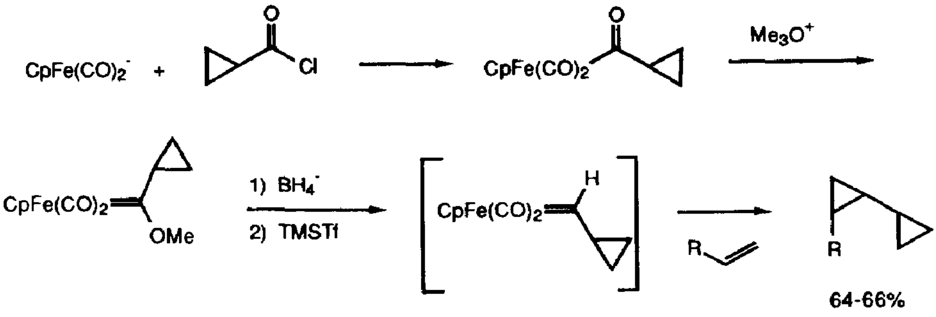
(161)

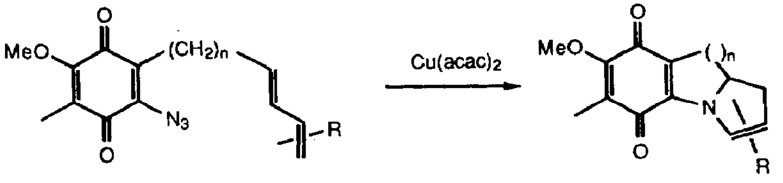
"Cyclopropanes from reactions of transition metal carbene complexes with olefins" was the topic of a review having 59 references [169]. The full details of the cyclopropanation of olefins by iron carbene complexes have been published (equation 162) [170]. Cyclopropyliron carbene complexes also cyclopropanated olefins (equation 163) [171]. Pyrroloindoloquinones were prepared by the copper(II) catalyzed decomposition of the appropriate azido quinone (equation 164) [172,173].

(162)



(163)





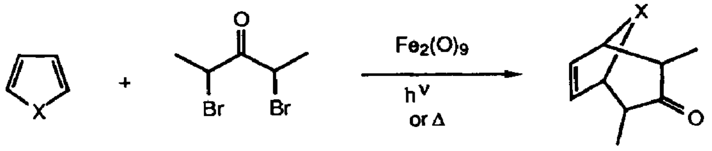
(164)

n = 1, 2

20-50%

## 5. Cycloaddition Reactions

Transition metals promote a number of different types of cycloaddition processes. The use of oxallyl complexes in organic synthesis has been reviwed (180 references) [174]. Organoiron-mediated syntheses of cyclohexanones via oxallyl cation chemistry and iron-promoted cycloaddition reactions of α,α'-dihaloketones to N-tosylenamines were topics of a dissertation [175]. Iron nonacarbonyl promoted the reaction of α,α'-dibromoketones with selenophane and telluorphane (equation 165) [176].

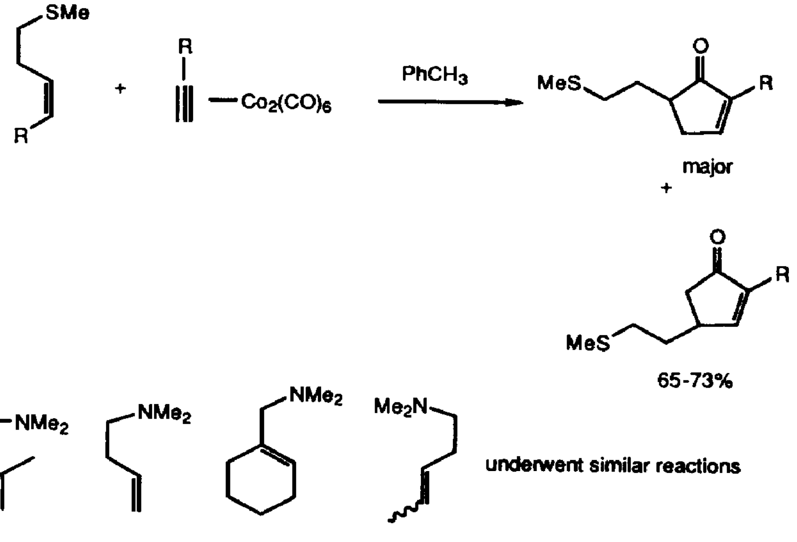


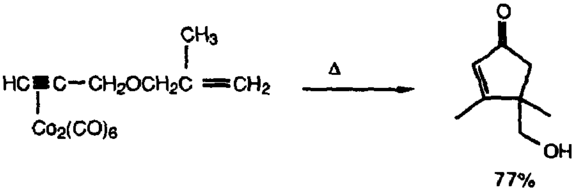
(165)

X = Te, Se

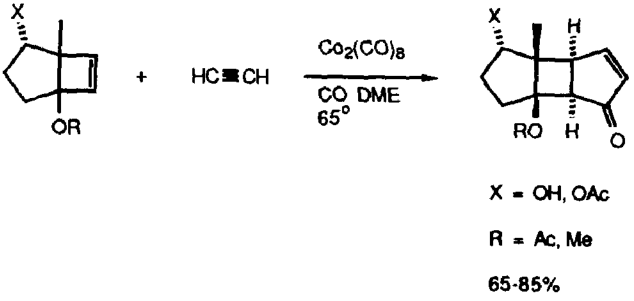
The Pauson-Khand reaction of alkynes, alkenes, and carbon monoxide with $Co_2(CO)_8$ to form cyclopentenones is finding increased use in organic synthesis. Using homoallylic sulfides and amines as the olefinic partner led to a large degree of control of regiochemistry (equation 166) [177]. $Co_2(CO)_6$ complexes of allyl propargyl ethers cyclized to cyclopentenones when heated (equation 167) [178]. Tricyclic compounds were made by intermolecular (equation 168) [179] and intramolecular versions (equation 169) [180], (equation 170) [181]. Allenic esters cyclodimerized when heated with dicobalt octacarbonyl (equation 171) [182].

(166)

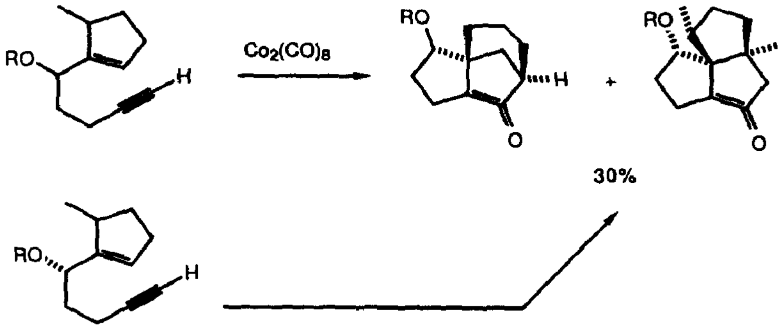




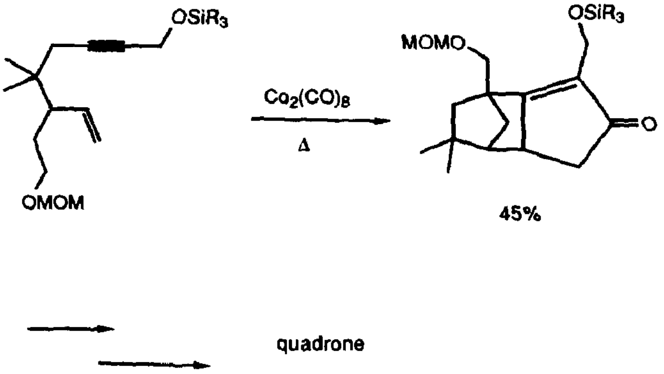
(167)



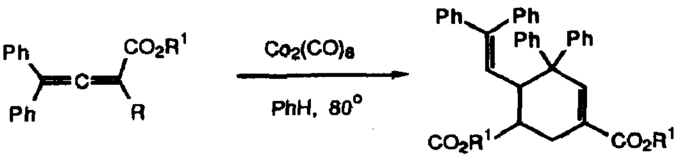
(168)



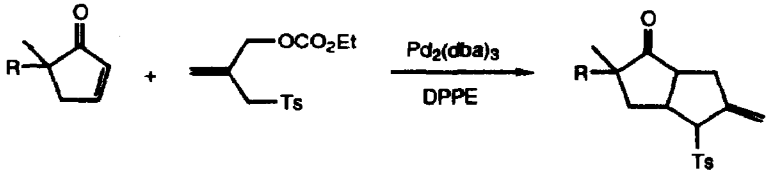
(169)



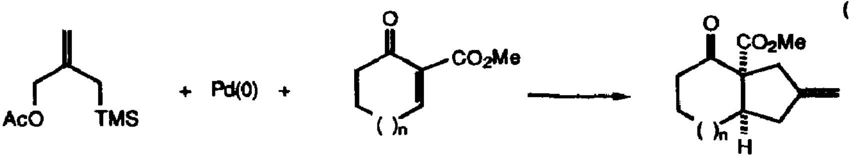
(170)


(171)

Palladium-catalyzed generation of trimethylenemethane derivatives in the presence of electron deficient olefins provided an efficient route to cyclopentanones (equation 172) [183], (equation 173) [184]. With tropanone as the acceptor, bridged bicyclic compounds formed (equation 174) [185], and in some instances (equation 175) [186], (equation 176) [187] addition to the carbonyl group was observed. Palladium(0) complexes also catalyzed the cycloaddition of methylenecyclopropanes to olefins (equation 177) [188].

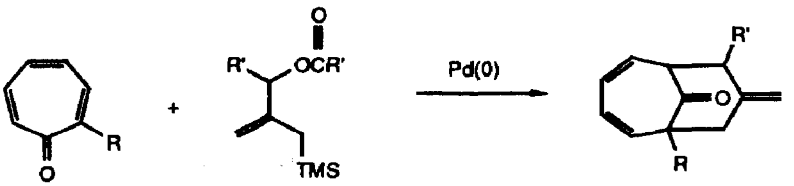

(172)

(Improved synthesis of starting material.)


(173)

n = 1, 46%

n = 2, 91%

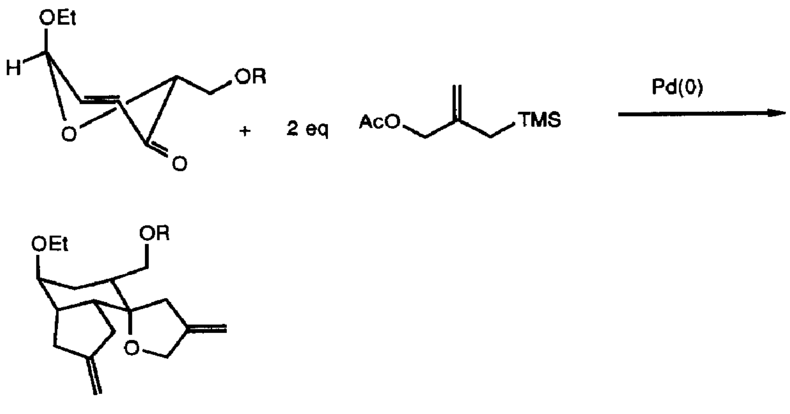

(174)

40-80%

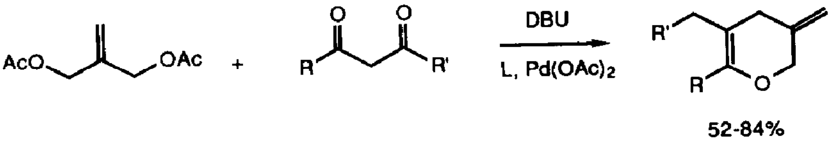
R = H, Me, OMe

R' = H, Me, $CO_2H$, $CO_2Et$

(175)



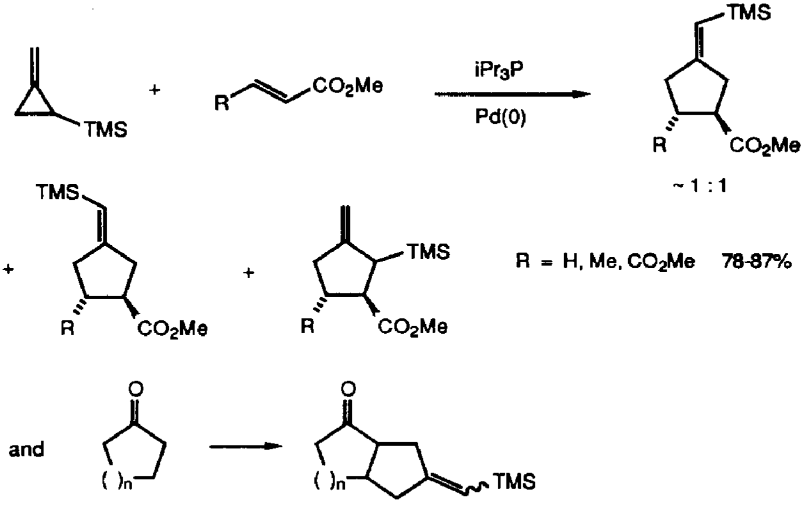
(176)



R = Me, Ph

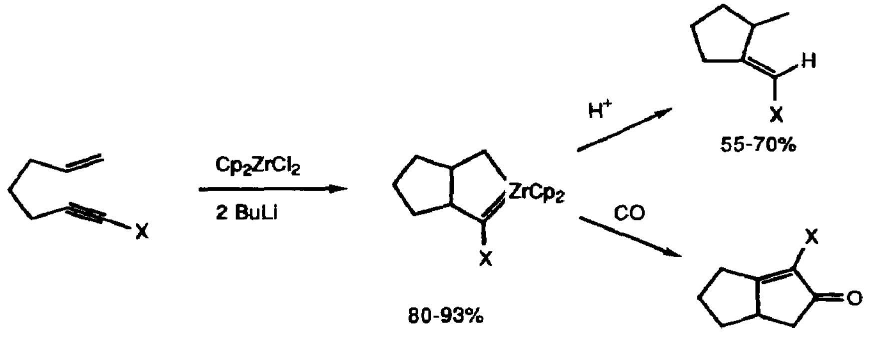
R' = Me, Ph, OMe

(177)

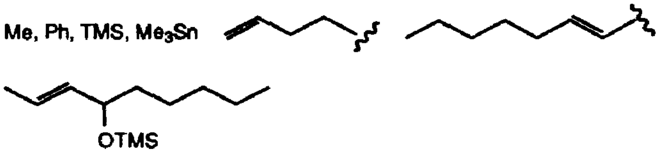


A number of methods to cyclize eneynes have been developed. Reduced zirconium complexes reacted to give a metallacycle which was converted into a variety of products (equation 178) [189]. Palladium catalyzed a related eneyne cyclization (equation 179) [190], (equation 180) [191], (equation 181) [192], as did low-valent nickel complexes (equation 182) [193]. Palladacyclopentadiene complexes catalyzed a co-cyclotrimerization of eneynes with alkynes, (equation 183) [194]. Palladium(0) complexes catalyzed the cycloaddition of diynes with alkenes and carbon monoxide (equation 184) [195].

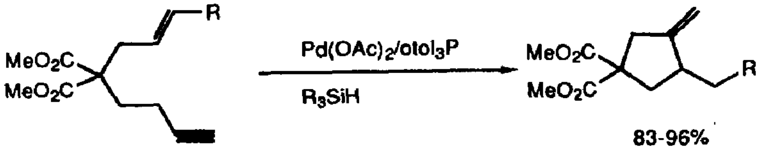
(178)



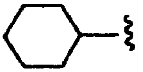
X = Me, Ph, TMS, Me$_3$Sn



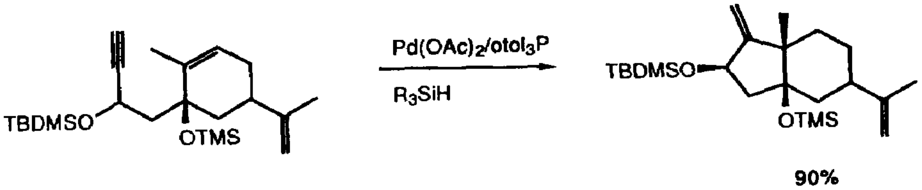
(179)



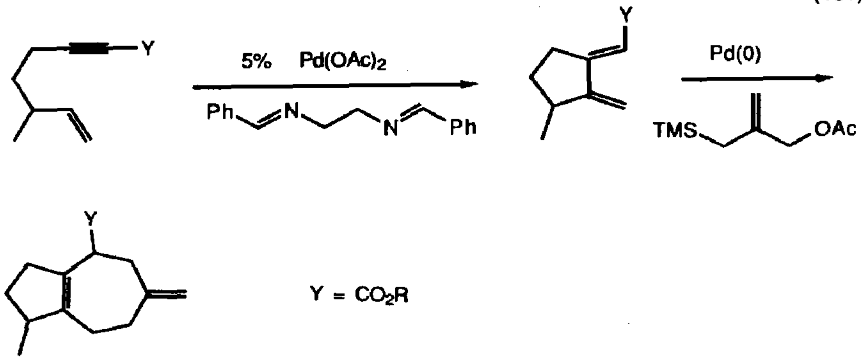
R = CH(OAc)$_2$, TBDMSOCH$_2$,



(180)

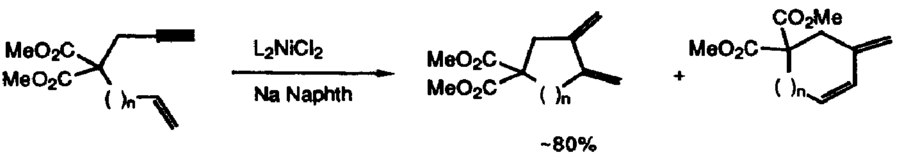


(181)



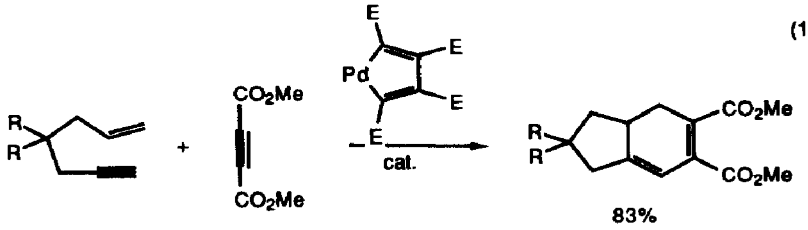
Y = CO$_2$R

(182)

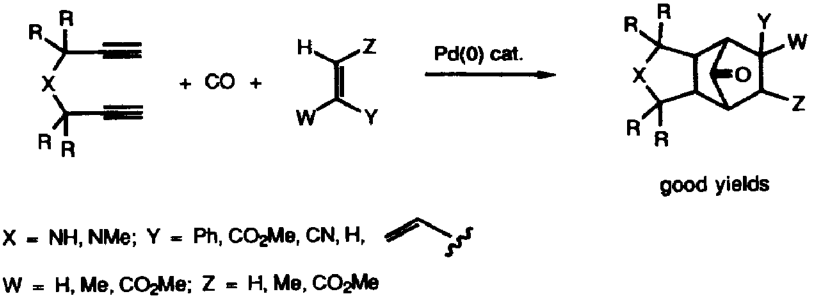


n = 1, 2

(183)



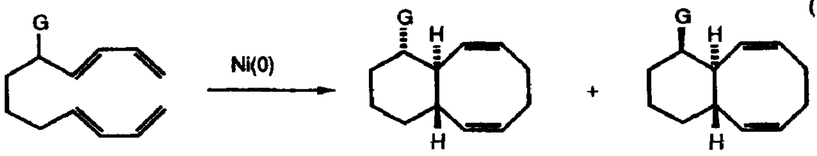
R = Me, $CO_2Me$



(184)

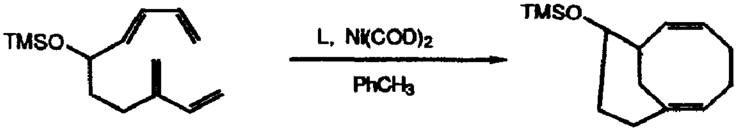
Nickel(0) complexes catalyzed the cyclization of tetraenes to bicyclodienes (equation 185) [196], (equation 186) [197], and α,ω-dienes to cyclopentenes (equation 187) [198]. The full details of the copper catalyzed photocyclization of α,ω-dienes to bicyclic compounds have been published (equation 188) [199].

Reduced iron species catalyzed intermolecular (equation 189) [200], (equation 190) [201] and intramolecular (equation 191) [202], (equation 192) [203] ene reactions.



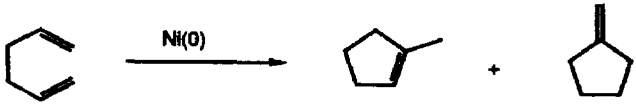
(185)

| | | |
|---|---|---|
| G = CN | 1.5/1 | 66% |
| $CH_3$ | 20/1 | 88% |
| $CH_2OAc$ | 21/1 | 92% |
| $CO_2Me$ | ≥70/1 | 84% |

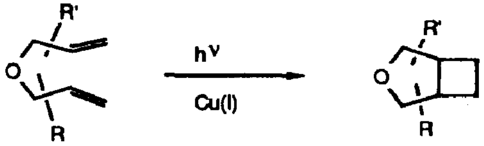


62%

(186)



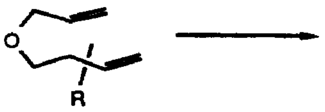
(187)



(188)

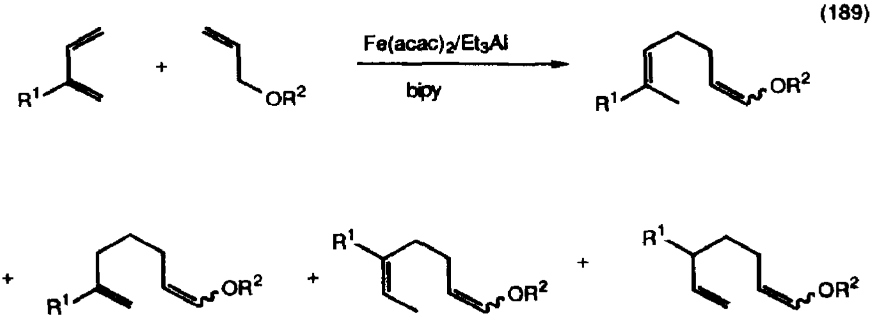
30-50% 14 cases

7 cases various rings

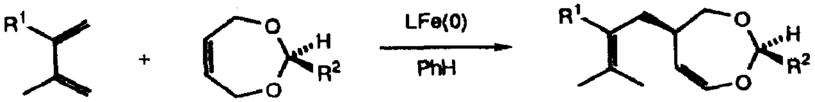


7 cases

(189)



(190)



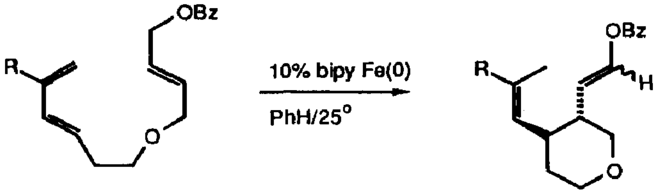
70%

>95% syn

ALSO:

40-60%

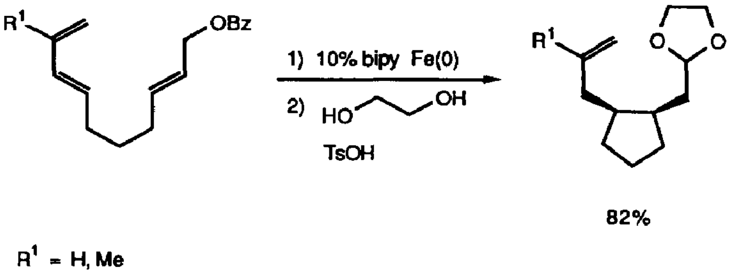
>95% syn



(191)

57%

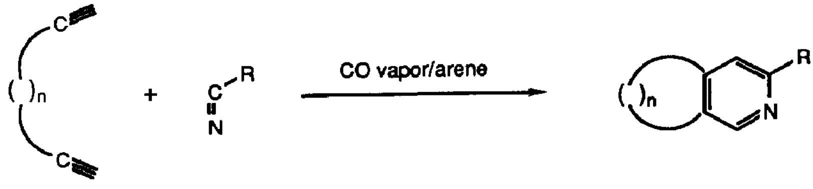
98 : 2 trans / cis



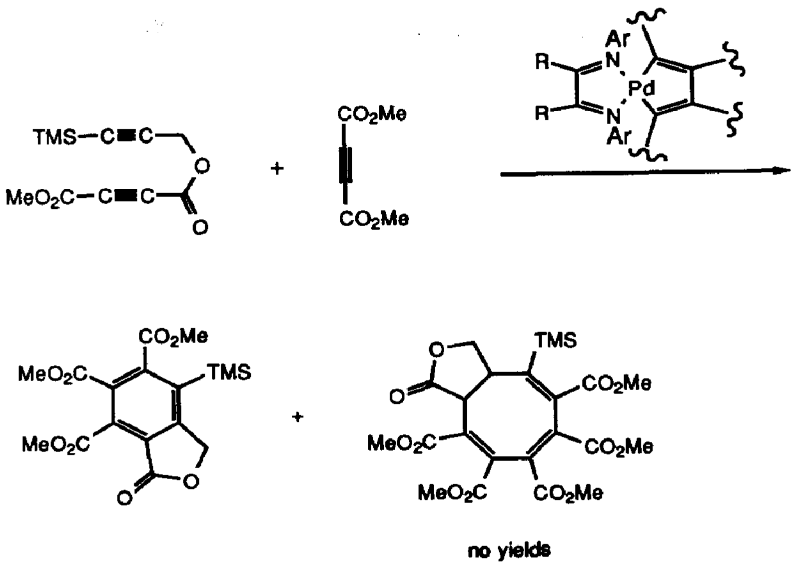
(192)

The cobalt-catalyzed cyclotrimerization of alkynes has been reviewed (36 references) [204]. Cobalt-mediated cyclization of acyclic endiynes was the topic of a dissertation [205]. Diynes cyclotrimerized with nitriles to form isoquinolines under cobalt catalysis (equation 193) [206]. Diynes co-cyclotrimerized with alkynes under palladium(0) catalysis (equation 194) [207]. Copper(II) acetate catalyzed a messy reaction between dimethylacetylene dicarboxylate and 2-iminoindolines to give seven different products [208].

(193)

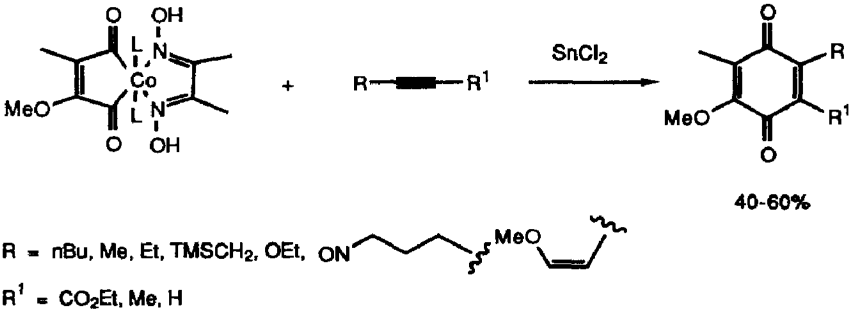


(194)

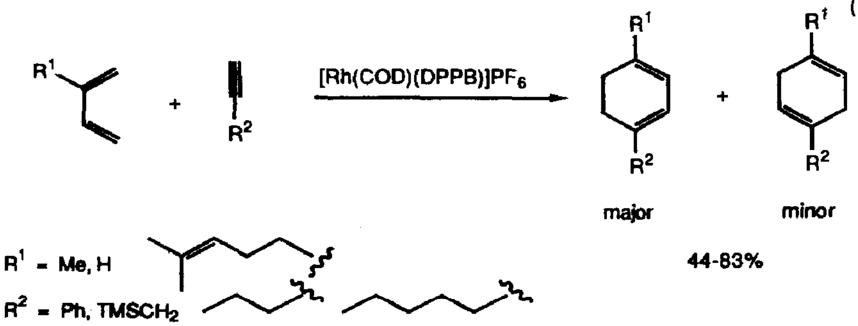


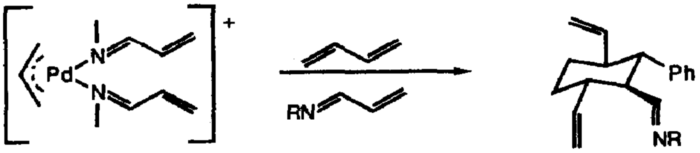
Alkynes cycloadded to maleoylcobalt complexes to give quinones (equation 195) [209]. Rhodium(I) complexes catalyzed the co-cyclodimerization of dienes with alkynes (equation 196) [210]. Palladium(II) catalyzed the cycloaddition of dienes to azadienes (equation 197) [211]. Iron carbonyl catalyzed the cyclization of allenes, alkynes and carbon monoxide to produce cyclopentenones (equation 198) [212]. Allyltin reagents cycloadded to α,β-unsaturated iron acyl complexes (equation 199) [213]. Isocyanates and isothiocyanates underwent 1,3-dipolar cycloaddition to metal-bound isonitrile anions (equation 200) [214].

(195)



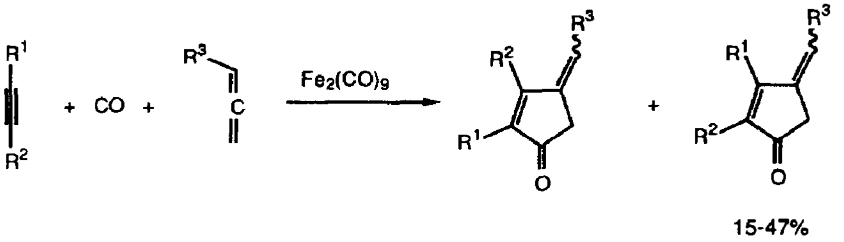
(196)





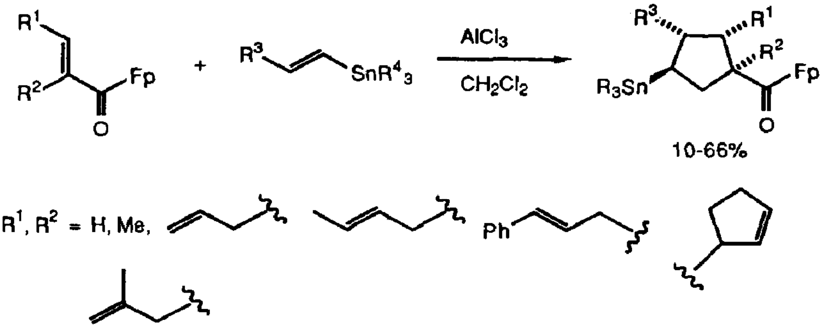
(197)

(198)

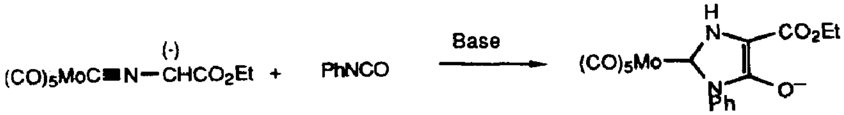


$R^1$ = H, Ph; $R^2$ = H, Ph; $R^3$ = H, Ph, $PhCH_2$

(199)



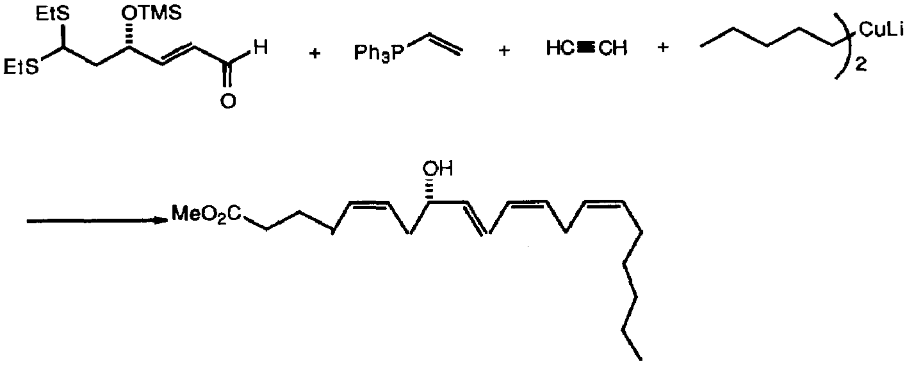
(200)



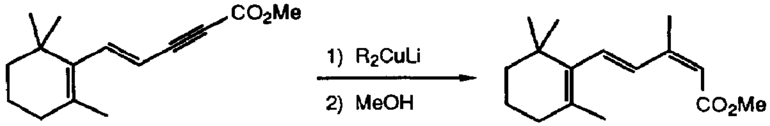
## 6. Alkylation of Alkynes

Alkylcuprates added to acetylene in the presence of aldehydes and vinylphosphonium salts to give long chain dienes (equation 201) [215]. Ethylcopper added to 1-octyne to give (E)-5-ethyl-1,4-undecadiene [216]. Organocuprates alkylated acetylenic esters with high regio- and stereoselectivities (equation 202) [217]. Eneynes were prepared by the reaction of vinyl cuprates with iodoalkynes (equation 203) [218].

(201)



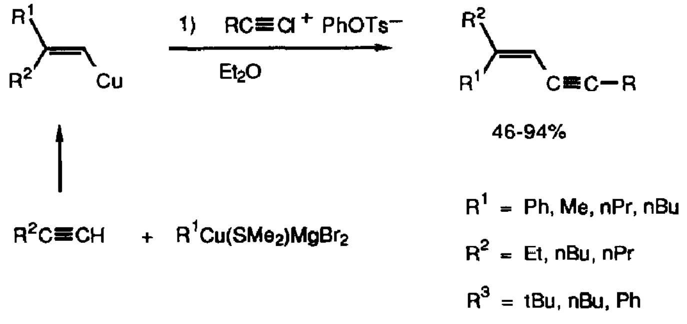
(202)



>99% Z

80% yield

(203)



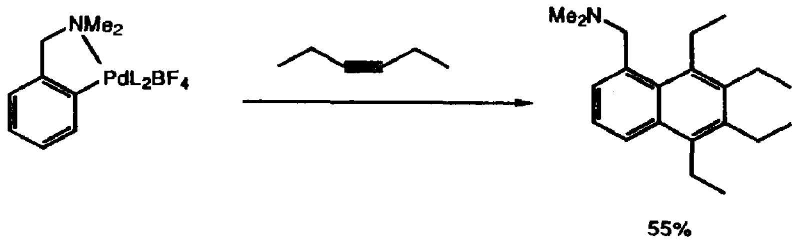
46-94%

$R^1$ = Ph, Me, nPr, nBu

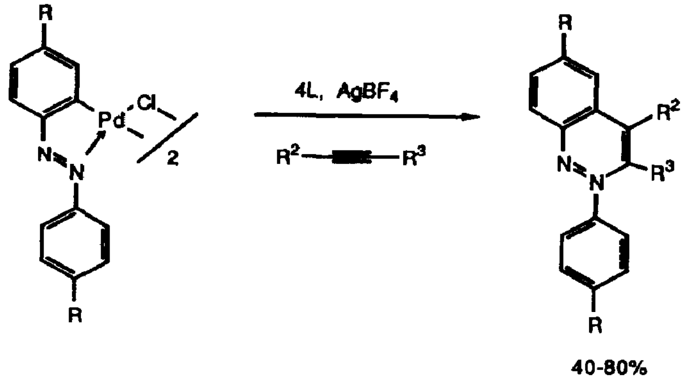
$R^2$ = Et, nBu, nPr

$R^3$ = tBu, nBu, Ph

Alkynes inserted into cyclopalladated arenes to give a variety of cyclic compounds (equation 204) [219], (equation 205) [220], (equation 206) [221], (equation 207) [222], (equation 208) [223]. Palladium(0) complexes catalyzed the arylation of propargyl alcohols (equation 209) [224].



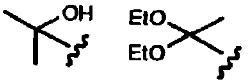
(204)

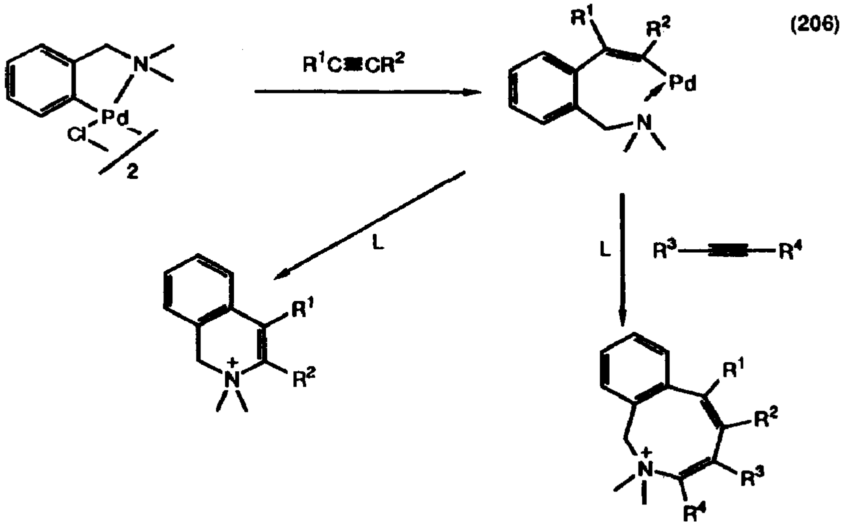


(205)

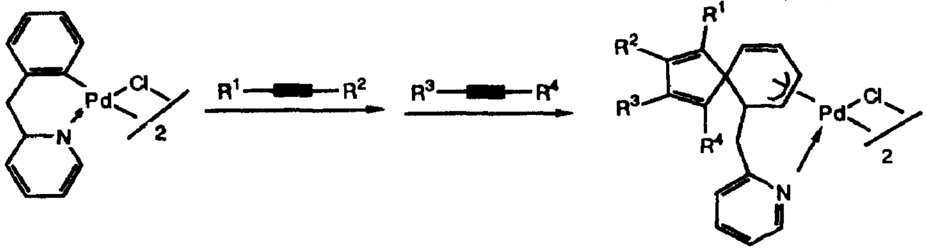
R = H, Me

R², R³ = Ph, MeO$_2$C, Et,

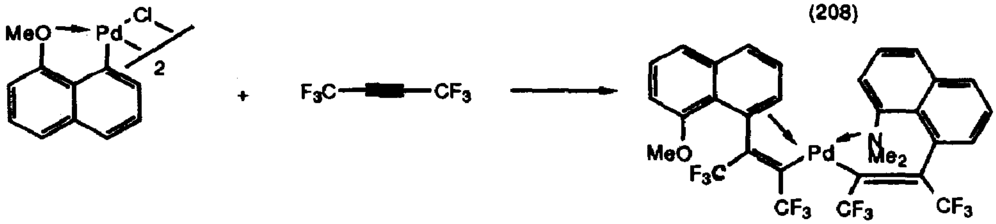




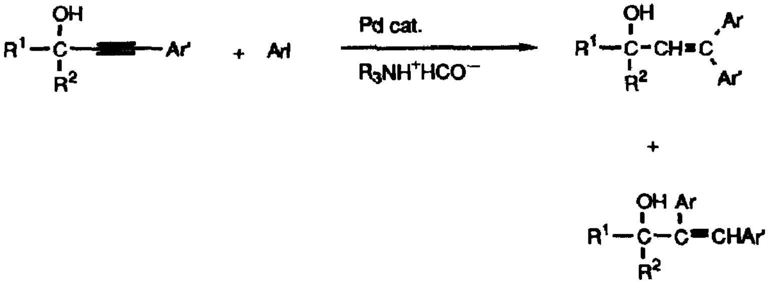
(206)



(207)

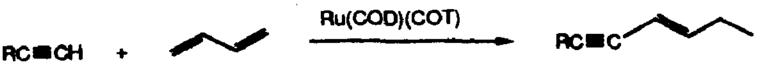


(208)



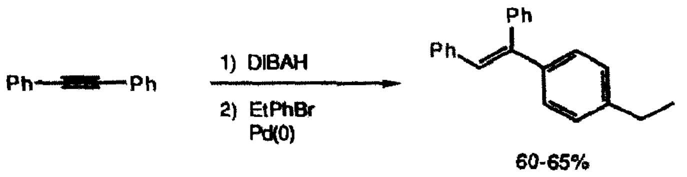
(209)

Ruthenium(0) complexes catalyzed the linear addition of terminal alkynes to dienes (equation 210) and the cycloaddition of internal alkynes to strained olefins (equation 211) [225]. Alkynes were arylated by a sequence involving hydroalumination/Pd(0) oxidative addition (equation 212) [226], (equation 213) [227]. Halopyrimidines (equation 214) [228] and purines (equation 215) [229] alkylated alkynes in the presence of palladium catalysts.



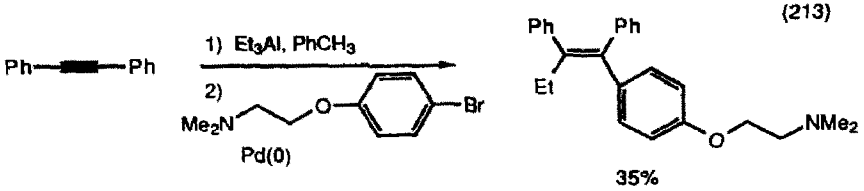
(210)

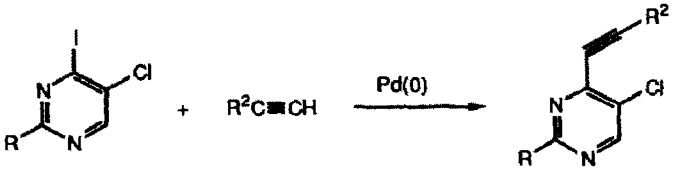
(211)



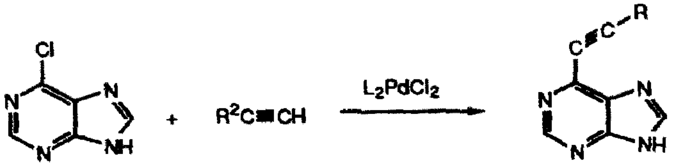
(212)

(213)





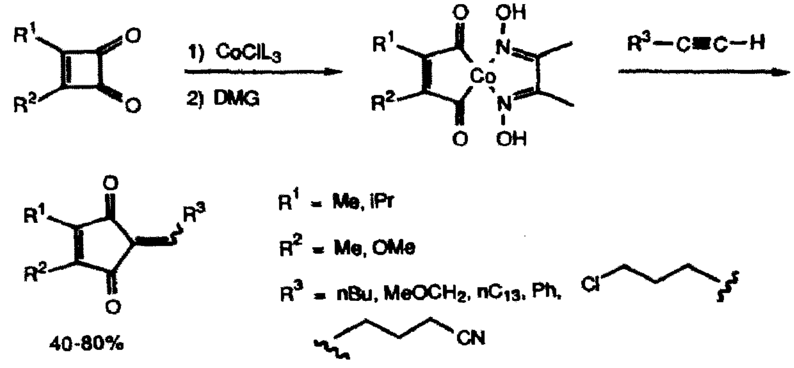
(214)



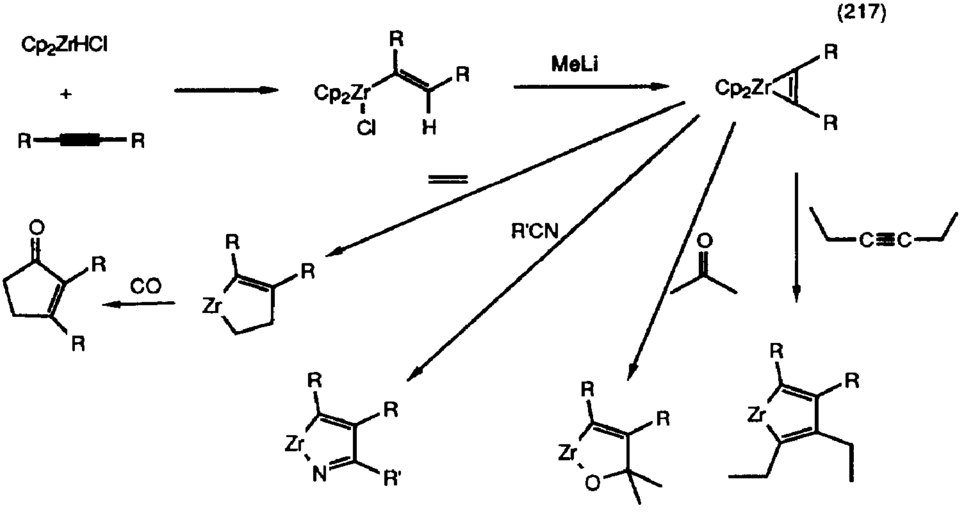
(215)

Cobalt maleoyl complexes combined with terminal alkynes to give vinyl cyclopentendiones (equation 216) [230]. Zirconacyclopropenes inserted a number of unsaturated species to give more elaborate metallacycles of potential use in organic synthesis (equation 217) [231].

(216)



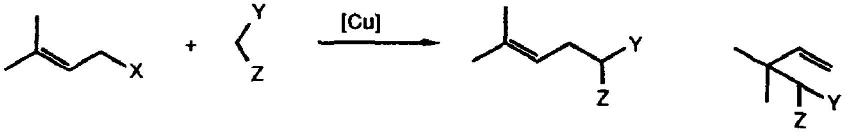
(217)



7. Alkylation of Allyl, Propargyl and Allenyl Systems.

Organocuprates continued to be used extensively to alkylate allylic systems. A dissertation dealing with cross-coupling of organocuprates in allylic systems has appeared [232]. The effects of the nature of the cuprate reagent on the regiochemistry and yield of the alkylation of allylic substrates by stabilized carbanions has been reported (equation 218) [233]. Copper(I) cyanide catalyzed the coupling of alkyl zincs with allylic halides (equation 219) [234]. γ-Alkoxy-α,β-unsaturated esters were alkylated by cyanocuprates in good yield with high stereoselectivity (equation 220) [235]. Copper-catalyzed reactions of Grignard reagents with 4-heteroatom substituted allylic acetates have been studied (equation 221), (equation 222) [236]. Allylic quaternary ammonium salts were alkylated by Grignard reagents in the presence of copper(II) chloride (equation 223) [237]. The regio- and stereochemistry of the ring opening of allylic epoxides by cyanocuprates have been clarified (equation 224) [238], (equation 225) [239], (equation 226) [240]. Propargyl ethers were converted to allenes by copper catalyzed Grignard reactions (equation 227) [241] while allenic halides were alkylated to allenes or propargyl systems, depending on the nature of the copper reagent (equation 228) [242].

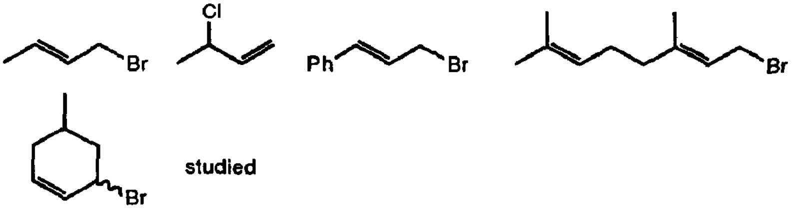
(218)



X = Br, Cl

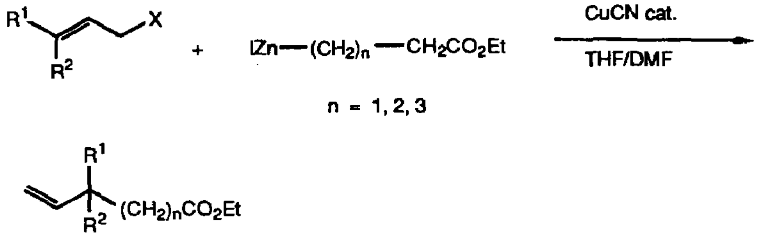
Y = $CO_2Et$, COMe, CN

Z = $CO_2Et$, COMe



studied

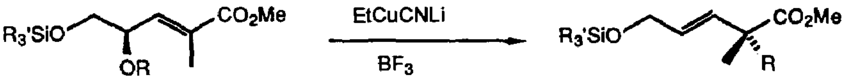
(219)



n = 1, 2, 3

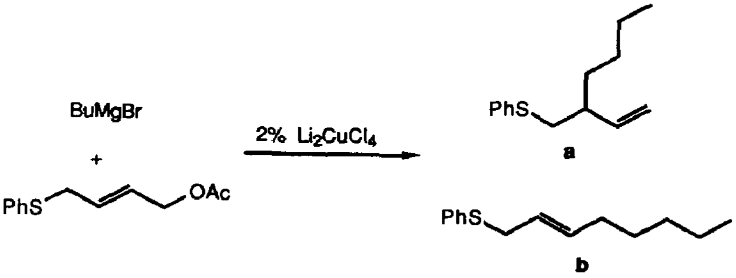
major 50-90%

(220)



(5 cases)

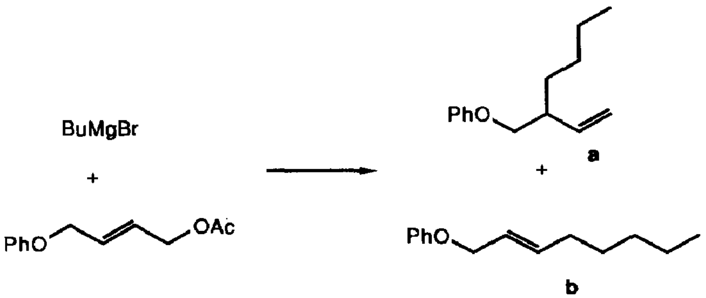
(221)



ADD TIME:

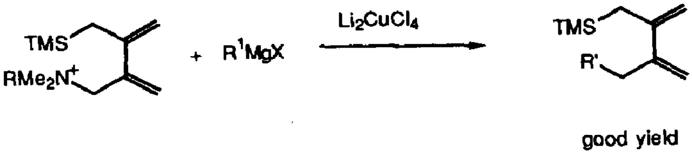
| 2 min | 4 min |
|---|---|
| a 25 | b 75 |
| a 13 | b 87 |

(222)



Cu$_2$Cl$_4$:

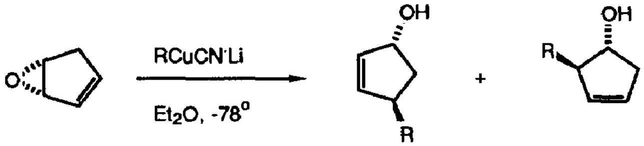
| 2%, 1.5min, -30° | 5%; 150 min, 0° |
|---|---|
| a 4 | b 96 |
| a 86 | b 14 |



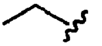
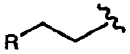
(223)

good yield

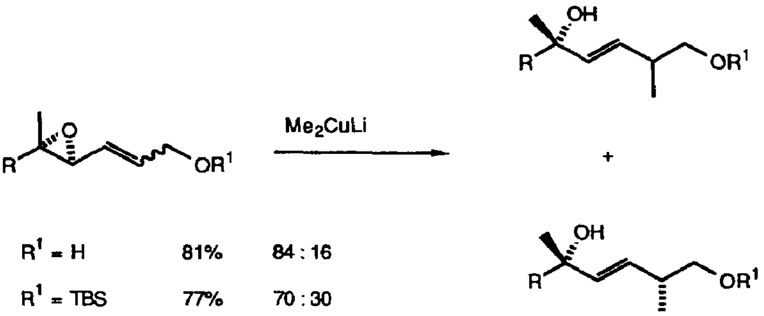
R$^1$ = Ph, TMSCH$_2$, PhCH$_2$, 



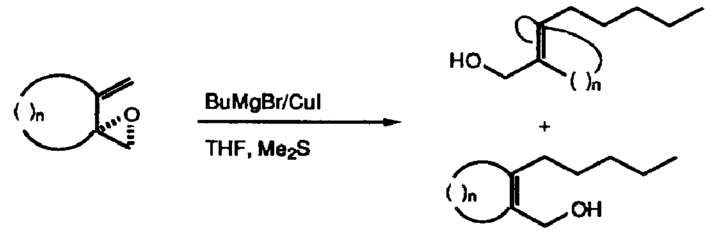
(224)

| R = Me, nBu, tBu | 75-95% | 0% |
|---|---|---|
| EtO$_2$CCH$_2$, Bu$_3$SnCH$_2$ | 5-17% | 0% |
|  | 75% | 1 : 1 |
|  | 80% | 4 : 1 |
| Ph | 50% | 2 : 1 |

(225)

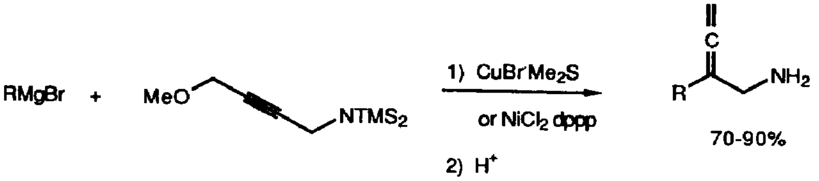


| E | R$^1$ = H | 81% | 84 : 16 |
|---|---|---|---|
| | R$^1$ = TBS | 77% | 70 : 30 |
| Z | R$^1$ = H | 75% | 3 : 97 |
| | R$^1$ = TBS | 78% | 18 : 72 |



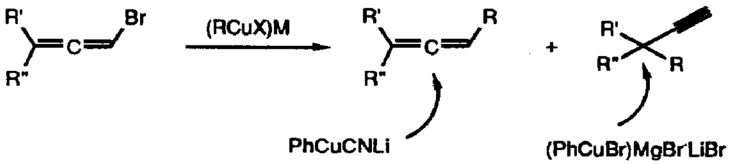
(226)

| | | | | |
|---|---|---|---|---|
| n = 10 | 99 : 1 | 68% | 92% ee | $S_N2'$ - syn |
| 12 | 94 : 6 | 59% | 82% ee | |
| 14 | 89 : 11 | 66% | 0% ee | (prior racemization) |



(227)

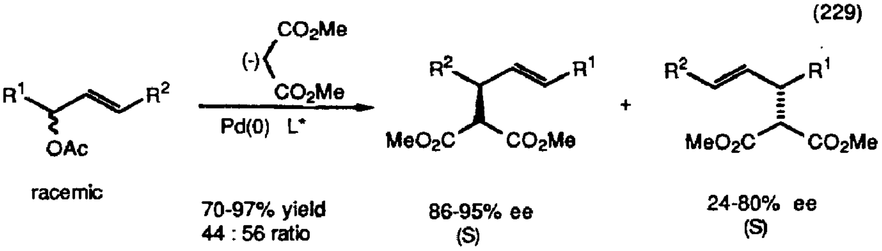
R = Ph, pClPh, pMeOPh, pMePh, $nC_6$



(228)

Palladium-catalyzed enantioselective allylation was the topic of a dissertation [243]. Allyl acetates were alkylated by stabilized carbanions in the presence of palladium(0) catalysts [244]. Allyl acetates and allyl phenyl ethers were alkylated by cyclohexanone enamines in the presence of palladium(0) catalysts [245]. Racemic allylic acetates were alkylated by malonate anions in the presence of chiral palladium(0) catalysts with high stereoselectivity (equation 229) [246]. Cyanoacetic ester enolates were allylated by allyl acetate in the presence of palladium(0) catalysts (equation 230) [247]. Intramolecular alkylation of a chiral steroidal allylic ester occurred with complete chirality transfer (equation 231) [248]. Phosphorous-substituted allyl acetates were alkylated (and aminated) under palladium catalysis (equation 232) [249][250]. Palladium catalyzed allylic alkylation was combined with palladium catalyzed ene reactions to produce cyclic compounds (equation 233) [251]. A reaction to classify nucleophiles for π-allylpalladium reactions has been developed [252].

(229)

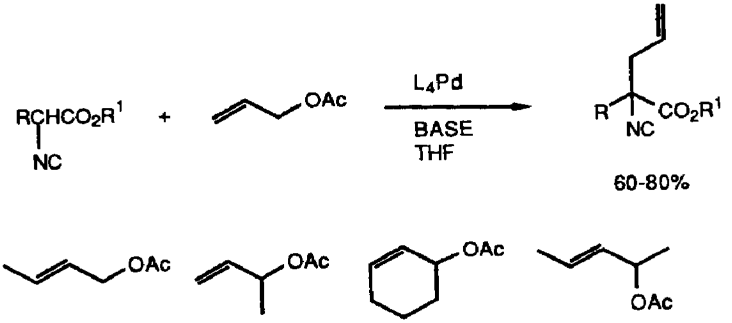


racemic

70-97% yield
44 : 56 ratio

86-95% ee
(S)

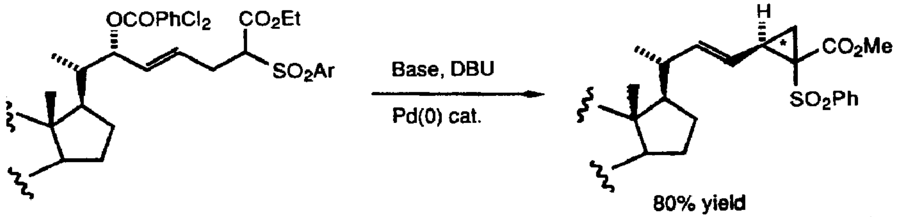
24-80% ee
(S)



(230)

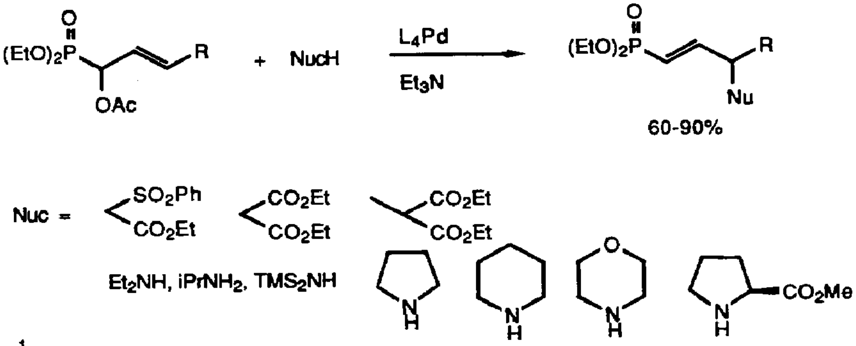
60-80%

(231)



80% yield
complete chirality transfer

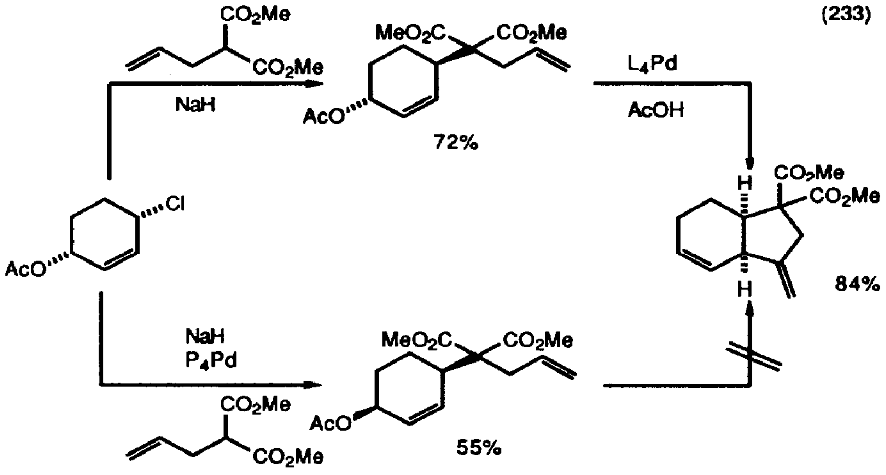
(232)



60-90%

$R^1$ = Me, H

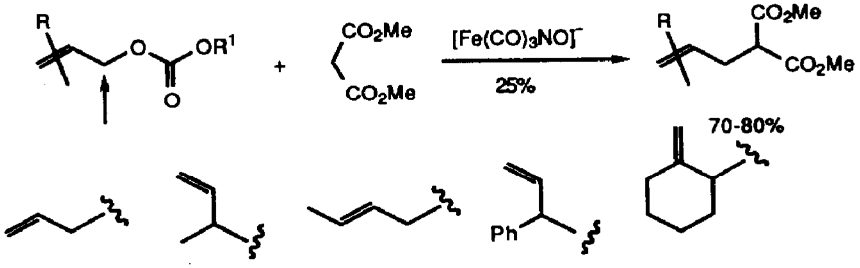
(233)



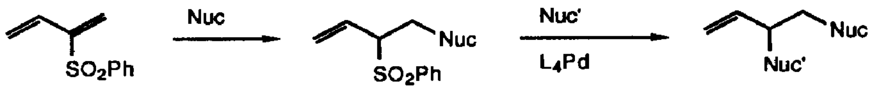
New synthetic reactions of allyl alkyl carbonates, allyl-β-ketocarboxylates and allylic vinylic carbonates catalyzed by palladium complexes was the subject of a review with 40 references [253]. Cationic iron nitrosyl carbonyl complexes catalyzed the alkylation of allyl carbonates (equation 234) [254]. Allyl sulfones were alkylated using palladium catalysis (equation 235) [255]. The full details of molybdenum(0) catalyzed allylic alkylations have been published (equation 236) [256]. A comparison of molybdenum with palladium catalysts (equation 237) [257] and tungsten with palladium catalysts (equation 238) [258] has been made. Molybdenum(0) complexes also catalyzed the alkylation of allyl sulfides (equation 239) [259] and allyl carbonates (equation 240) [260].

Propargyl epoxides underwent reaction with acetoacetates under palladium(0) catalysis to form furans (equation 241) [261]. Allenic acetates were alkylated by malonates in the presence of palladium(0) catalysts (equation 242) [262]. Palladium(0) catalyzed reactions of vinyl halides with allenic malonates led to cyclic products (equation 243) [263]. π-Allylpalladium chloride dimers catalyzed the alkylation of allylic halides by $RTi(OR')_3$ species [264]. Propargyl acetates reacted under the same conditions to give alkylated allenes [265].

(234)

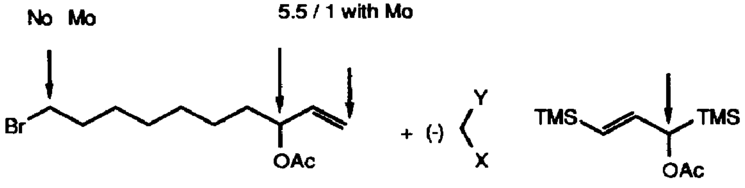


(235)



Nuc' = $^{-}CH(CO_2Me)_2$, $R_2CuLi$, $R_2NH$

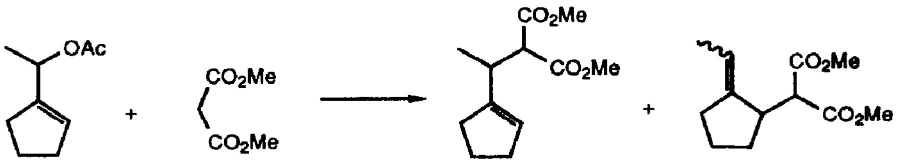
(236)



E-geometry in product

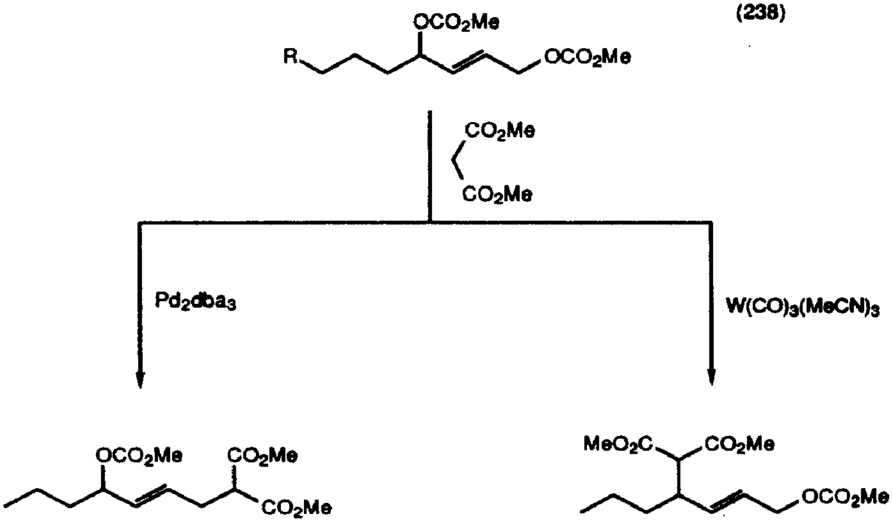
Retention of stereochemistry

(237)

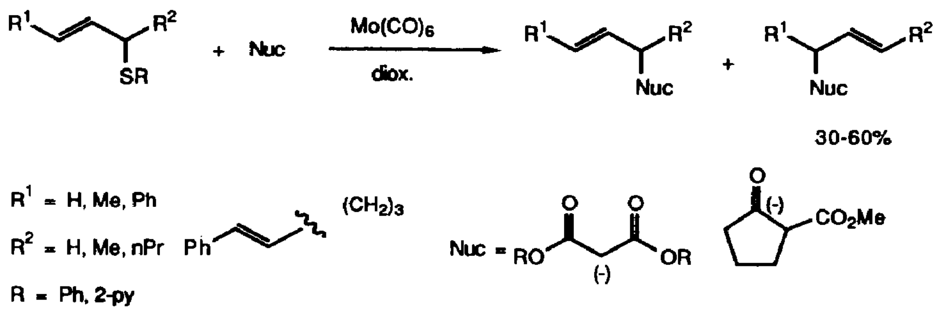


| | | |
|---|---|---|
| $L_4Pd$ | 100 | 0 |
| $Mo(CO)_6$ | 15 | 85 |
| $Mo(CO)_4bipy$ | 100 | 0 |

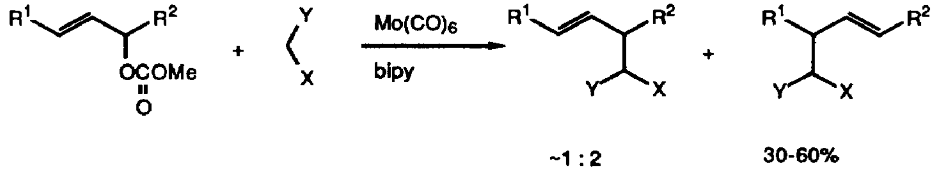
(238)



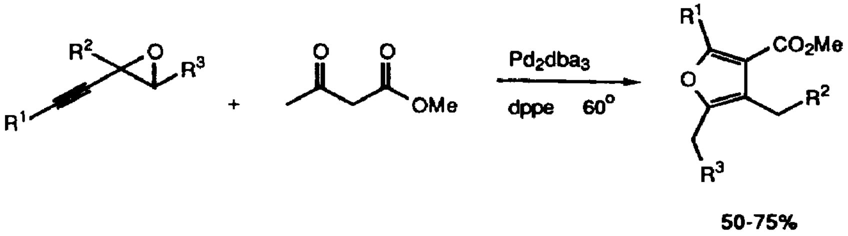
(239)



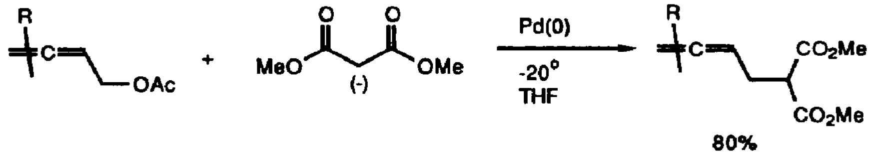
(240)



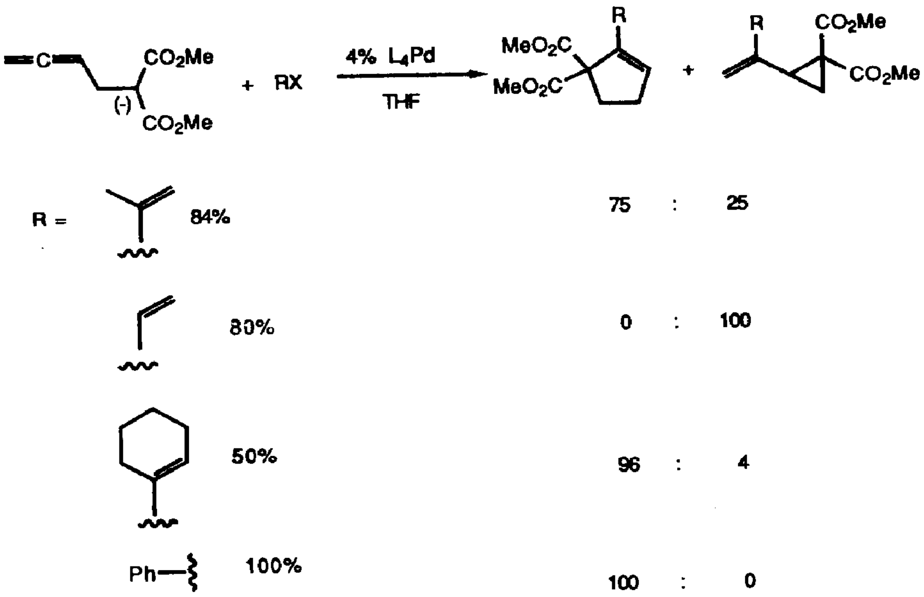
(241)



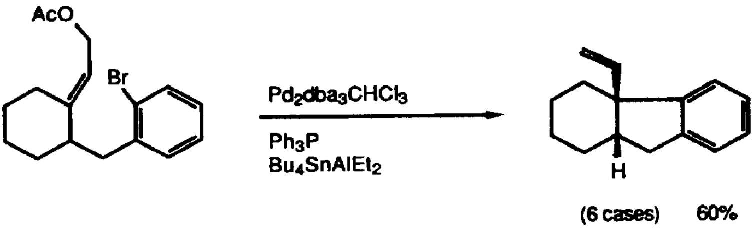
(242)



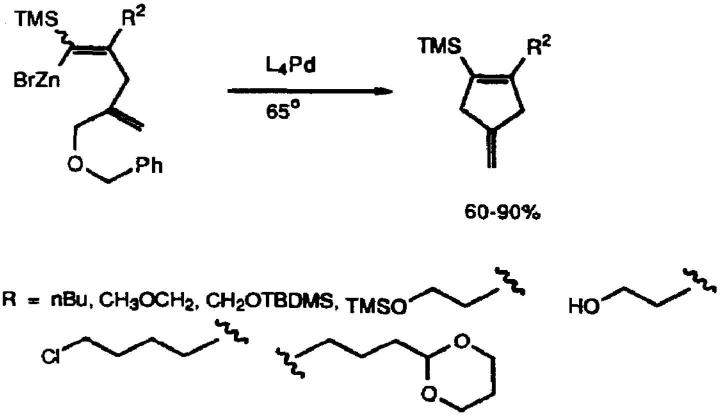
(243)



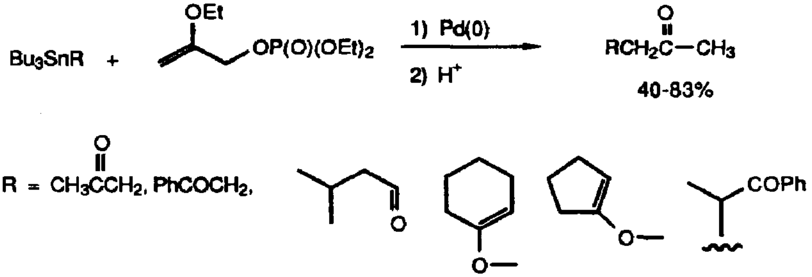
Allylic acetates also underwent a variety of palladium(0) catalyzed oxidative addition/insertion reactions (equation 244) [266] and oxidative addition/transmetallation reactions (equation 245) [267], (equation 246) [268], (equation 247) [269]. Finally enol ethers and allylic alcohols underwent efficient palladium(II) catalyzed Claisen rearrangements (equation 248) [270], while palladium(0) complexes catalyzed the reductive alkylation of ketones with propargyl acetates (equation 249) [271].



(244)

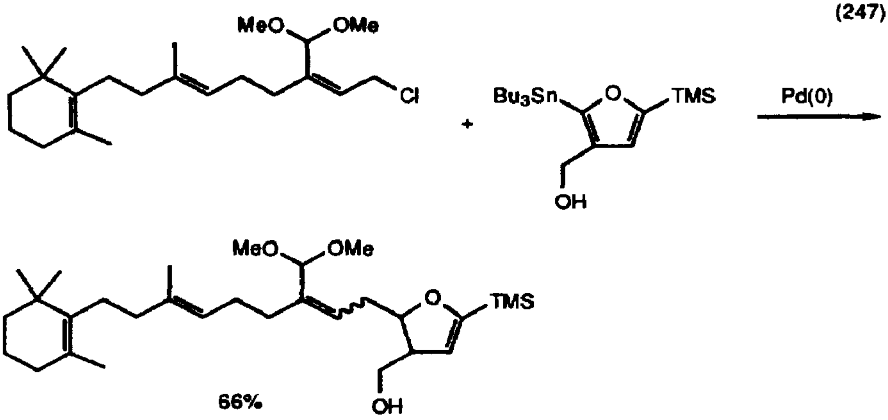

TMS
R$^2$
BrZn
O
Ph
$L_4Pd$
65°
TMS
R$^2$
60-90%
R = nBu, $CH_3OCH_2$, $CH_2OTBDMS$, TMSO
HO
Cl
O
O

(245)

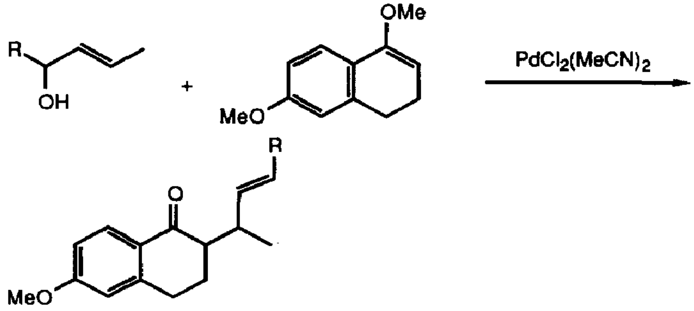

$Bu_3SnR$ +
OEt
$OP(O)(OEt)_2$
1) Pd(0)
2) H$^+$
$RCH_2\overset{O}{\overset{\|}{C}}-CH_3$
40-83%
R = $CH_3\overset{O}{\overset{\|}{C}}CH_2$, $PhCOCH_2$,
O
O
O
COPh

(246)

(247)

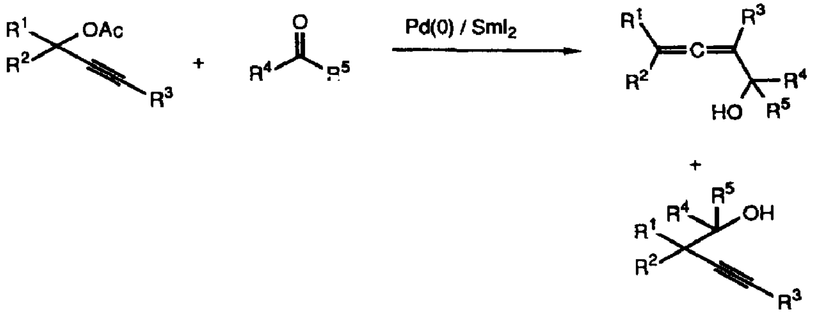

MeO
OMe
Cl
+
$Bu_3Sn$
O
TMS
OH
Pd(0)
MeO
OMe
O
TMS
66%
OH

(248)



95% 13 : 87 syn / anti

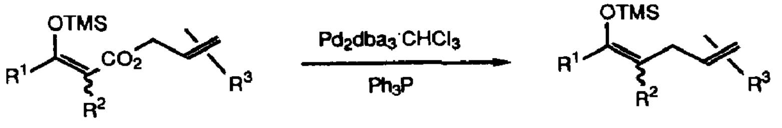
(249)



good yields

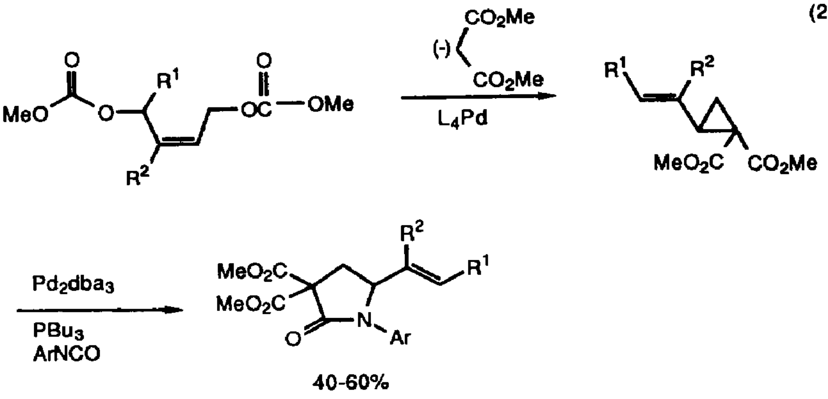
β-Ketoallyl carbonates underwent a number of interesting palladium catalyzed reactions (equation 250) [272], (equation 251) [273], (equation 252) [274], (equation 253) [275]. This topic has been reviewed (51 references) [276]. β-Acetoxy allyl esters underwent palladium catalyzed elimination (equation 254) [277].

(250)

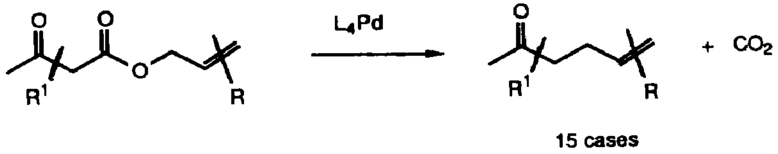


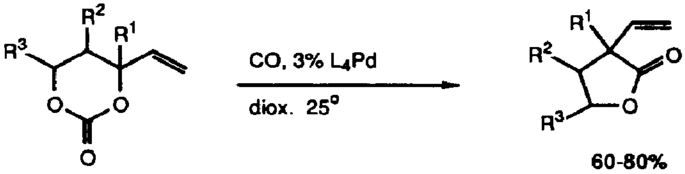
(6 cases) 60-80%

(251)



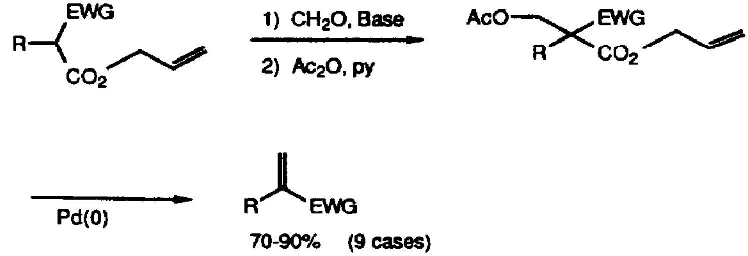
(252)





(253)

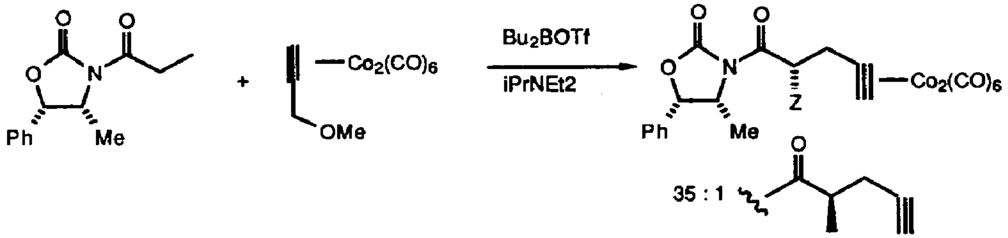
(254)



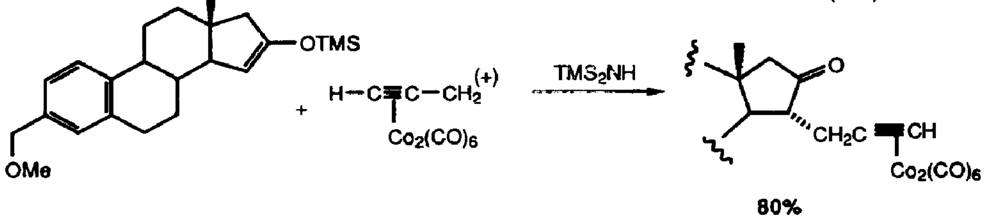
EWG = $CO_2R$, $CONR_2$, $NO_2$, $SO_2R$, CN, COR

Phenylmanganese iodide phenylated allyl and propargyl acetates with $S_N2'$ regiochemistry [278]. The chemistry and synthetic utility of cobalt-complexed propargyl cations has been reviewed (75 references) [279]. This complex chemistry has been used to introduce the propargyl group into electron-rich substrates (equation 255) [280], (equation 256) [281].

(255)

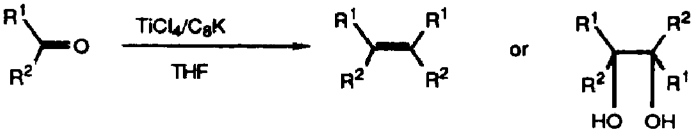


(256)



## 8. Coupling Reactions.

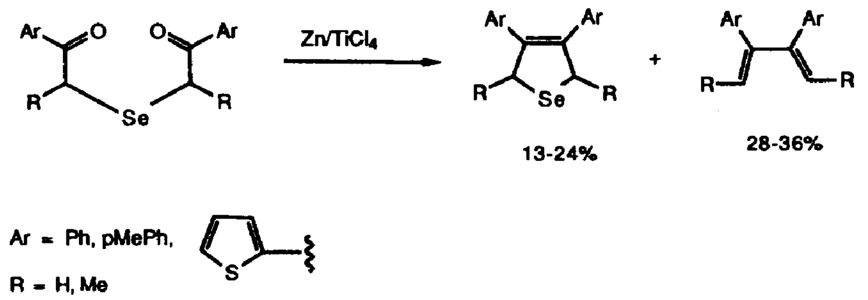
Low-valent titanium reagents remain the compounds of choice for reductive coupling of ketones to give olefins. Titanium(IV) chloride/potassium graphite was an efficient reagent for this type of coupling (equation 257) [28]. Selenium (equation 258) [283] and sulfur heterocycles were made from appropriate diketones (equation 259) [284]. Aromatic ketones were coupled to give alkanes in the presence of titanium(IV) chloride/zinc (equation 260) [285]. Dithioketals were reductively coupled to give olefins by tungsten hexacarbonyl (equation 261) [286].



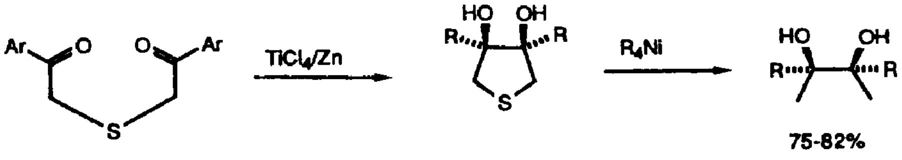
(257)

high yields

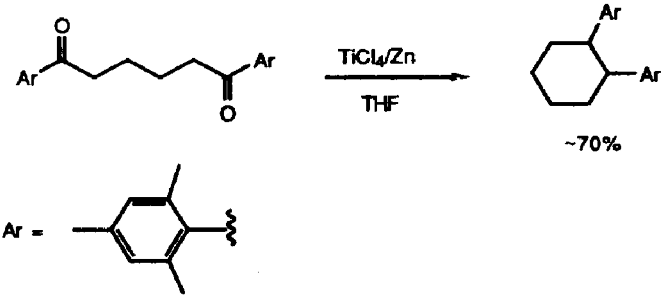
(258)



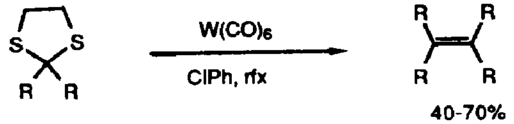
(259)



R = Ph, pMePh, pMeOPh



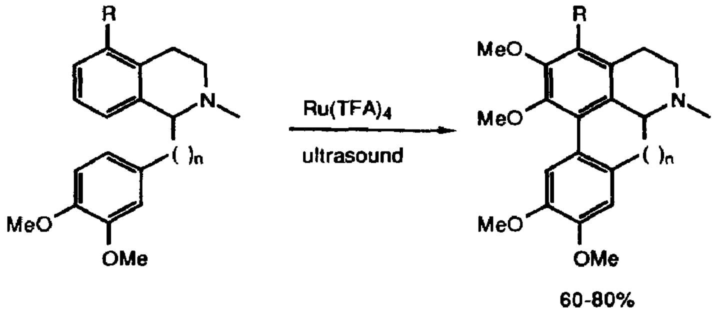
(260)



(261)

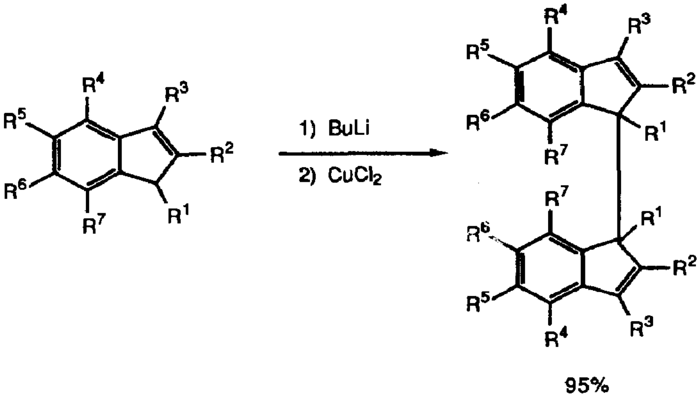
R = Ph, pMePh, adamantyl, fluorene

Chiral oxidative coupling of naphthol and phenol derivatives by copper complex catalysts has been reviewed (39 references) [287]. Phenol couplings were efficiently carried out using ruthenium(IV) trifluoroacetate and ultrasound (equation 262) [288]. Copper salts promoted the oxidative coupling of a number of main group organometallic compounds (equation 263) [289], (equation 264) [290], (equation 265) [291], (equation 266) [292]. Cationic iron dienyl complexes were reductively coupled (equation 267) [293].



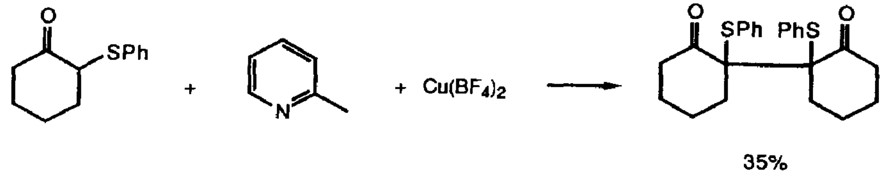
(262)

n = 1, 2, 0; R = H, OMe



(263)

(264)



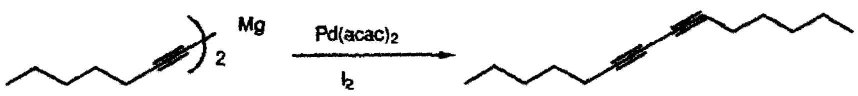
$$RC\equiv CSnBu_3 \xrightarrow{Cu(NO_3)_2 \cdot 3\,H_2O} RC\equiv C-C\equiv C-R \qquad (265)$$

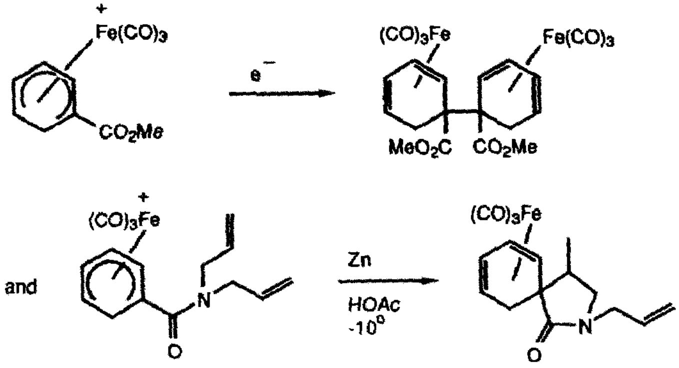
50-80%

R = nBu, nPr, Ph, $THPOCH_2$

also $ArSnBu_3 \longrightarrow ArAr$

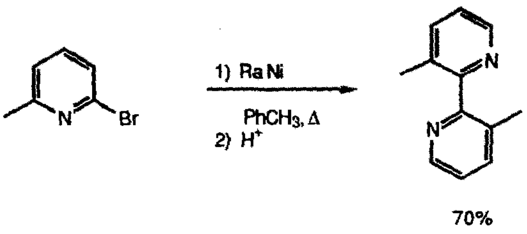
(266)



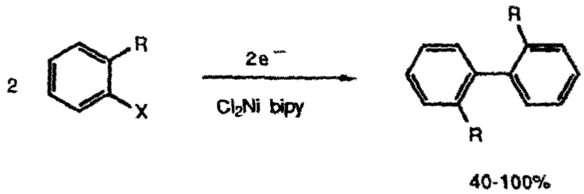


(267)

Ultrasound was used to enhance classic Ullmann coupling reactions of aryl halides [294][295]. The mechanism of the reductive coupling of aryl halides by hydrazine and a palladium amalgam catalyst was studied [296]. Aryl halides were reductively coupled by low valent nickel complexes (equation 268) [297], (equation 269) [298], (equation 270) [299]. Conjugated aryl ketones were reductively coupled electrochemically in the presence of transition metal salts (equation 271) [300].

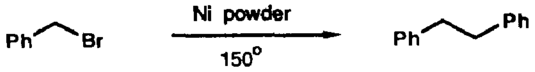


(268)



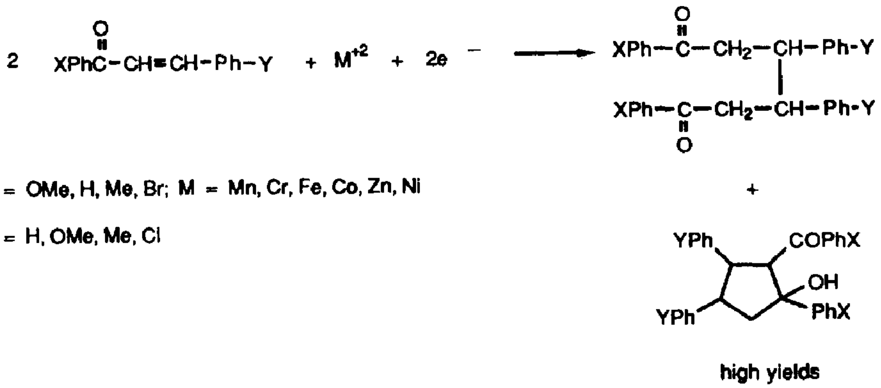
(269)

R = OMe, Me, Cl, $CH_3CO$, F, $CF_3$, $NH_2$, CN



(270)

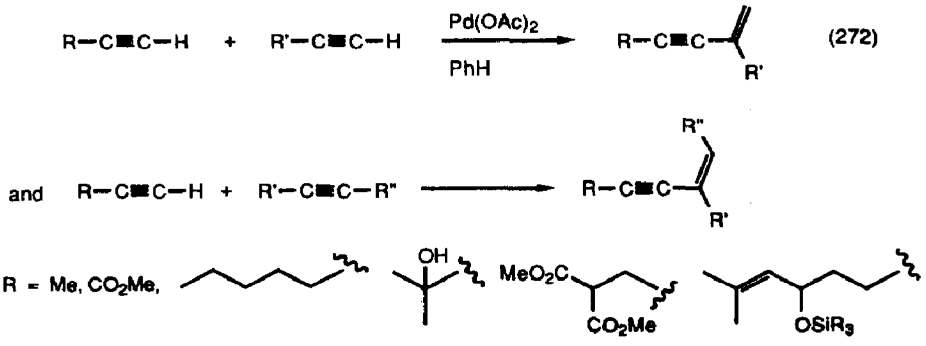
(271)



X = OMe, H, Me, Br; M = Mn, Cr, Fe, Co, Zn, Ni

Y = H, OMe, Me, Cl

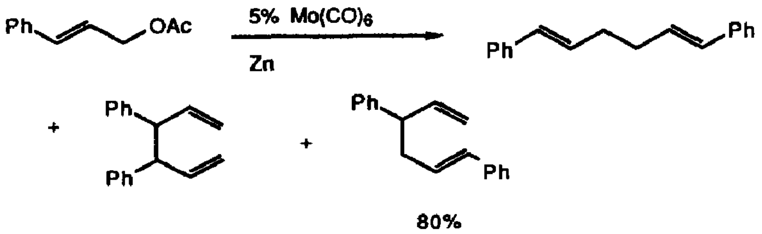
Palladium(II) acetate dimerized alkynes to eneynes (equation 272) [301]. Allyl acetates were coupled by molybdenum hexacarbonyl in the presence of zinc (equation 273) [302]. Vinyl tins coupled to give dienes when treated with palladium(II) acetate and t-butyl hydroperoxide (equation 274) [303]. Lithium acetylides dimerized when treated with palladium(II) salts (equation 275) [304]. Copper(I) chloride/sodium hydride reductively dimerized alkynes to dienes (equation 276) [305].



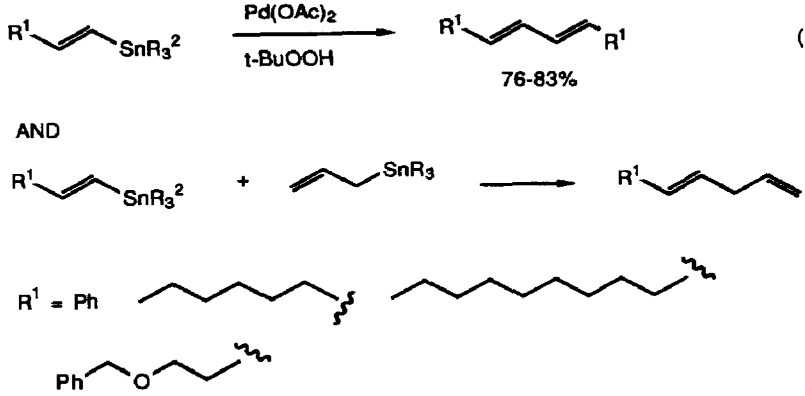
(272)

R = Me, CO$_2$Me,

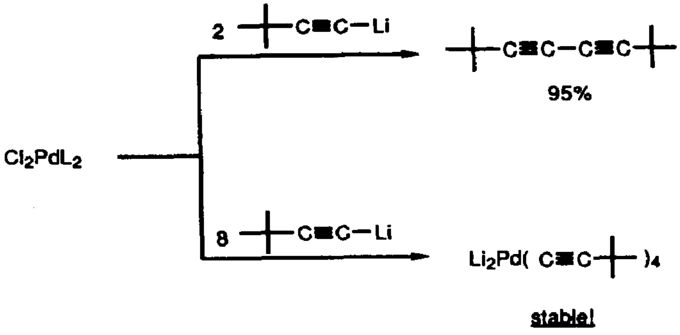
(273)



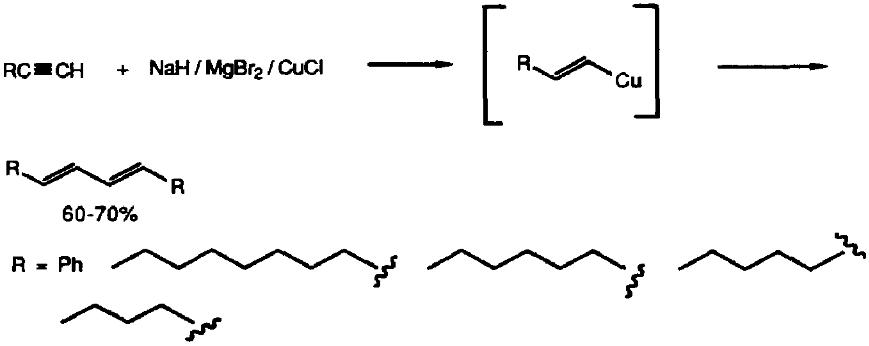
(274)



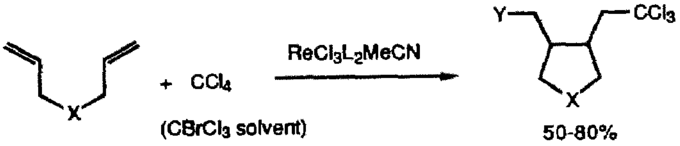
(275)



(276)



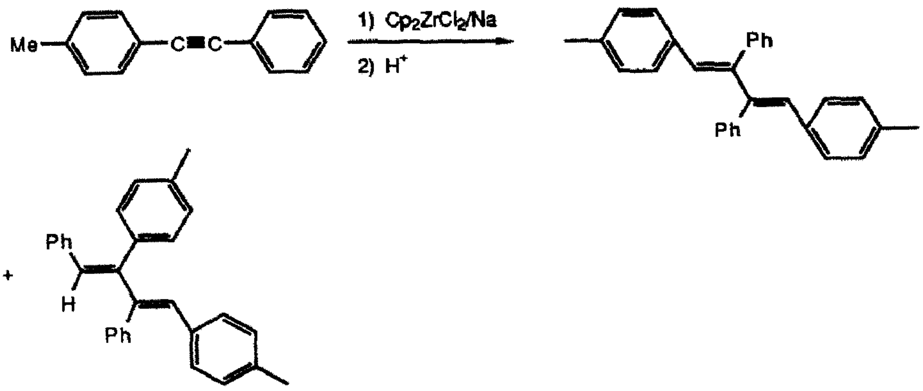
1,6-Dienes were coupled to cyclopentanes by rhenium catalyzed addition of haloforms to the diene system via radical processes (equation 277) [306]. Alkynes were coupled to give butadienes by zirconocene dichloride/sodium (equation 278) [307]. Cobalt complexes cyclodimerized the diyne in equation 279 in low yield (equation 279) [308].



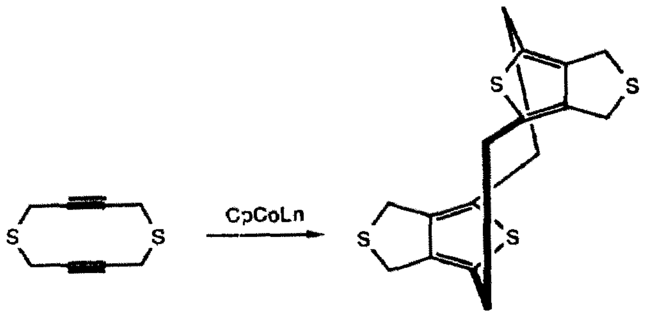
(277)

X = $C(CO_2Et)_2$, $C(COPh)_2$, $C(COMe)_2$, O, NCOMe

(278)



5 : 95 63%



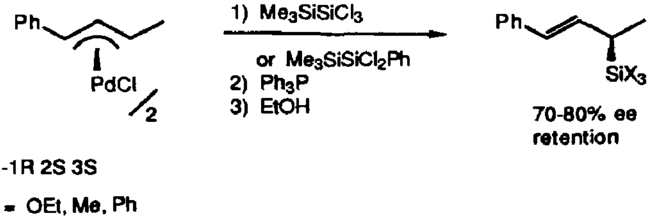
(279)

low yield

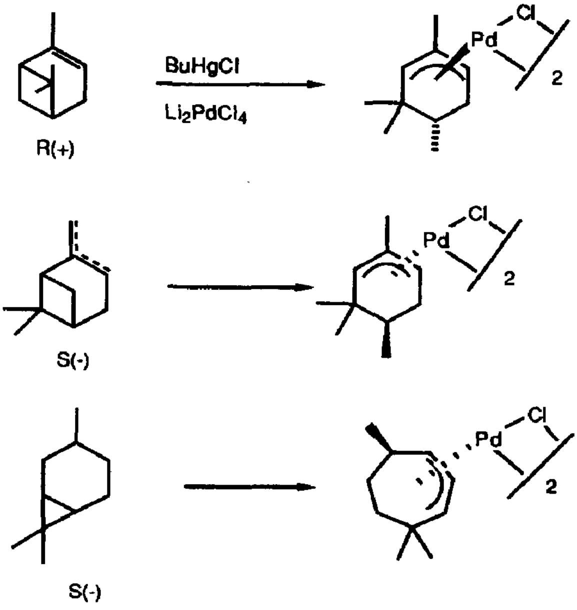
proven by X-ray

## 9. Alkylation of π-Allyl Complexes.

"New developments of chemistry of π-allyl complexes" is the topic of a review with 12 references [309], as was "Catalytic carbon-carbon bond formation via π-allyl complex intermediates" (81 references) [310]. A review entitled "Molecular basis of catalytic reactions involving η³-allyl complexes of Group 10 metals in key intermediates" (39 references) has appeared [311]. Asymmetric allylation reactions have been reviewed (28 references) [312]. Chiral π-allylpalladium complexes were converted to allyl silanes with retention of configuration (equation 280) [313]. Chiral olefins such as α-pinene were converted to chiral π-allylpalladium complexes upon reaction with butylmercuric chloride and lithium chloropalladate (equation 281) [314]. A carbon-13 nmr study of the effects of ligands on nucleophilic addition to π-allylpalladium complexes has been completed [315]. The process of reductive elimination of allylarenes from π-allyl-σ-aryl palladium complexes has been subjected to both experimental and theoretical consideration. The rate of elimination was insensitive to added donor ligands and was enhanced by π-acceptor olefins [316]. The influence of ligands on rates and regiochemistry of nucleophilic attack on π-allylpalladium complexes has been reported [317]. π-Allyl palladium complexes



(280)

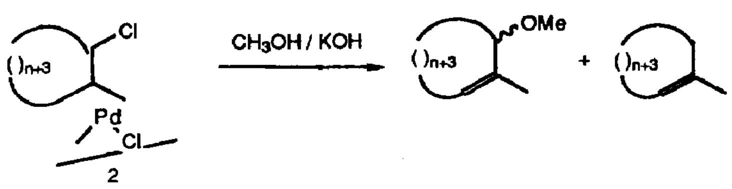


(281)

enantiomerically pure

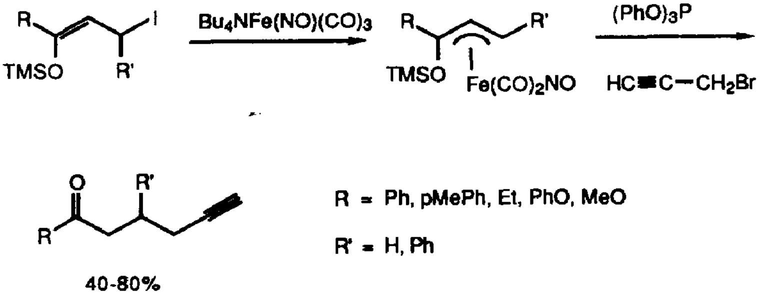
having a chloromethyl group on the 2-carbon underwent reaction with methoxide to give both substitution and elimination products (equation 282) [318].

(282)



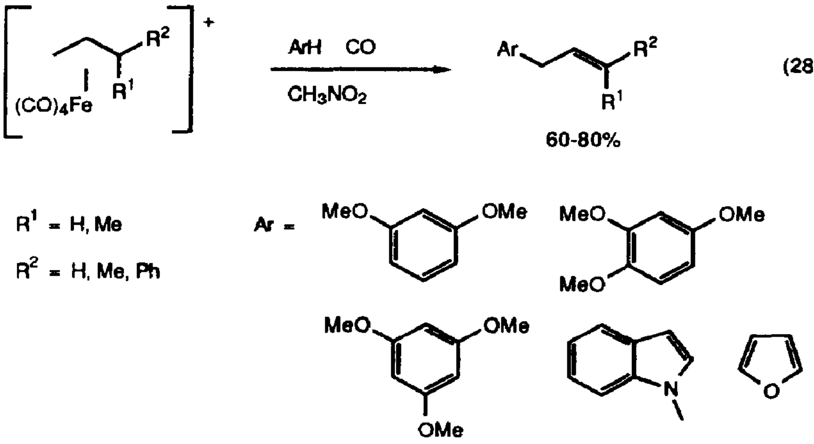
$n = 0, 1, 2, 3, 7\ 11$

(283a)



R = Ph, pMePh, Et, PhO, MeO

R' = H, Ph



(283b)

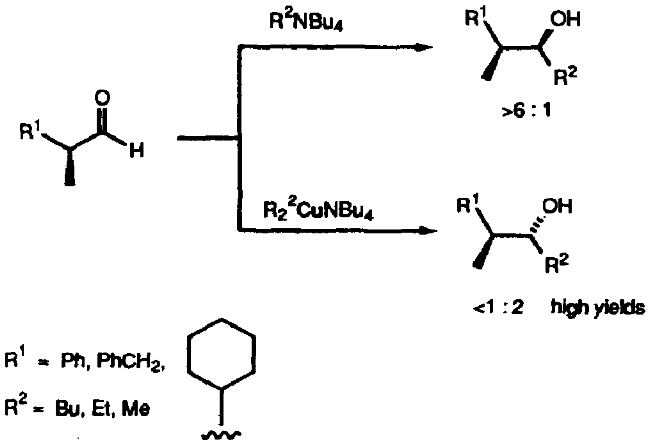
$R^1$ = H, Me

$R^2$ = H, Me, Ph

The subject, oxallyl complexes in organic synthesis has been reviewed (180 references) [319]. π-Allyliron complexes underwent reaction with a variety of carbon electrophiles (equation 283a) [320] and nucleophiles (equation 283b) [321].

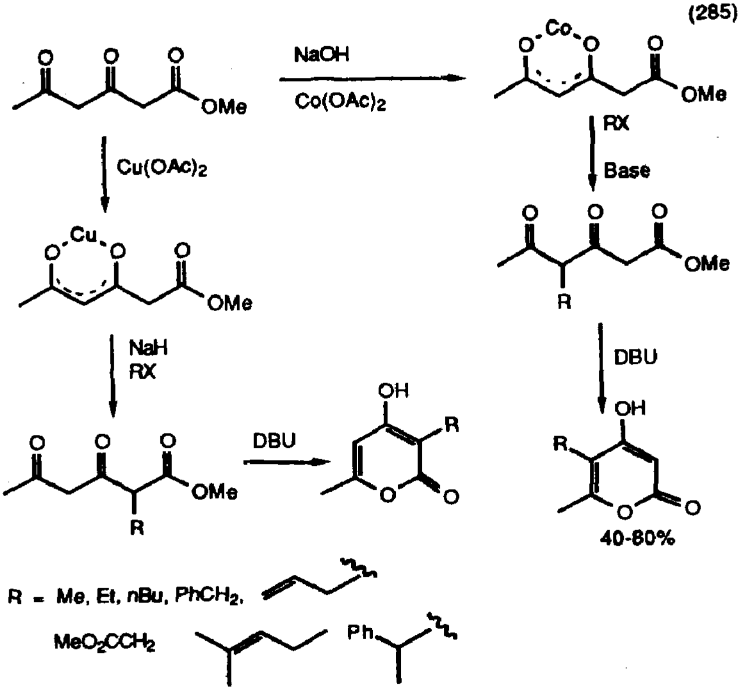
## 10. Alkylation of Carbonyl Compounds.

Acyclic stereocontrol in the reaction of allylic organometallic reagents with aldehydes was the subject of a review with 59 references [322]. Aldehydes underwent alkylation by organometallic reagents with varying degrees of stereocontrol, depending on the metal (equation 284) [323]. 3,5-Dioxoesters were alkylated at the 4-position exclusively by complexing the β-diketonate

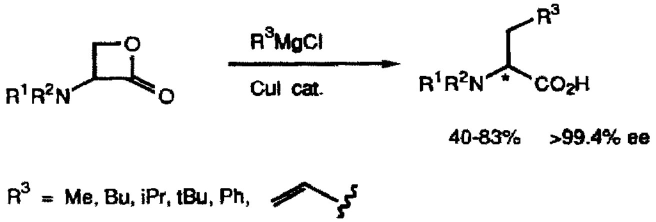


(284)

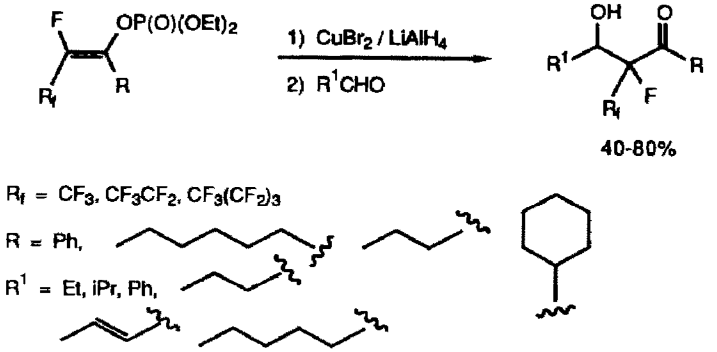
(285)



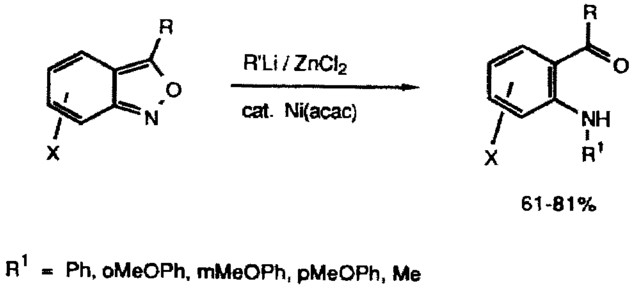
subunit to cobalt [324] or the 2-position by complexing to copper salts (equation 285) [325]. Serine β-lactone underwent alkylation α- to the lactone oxygen with very high enantioselectivity (equation 286) [326]. Copper(I) bromide promoted the alkylation of aldehydes by fluorinated enol ethers (equation 287) [327]. Nickel(II) acetylacetonates catalyzed the reactions of organozinc reagents with nitrogen heterocycles (equation 288) [328].



(286)

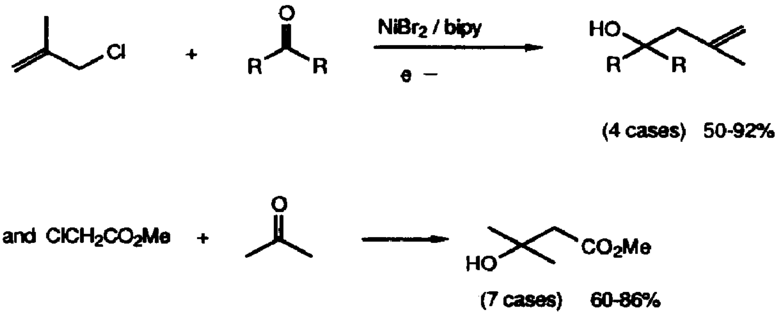


(287)

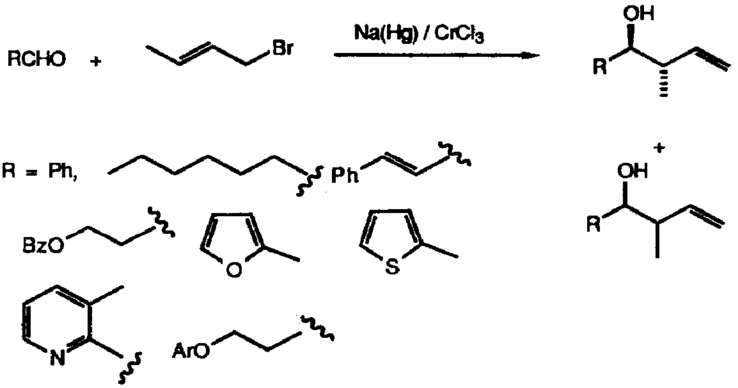


(288)

Nickel bromide catalyzed the reductive alkylation of ketones by allyl chloride (equation 289) [329]. Chromium(II) chloride promoted a similar reaction (equation 290) [330]. Aldehydes were olefinated by the reductive coupling of gem dihalides promoted by chromium(II) salts (equation 291) [331], (equation 292) [332].



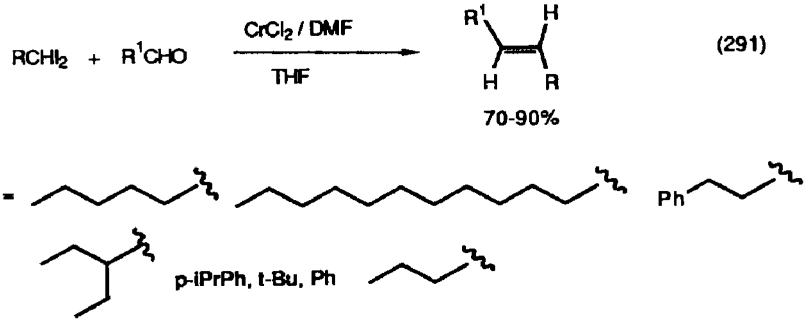
(289)



(290)

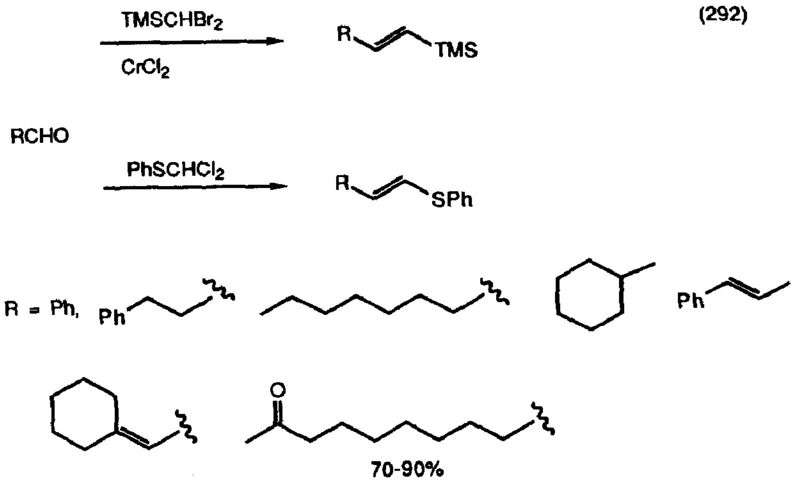
70-90%

5 : 1 to 10 : 1
erythro/threo



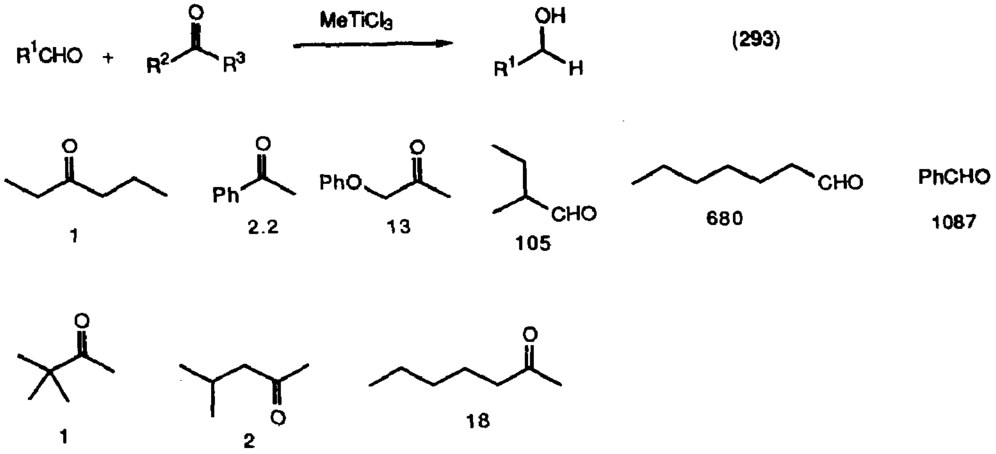
(291)

$R^1$ = Me, nPr, iPr, tBu, H



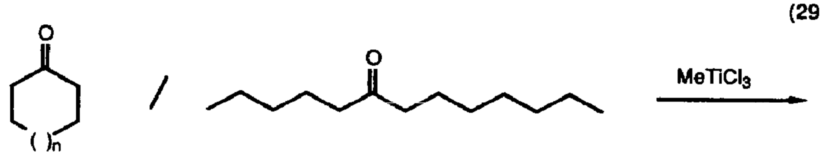
(292)

The relative rates of the reaction of $RTiX_3$ species with aldehydes and ketones are shown in equation 293 [333]. Similar comparisons of cyclic and acyclic ketones are given in equation 294 [334]. Aldehydes were dialkylated by dimethyltitanium dichloride (equation 295) [335]. Titanocene dichloride controlled the stereochemistry of aldol condensation between amide enolates and aldehydes (equation 296) [336].



(293)

(294)

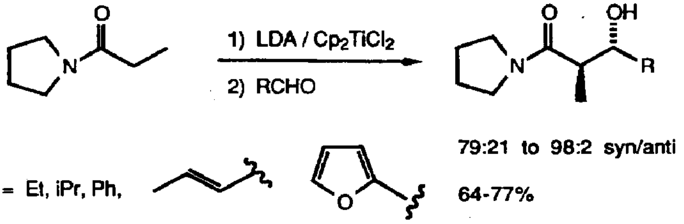


| n = 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 12 |
|---|---|---|---|---|---|---|---|---|
| 1 | 0.07 | 1.2 | 9.4 | 17.9 | 71.1 | 39.6 | 13.2 | 2.6 |

$ArCHO + Me_2TiCl_2 \rightarrow ArCH(CH_3)_2$ (295)

87-98%

Ar = Ph, pMeOPh, mMeOPh, pClPh, mClPh



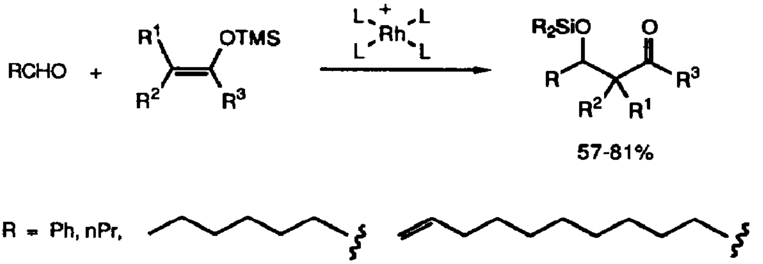
(296)

R = Et, iPr, Ph,

79:21 to 98:2 syn/anti

64-77%

Rhodium(I) complexes catalyzed the reaction between aldehydes and silylenol ethers (equation 297) [337], and between ketals and silylenol ethers (equation 298) [338], while rhodium(III) chloride effected the condensation of methyl methacrylate with ketones and trimethylsilane (equation 299) [339].



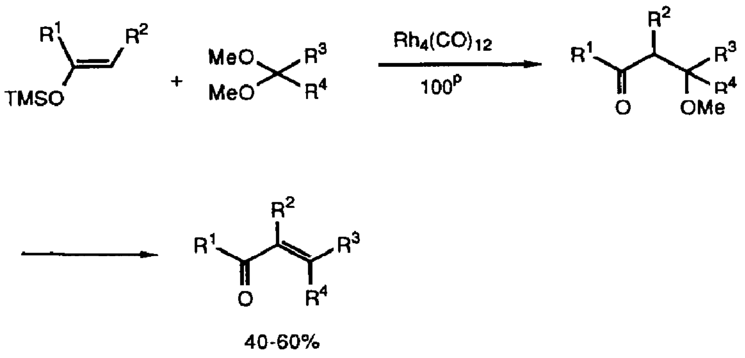
(297)

57-81%

R = Ph, nPr,

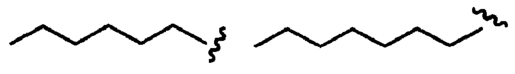
$R^1$ = Me, OEt, H; $R^2$ = Me, OTMS, H; $R^3$ = OMe, OEt, Ph

(298)

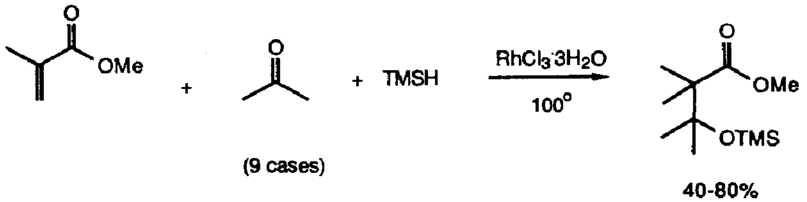


$R^1$ = Me; $R^2$ = H; $R^4$ = H, Me, OMe

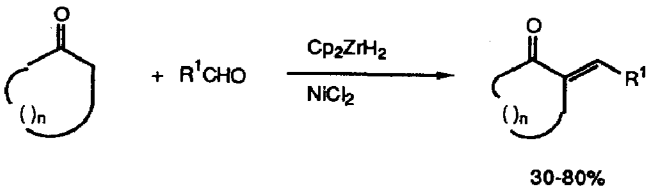
$R^3$ = Ph, H, Me,



(299)

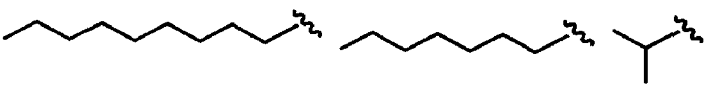


Zirconium(IV)/nickel(II) catalysts promoted the aldol condensation of cyclic ketones with aldehydes (equation 300) [340]. Zirconacyclopentenes alkylated conjugated enones and epoxides (equation 301) [341], while related hafnium compounds incorporated two equivalents of ketone (equation 302) [342]. Niobium(III) chloride promoted the reaction between imines and ketones (equation 303) [343], while zirconium diazene complexes olefinated aldehydes (equation 304) [344].

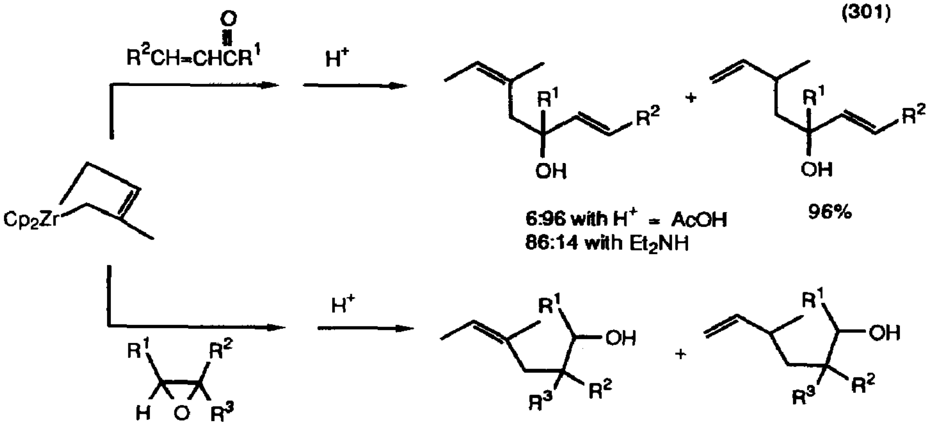


(300)

n = 1, 2; R = H, Me, nPr, nBu

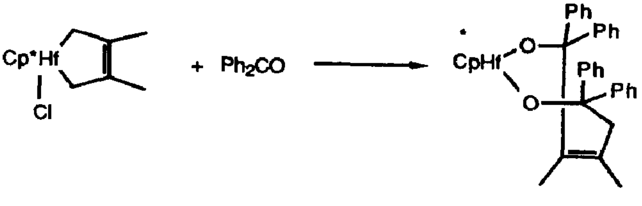


(301)



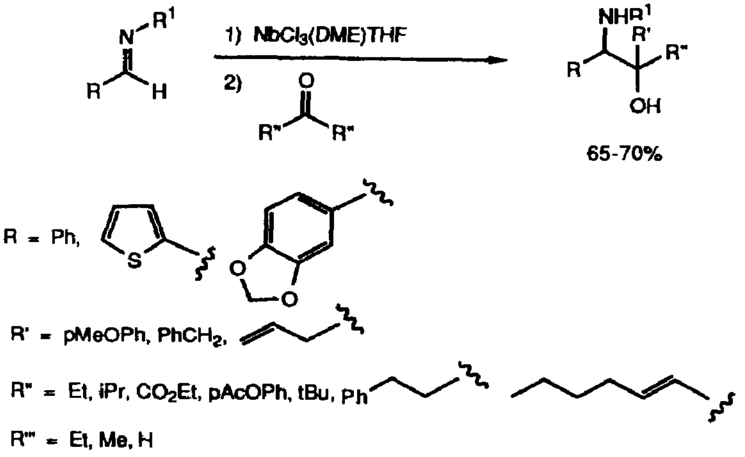
6:96 with $H^+$ = AcOH
86:14 with $Et_2NH$

96%



(302)

no yields



(303)

65-70%

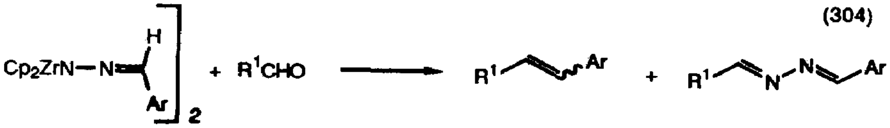
R = Ph,

R' = pMeOPh, $PhCH_2$,

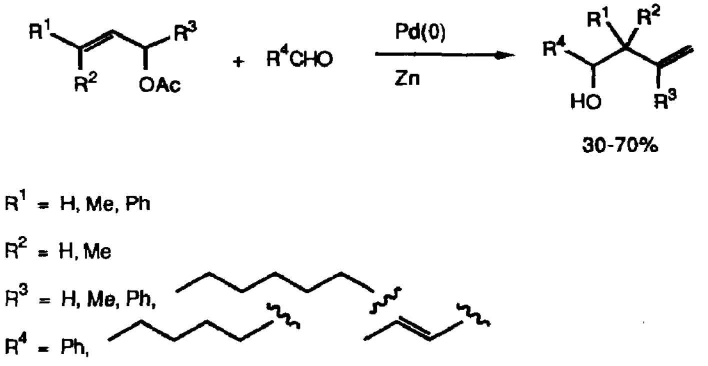
R'' = Et, iPr, $CO_2Et$, pAcOPh, tBu,

R''' = Et, Me, H

(304)

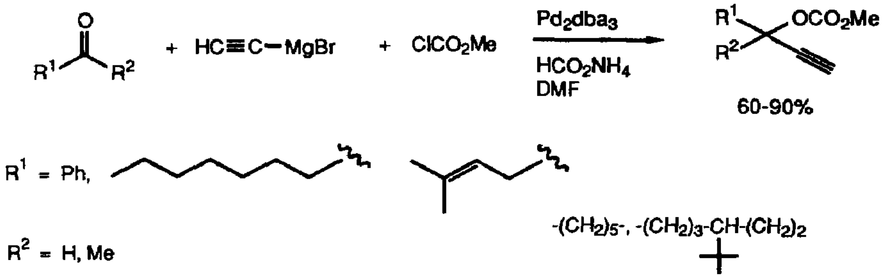


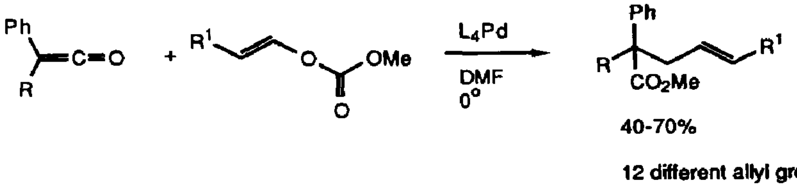
Palladium(0)/zinc systems promoted the alkylation of aldehydes by allyl acetates (equation 305) [345], while palladium(0)/formate systems catalyzed the reactions of acetylides with ketones (equation 306) [346]. Ketenes condensed with allyl carbonates in the presence of palladium(0) catalysts (equation 307) [347]. Palladium(0) also catalyzed the Wittig reaction between allylic alcohols and aldehydes [348]. Cobalt(II) chloride catalyzed the reaction between aldehydes and acetic anhydride (equation 308) [349].



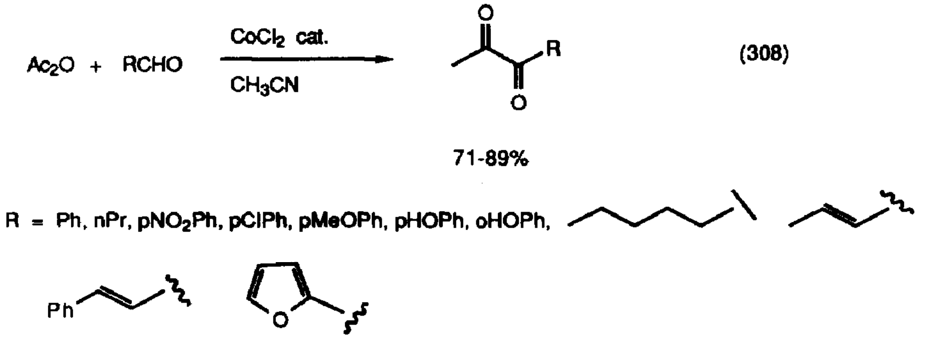
(305)

(306)





(307)

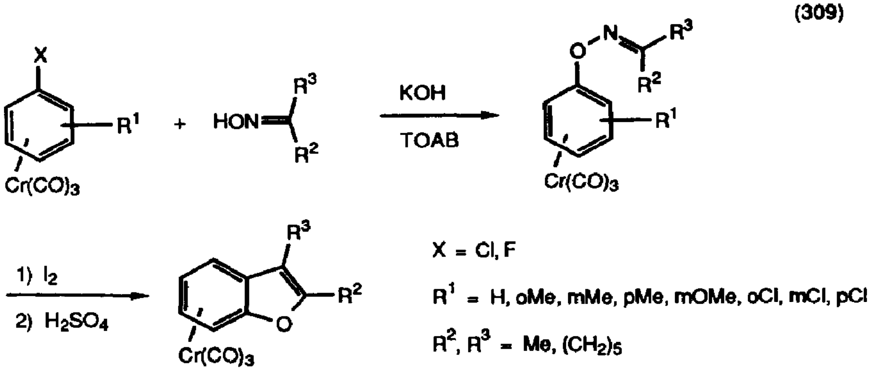


(308)

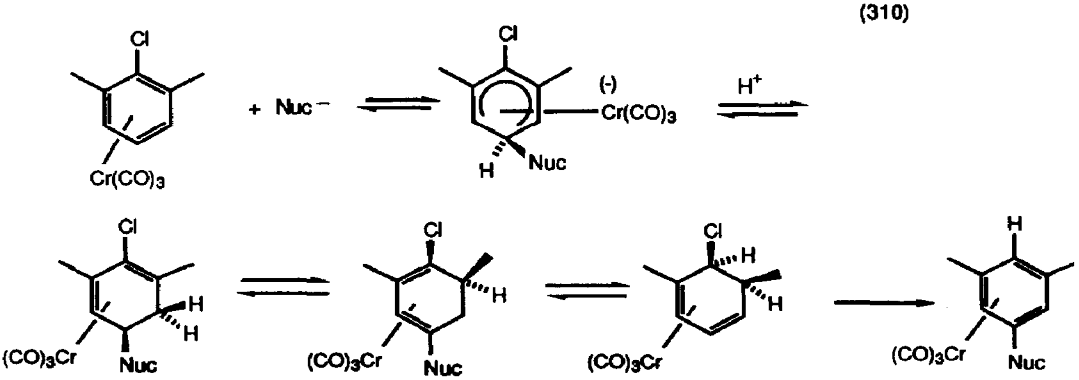
## 11. Alkylation of Aromatic Compounds.

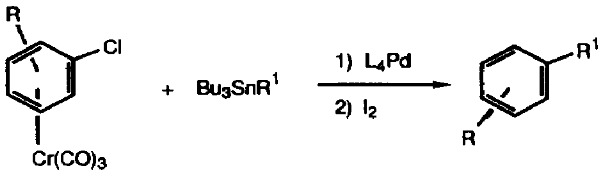
A review dealing with *ipso* and *tele* nucleophilic aromatic substitution on arenechromium tricarbonyl complexes (44 references) has appeared [350]. Benzofurans were prepared by nucleophilic attack of oximes on chromium-complexed aryl halides (equation 309) [351]. Nucleophilic attack on 2,6-dimethylchlorobenzene complexed to chromium occurred by a complex mechanism (equation 310) [352]. Substituent effects on the rate of replacement of chloride by methoxide on chromium complexed substituted chlorobenzenes were studied and yielded a Hammett plot with $\sigma^+_p = 4.39$ [353]. Chromium complexed aryl chlorides underwent facile Heck alkylation (equation 311) [359].

(309)



(310)





(311)

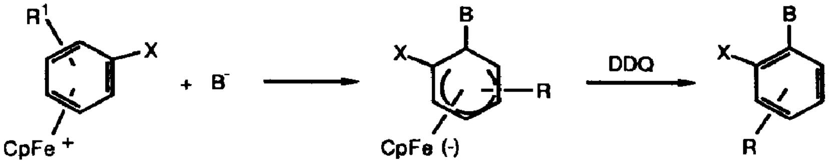
68-82%

R = pMe, pMeO, oMeO

$R^1$ = Bu,


Cationic iron arene complexes (equation 312) [355][356] and ruthenium complexes (equation 313) [357] as well as neutral rhenium pyrrole complexes (equation 314) [358] underwent nucleophilic aromatic substitution.

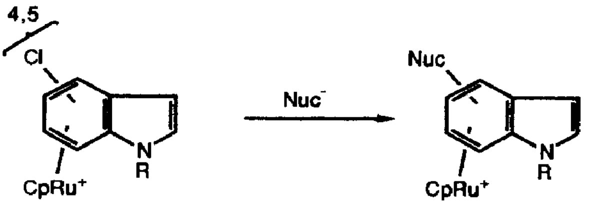
(312)



60-80%

B = $CN^-$, $^-CH_2COCH_3$

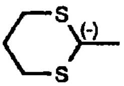
| | | | | | |
|---|---|---|---|---|---|
| X = 1-Cl | 1-Cl | 1-CN | $NO_2$ | COPh | $NO_2$ |
| $R^1$ = 4-Cl | 2-Cl | H | 4Me | H | H |



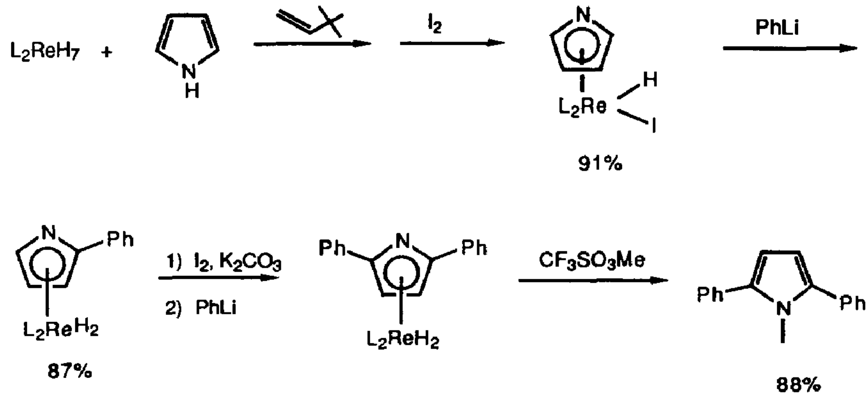
(313)

72-80%

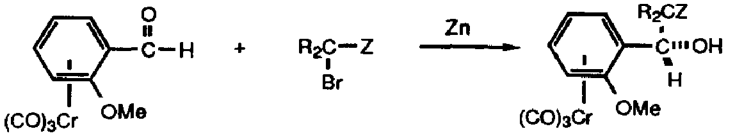
Nuc = $MeO^-$, $PhCH_2O^-$, $^-SCH_2CO_2^-$, $Me_2NH$,



(314)



Chromium-complexed arenes underwent a number of useful reactions at the benzylic position. Complexed benzaldehydes were alkylated by organozinc reagents and nitronates with a high degree of stereocontrol (equation 315) [359], (equation 316) [360]. Complexed chiral β-phenethyl amines were α-alkylated stereoselectively (equation 317) [361]. The regiospecificity of benzylic activation of chromium complexed arenes could not be explained by preferential attack at the carbon eclipsed by a carbon monoxide, but rather was determined by electronic effects [362]. Chromium complexed arenes underwent aldol condensations at the benzylic position (equation 318) [363], as well as cationic alkylations (equation 319) [364].



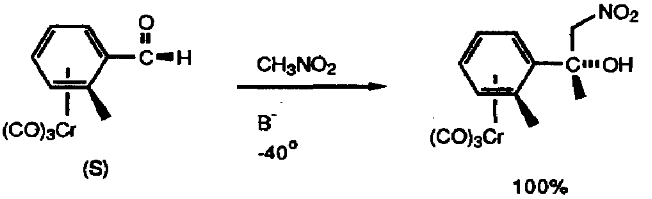
(315)

R = H, Me

Z = $CO_2Me$, $CO_2tBu$, $CO_2Et$, CN

70-80%

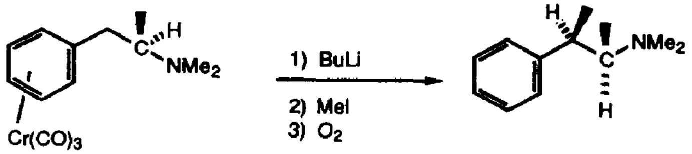
RR and SS only



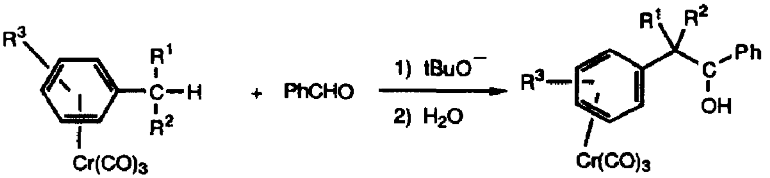
(316)

100%

97 / 3 1S,2S / 1S, 2R

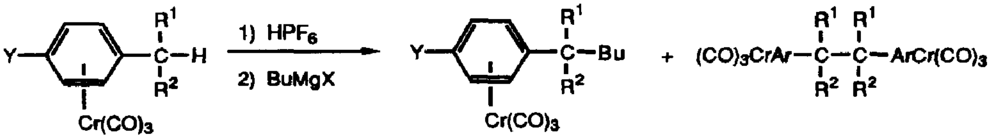


(317)

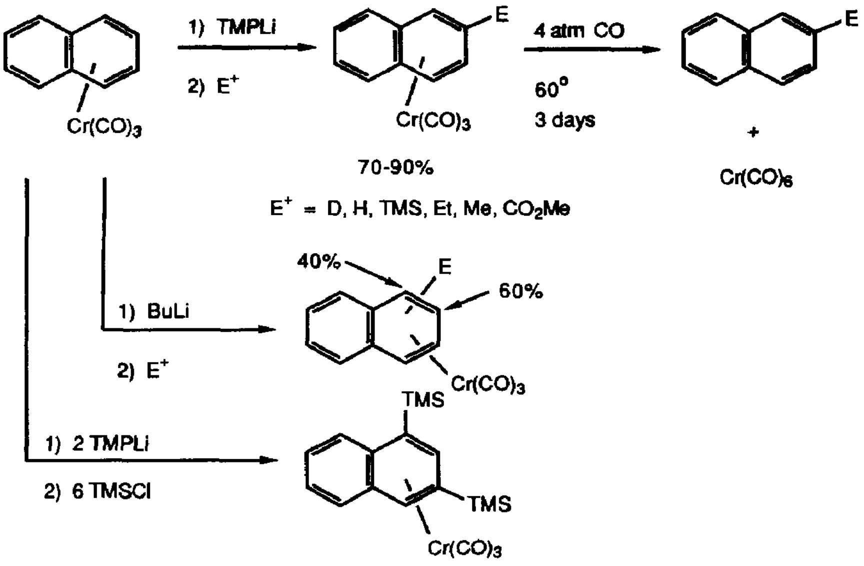


(318)

(319)

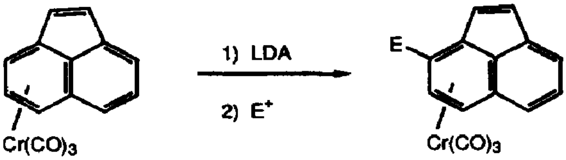


(320)



70-90%

$E^+$ = D, H, TMS, Et, Me, $CO_2Me$

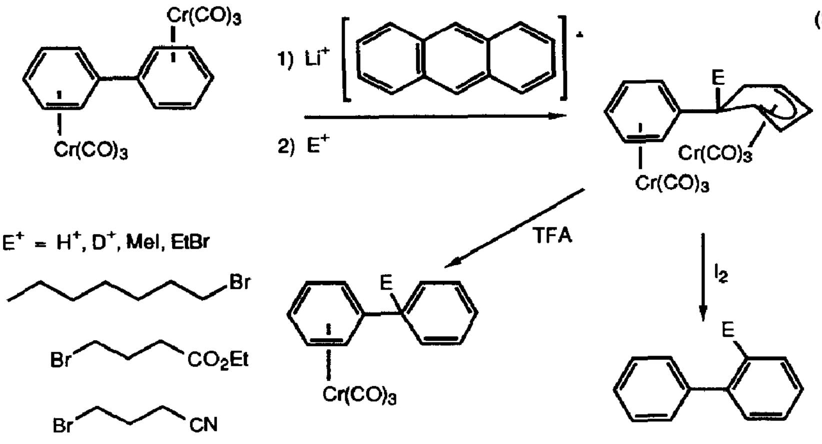


(321)

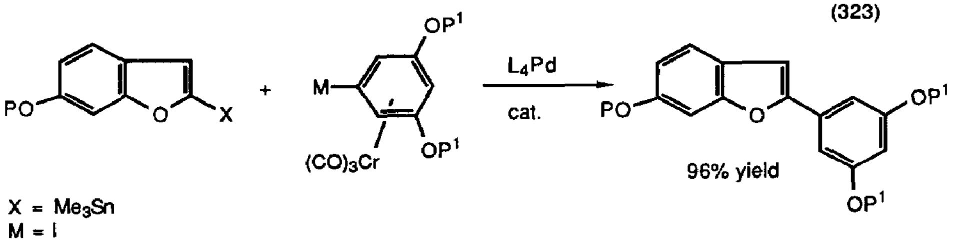
E = RX, RCHO, $Br_2$

Lithiation/alkylation of arenechromium tricarbonyl complexes has also been used to functionalize arenes (equation 320) [365], (equation 321) [366], (equation 322) [367], (equation 323) [368].

(322)

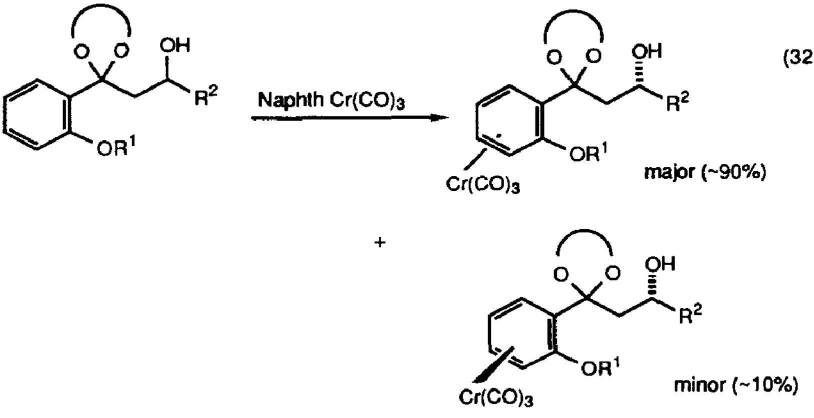


(323)



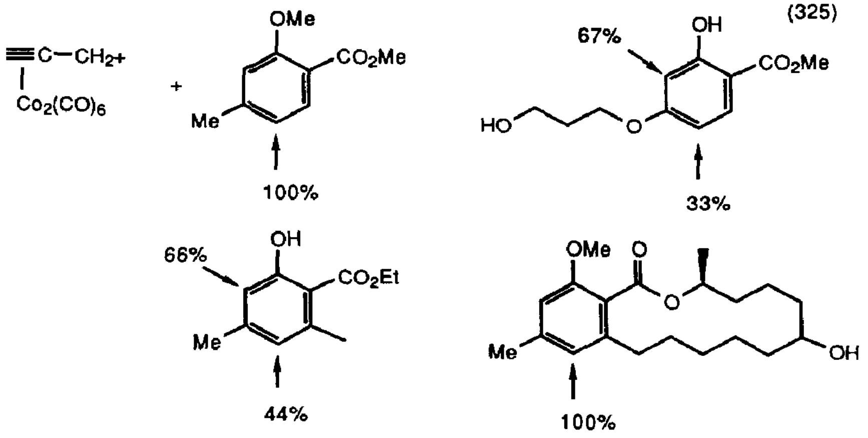
Remote hydroxyl groups controlled the diastereoselectivity of arene complexation by chromium hexacarbonyl (equation 324) [369]. The barriers to rotation of substituted diphenylmethanes and diphenyl ethers, and their mono- and bis $Cr(CO)_3$ complexes were studied [370].

(324)

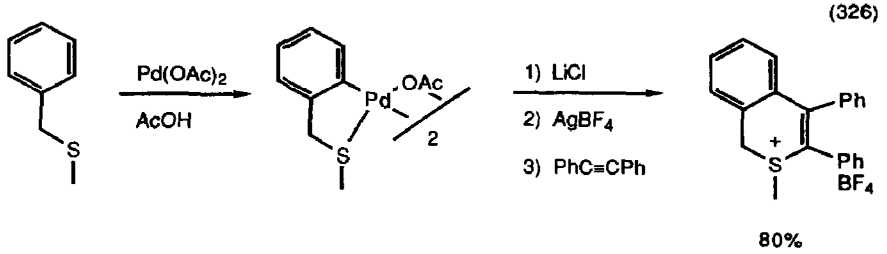


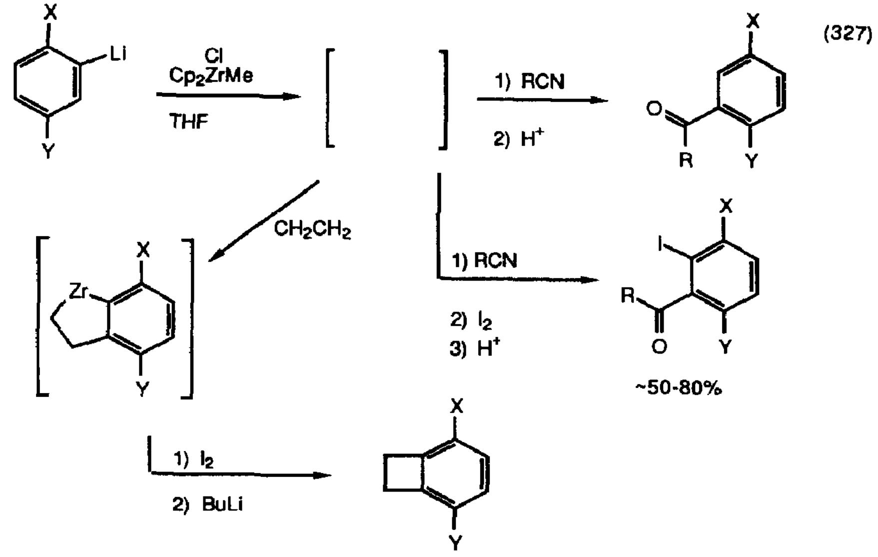
Except when ketal became very large.

Electron rich arenes were alkylated by cobalt-complexed propargyl cations (equation 325) [371]. Palladium acetate in acetic acid catalyzed the arylation of naphthoquinones [372]. Arenes were alkylated by o-palladation alkyne insertion (equation 326) [373]. Arylzirconium species were converted to a variety of substituted arenes (equation 327) [374].


(325)

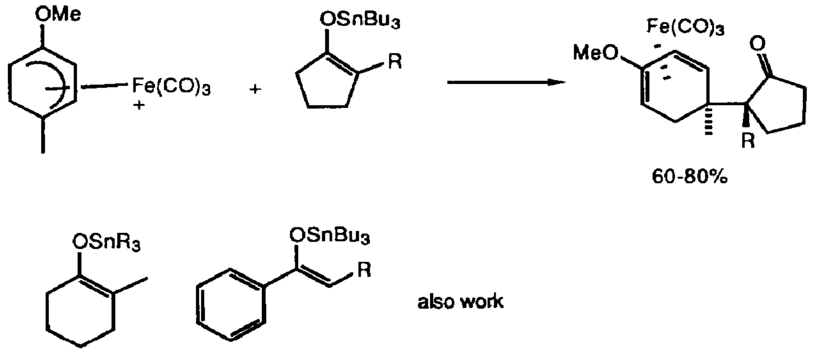
(326)



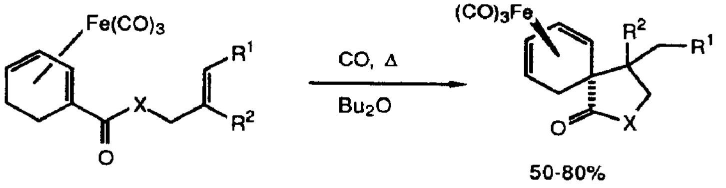

(327)

## 12. Alkylation of Diene and Dienyl Complexes

Transition metal pentadienyl chemistry has been reviewed (15 references) [375]. A nucleophilicity scale for the addition of forty different nucleophiles to cyclohexadienyliron cations has been developed [376]. A dissertation entitled "Synthesis of 3α,14-dihydroxy-12,13-epoxytrichothecanes via organoiron complex intermediates" has appeared [377]. Cyclohexadienyl iron complexes were alkylated by tin enolates (equation 328) [378]. Spirocyclic compounds were made by thermolysis of cyclohexadieneiron complexes having olefinic side chains (equation 329) [379][380]. Cationic cyclohexadiene molybdenum complexes were alkylated with modest asymmetric induction by chiral sulfilimine anions (equation 330) [381]. Cyclohexadienyl complexes of iron were prepared by oxidation of cyclohexadiene complexes (equation 331) [382].



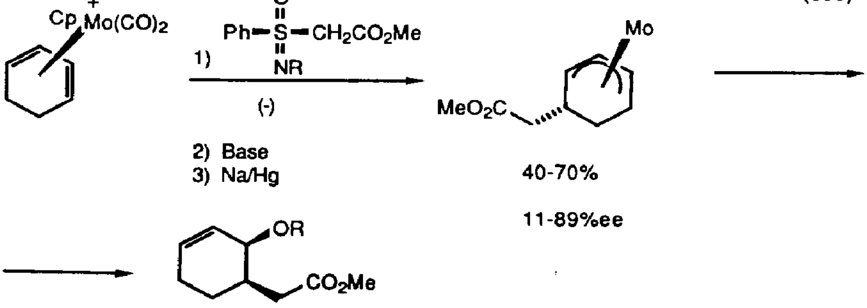
(328)



(329)

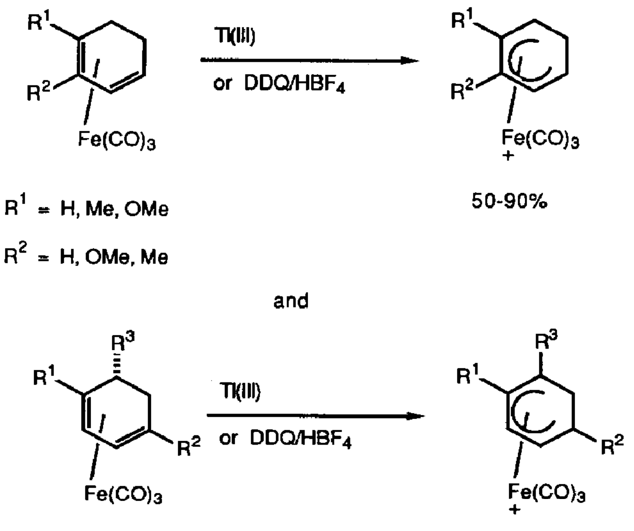
X = NPh, O

$R^1, R^2$ = H, Me



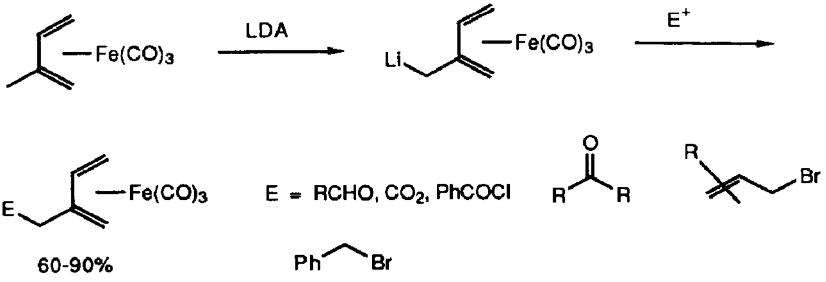
(330)

(331)

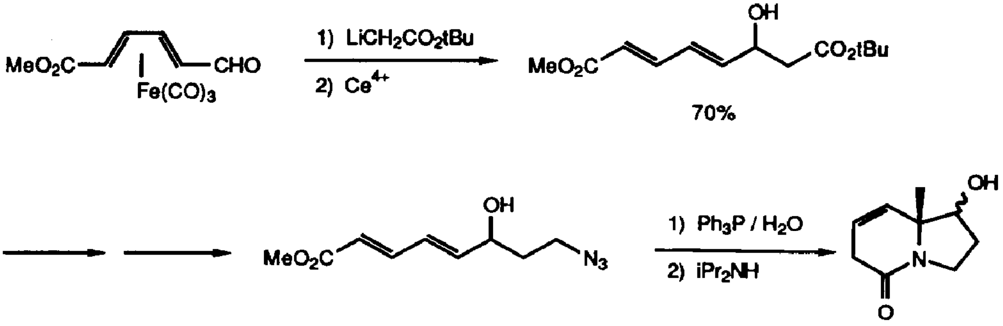


Isoprene complexes of iron were lithiated on the methyl group and alkylated by a variety of carbon electrophiles (equation 332) [383]. Iron-complexed diene aldehydes (equation 333) and ketones (equation 334) were homologated into bicyclic lactams [384]. Butadiene iron complexes underwent clean Friedel-Crafts acylation (equation 335) [385] and complexed dienylacetates underwent nucleophilic attack with retention (equation 336) [386]. A 1,3-diene portion of a polyene was protected from hydrogenation by complexation to iron (equation 337) [387]. An oxo bridged bicyclic diene iron complex was converted into 2-indanones (equation 338) [388]. Dienes inserted into iron carbene complexes to produce functionalized iron diene complexes (equation 339) [389]. Iron complexes of conjugated enones underwent a strange reaction with Grignard reagents (equation 340) [390].

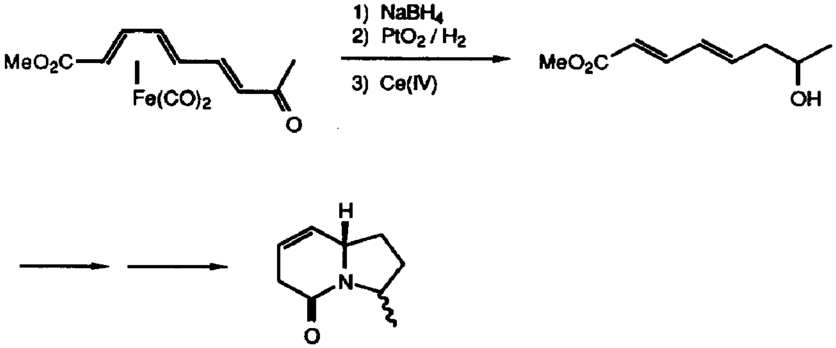
(332)



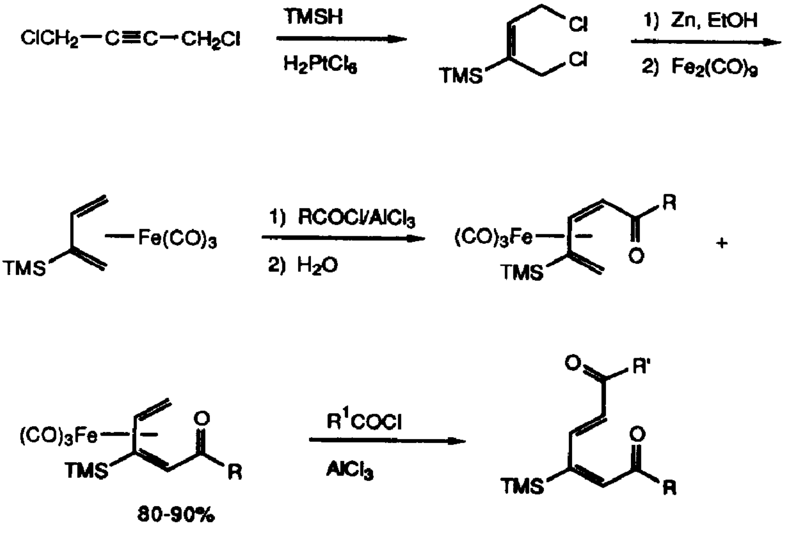
(333)

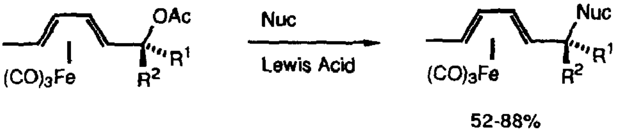

MeO2C
Fe(CO)3
CHO
1) LiCH2CO2tBu
2) Ce4+
OH
MeO2C
CO2tBu
70%
OH
MeO2C
N3
1) Ph3P / H2O
2) iPr2NH
OH
N
O

(334)


MeO2C
Fe(CO)2
O
1) NaBH4
2) PtO2 / H2
3) Ce(IV)
MeO2C
OH
H
N
O

(335)

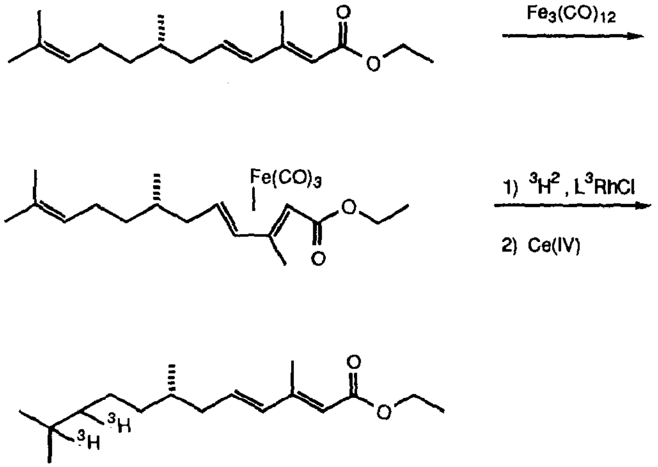

ClCH2—C≡C—CH2Cl
TMSH
H2PtCl6
Cl
Cl
TMS
1) Zn, EtOH
2) Fe2(CO)9
TMS
Fe(CO)3
1) RCOCl/AlCl3
2) H2O
(CO)3Fe
TMS
R
O
+
(CO)3Fe
TMS
O
R
80-90%
R1COCl
AlCl3
O
R'
TMS
O
R



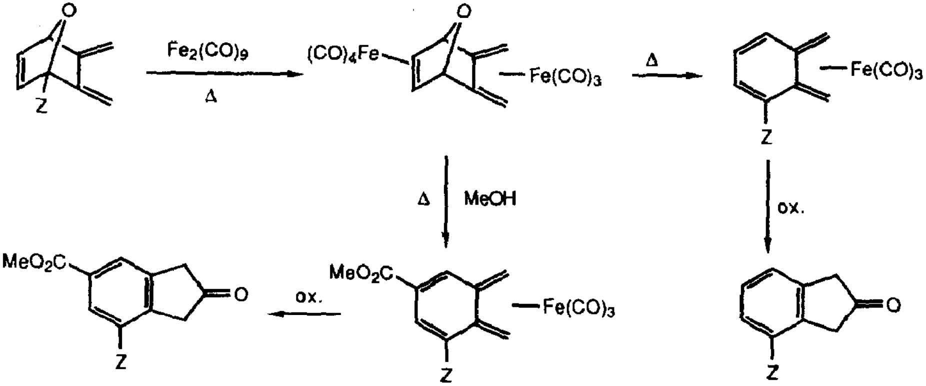
(336)

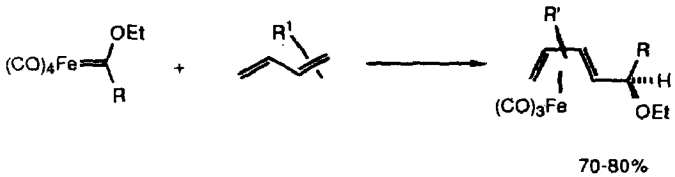
$R^1$ = H, Me, Et; $R^2$ = H, Me, Et; Nuc = $Me_3Al$,

(337)



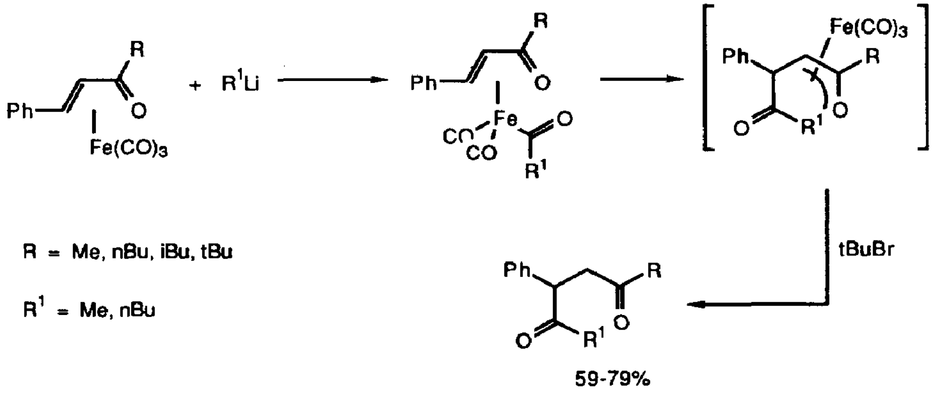
(338)





(339)

(340)



## 13. Metal-Carbene Reactions

Transition metal carbenes are finding increased use in organic synthesis, and several reviews of the field have appeared. They are listed below:

"Organic Synthesis using Metal Carbene Complexes" (10 references) [391].

"Carbene-metal Complexes. New Processes and Applications in Organic Synthesis" (20 references) [392][393].

"Reactions of Alkenes and Alkynes with Metal Carbenes, Metal Carbynes, and Other Metal Initiators" (dissertation) [394].

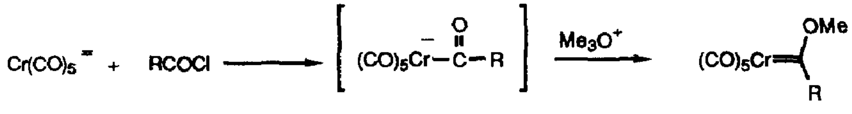
"Catalytic Reactions Containing Carbene or Carbyne Complexes" (43 references) [395].

"Selective Carbon-Carbon Bond-Forming Reaction of Carbenes. I" (15 references) [396].

(Methoxy)(methyl)(triphenylphosphine)(tetracarbonyl)chromium carbene complexes were more reactive toward alkynes than were pentacarbonyl complexes because the phosphine dissociated faster than carbon monoxide. The $\alpha$-protons were $10^6$ less acidic than the corresponding pentacarbonyl complex, and the conjugate base more reactive toward electrophiles [398]. Solvent, chelation, and concentration effects on the benzannulation reaction of chromium carbene complexes with acetylenes were studied in detail [399]. The development of carbene-like chemistry with terminal phosphinidene complexes has been reviewed (62 references) [400].

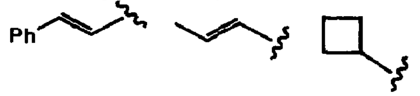
A number of new synthetic approaches to chromium carbene complexes have been developed. Acid chlorides were attacked by disodium pentacarbonyl chromate to give modest yields of alkoxy carbene complexes (equation 341) [401]. Aldol condensations at carbene $\alpha$-positions were promoted by trimethylsilyl chloride (equation 342) [402]. $\omega$-Acetylenic alcohols combined with unsaturated metal carbonyls to give cyclic alkoxy carbenes (equation 343) [403]. Phenyl cyclopropenes were converted to naphthols by chromium carbonyl (equation 344) [404].

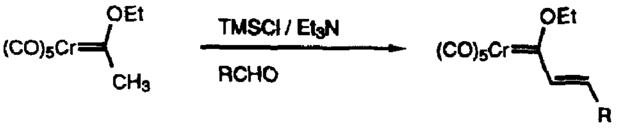
(341)



40-60%

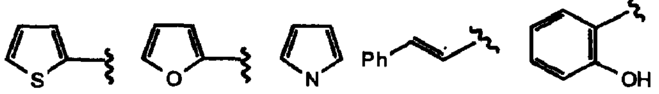
R = Ph, oMePh, 2,6-$Cl_2$Ph, 3,4,5-$MeO_3$Ph, 3-$ClCH_2$Ph, oAcOPh,
2,5-$(AcO)_2$Ph, Me, tBu,



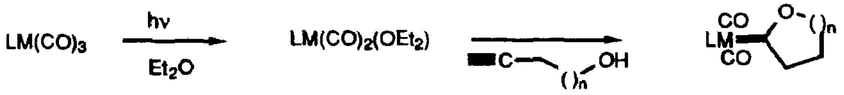


(342)

R = Ph



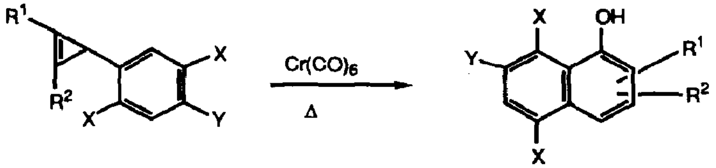
(343)



n = 1, 2

L = $(CO)_3$, $\eta^5C_5H_4Me$

M = Cr, W, Mn



(344)

35-76%

$R^1$ = Et, tBu, iPr, Ph, Me

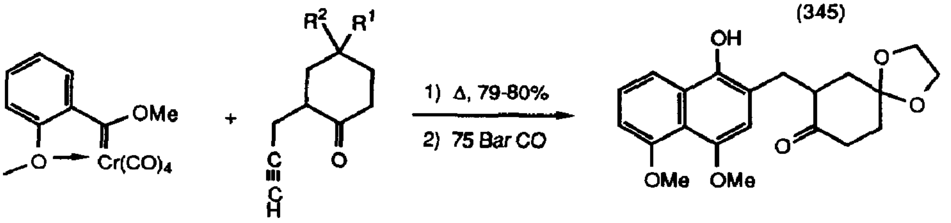
$R^2$ = Et, Me, H

X = H, OMe

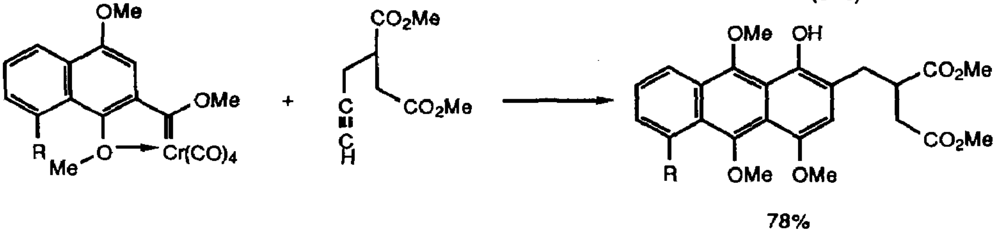
Y = OMe, H

Applications of chromium carbene chemistry to the synthesis of naphthoquinone derivatives (equation 345) [405], (equation 346) [406], and β-lactams (equation 347) [407] have been reported.

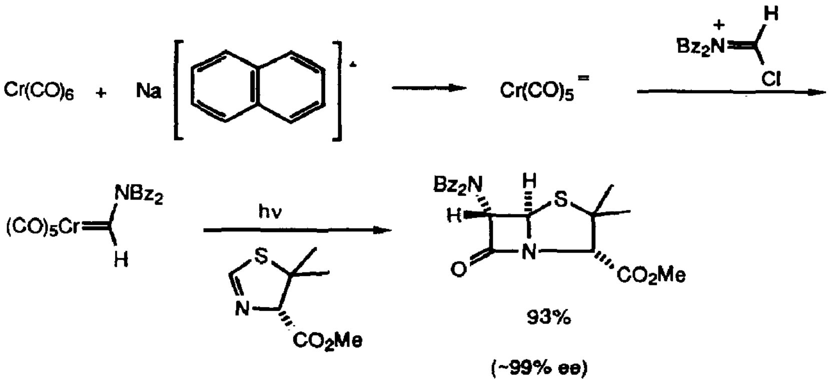
(345)



(346)

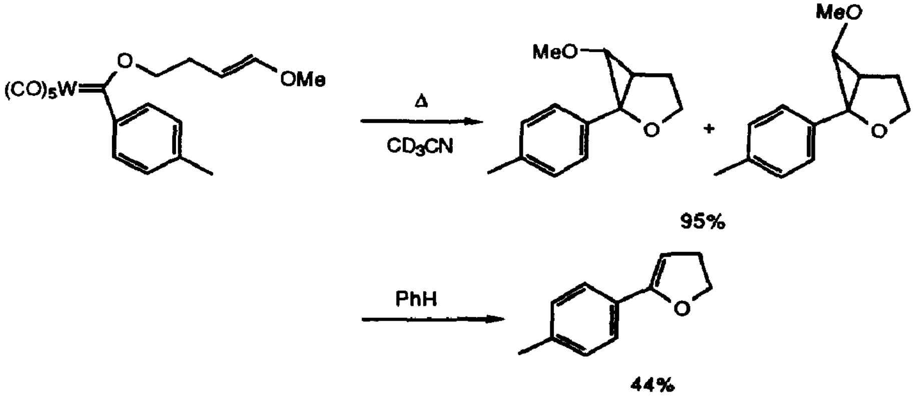


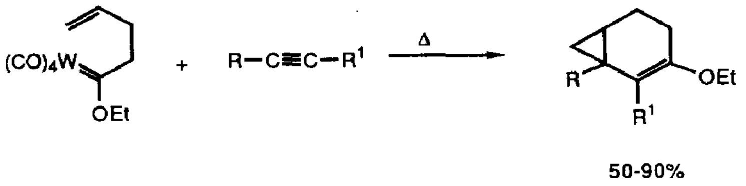
(347)



Carbene complexes containing remote olefins underwent thermal cyclopropanation reactions (equation 348) [408], (equations 349 and 350) [409].

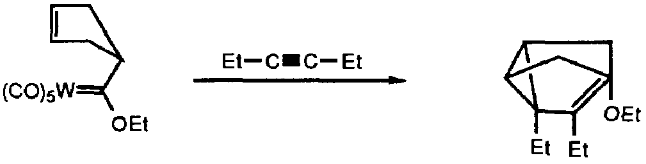
(348)





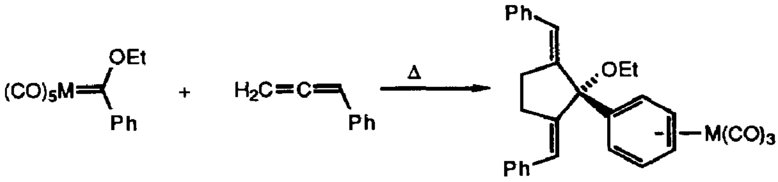
(349)

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

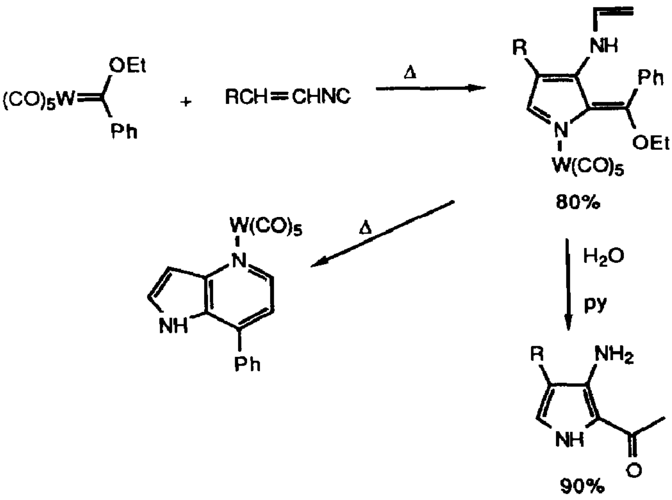


(350)

Allenes (equation 351) [410] and isonitriles (equation 352) [411], (equation 353) [412], (equation 354) [413], (equation 355) [414], (equation 356) [415] underwent reaction with carbene complexes to give a variety of unusual organic compounds.



(351)



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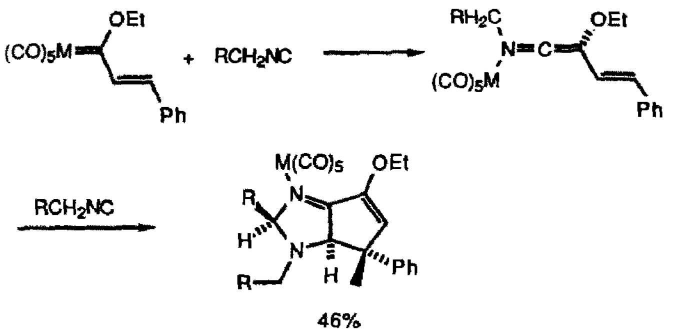

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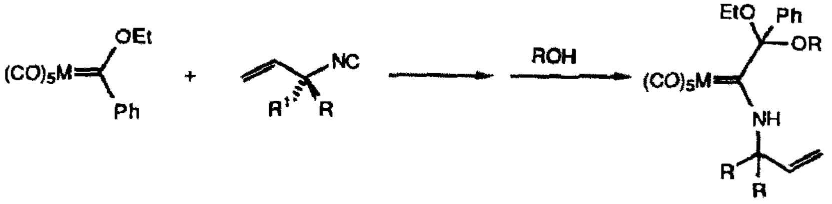

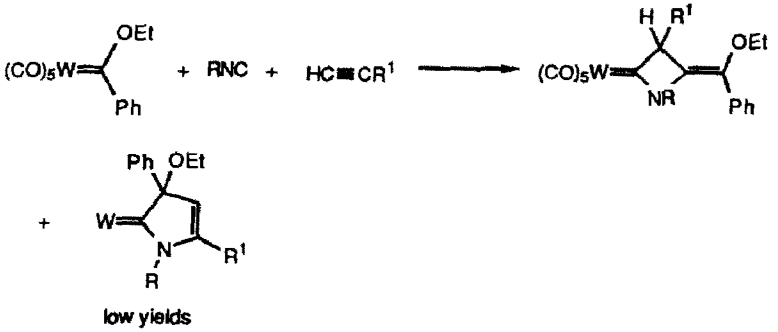

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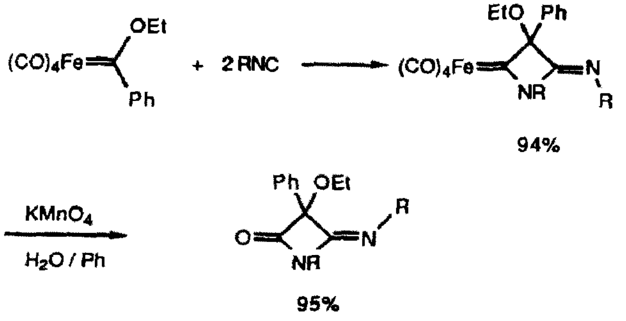
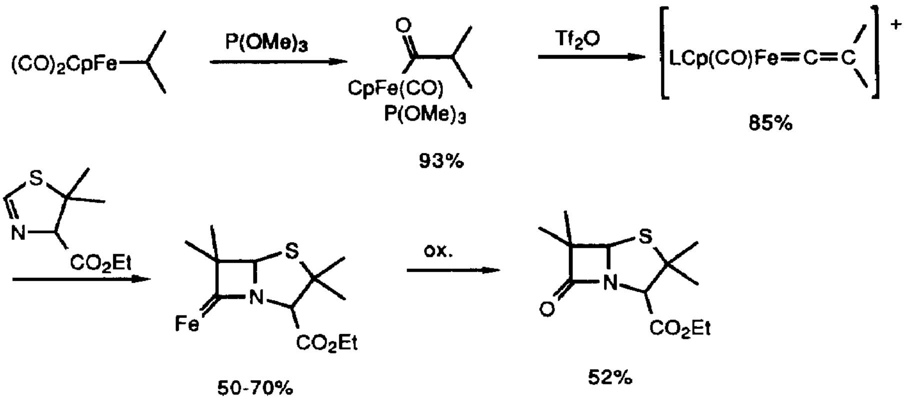


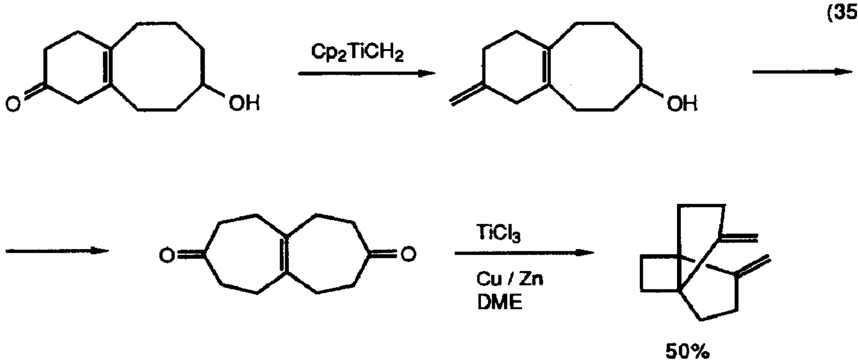
(356)

β-Lactams were prepared by cycloaddition of imines to iron vinylidene complexes, followed by oxidation (equation 357) [416]. Tebbe's reagent was used to methylenate a β,γ-unsaturated ketone without rearrangement (equation 358) [417]. The mechanism of titanocene metallocyclobutane cleavage and the nature of the reactive intermediate was studied [418].

(357)



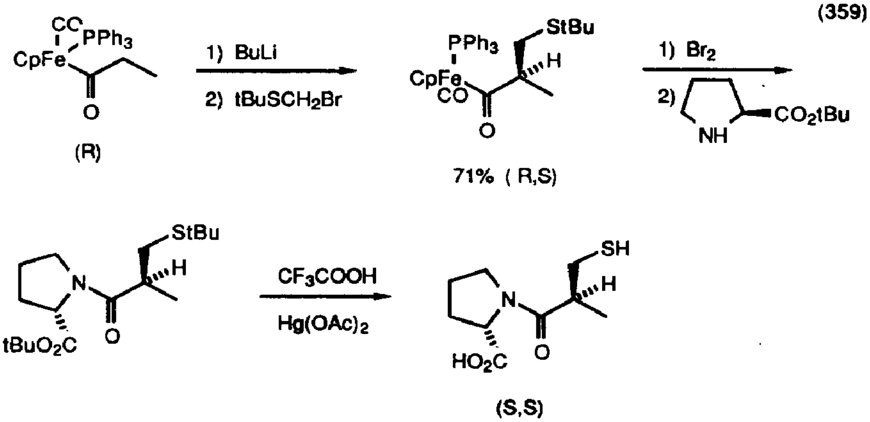
(358)



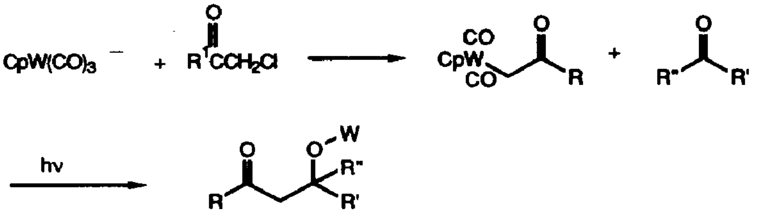
## 14. Alkylation of Metal Acyl Enolates

A review entitled "Synthesis and stereoselectivity reactions of α,β-unsaturated acyl ligands bound to the chiral auxilliary CpFe(CO)PPh3" has appeared (76 references) [420]. (-)-Captopril was synthesized using this chemistry (equation 359) [421]. Tungsten enolates were useful in aldol condensation chemistry (equation 360) [422].

(359)



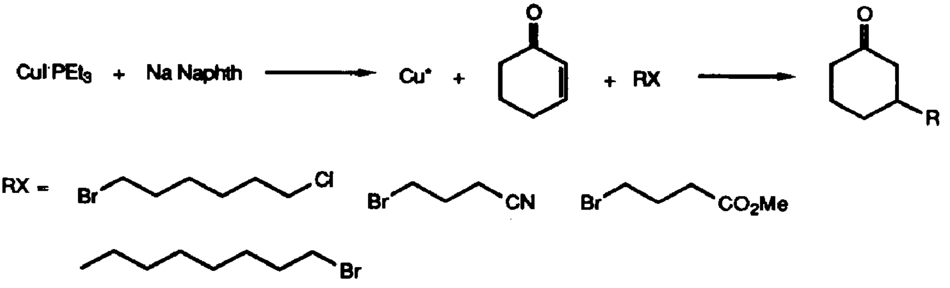
(360)



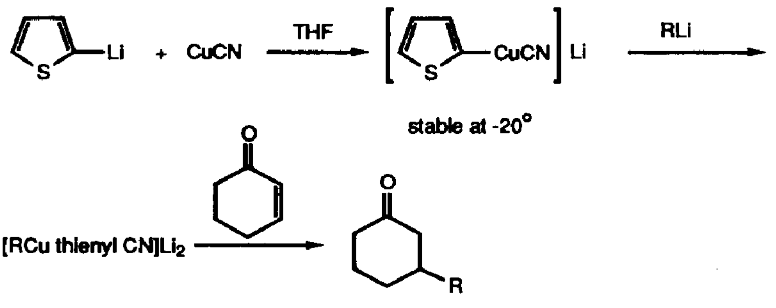
## B. Conjugate Addition

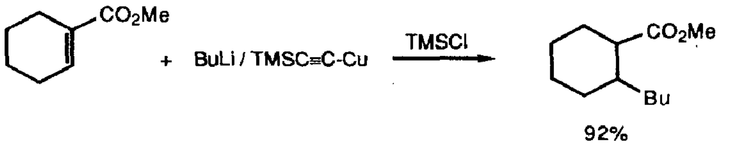
Organocopper chemistry continues to provide the method of choice for the 1,4-alkylation of conjugated enones. A variety of copper(I) salt precursors to dialkylcuprates were examined, and, in general, copper(I) cyanide and copper(I) bromide dimethylsulfide were superior [423]. Active copper species were generated *in situ* by the reaction of halides with copper(I) iodide triethylphosphine reduced with sodium naphthalenide (equation 361) [424]. A variety of active functional groups were tolerated. A thienyl copper cyanide precursor was stable at -20° for over 2 months (equation 362) [425]. The most efficient reagent for conjugate alkylation of α,β-unsaturated esters was that derived from copper(I) trimethylsilyl acetylide, organolithium, and trimethylsilyl chloride (equation 363) [426].

(361)



(362)





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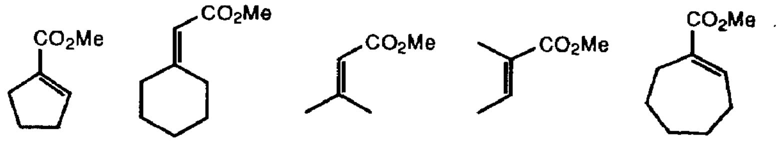
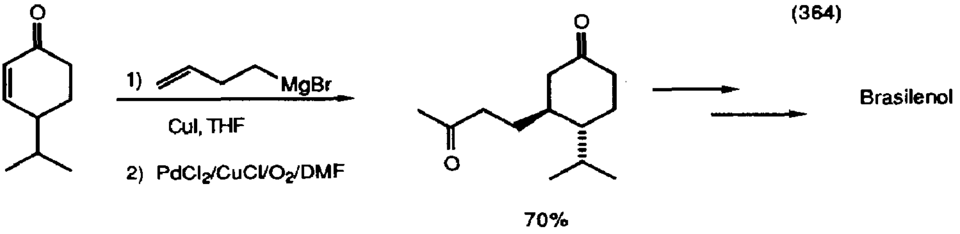
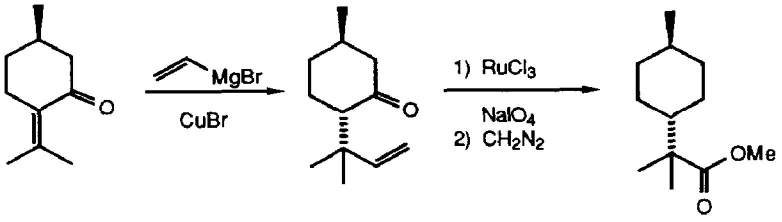


work

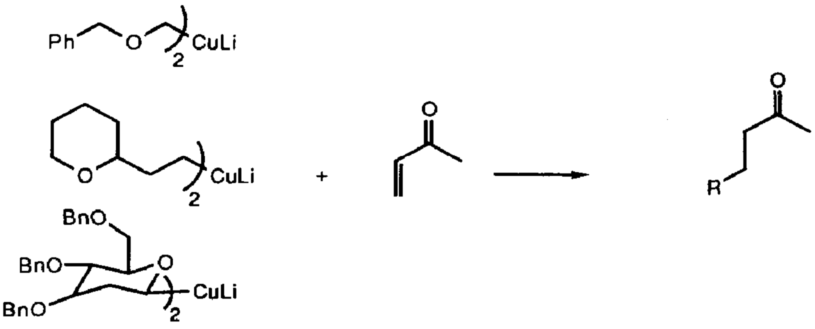
Copper catalyzed Grignard reactions efficiently alkylated unsaturated terpenoid enones (equation 364) [427], (equation 365) [428]. Alkoxymethyl cuprates have been prepared, and they were effective conjugate alkylation reagents (equation 366) [429], (equation 367) [430]. β-Stannylation of conjugated enones was carried out using tin-copper reagents (equation 368) [431].



(364)

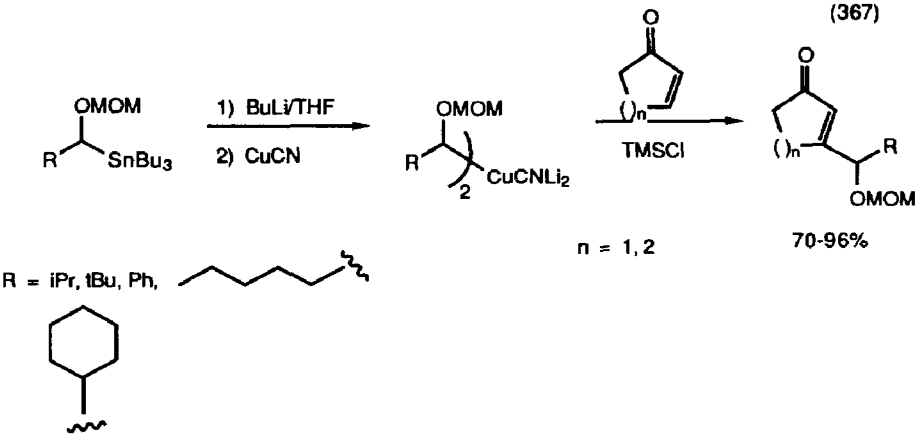


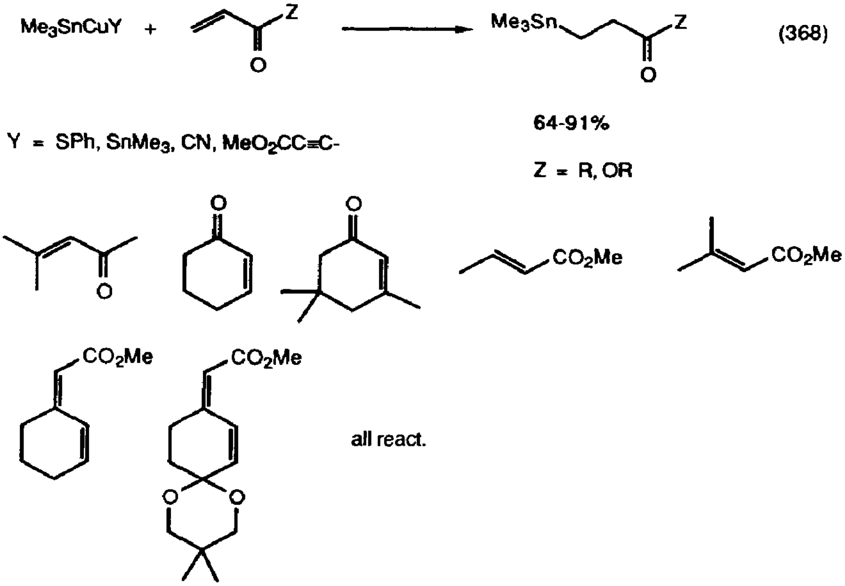
(365)



(366)

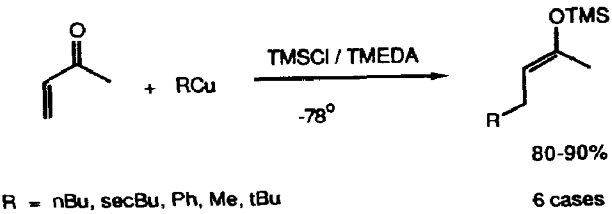
(367)



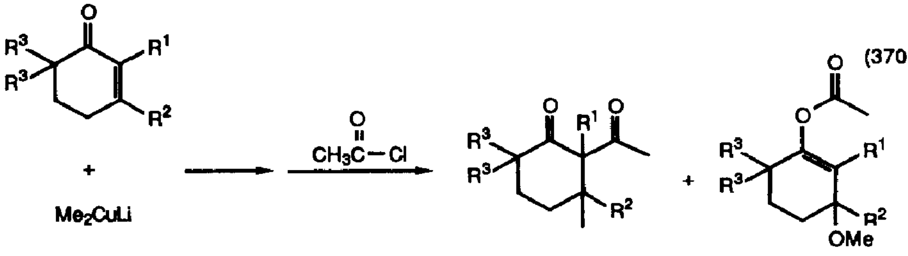


(368)

Conjugate addition followed by trapping of the enolate by an electrophile continued to be a useful process. Trimethylsilyl enol ethers (equation 369) [432], β-dicarbonyl compounds (equation 370) [433] and prostaglandins (equation 371) [434] were prepared this way.



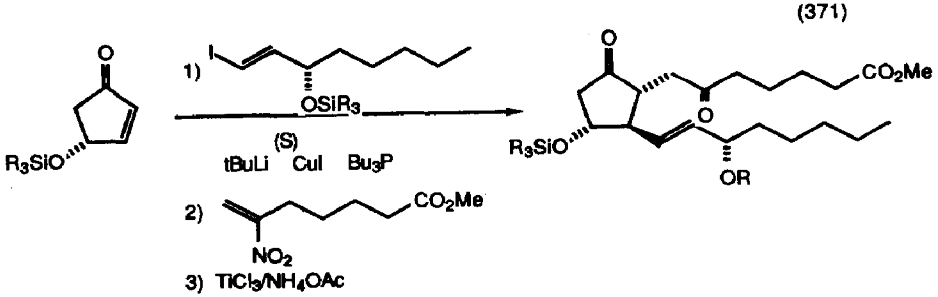
(369)



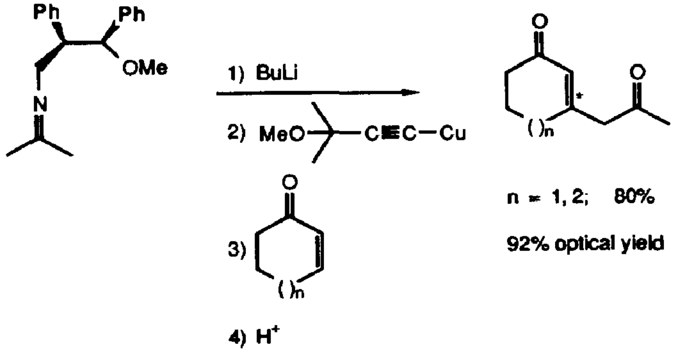
(370)

| | | |
|---|---|---|
| R's = H | 87 | 0 |
| $R^1$ = Me, $R^2$, $R^3$ = H | 43 | 40 |
| $R^2$, $R^3$ = alkyl | | 90 |

(371)



Chiral induction in the conjugate addition process has been achieved by the use of chiral ligands on copper (equation 372) [435], (equation 373) [436] and by the use of chiral substrates (equation 374) [437], (equation 375) [438], (equation 376) [439].



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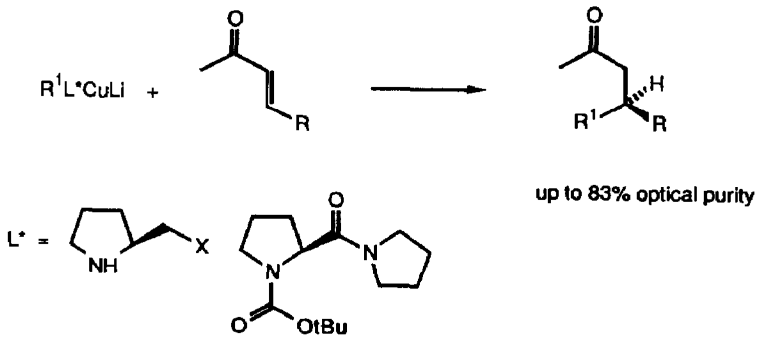
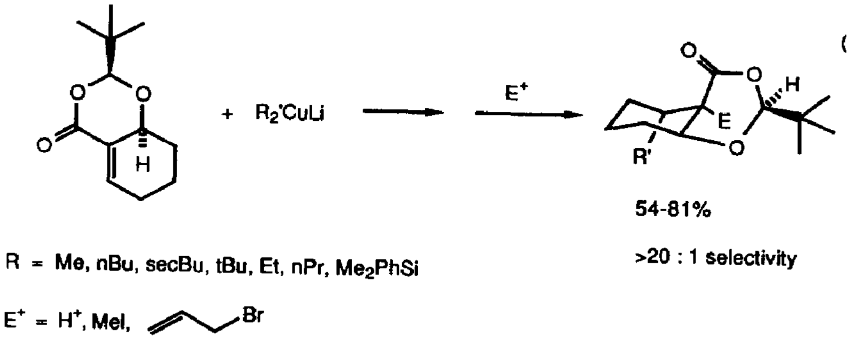


(373)

up to 83% optical purity



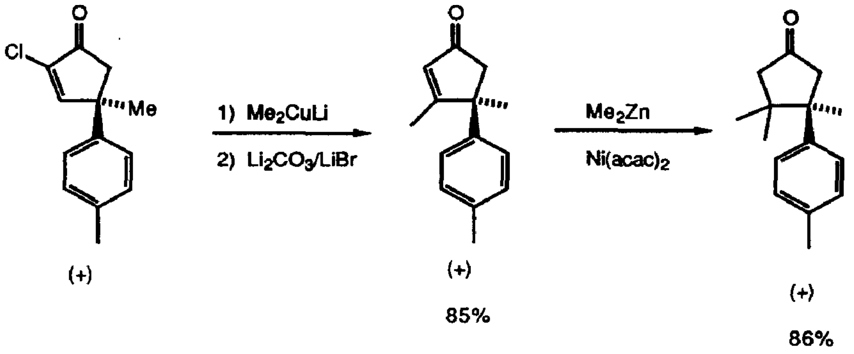
(374)

54-81%

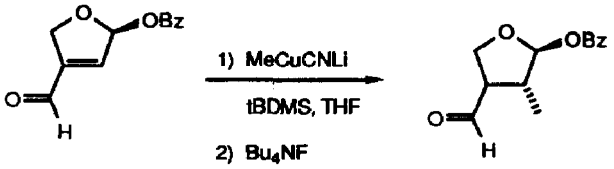
>20 : 1 selectivity

R = Me, nBu, secBu, tBu, Et, nPr, Me$_2$PhSi

E$^+$ = H$^+$, MeI,



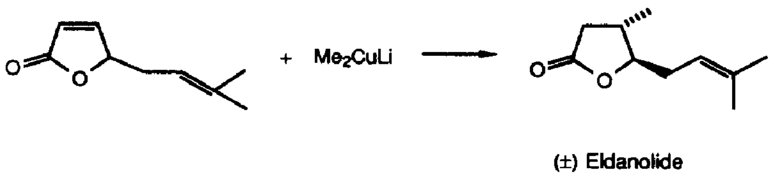
(375)



(376)

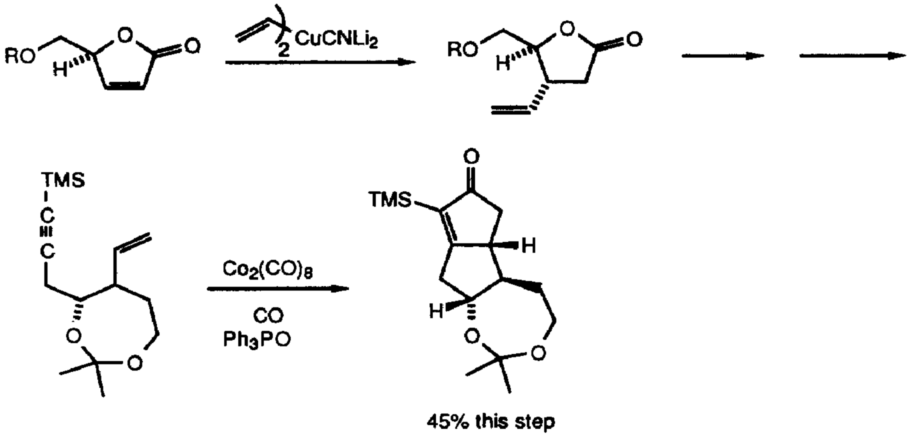
94% de

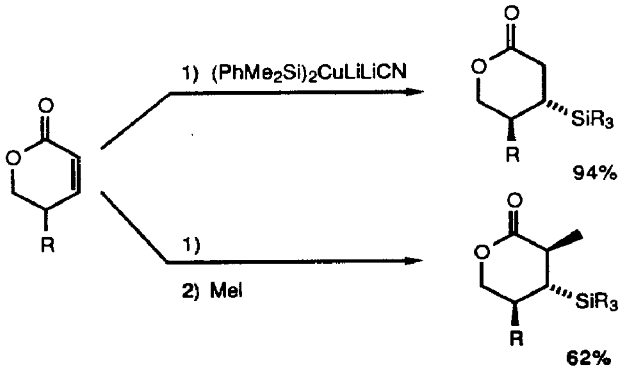
α,β-Unsaturated lactones underwent efficient conjugate alkylation by organocuprate reagents (equation 377) [440], (equation 378) [441], (equation 379) [442].



(377)

(378)

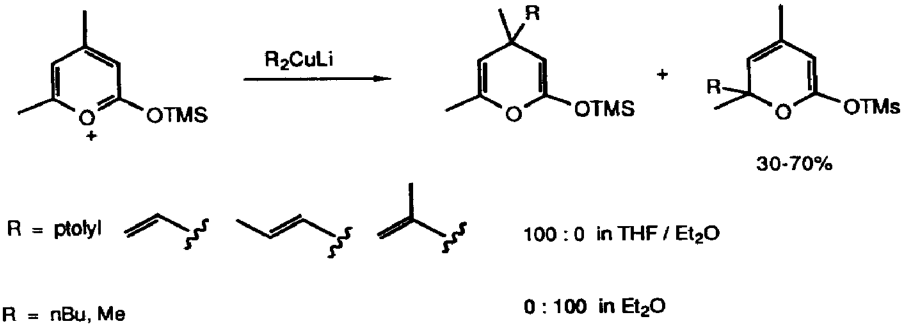


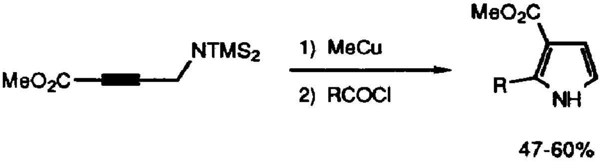


(379)

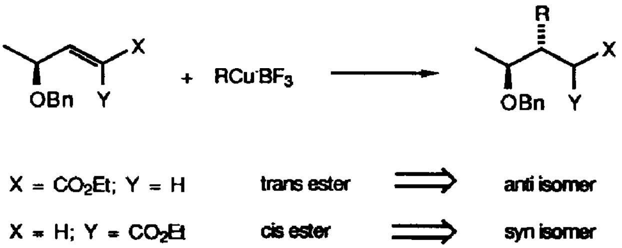
Pyrylium salts underwent 1,4- and 1,6-alkylation by organocuprates (equation 380) [443][444]. Pyrroles were made by a process involving conjugate alkylation of an ynone (equation 381) [445]. γ-Benzyloxyenones underwent stereoselective conjugate addition with alkylcopper species (equation 382) [446], (equation 383) [447].

(380)



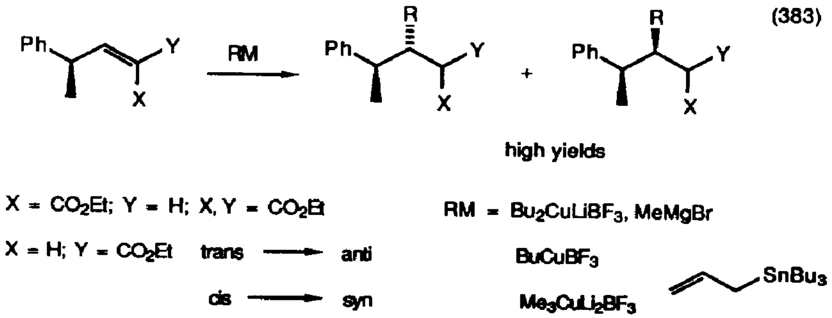


(381)



(382)

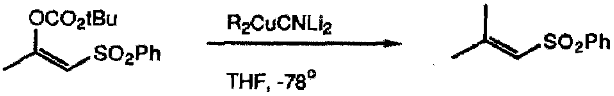
(383)



The reagent $Li_2R_2CuCN$ was the best complex for 1,4-addition to acetylenic trifluoromethyl ketones (equation 384) [448]. Vinyl sulfones underwent efficient 1,4-alkylation by organocopper reagents (equation 385) [449], (equation 386) [450]. Diastereoselectivity of γ-chiral-α,β-unsaturated sulfoxides and sulfones in the Michael reaction with lithium dimethylcuprate was the topic of a dissertation [451]. Chiral α,β-unsaturated aldehyde acetals underwent ring opening by vinyl cuprates with high diastereoselectivity (equation 387) [452].

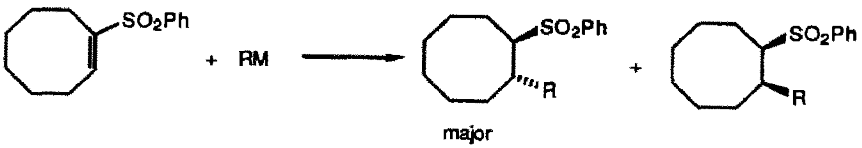
(384)

R = Me, nBu, tBu



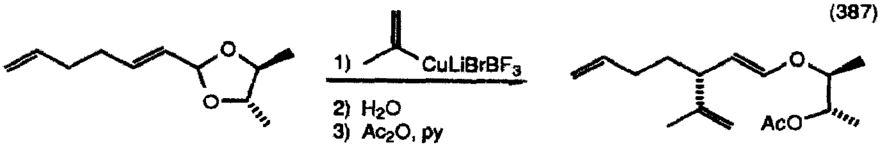
(385)

(386)



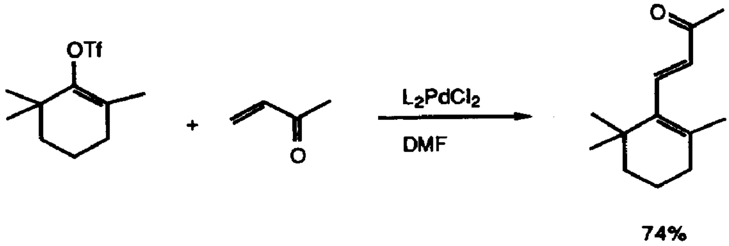
RM = MeLi, PhLi, BuLi, tBuLi, MeMgBr, $Me_2CuLi$, $Ph_2CuLi$, $MeCuCl_2$, $MeTi(OiPr)_3$

(387)

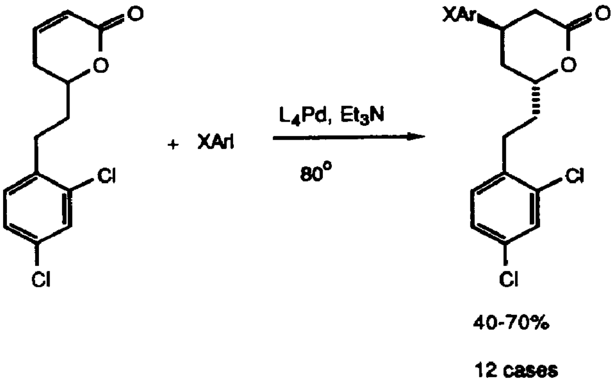


79% 60-95% de

Palladium catalyzed the 1,4-addition of vinyl triflates (equation 388) [453], aryl halides (equation 389) [454], and acetylenic iodides (equation 390) [455], to conjugated enones. Organolithiums added in a Michael sense to iron-complexed methyl acrylate (equation 391) [456]. Alkynes (equation 392) [457] and alkenes (equation 393) [458] were converted to conjugated enones via manganese chemistry. Molybdenum hexacarbonyl catalyzed the conjugate reduction of α,β-unsaturated carbonyls by $PhSiH_3$ (equation 394) [459].

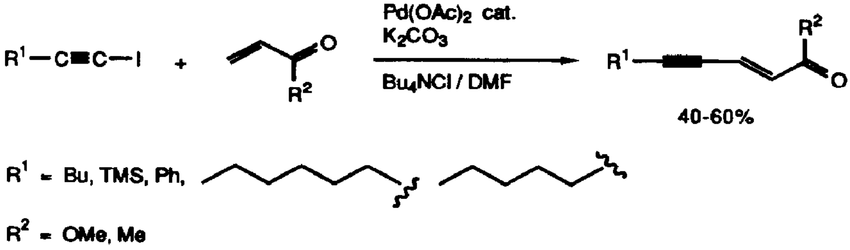


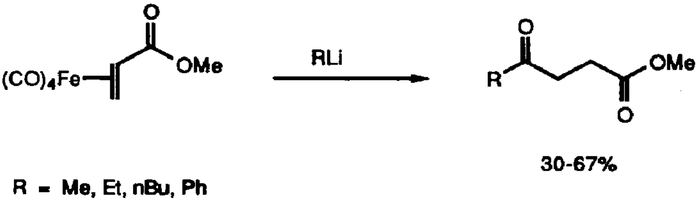
(388)



(389)

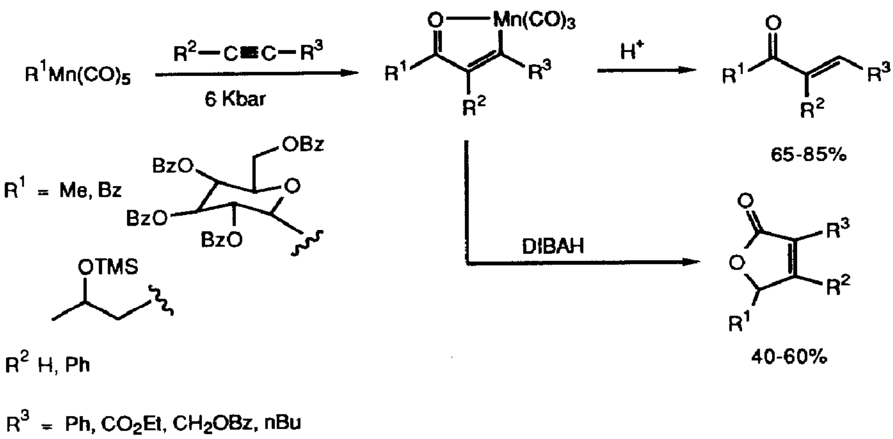
(390)



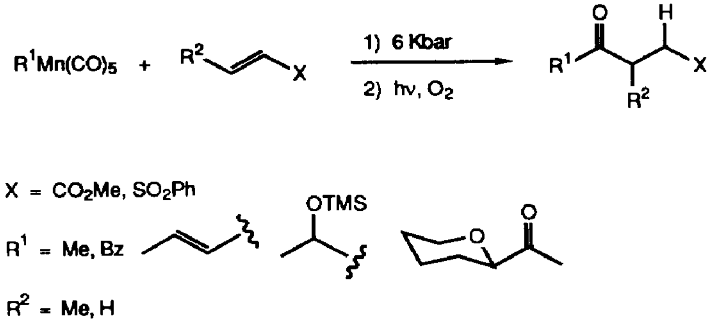


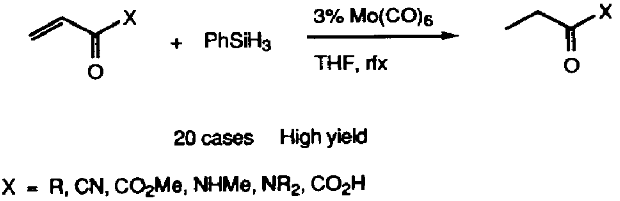
(391)

(392)



(393)





(394)

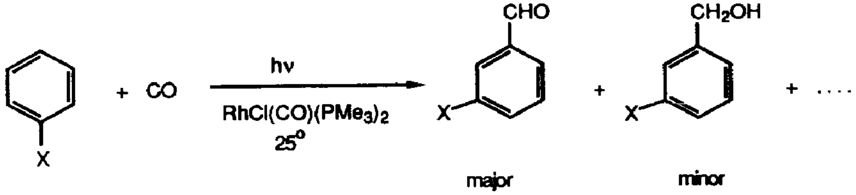
## C Acylation Reactions

### 1. Carbonylation of Alkenes, Alkanes, and Alkynes

Reviews entitled "Liquid phase carbonylation reactions using cobalt catalysts" (97 references) [460]; "Study of carbonylation for the synthesis of α-amino acids" (110 references) [461]; and "Recent developments in cobalt-catalyzed carbonylation" (38 references) [462] have appeared.

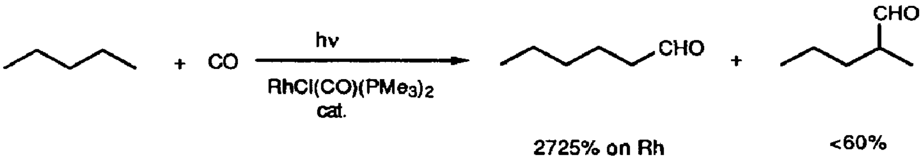
Photolysis of arenes (equation 395) [463][464] and alkanes (equation 396) [465] in the presence of carbon monoxide and a rhodium(I) catalyst led to formylation in low yield.

(395)

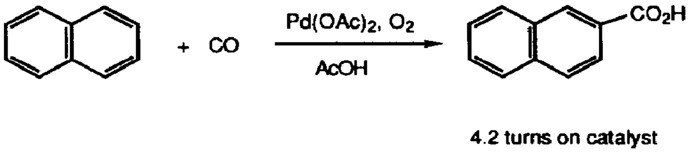


X = H, Me, MeO, Cl, CN

(396)



Naphthalene was catalytically carboxylated and arenes were stoichiometrically formylated using palladium(II) catalysts (equation 397) [466], [467].



(397)

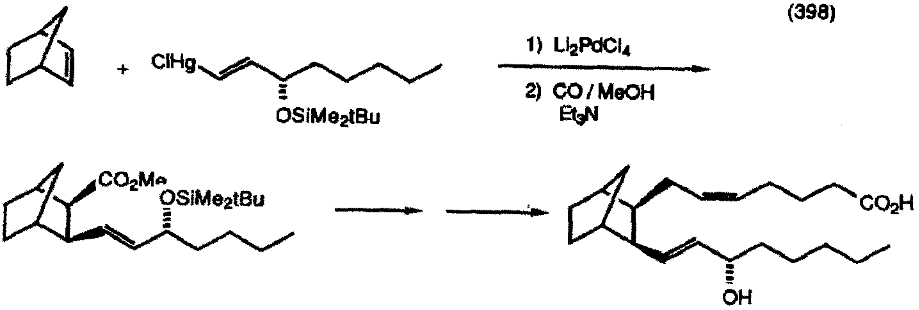
$$\mathrm{ArH} + \mathrm{CO} \xrightarrow[\mathrm{TFA}\quad 25^{\circ}]{1\ \mathrm{eq.}\ \mathrm{Pd(OAc)_2}} \mathrm{ArCO_2H}$$

28-60%

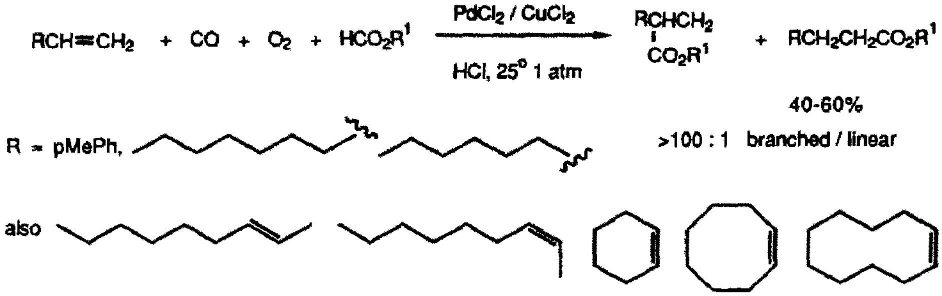
Ar = PhH, $PhCH_3$, PhOMe, naphth

Olefins were oxidatively carbonylated over heterogeneous palladium catalysts [468]. Norbornene was carboacylated to produce prostaglandin analogs using palladium(II) complexes (equation 398) [469]. Palladium(II) chloride/copper(II) chloride systems oxidatively carbonylated olefins to give a high branched to linear ratio (equation 399) [470].

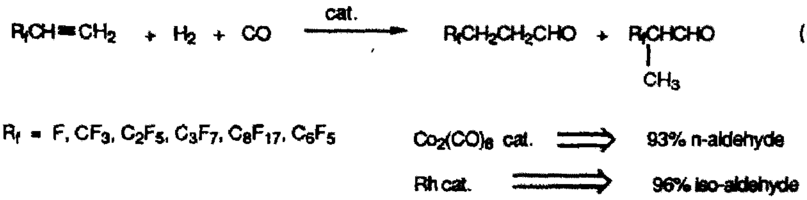
(398)



(399)



Cobalt and rhodium catalyzed hydroformylation of fluorinated alkenes has been extensively investigated (equation 400) [471]. Hydroformylation of substituted styrenes using a chiral platinum catalyst resulted in good asymmetric induction, but poor branched to linear ratios (equation 401) [472]. A new olefin homologation procedure involving hydroformylation followed by reaction with polymer bound phosphorous ylids has been devised (equation 402) [473].

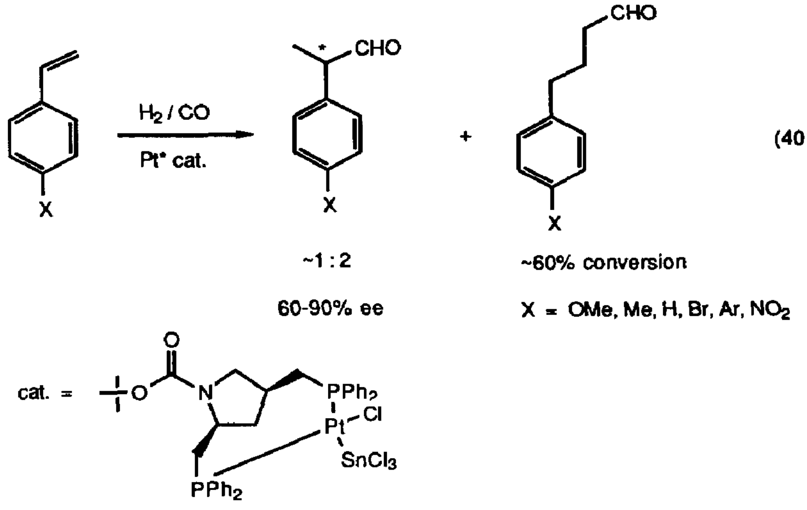


$$R_fCH{=}CH_2 + H_2 + CO \xrightarrow{\text{cat.}} R_fCH_2CH_2CHO + \underset{\displaystyle CH_3}{R_fCHCHO} \qquad (400)$$

$R_f$ = F, $CF_3$, $C_2F_5$, $C_3F_7$, $C_8F_{17}$, $C_6F_5$

$Co_2(CO)_8$ cat. $\Longrightarrow$ 93% n-aldehyde

Rh cat. $\Longrightarrow$ 96% iso-aldehyde



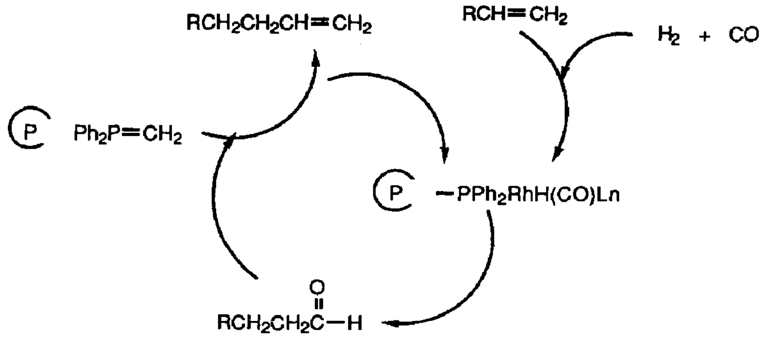
(401)

~1 : 2

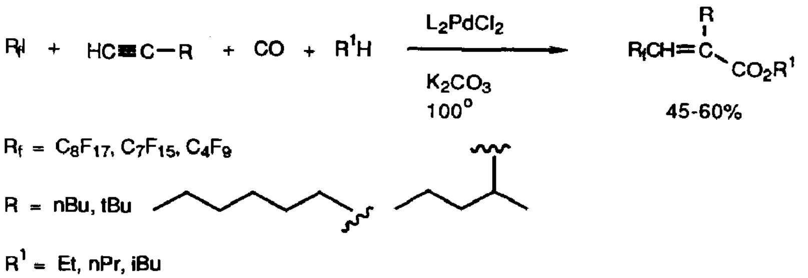
60-90% ee

~60% conversion

X = OMe, Me, H, Br, Ar, NO2



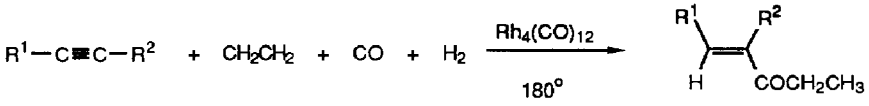
(402)



(403)

A mathematical model for the palladium-catalyzed methoxycarbonylation of acetylenic alcohols has been devised [474]. The complex $[Pd(Ph_3P)Cl]_2Co$ catalyzed the carboxylation of acetylene to methyl propiolate [475]. Homogeneous platinum complexes in alcoholic media catalyzed the carbonylation of acetylene [476]. Palladium complexes catalyzed the perfluoroalkylation/ carbonylation of alkynes (equation 403) [477]. Rhodium carbonyls catalyzed the conversion of alkynes, ethylene and carbon monoxide into ethyl acrylates (equation 404) [478].

(404)

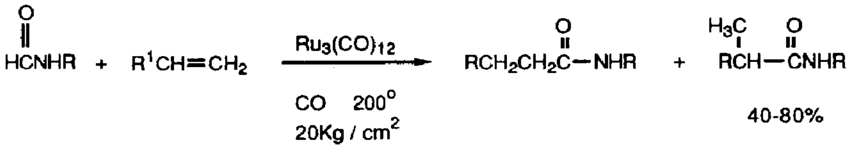


$R^1$ = Ph, Me, MeOCH$_2$, nBu

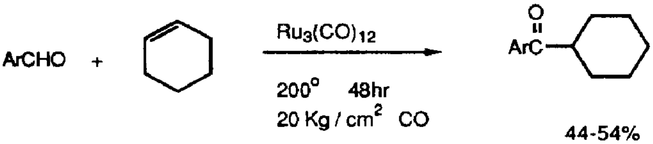
$R^2$ = tBu, TMS, CO$_2$Me, secBu

Ruthenium carbonyls catalyzed the addition of formamides (equation 405) [479] and aldehydes (equation 406) [480] to olefins. Other, less straightforward carbonylations of alkynes and alkenes are shown in equation 407 [481], equation 408 [482].

(405)



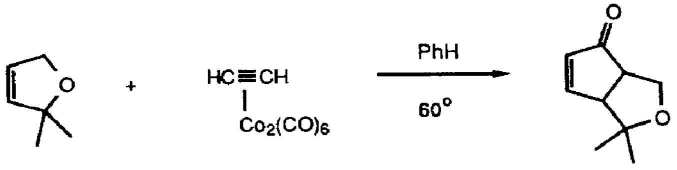
R = PhCH$_2$, Me,



(406)

44-54%

Ar = Ph, pClPh, pMePh



(407)

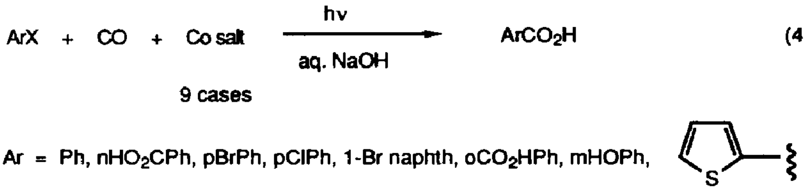
53%

(408)

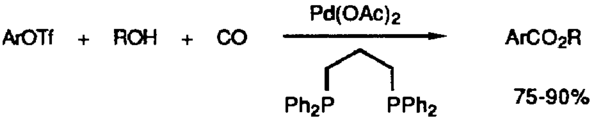
7%

## 2. Carbonylation of Halides

The factors influencing the outcome and product distribution as well as the scope and limitations of the phase-transfer catalyzed reaction of aryl and vinyl halides with dicobalt octacarbonyl and methyl iodide to give methyl ketones and carboxylic acids were reported [483][484]. Aryl halides were converted to carboxylic acids by photolysis under basic conditions in the presence of carbon monoxide and a wide range of cobalt salts (equation 409) [485]. Aryl triflates were carbonylated to esters using palladium catalysts (equation 410) [486]. Aryl diazonium salts were converted to ketones by reaction with alkyltin reagents and carbon monoxide in the presence of palladium catalysts (equation 411) [487]. Aryl halides were converted to acyl fluorides by palladium catalyzed carbonylation in the presence of cesium fluoride (equation 412) [488]. Aryl halides were alkoxycarbonylated by carbon monoxide and alcohols in the presence of palladium(0) catalysts [489]. Under appropriate conditions, double carbonylation occurred (equation 413) [490].

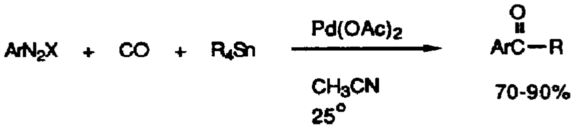


(409)



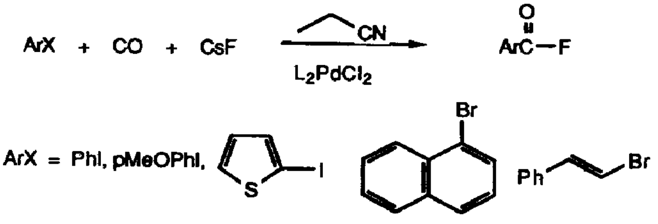
(410)

12 cases including steroidal triflates

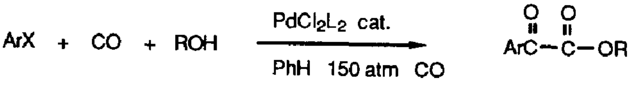


(411)

R = Me, Et, Bu, Ar'; 15 cases Me, Br, I, NO2, MeO substituent on Ar



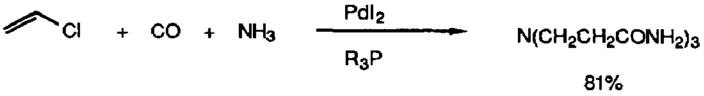
(412)



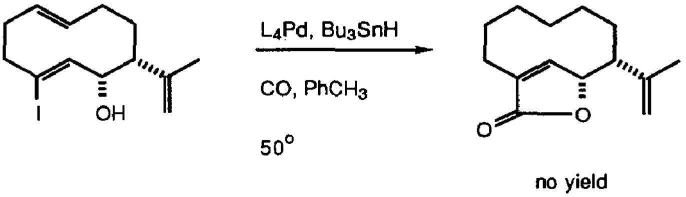
$$ArX + CO + ROH \xrightarrow[\text{PhH 150 atm CO}]{PdCl_2L_2 \text{ cat.}} ArC(=O)-C(=O)-OR$$

(413)

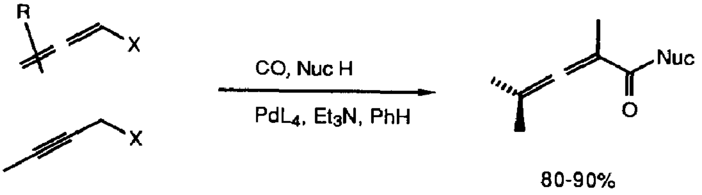
Vinyl halides were aminoacylated (equation 414) [491], and alkoxyacylated (equation 415) [492], and allenic halides were converted to allenic esters using palladium catalysts (equation 416) [493]. Allylic halides were converted to ketones by acyltin reagents in the presence of palladium catalysts (equation 417) [494]. Benzylic halides (equation 418) [495] and aliphatic halides [496] were converted to carboxylic acid derivatives using cobalt catalysts.



(414)

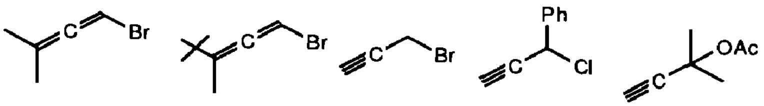


(415)



(416)

Nuc = MeOH, EtOH, Et$_2$NH



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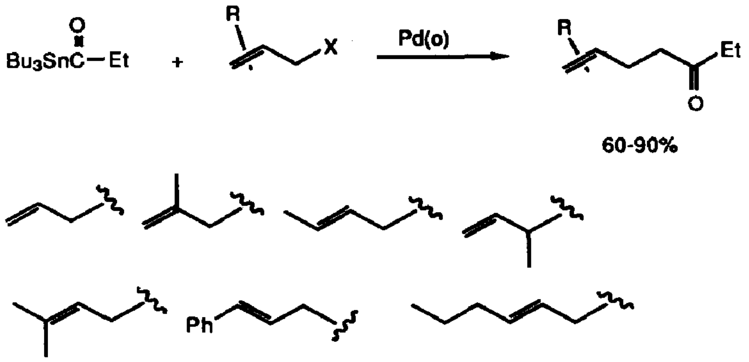


(417)

(418)

$$\mathrm{CoCl_2}\xrightarrow[\text{2) NaOH}]{\text{1) }\mathrm{BH_4^-/CO/THF}}\mathrm{Co(CO)_4^-}\xrightarrow[\mathrm{CO,\ NaOH}]{\mathrm{ArCH_2Br}}\mathrm{ArCH_2CO_2^-}$$

72-88%

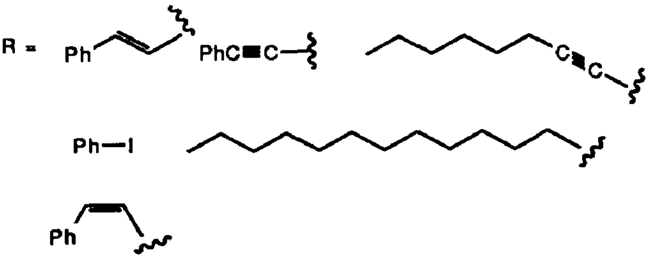
Ar = Ph, pClPh, pMePh

Palladium catalyzed the carbonylation of organotellurium species (equations 419, 420, and 421) [497], as well as the double carbonylation of aryl and aliphatic bromides (equation 422) [498], (equation 423) [499]. Double carbonylation reactions have been reviewed [500].

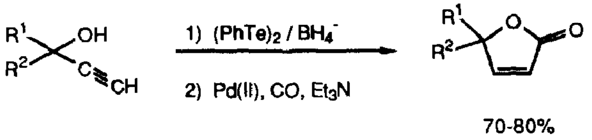
$$\mathrm{RTePh}\xrightarrow[\mathrm{PdCl_2\ Et_3N\ MeOH}]{\text{1 atm CO}}\mathrm{RCO_2Me}\qquad(419)$$

$\mathrm{CuCl_2}$

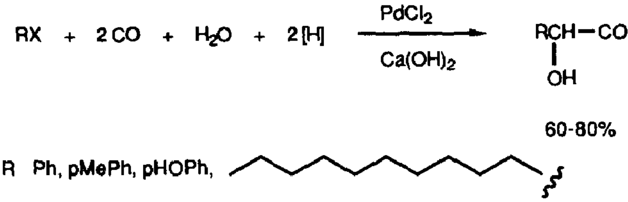
60-90%



(420)

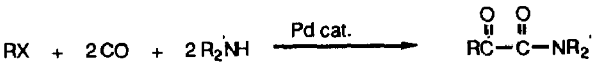


(421)



$$RX + 2CO + H_2O + 2[H] \xrightarrow[Ca(OH)_2]{PdCl_2} \underset{\displaystyle OH}{RCH}{-}CO_2H$$

(422)

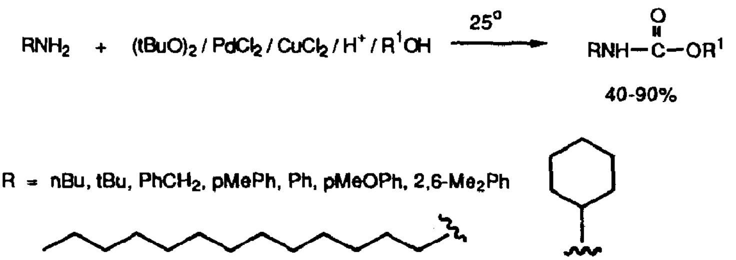


$$RX + 2CO + 2R_2NH \xrightarrow{Pd\ cat.} R\overset{O}{\overset{\|}{C}}{-}\overset{O}{\overset{\|}{C}}{-}NR_2$$

(423)

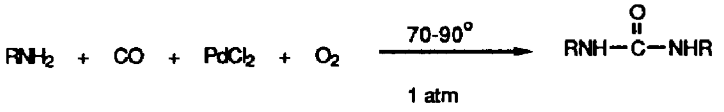
### 3. Carbonylation of Nitrogen Compounds

Primary and seconary amines were oxidatively carbonylated to carbamates, with di-t-butyl/peroxide/palladium/copper catalyst systems (equation 424) [501]. Ureas were formed by a similar process (equation 425) [502]. Under appropriate conditions similar catalyst systems resulted in double carbonylation (equations 426 and 427) [503]. In contrast, ruthenium(III) catalysts converted amines to formamides (equation 428) [504]. "Homogeneous mixed metal catalyst systems: new and effective routes to N-acyl-α-amino acids via carbonylations" was the subject of a review with 21 references [505]. Dicobalt octacarbonyl catalyzed the carbonylation of aldehydes and amides to α-amino acid derivatives (equation 429) [506]. Nitrosobenzene was carbonylated to an amide using palladium(II) catalysts (equation 430) [507].



$$RNH_2 + (tBuO)_2/PdCl_2/CuCl_2/H^+/R^1OH \xrightarrow{25^\circ} RNH{-}\overset{O}{\overset{\|}{C}}{-}OR^1$$

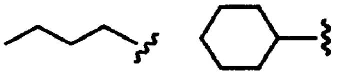
(424)

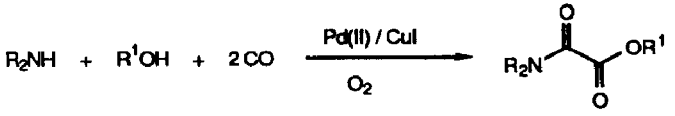


$$RNH_2 + CO + PdCl_2 + O_2 \xrightarrow[\text{1 atm}]{70\text{-}90^\circ} RNH-\overset{O}{\overset{\|}{C}}-NHR$$

(425)

R = Ph, pClPh, pMeOPh,





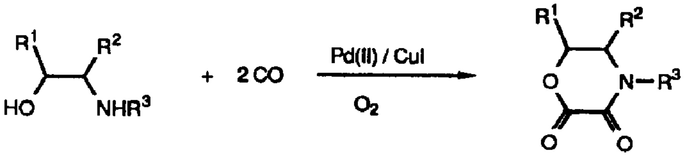
$$R_2NH + R^1OH + 2CO \xrightarrow[O_2]{Pd(II)/CuI}$$

(426)

60-90%

R = Et, $(CH_2)_4$, $(CH_2)_2O(CH_2)_2$

$R^1$ = Me



$$+ 2CO \xrightarrow[O_2]{Pd(II)/CuI}$$

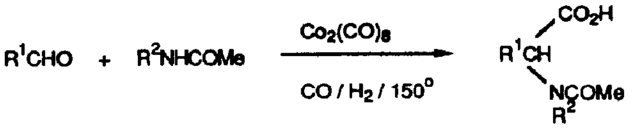
(427)

6 cases

82-86%

$$\begin{array}{l} R_2NH \\ RNH_2 \end{array} + CO \xrightarrow[\begin{array}{l}\text{220 Bar} \\ 200^\circ\end{array}]{RuCl_3 \cdot 3H_2O} \begin{array}{l} R_2NCHO \\ RNHCHO \end{array}$$

(428)



$$R^1CHO + R^2NHCOMe \xrightarrow[CO/H_2/150^\circ]{Co_2(CO)_8} R^1CH(CO_2H)NR^2COMe$$

(429)

$R^2$ = H; 70%

$R^2$ = Me, Et; 0-30%

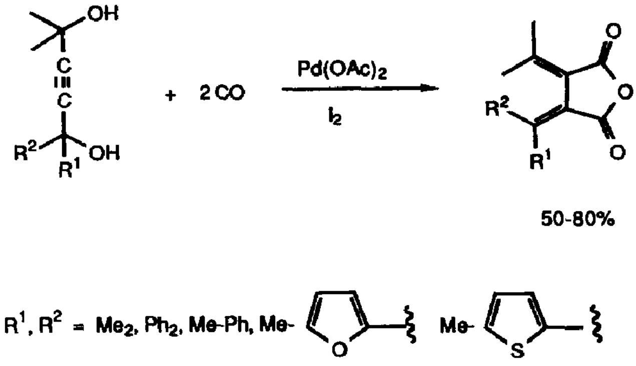
$$ArNO + ROH + CO \xrightarrow[O_2, HCl]{Pd(OAc)_2 / Cu(OAc)_2} ArNHCO_2R + CO_2$$

(430)

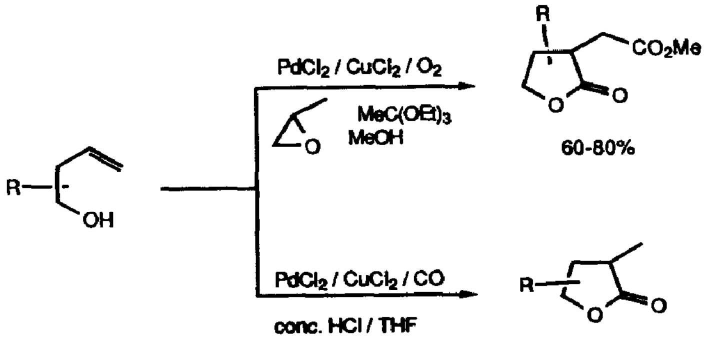
89%

## 4. Carbonylation of Oxygen Compounds

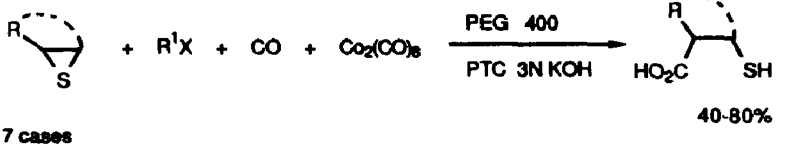
Bis-propargyl alcohols were cyclocarbonylated to anhydrides (equation 431) [508] and allylic alcohols were cyclized to lactones using palladium catalysts (equation 432) [509]. Thiirans were carboxylated to α-thioacids by cobalt carbonyl under phase transfer conditions (equation 433) [510]. Cobalt(II) iodide catalyzed the reductive carbonylation of methyl esters to acetaldehyde and carboxylic acids [511].



(431)



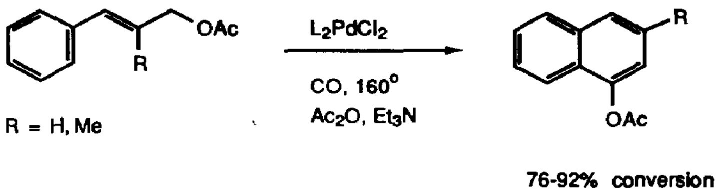
(432)



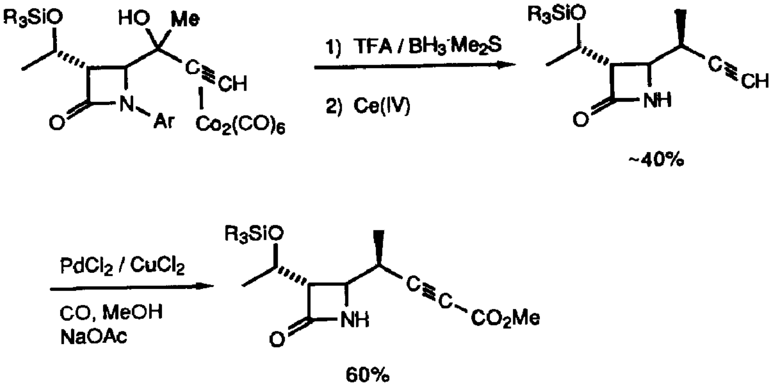
(433)

### 5. Miscellaneous Carbonylations

Cinnanyl acetate was cyclocarbonylated to an α-naphthol under palladium catalysis (equation 434) [512]. A terminal alkyne was converted to an acetylenic ester using a similar catalyst (equation 435) [513].



(434)



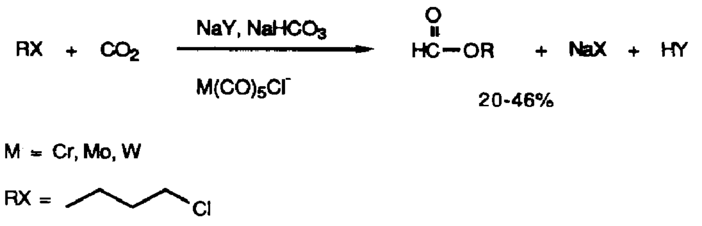
(435)

### 6. Decarbonylation Reactions

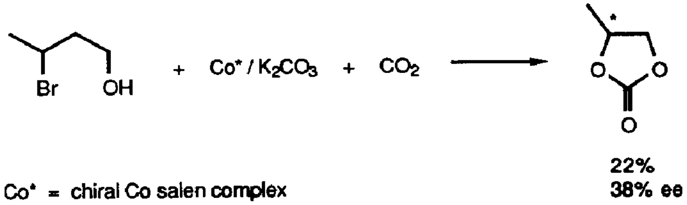
Rhodium(I) catalyzed decarbonylation of chiral 4-methyl-[1-$^3$H,2-$^2$H] pentanal proceeded with net retention accompanied by some racemization [514].

### 7. Reactions of Carbon Dioxide

Reviews entitled "Homogeneous catalytic reactions of carbon dioxide with unsaturated substrates, reversible $CO_2$ carriers, and transcarboxylation reactions" (166 references) [515], and "Catalytic reactions involving carbon dioxide" (34 references) [516] have appeared. Rhodium and palladium complexes catalyzed a variety of reactions between carbon dioxide and butadiene [517]. Palladium(II) acetylacetonate catalyzed the reaction between butadiene, carbon dioxide, and ammonia to give 2,7-octadienyl formamide [518]. Organic halides were carboxylated to formate esters by reaction with carbon dioxide under chromium catalysts (equation 436) [519]. Cobalt complexes catalyzed the reaction between 3-bromobutanol and carbon dioxide to give the cyclic carbonate (equation 437) [520]. Low valent nickel complexes coupled olefins to carbon dioxide to give a metallacycle which was further converted to a number of interesting organic compounds (equation 438) [521].

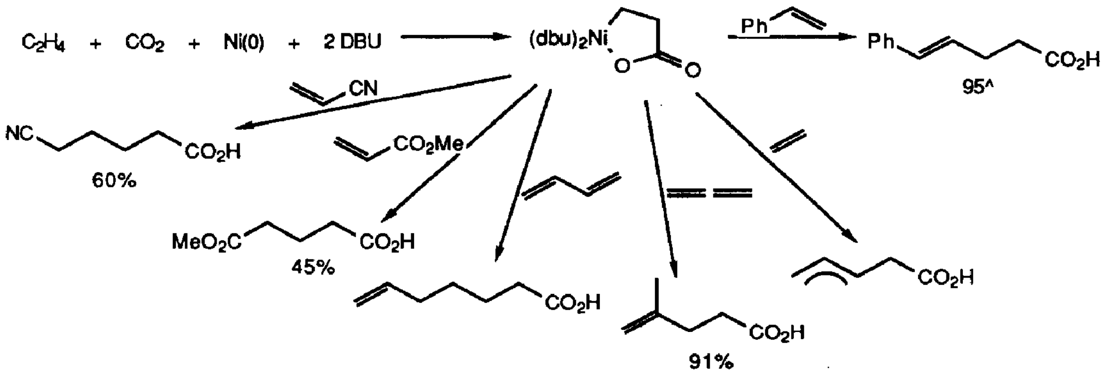


(436)

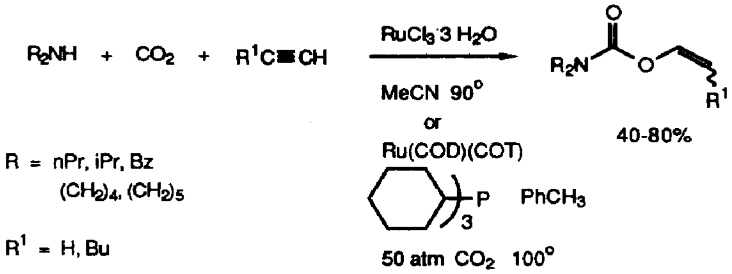


(437)

(438)

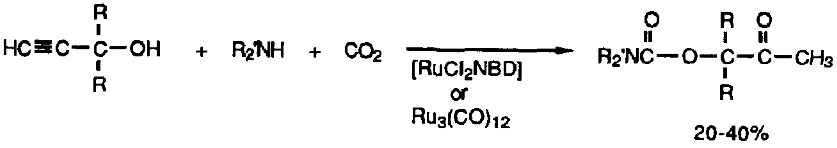


Ruthenium complexes catalyzed the reaction between amines, alkynes and carbon dioxide to give o-vinyl carbamates (equation 439) [522][523] while propargyl alcohols produced β-acetyl carbamates under similar conditions (equation 440) [524][525]. With cobaltocene as catalyst, propargyl alcohols underwent reaction with carbon dioxide to give cyclic vinyl carbonates (equation 441) [526]. Diynes were cyclocarboxylated to pyrones under nickel(0) catalysis (equation 442) [527].



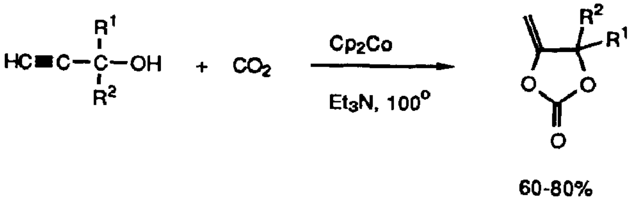
(439)

(440)



R = H, Me

$R^1$ = Et, $(CH_2)_4$, $(CH_2)_5$

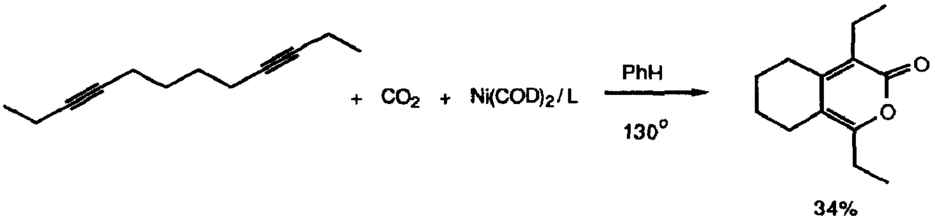


(441)

$R^1$, $R^2$ = Me, Et

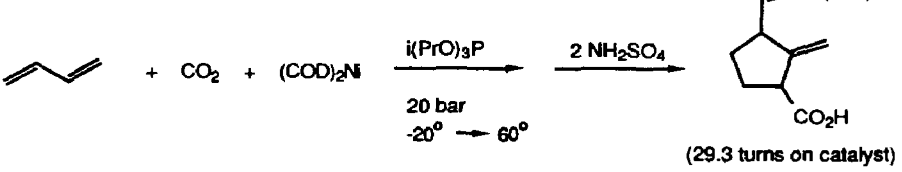
$R^1$ = Me; $R^2$ = Et, iBu

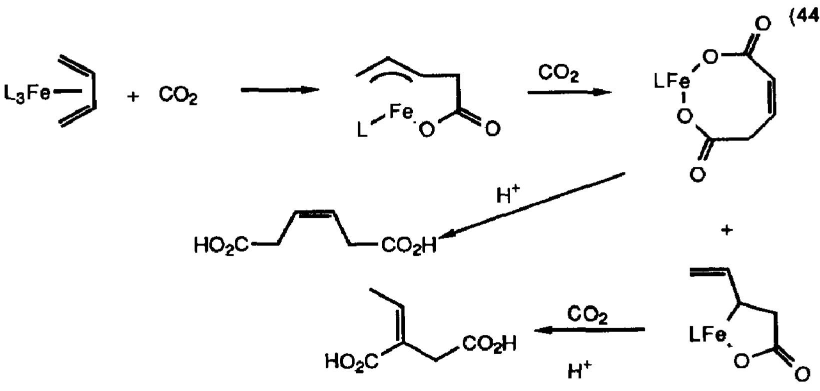
(442)



Butadiene and carbon dioxide were cyclodimerized to a cyclopentene carboxylic acid by nickel bis cyclooctadiene (equation 443) [528]. Butadiene iron(0) complexes incorporated carbon dioxide to give a π-allyl iron complex which was further converted to organic products (equation 444) [529].

(443)

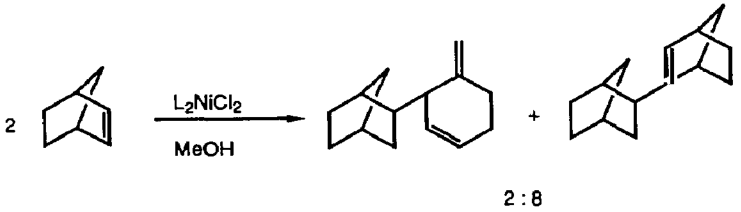




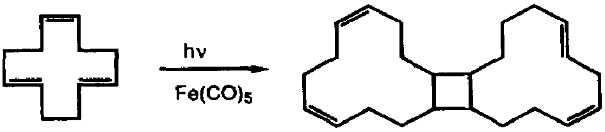
(444)

## D. Oligomerization

Propylene was dimerized by a catalyst containing palladium(II) acetylacetonate, trialkylphosphine, and boron trifluoride etherate [530]. Bisphosphine nickel(II) complexes catalyzed the dimerization of norbornene (equation 445) [531]. Terminal alkynes and 1,3-butadienes were linearly codimerized by the ruthenium(0) catalyst, Ru(COD)(COT)R$_3$P [532]. Acetylene was dimerized to CH$_2$OHC≡CH by ruthenium(III) chloride in the presence of sodium hydroxide [533]. Nickel(0) catalysts linearly dimerized butadiene to mixed octatrienes [534]. Butadiene was also dimerized by nickel(0) catalysts having chiral diphosphine ligands made from α-amino acids [535]. Iron pentacarbonyl cyclodimerized 1,5,9-cyclododecatriene under photolysis conditions (equation 446) [536], while nickel(0) complexes dimerized dimethylenecyclobutane (equation 447) [537].

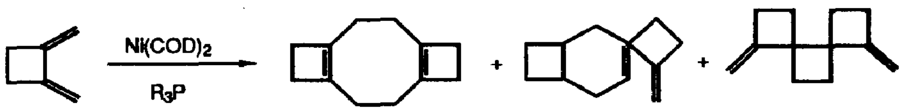


(445)

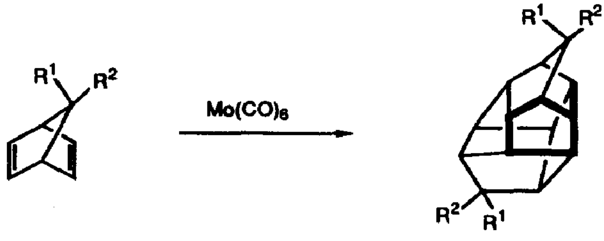


(446)

(447)



3,3-Dimethylcyclopropene was cyclodimerized by nickel(0) ($R_3Al$) catalyst systems [538]. Molybdenum hexacarbonyl dimerized norbornadiene to the cage compound shown in equation 448 [539]. Rhodium(II) carboxylates catalyzed the cyclodimerization of norbornadiene [540]. Methyl methacrylate was dimerized by palladium(II) catalysts (equation 449) [541][542] and by cobalt-dimethylglyoxime catalysts (equation 450) [543]. Allyl acetate and norbornene (equation 451) [544] and norbornadiene (equation 452) [545] were codimerized by nickel(0) catalysts.



(448)

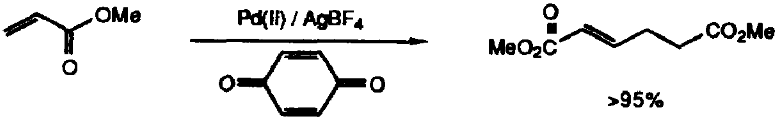
~50%

$R^1$ = H, $PhCO_2$, tBuO

$-(CH_2)_4-$

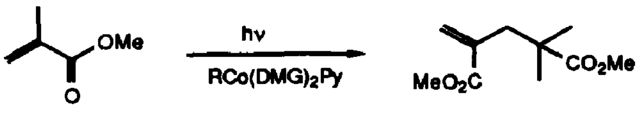
$R^2$ = H

(449)



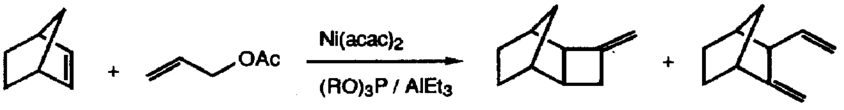
>95%

340 turnovers



(450)

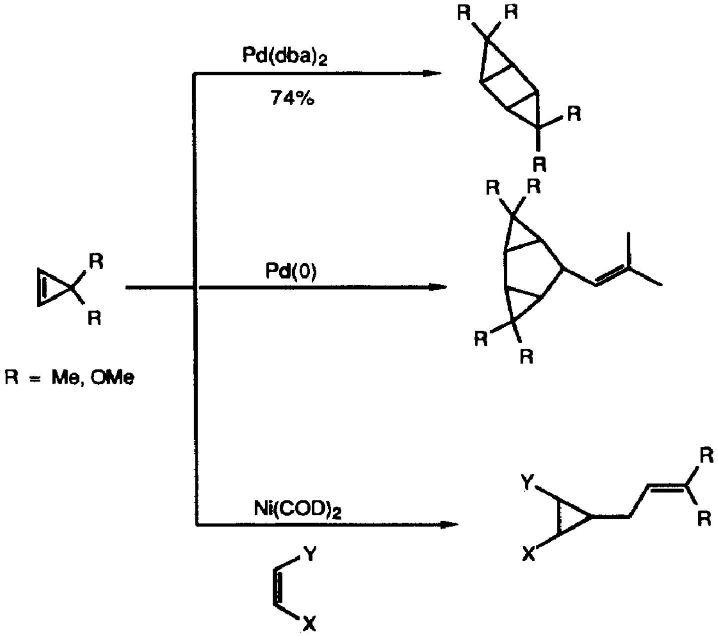
48%, 4800 turnovers



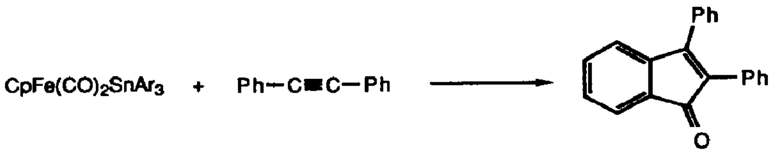
(451)

(452)

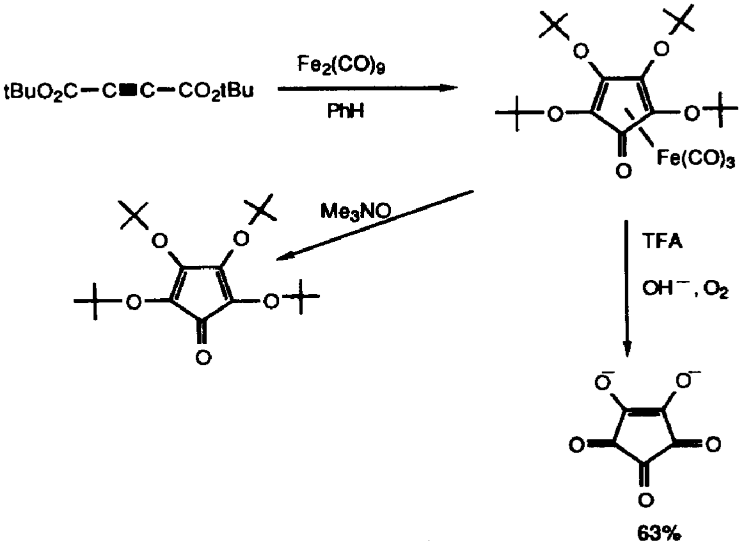
Dimethoxy- [546] and dimethylcyclopropene [547] were cyclodimerized, cyclotrimerized and cocyclotrimerized with acrylates, by nickel(0) and palladium (0) complexes (equation 453). Alkynes cyclo added to arenes in the presence of iron-tin complexes (equation 454) [548]. Acetylene dicarboxylates cyclodimerized to cyclopentadienones (equation 455) [549]. Alkynes were cocyclooligomerized with carbon dioxide to give pyrones (equation 456) [550].



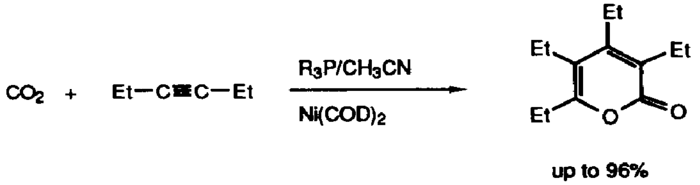
(453)



(454)



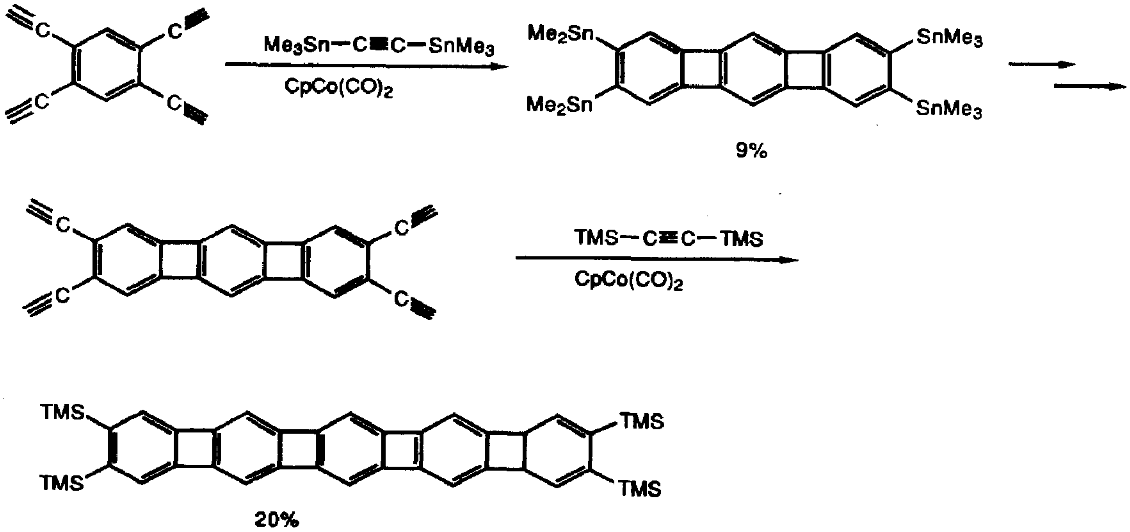
(455)



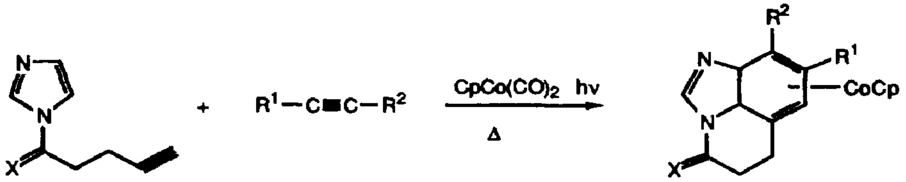
(456)

Polybenzocyclobutanes were made by cobalt-catalyzed cyclotrimerization of alkynes (equation 457) [551]. Cyclotrimerization was used to prepare a variety of heterocycles (equation 458) [552], (equation 459) [553], (equation 460) [554]. Palladium on carbon cyclotrimerized alkynes to benzenes (equation 461) [555]. Nickel(0) complexes cyclotrimerize alkynes via nickelirene and nickelole intermediates (equation 462) [556][557]. Nickel(II) salts cyclotrimerized 1,4-dimethoxy-2-butyne in the presence of sodium borohydride and phosphines [558]. Propargyl alcohol was cyclotetramerized by 1-azadiene nickel complexes (equation 463) [559].

(457)



(458)

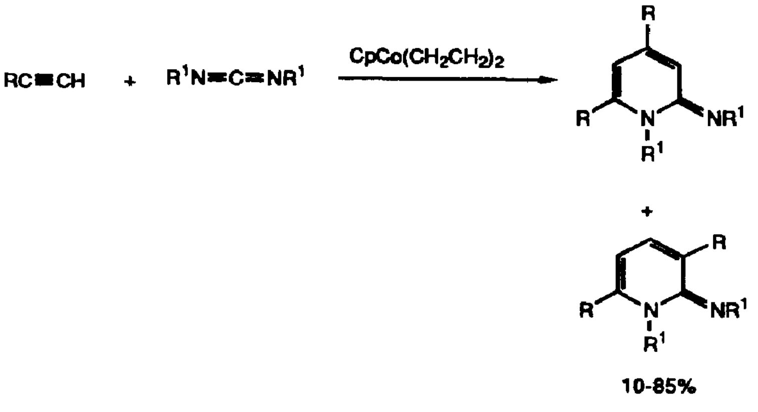


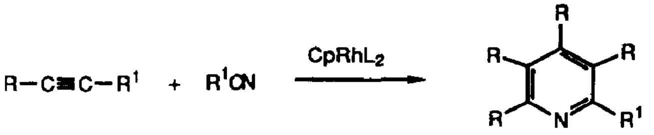
X = 0

$R^1, R^2$ = TMS, 72%; $R^1$ = Et, $R^2$ = TMS, 22%; $R^1$ = OMe, $R^2$ = TMS, 25%

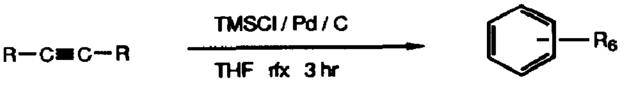
$R^1$ = $CO_2Me$, $R^2$ = TMS, 35%

(459)





(460)

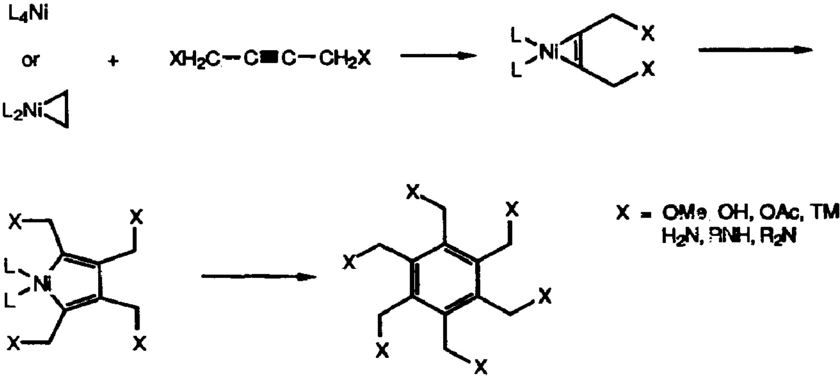


(461)

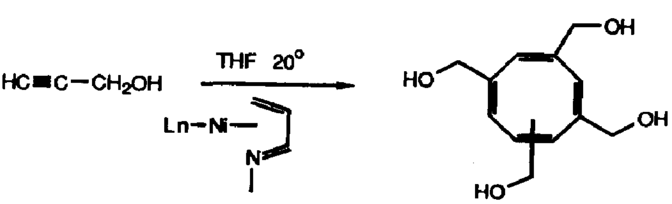
R = Et, nBu, Ph

100%

(462)



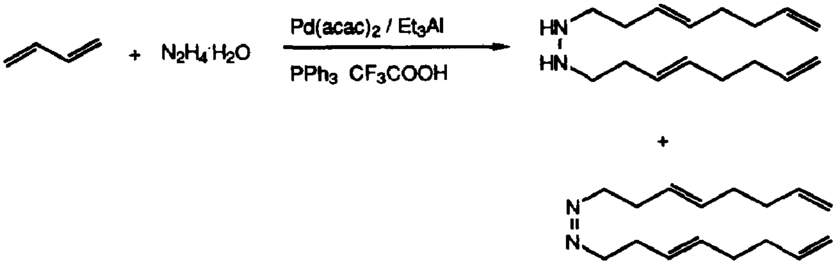
X = OMe, OH, OAc, TMSO, H₂N, RNH, R₂N



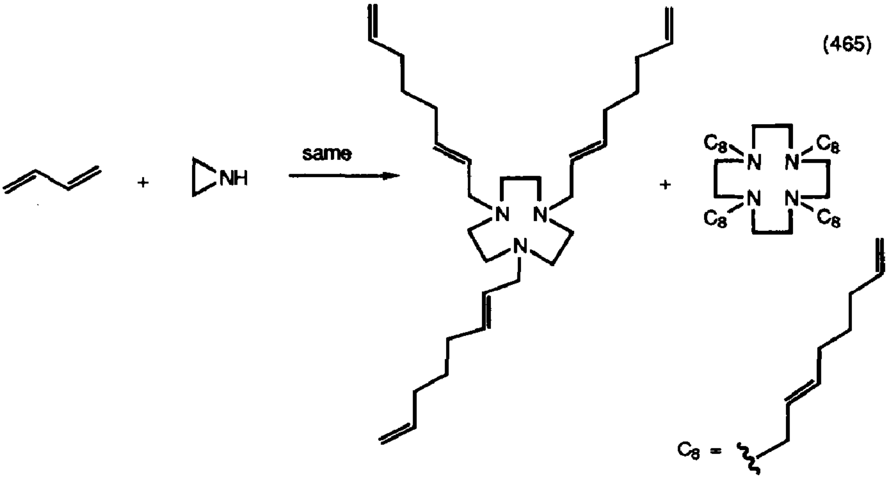
(463)

Palladium-phosphine complexes telomerized butadiene with heteroaromatic compounds [560], with N-methylaniline [561], with hydrazine (equation 464) [562], with hydroxylamine [563], with aziridines (equation 465) [564], with amino acids [565] and with hydroxy and carboalkoxysulfides [566].

(464)

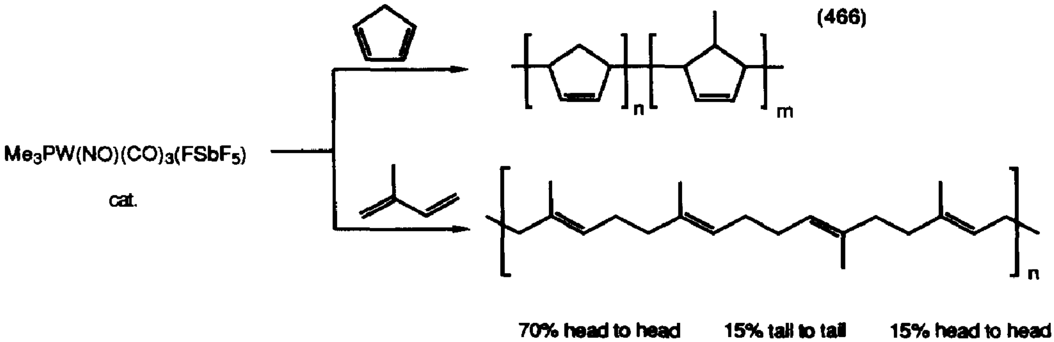


(465)

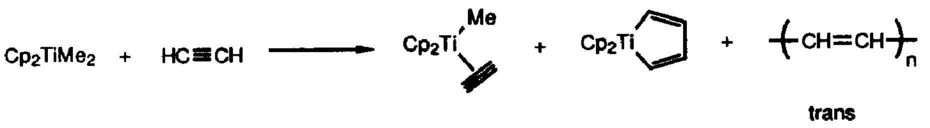


1,3-Diketones were active ligands in the nickel-catalyzed linear oligomerization of olefins [567][568]. Tungsten catalysts were effective for the polymerization of dienes (equation 466) [569], while titanium reagents polymerized acetylene (equation 467) [570]. Propylene was polymerized with a high degree of stereocontrol by chiral zirconium catalysts (equation 468) [571].

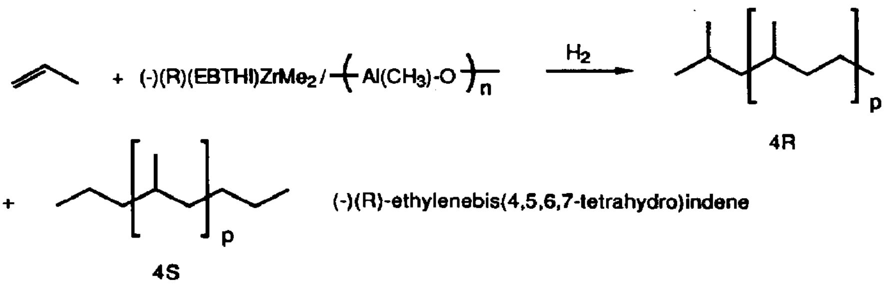
(466)



(467)



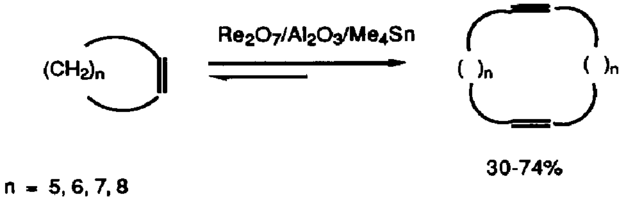
(468)



## E. Rearrangements

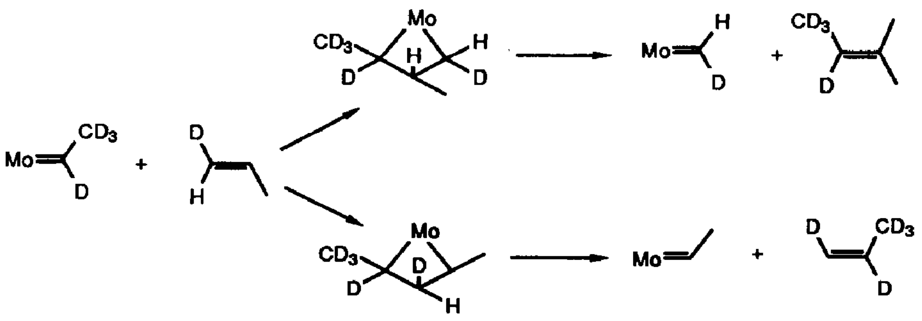
### 1. Metathesis

A dissertation dealing with the induction and quenching of olefin metathesis by acetylenes has appeared [572]. The complex Mo(CHtBu)(NAr)(OCMe(CF$_3$)$_2$)$_2$ was a stable compound and an active metathesis catalyst [573]. The role of the tungsten hexachloride/cycloolefin reaction in metathesis has been studied [574]. The behavior of W(OAr)$_2$Cl$_4$ complexes associated with tetraalkyl tin or lead compounds in olefin metathesis reactions was studied [575]. Polar organic polymers were used as supports for olefin metathesis catalysts [576]. Rhenium complexes catalyzed the ring opening dimerization of cyclic olefins (equation 469) [577][578]. Olefin metathesis mechanisms were probed by deuterium labeling studies (equation 470) [579].



(469)

(470)



Tungsten complexes catalyzed metathesis and cometathesis reactions of ethyl 4-pentenoate [580]. The metathesis reactions of fatty acid esters have been reviewed (90 references) [581]. Rhenium on alumina catalyzed metathesis of unsaturated ethers [582]. The effects of formamide on the ring opening metathesis polymerization of norbornene were studied [583]. 7-Oxobicycloheptenes were ring opening metathesis polymerized by a variety of catalyst systems (equation 471) [584]. Tebbe's reagent catalyzed the polymerization of divinylcyclobutenes to conductive polymer precursors (equation 472) [585]. Molybdenum/tin catalysts gave living polymers from alkynes (equation 473) [586].

(471)

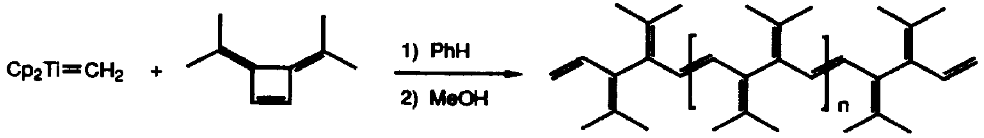
R = H, Me, Et

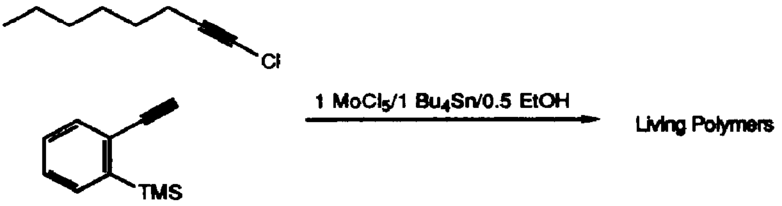
R' = H, $CH_2OH$, $CH_2OTMS$, $CH_2OMe$

R" = Me, $CH_2OCH_3$, $CH_2OH$, $CH_2OTMS$

cat. = $RuCl_3$, $OsCl_3$, $(COD)RuCl_3$

(472)



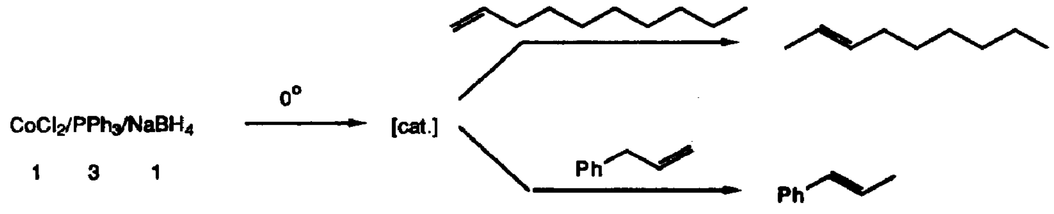


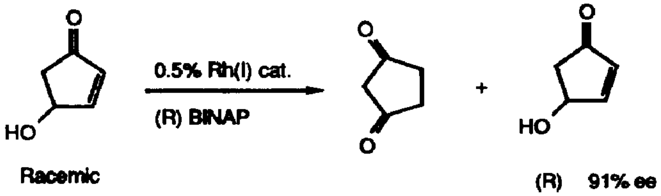
(473)

## 2. Olefin Isomerization

Terminal olefins were isomerized to internal olefins by $Rh(PPh_3)_3(SnCl_3)$ [587]. Nickel(II) acetylacetonate/$Et_3Al_2Cl_3$ catalysts isomerized 1,5-cyclooctadiene to 1,4- and 1,3-cyclooctadiene, as well as bicyclo[3.3.0]oct-2-ene, depending on conditions [588]. Rhodium and nickel complexes catalyzed the isomerization of 1,4-dichloro-2-butene to 3,4-dichloro-1-butene [589]. Titanocene dichloride/Grignard reagent mixtures isomerized 1,5-hexa-diene to 2,4-hexadiene, as well as to methylenecyclopentane [590]. Reduced cobalt catalyst isomerized terminal olefins to internal positions (equation 474) [591]. Chiral rhodium BINAP catalysts discriminated between enantiomers of 4-hydroxycyclopent-2-enone (equation 475) [592].

(474)

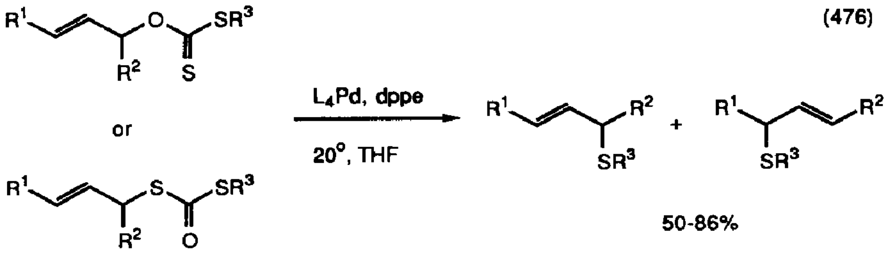




(475)

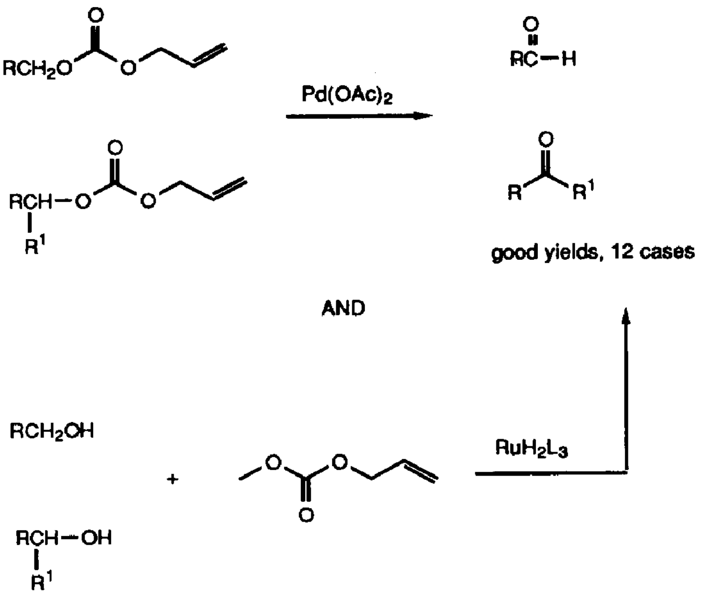
### 3. Rearrangements of Allylic and Propargylic Oxygen and Nitrogen Compounds

A review dealing with homogeneous catalysis - mechanisms of metal-catalyzed Claisen rearrangements of O-allylic-S-methyldithiocarbonates (17 references) has appeared [593]. Palladium(0) complexes efficiently catalyzed a number of rearrangements of allylic thiocarbonates and carbonates which resulted in the expulsion of COS or $CO_2$(equation 476) [594], (equation 477) [595], (equation 478) [596]. This process was used to remove allyloxycarbonyl protecting groups from carbohydrates [597]. Zirconocene dichloride catalyzed a Claisen rearrangement (equation 479) [598]. Palladium(II) triflate catalyzed rearrangements of propargyl alcohols to α,β-unsaturated carbonyl compounds (equation 480) [599]. Palladium(II) acetate isomerized azepines to azabicycloheptenols (equation 481) [600].

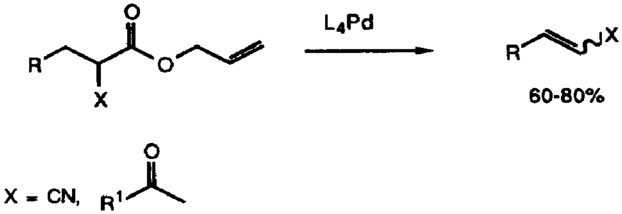


(476)

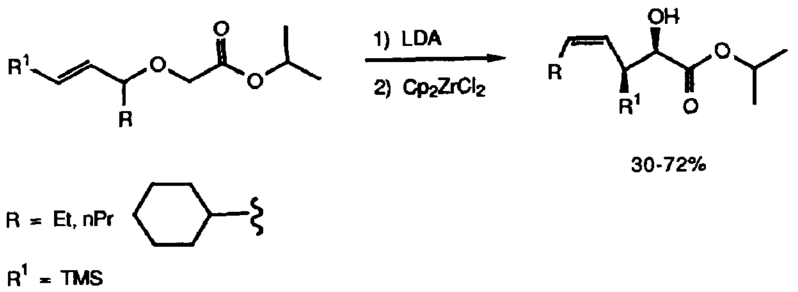
$R^1$ = H, Ph, Me; $R^2$ = H, Me, Ph; $R^3$ = PhCH$_2$, nPr, Me



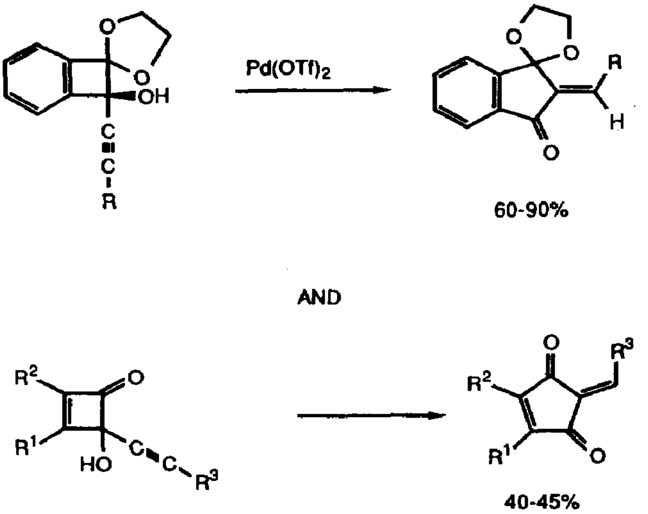
(477)



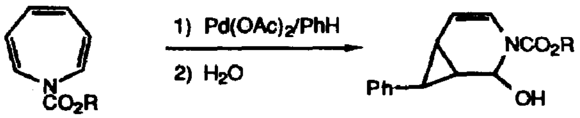
(478)



(479)



(480)



(481)

### 4. Skeletal Rearrangement

Skeletal rearrangements were catalyzed by molecular metal clusters on supports [601], by unsupported films and powders [602] and by alumina supported molybdenum complexes [603]. Quadricyclane was isomerized to norbornadiene by polymer-supported palladium(II) and cobalt(II) catalysts [604].

### 5. Miscellaneous Rearrangements

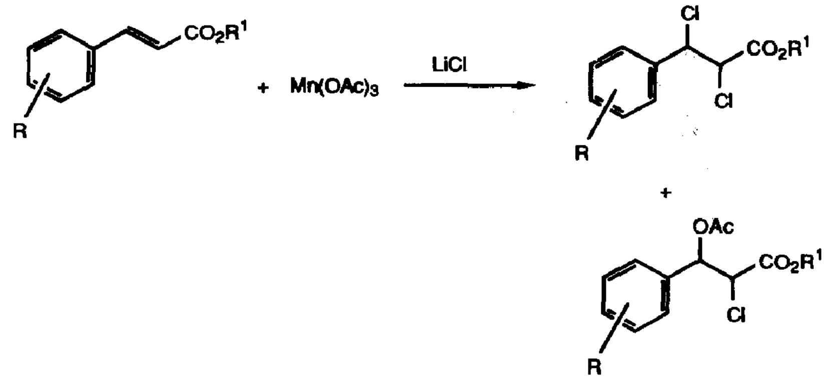
Steroidal alcohols (seven cases studied) were epimerized by $Pd(PPh_3)_4$/p-benzoquinone systems [605].

# III. Functional Group Preparations

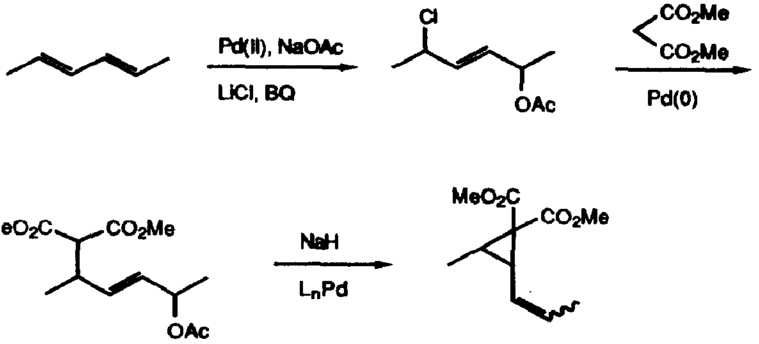
## A. Halides

Chloro- and bromobenzene were converted to iodobenzene in 60-90% yield by treatment with copper(I) iodide on alumina or charcoal at 150° [606]. Manganese(III) acetate chlorinated cinnamate esters in the presence of lithium chloride (equation 482) [607]. 1,3-Dienes were chloro-acetoxylated by palladium catalysts (equation 483) [608], (equation 484) [609].

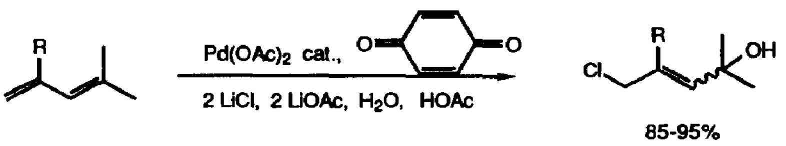
(482)



(483)

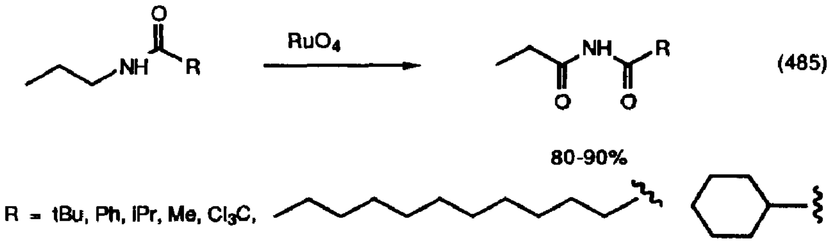


(484)



## B. Amides, Nitriles

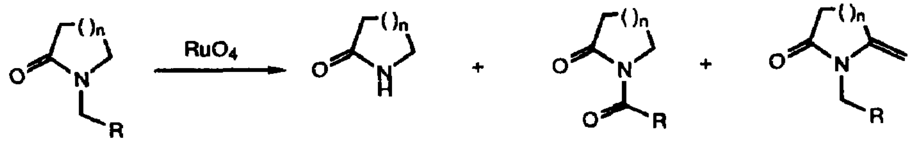
Imides (equation 485) [610], cyclic imides (equation 486) [611], amides (equation 487) [612], N-hydroxyquinolones (equation 488) [613], and β-lactams (equation 489) [614] were prepared by the ruthenium oxidation of the corresponding amines.



(485)

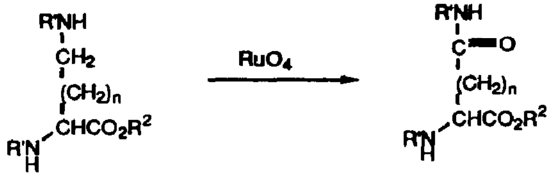
80-90%

R = tBu, Ph, iPr, Me, Cl$_3$C,



(486)

| R = Me, n = | | |
|---|---|---|
| 1 | 86% | |
| 2 | | 88-93% |
| 4 | 35% | 35% |
| 5 | 77% | |



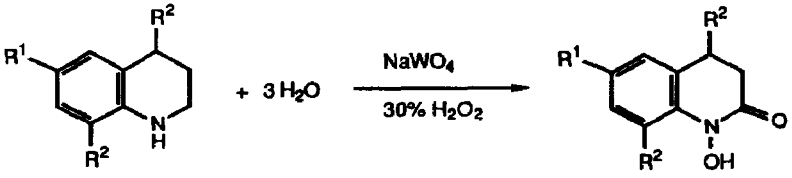
(487)

60-90%

n = 1, 2, 3

R$^1$ = BOC, tBOC

R$^2$ = Bz, tBu, Me

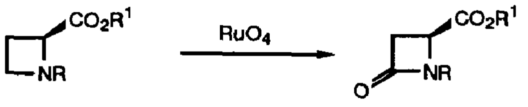


(488)

50-87%

R$^1$ = H, Me, OMe, NHAc, Cl, AcO

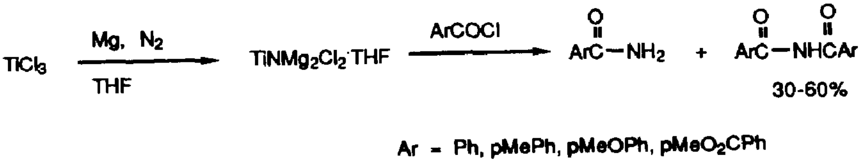
R$^2$, R$^3$ = H, Me

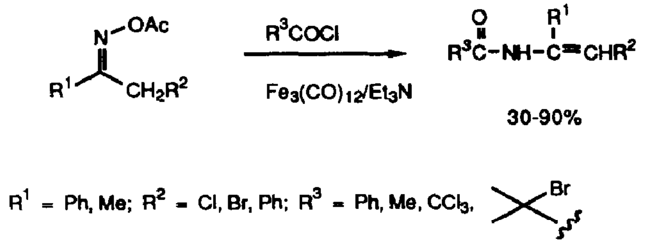


(489)

Acid chlorides were converted to amides and imides by reaction with titanium nitrides (equation 490) [615]. Iron carbonyls catalyzed the reaction between oxime O-acetates and acid chlorides to give amides (equation 491) [616]. Anilines were converted to amides by nickel(0) catalyzed reaction with carbon monoxide and iodobenzene (equation 492) [617]. Molybdenum complexed thioketones were converted thioamides by reaction with ynamines and carbon monoxide (equation 493) [618]. $RhCl_3$, $FeCl_3$, $CoCl_2$, $PdCl_2$ and $CuCl_2$ catalyzed the transamidation reaction between allyl and butyl amines and DMF [619]. Copper(I) iodide coupled leucine to β-keto thioesters (equation 494) [620]. A variety of amides were prepared by the nickel(0) promoted reaction between butadiene and phenyl isocyanide (equation 495) [621].

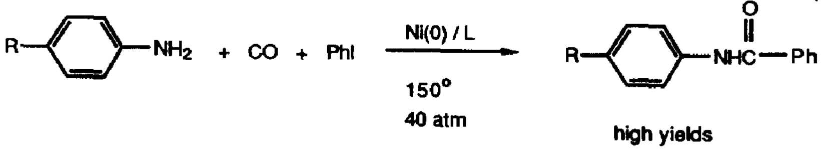
(490)



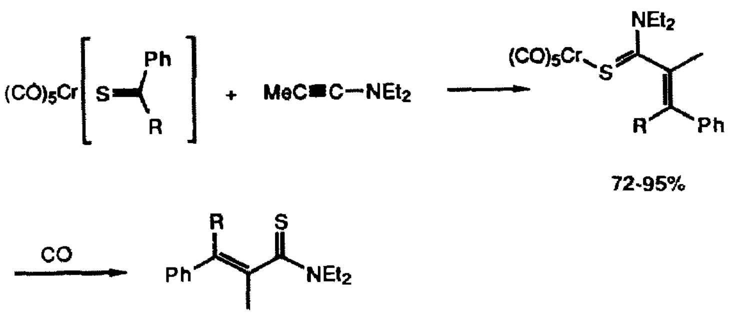


(491)

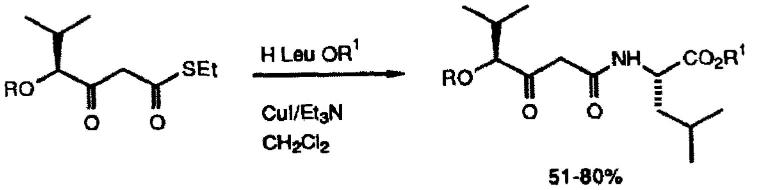
(492)



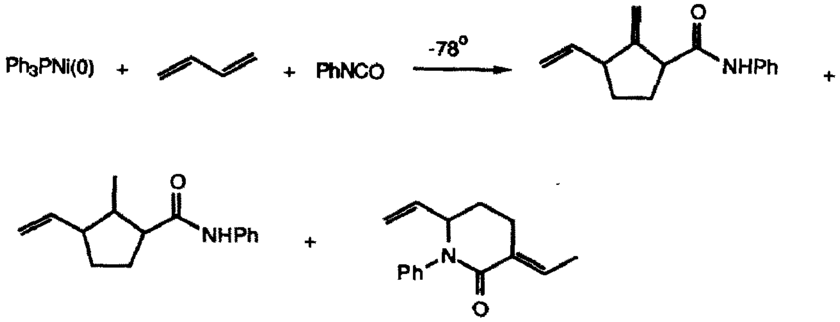
(493)



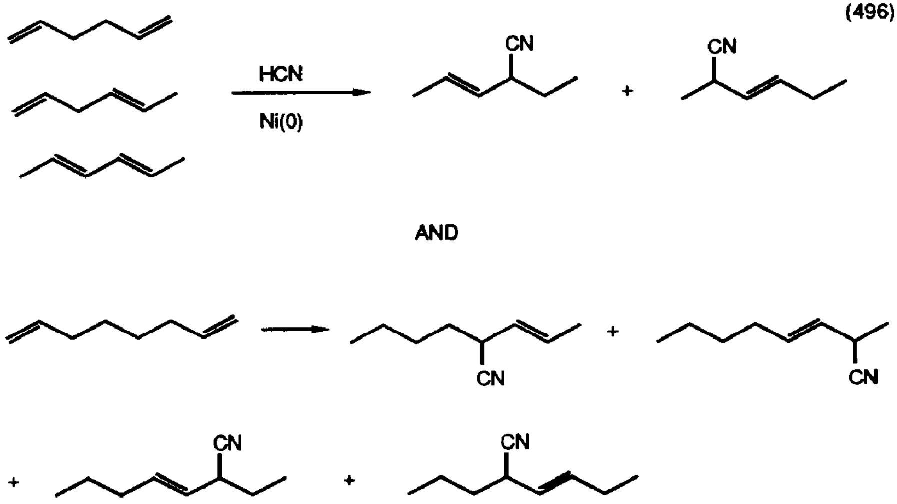
(494)



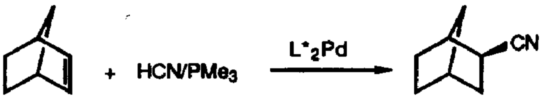
(495)



The effects of ligands on the product distribution in the nickel catalyzed hydrocyanation of dienes were studied (equation 496) [622]. Norbornene was hydrocyanated with low enantiomeric excess by chiral nickel(0) catalysts (equation 497) [623]. Nickel(0) catalysts also promoted the hydrocyanation of alkynes (equation 498) [624]. Alkenes and alkynes were converted to alkyl and alkenyl nitriles via zirconium chemistry (equation 499) [625]. Palladium(0) complexes catalyzed the addition of sulfonamides to dienes to give N-alkenylsulfonamides [626].

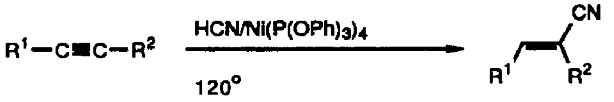


(496)



(497)

60% yield 10% ee
or
6% yield 40% ee

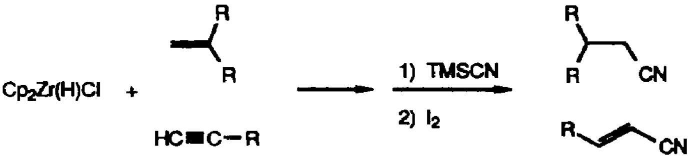


(498)

79-88%

$R^1$ = TBDMS, Ph, H

$R^2$ = Me, nBu, Ph,



(499)

good yields

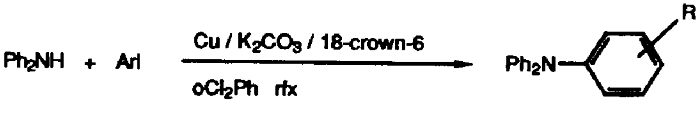
12 cases

## C Amines, Alcohols

Complex catalysis of aminomethylation and amination of olefins has been reviewed (110 references) [627], as has amination and carboamination of olefins and arenes (65 references) [628]. Copper(II) acetate catalyzed the arylation of anilines by aryllead(IV) acetate (equation 500) [629], and by aryl iodides (equation 501) [630]. Epoxy nitro compounds were aminated using palladium(0) catalysts (equation 502) [631]. Allyl acetates and carbonates were aminated using similar catalysts (equation 503) [632]. Chiral η$^5$-dienyliron complexes were resolved by an amination procedure (equation 504) [633].

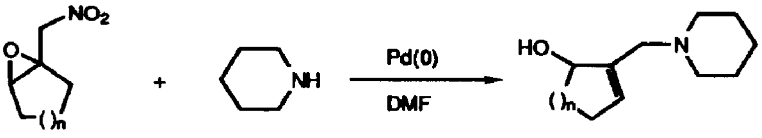
$$PhPb(OAc)_3 + Cu(OAc)_2 + ArNH_2 \longrightarrow \underset{85\text{-}94\%}{ArNHPh} \quad (500)$$

Ar = pMePh, Ph, pMeOPh, 2,6-Me$_2$Ph; pNO$_2$Ph fails



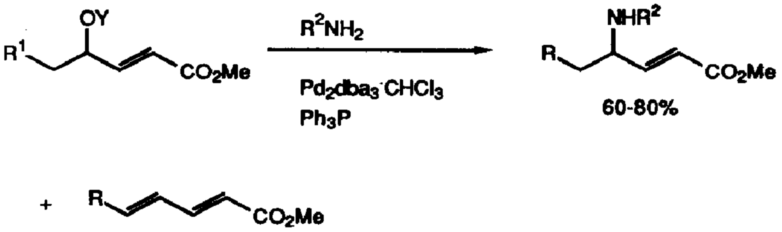
(501)

R = OMe; pMe; nMe; o, m, pCl; o, m, pOMe



(502)

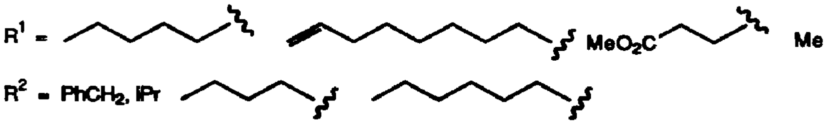
50-80%



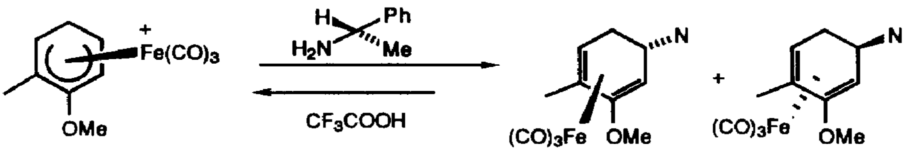
(503)

60-80%

Y = Ac, COCH$_2$Cl, COCF$_3$, CO$_2$Et

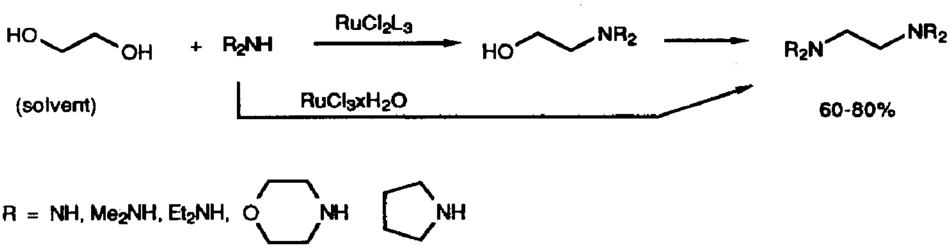


(504)

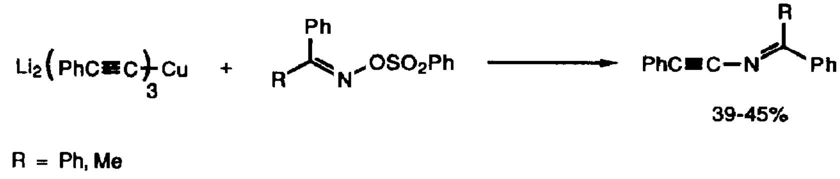


Ruthenium complexes catalyzed the amination of diols (equation 505) [634]. Pyridine oxides were reduced to pyridines by low-valent titanium [635][636]. Imines were reduced to amines using Wilkinsons catalyst, $RhCl(PPh_3)_3$, and hydrogen [637]. Nitroaromatics and nitroalkanes were reduced using hydrogen and palladium catalysts [638]. Copper acetylides alkylated O-tosyl oximes (equation 506) [639], while copper(I) bromide catalyzed the reaction of Grignard reagents with nitriles to give amines, imines and ketones (equation 507) [640].

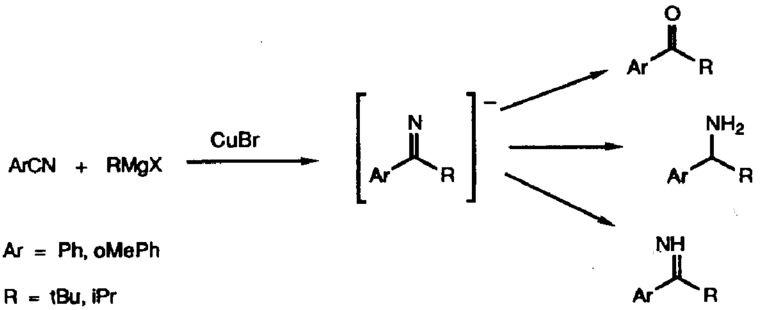
(505)



(506)

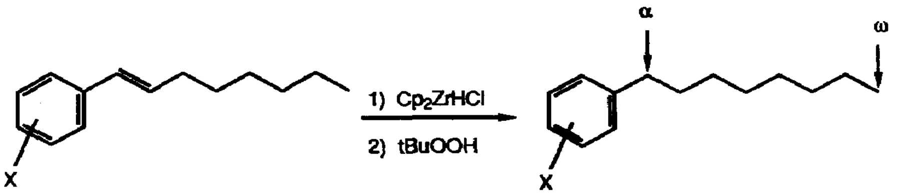


(507)



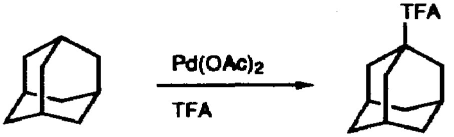
Hydrozirconation/oxidation of aryl-terminated long chain olefins gave mixtures of alcohols (equation 508) [641]. Cyclic, saturated hydrocarbons were oxidized to alcohols [642] or triflates (equation 509) [643] by ruthenium tetroxide or palladium acetate in trifluoracetic acid respectively. Palladium(II) trifluoroacetate catalyzed the allylic oxidation of allyl sulfones by t-butyl hydroperoxide (equation 510) [644]. Cyclohexane was oxidized to cyclohexanol and cyclohexanone by t-butylhydroperoxide and transition metal substituted heteropolytungstates [645]. Arene chromium tricarbonyl complexes of steroids were oxygenated (equation 511) [646]. Competitive ketonization, allylic oxidation, and epoxidation of olefins by air oxidation in the presence of cationic rhodium(I) complexes was studied [647]. Olefins were reductively oxygenated by $O_2$ and rhodium porphyrin catalysts (equation 512) [648]. Aliphatic olefins were converted saturated alcohols by air, borohydride and cobalt(III) catalysts [649].

(508)



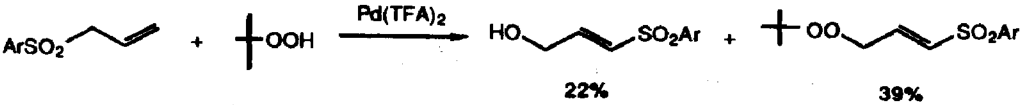
4h 25° 50% α, 0% ω, 50% SM

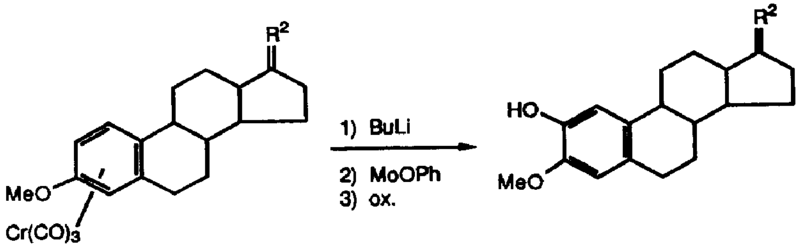
72h 40° 19% α, 24% ω, 59% SM



(509)

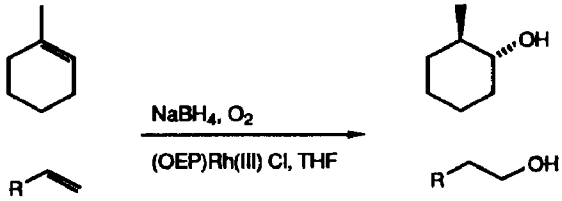
(510)





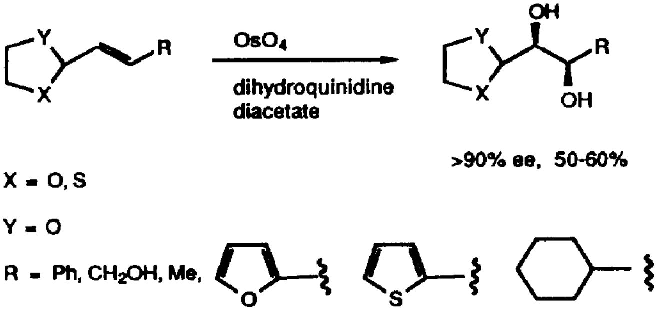
(511)

$R^2$ = OH, H; $R_3Si$, H; $OCH_2CH_2O$

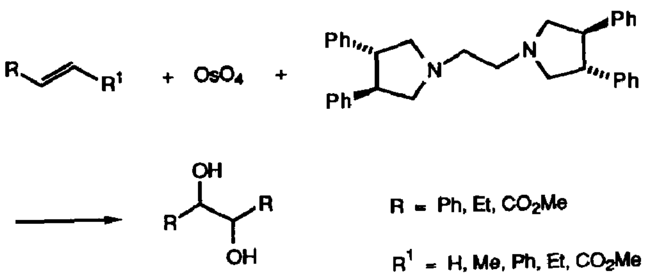


(512)

Olefins were converted to diols with high enantiomeric excess by osmium tetraoxide with chiral acetals as substrates (equation 513) [650] or with chiral diamines as ligands (equation 514) [651]. α-Ketolactones were reduced to α-hydroxylactones with high optical yields by chiral rhodium(I) catalysts (equation 515) [652]. Chiral ruthenium BINAP catalysts reduced β-dicarbonyl compounds to β-hydroxy esters with high ee (equation 516) [653]. Formate esters were decarbonylated to alcohols by $Rh_6(CO)_{16}$ [654]. Anhydrous ferric chloride removed O-benzyl protecting groups from carbohydrates <u>without</u> removing O-methyl or O-acetyl protecting groups [655]. t-Butylhydroperoxide in the presence of $MoO_2(acac)_2$ efficiently removed OTBDMS protecting groups [656].



(513)



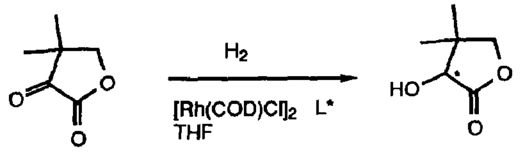
(514)

R = Ph, Et, $CO_2Me$

$R^1$ = H, Me, Ph, Et, $CO_2Me$

83-97% ee

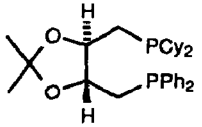
67-83% yield



(515)

70% opt. yield
100% conversion

L* =



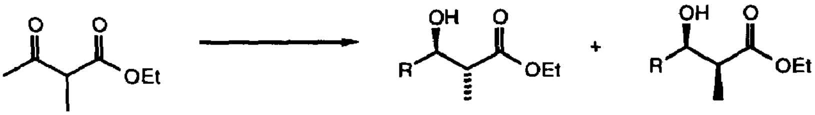
(516)

99% yield, 99% ee

R = Me, Et, nBu, iPr, Ph

$R^1$ = Me, iPr, tBu, Et

AND



racemic

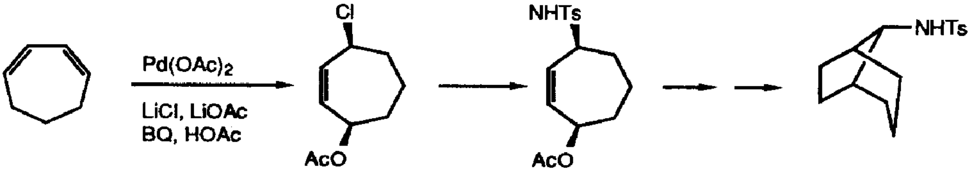
49%, 97% ee

51%, 96% ee

## D. Ethers, Esters, Acids.

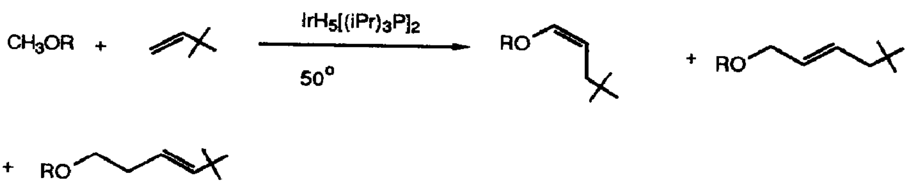
Palladium-catalyzed oxidation of 1,3-dienes to 1,4-diacetoxy-2-enes has been coupled to an oxygen reoxidation step via benzoquinone and cobalt tetraphenyl porphyrin [657] and also to an electrochemical reoxidation step [658], obviating the need for use of stoichiometric amounts of reoxidant. Palladium catalyzed chloroacetoxylation of cycloheptadiene was used to make tropane ring systems (equation 517) [659]. The copper catalyzed acetoxylation of butadiene was compared with the hydrocyanation of butadiene [660]. Palladium acetate/copper acetate mixed catalysts effected benzylic acetoxylations of alkyl aromatics [661], and allylic acetoxylation of 2-methylenebicyclo[2.2.1]heptanes [662]. The mechanism of catalytic oxidation of olefins by periodic acid in acetic acid solutions of palladium acetate has been studied [663].

(517)

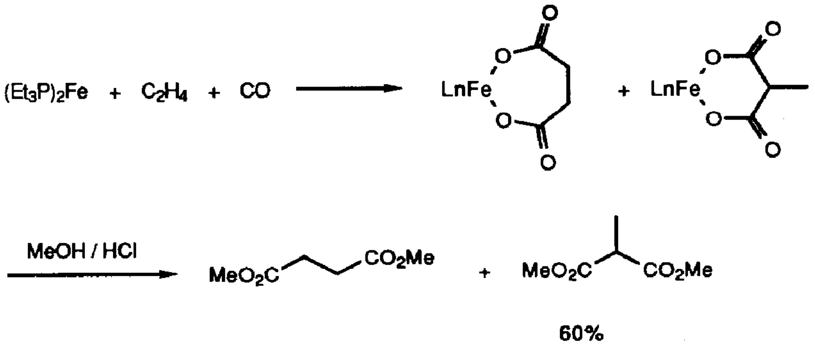


Iridium complexes catalyzed the addition of methyl ethers to t-butylethylene (equation 518) [664]. Low valent iron coupled ethylene and carbon dioxide to give diesters (equation 519) [665]. Ruthenium tetroxide/periodate oxidatively cleaved a number of cyclic compounds to keto acids (equation 520) [666]. Allyl acetates were converted to allyl phenyl ethers by reaction with phenols in the presence of palladium(0) catalysts (equation 521) [667]. Copper catalysts promoted the reaction of allylic chlorides and acetate with alcohols to give ethers (equation 522) [668]. Cobalt(II) chloride catalyzed the acetoxylation of β-hydroxyketones (equation 523) [669]. Copper(I) catalyzed the alkoxylation of aryl halides (equation 524) [670]. Ruthenium complexes catalyzed the addition of carboxylic acids to alkynes (equation 525) [671]. Cobalt(II) chloride catalyzed the reaction of acid chlorides with ether to give esters (equation 526) [672]. Ruthenium(II) hydrides catalyzed the disproportionation of alcohols to esters (equation 527) [673].

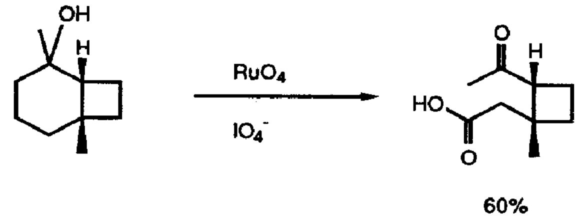
(518)



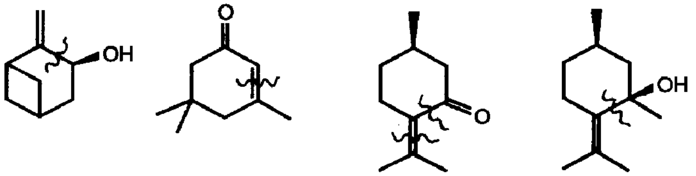
12 turns / 24 hr

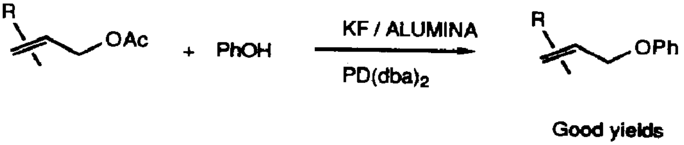

(Et3P)2Fe + C2H4 + CO
LnFe
O
O
O
O
+ LnFe
O
O
O
O
MeOH / HCl
MeO2C
CO2Me
+ MeO2C
CO2Me
60%

(519)

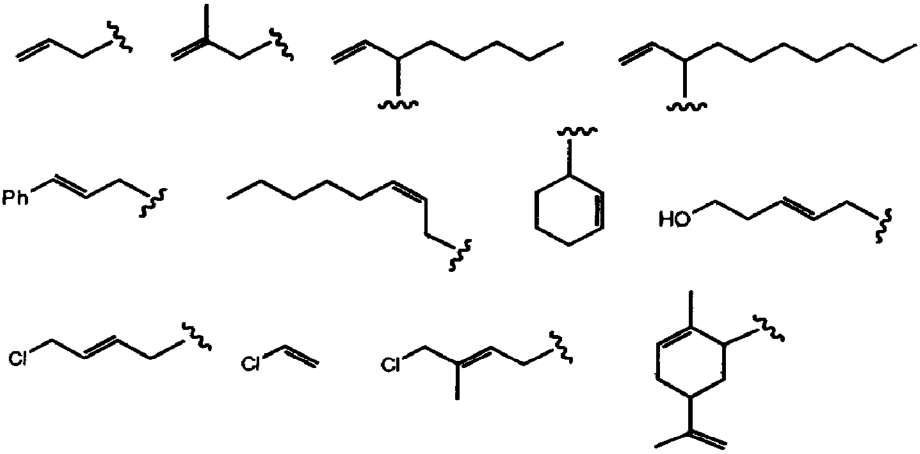

OH
H
RuO4
IO4-
O
H
HO
O
60%

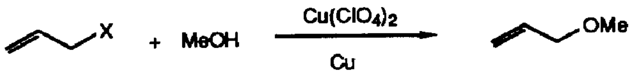
(520)


OH
O
O
OH


R
OAc
+ PhOH
KF / ALUMINA
PD(dba)2
R
OPh
Good yields

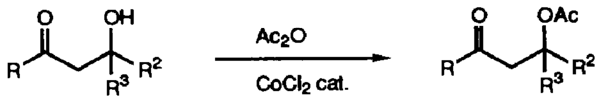
(521)


Ph
HO
Cl
Cl
Cl



(522)

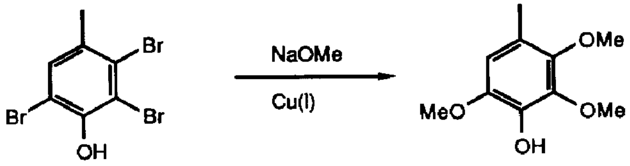
X = Cl, OAc



(523)

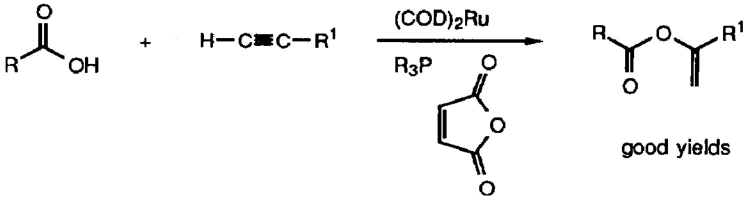
23 cases ROH ⟶ ROAc

more selective than DMAP RNH$_2$ ⟶ RNHAc



(524)

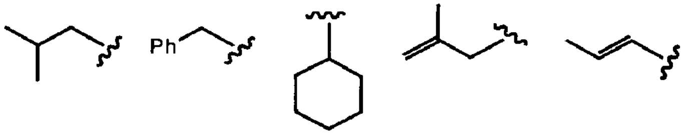
94%



(525)

good yields

R = C$_{18}$, Me, Et, iPr, adamantyl, ptolyl, pFPh, pClPh, pBrPh, pNH$_2$Ph

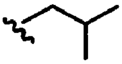


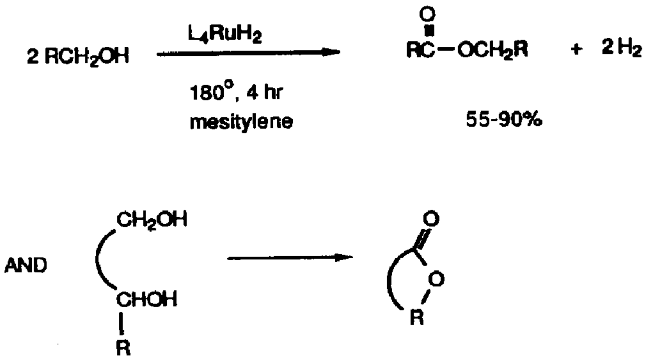
$$ROR + R^1COCl \xrightarrow[\text{MeCN}]{\text{CoCl}_2\text{ cat.}} R^1\overset{\overset{O}{\|}}{C}-OR$$

(526)

40-90%

R$^1$ = Me, Ph, nPr

R = Bu, Et, PhCH$_2$, Ph, 



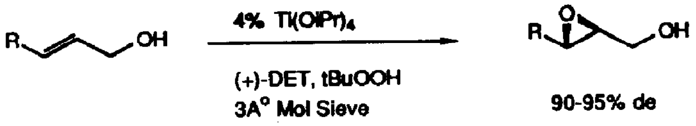
(527)

$$2\,RCH_2OH \xrightarrow[\text{180°, 4 hr, mesitylene}]{L_4RuH_2} R\overset{O}{\overset{\|}{C}}{-}OCH_2R + 2H_2$$

## E. Heterocycles

Metal-catalyzed epoxidations continue to be intensively studied, although conceptually new approaches are minimal. Asymmetric epoxidation has been the topic of four recent reviews: "Calculations on electronic and steric factors determining the asymmetric epoxidation of allylic alcohols" (Sharpless epoxidation) [674]; "Recent developments in asymmetric epoxidation" (48 references) [675]; "Asymmetric oxidation mediated by organometallic species" (47 references) [676]; "Asymmetric epoxidation by molybdenum(III) peroxo species" [677].

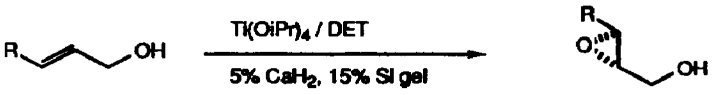
The full experimental details of the catalytic asymmetric epoxidation of allylic alcohols have been published (equation 528) [678]. A modified system which reacts faster than the Sharpless system in asymmetric epoxidation has been developed (equation 529) [679].



(528)

90-95% de

20 cases

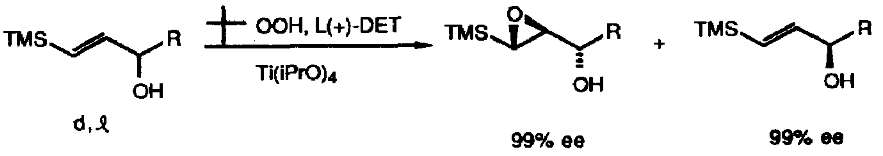


(529)

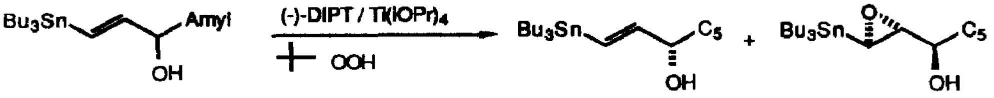
95% ee faster than Sharpless

A number of very very closely related kinetic resolutions of substituted allylic alcohols using Sharpless epoxidations have been reported (equations 530-533) [680-683].

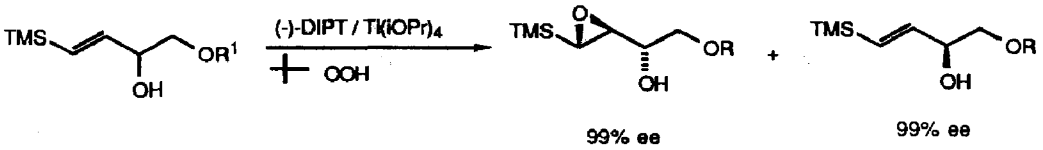
(530)

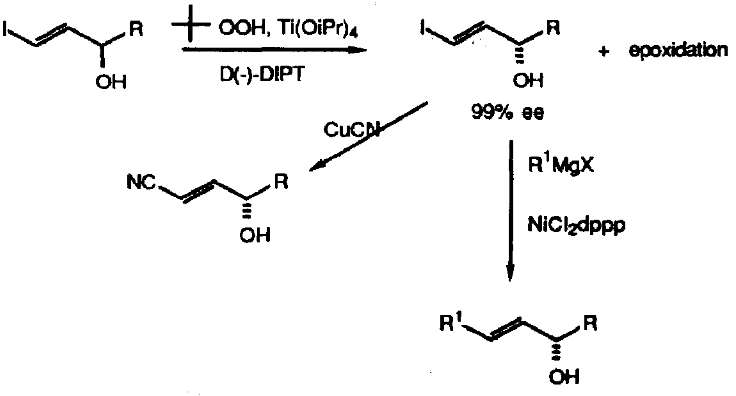


(531)



(532)

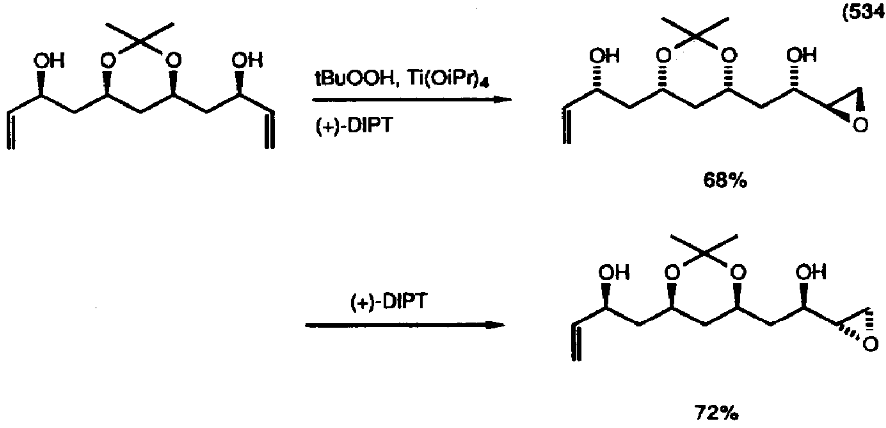




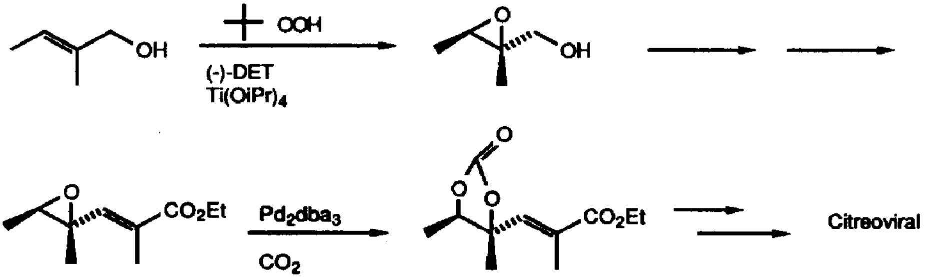
(533)

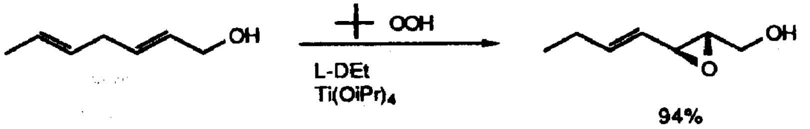
Sharpless asymmetric epoxidation continues to be used extensively in total synthesis (equation 534) [684], (equation 535) [685], (equation 536) [686].

(534)



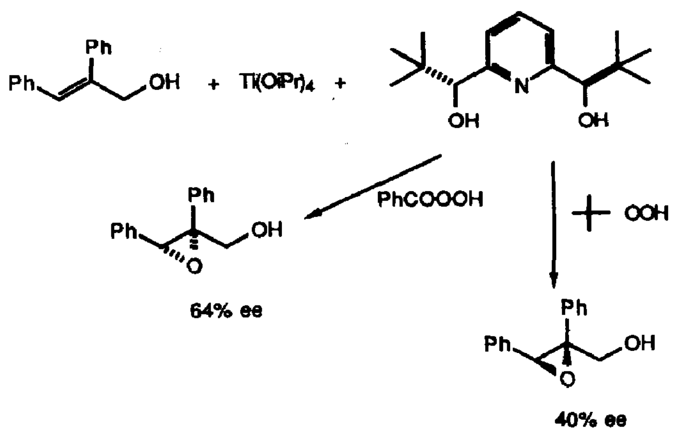
(535)



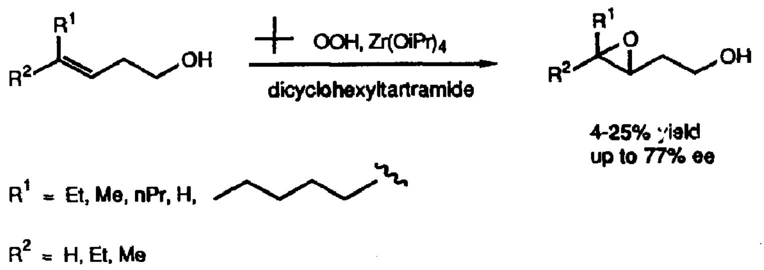


(536)

Notwithstanding the efficiency of Sharpless epoxidation, new systems continue to be studied (equation 537) [687], (equation 538) [688].



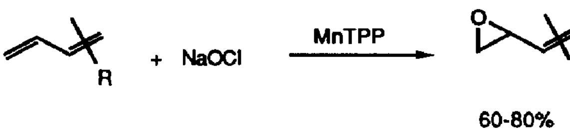
(537)



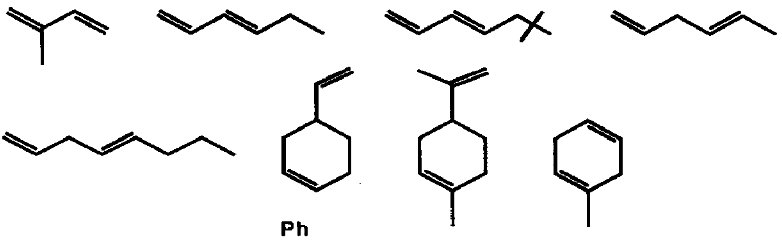
(538)

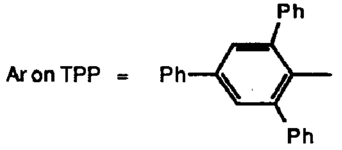
Many types of molybdenum and tungsten catalysts have been developed for epoxidation. These include molybdenum peroxide/t-butylhydroperoxide and polymer immobilized reagents [689], $MoO_2(amine)_2$ catalysts/t-butylhydroperoxide [690], ammonium tetrabromooxomolybdate [691], molybdenum/bisphosphine complexes [692], tungsten boride/hydrogen peroxide [693], heteropolytungstate hydrogen peroxide [694], peroxotungstate/HMPT [695] and bimetallic boron(III)/molybdenum(VI) catalysts [696].

Sterically-hindered porphyrins were used to catalyze the epoxidation of the less hindered olefin of dienes (equation 539) [697]. Ruthenium(III) chloride catalyzed the epoxidation of cyclohexene and 1-octene by N-methylmorpholine-N-oxide [698]. Iodosylbenzene, PhIO, and t-butylhydroperoxide were compared as epoxidizing agents for olefins in the presence of cobalt(I) catalysts. The former underwent reaction by a two electron process while the latter proceded by a radical chain process [699].

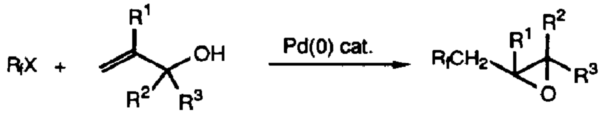


(539)





Allyl alcohols underwent reaction with perfluoroalkyl halides in the presence of palladium catalysts to give epoxides (equation 540) [700]. Norbornenes were epoxidized by palladium(II) nitrile complexes (equation 531) [701]. Nickel(II) Shiff's base complexes catalyzed the epoxidation of olefins by iodosylbenzene [702]. Chromium complexed benzaldehydes were converted to epoxides with high enantiomeric excess (equation 542) [703]. α-Pinene and car-3-ene were epoxidized by cobalt naphthenate catalyzed autoxidation [704].



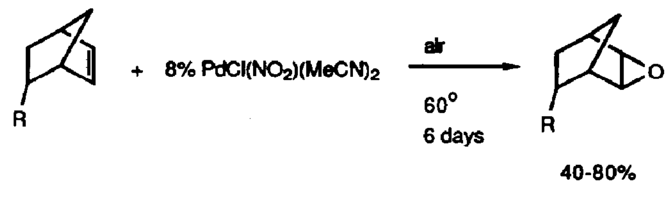
(540)

$R_f = C_8F_{17}, CF_3, C_2F_5, C_3F_2, iC_3F_7, C_4F_9, C_6F_{13}, CF_2BrCF_2, CF_2ClCFCl$

$R^1 = H, Me$

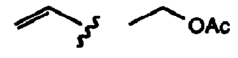
$R^2 = H, Me$

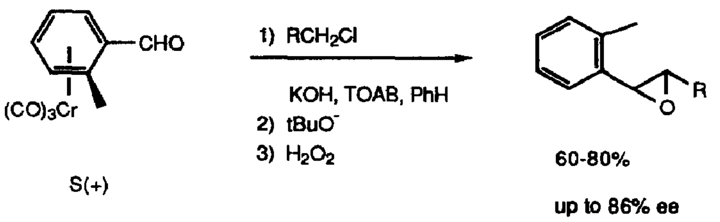
$R^3 = H, Me, Et, C_6$



(541)

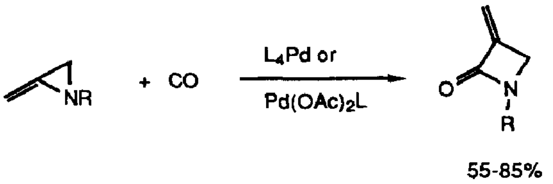
R = H, OAc,



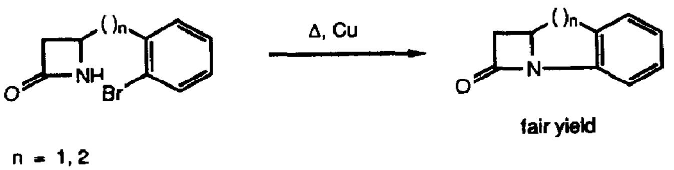


(542)

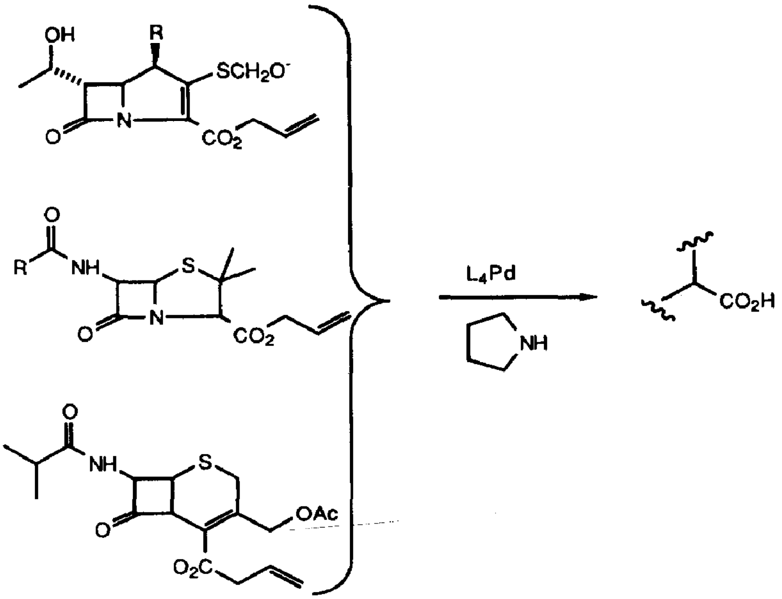
Methyleneaziridines were converted to β-lactams by carbonylation under palladium catalysis (equation 543) [705]. β-Lactams were N-arylated by aryl halides when treated with copper (equation 544) [706]. Allyl ester protecting groups were removed from β-lactams using palladium(0) catalysts (equation 545) [707]. Lactams were produced from the reaction of alkynes with ruthenium carbonyl nitrene complexes (equation 546) [708].



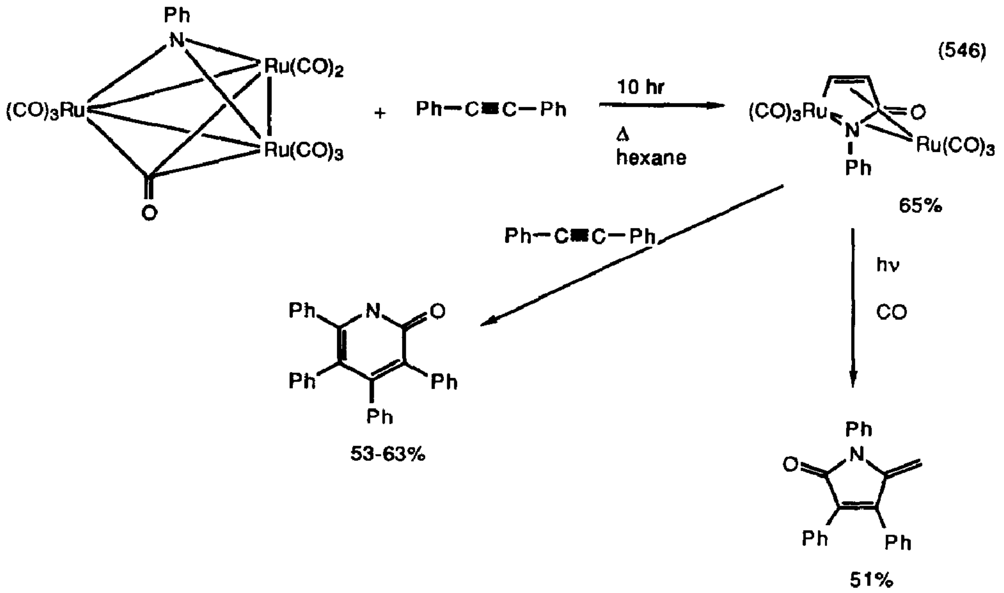
(543)



(544)

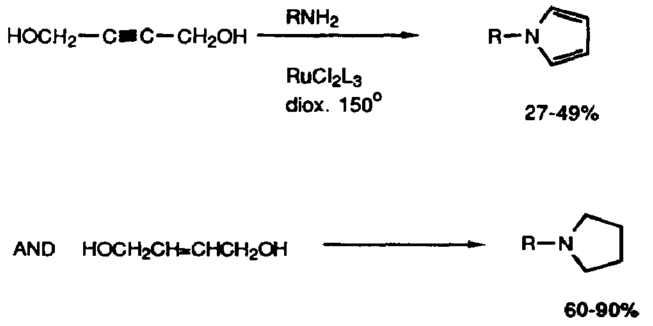


(545)



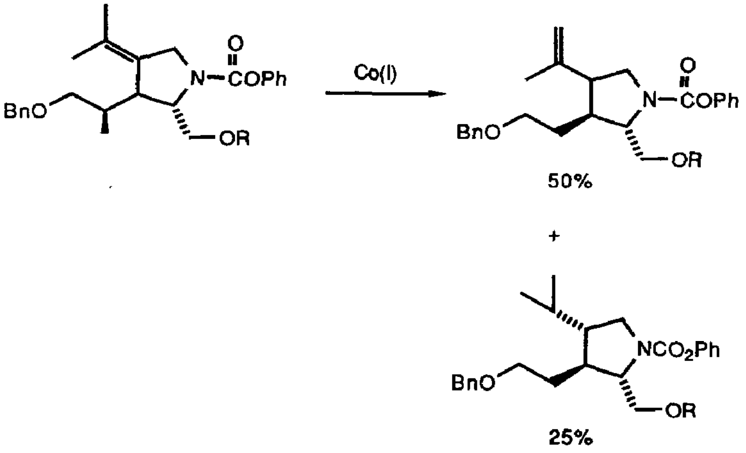
(546)

Palladium catalyzed intramolecular additions of OH, NH and $CO_2H$ to acetylenes in the synthesis of heterocycles has been reviewed (929 references) [709], as has heterocyclic synthesis by using the oxidating potential of palladium(II) (24 references) [710]. Nickel catalyzed reactions of trimethylene methane derivatives with imines gave pyrrolidines [711]. Ruthenium(II) complexes converted 1,4-dihydroxy-2-butynes and -2-butenes into pyrroles and pyrrolidines (equation 547) [712]. Cobalt(I) radical cyclization was used to make substituted pyrrolidines (equation 548) [713]. Rhenium π-complexes of pyrroles were alkylated by organolithium reagents (equation 549) [714]. Anilines reacted with diols to give indoles or quinolines in the presence of ruthenium(III) chloride (equation 550) [715]. An indoline system was prepared by palladium catalyzed β-amination of a conjugated enone (equation 551) [716].

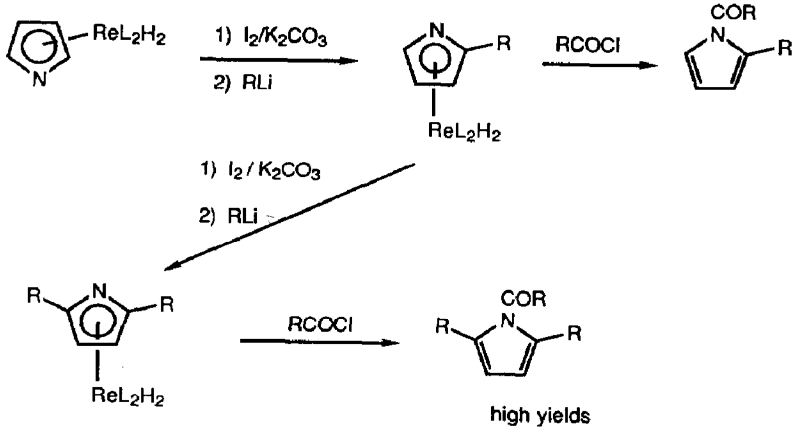


(547)

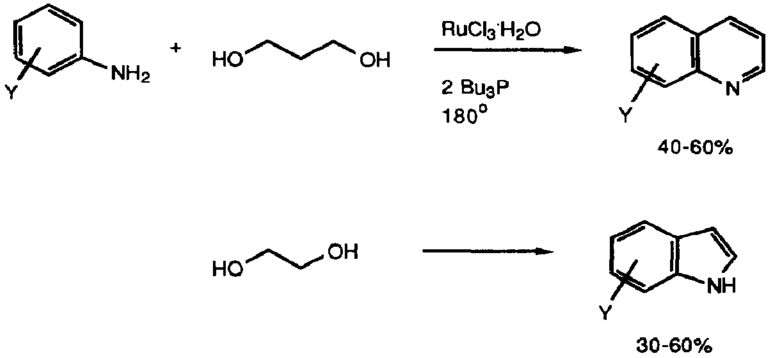
(548)

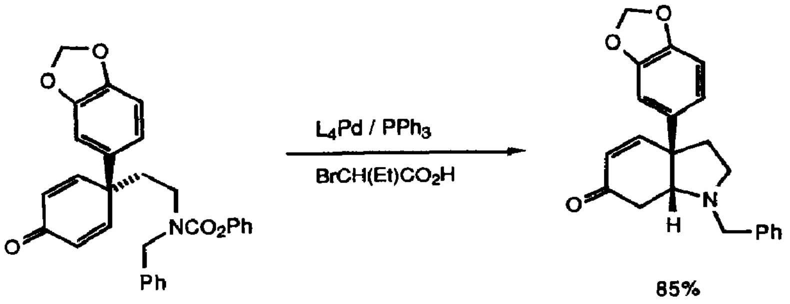

Co(I)
BnO
N—COPh
OR
50%
+
NCO2Ph
25%

(549)


ReL2H2
1) I2/K2CO3
2) RLi
RCOCl
COR
R
1) I2 / K2CO3
2) RLi
RCOCl
high yields

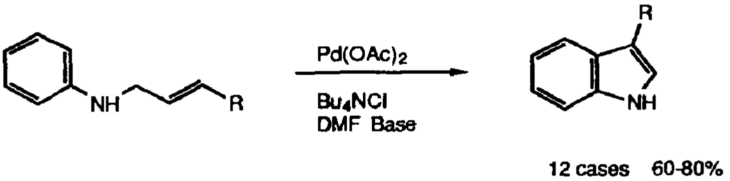
(550)


NH2
Y
+
HO
OH
RuCl3·H2O
2 Bu3P
180°
N
40-60%
NH
30-60%



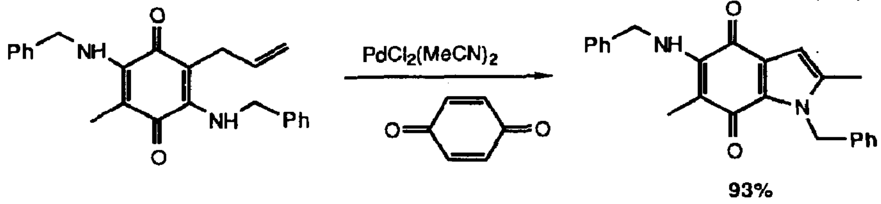
(551)

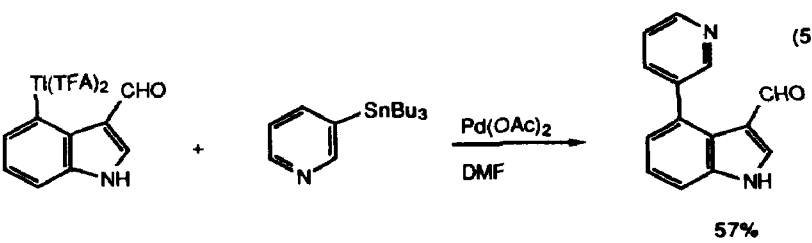
o-Iodo-N-allyl anilines were cyclized to indoles using mild conditions and palladium catalysts (equation 552) [717]. Indoloquinones were prepared by palladium(II) catalyzed olefin amination (equation 553) [718]. Indoles were arylated at the 4-position by palladium(II) transmetallation chemistry (equation 554) [719]. Palladium catalysis figured extensively in the synthesis of clavicipitic acid (equation 555) [720] and aurantioclavine (equation 556) [721].



(552)

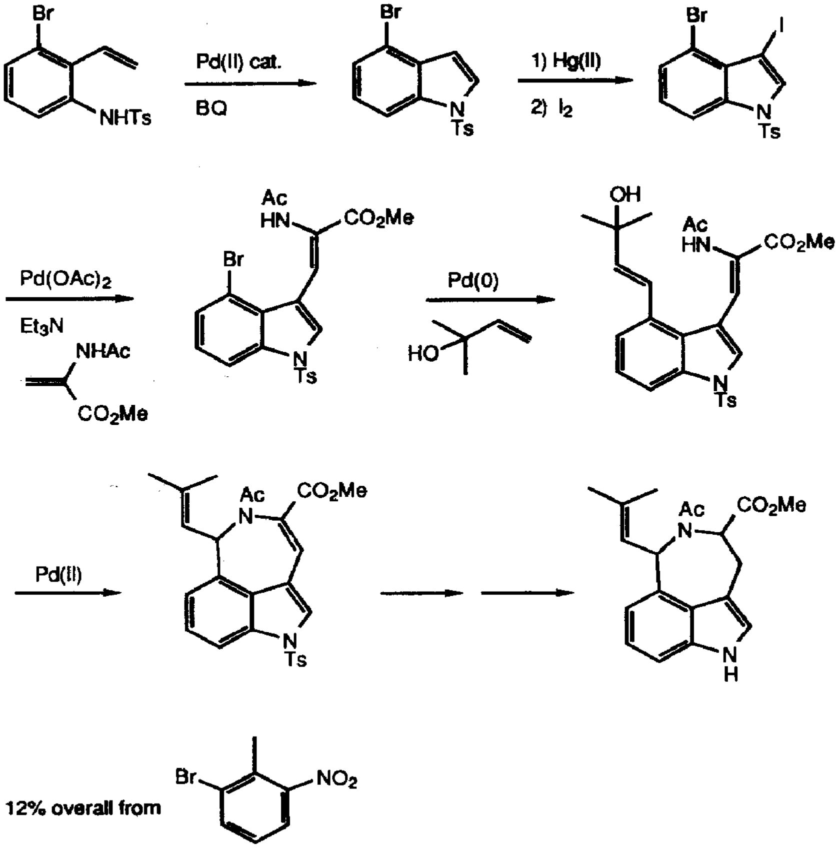
(553)



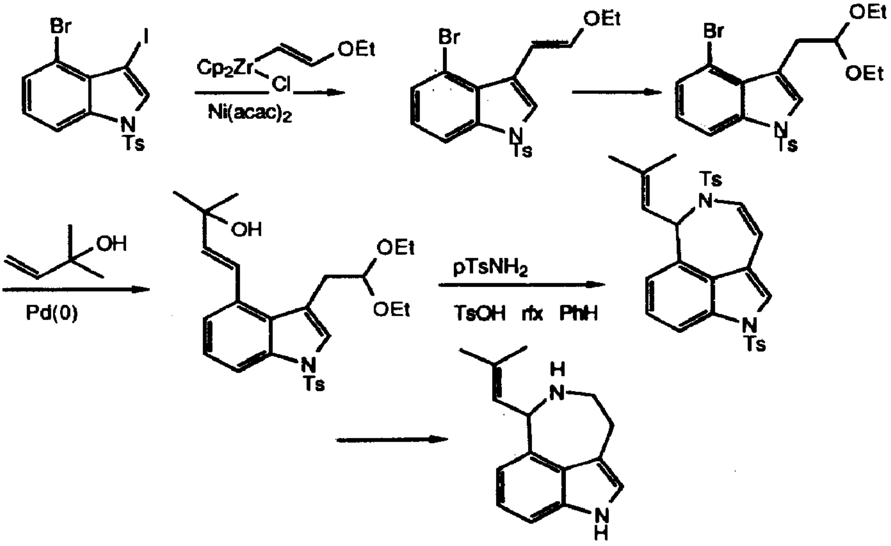


(554)

(555)

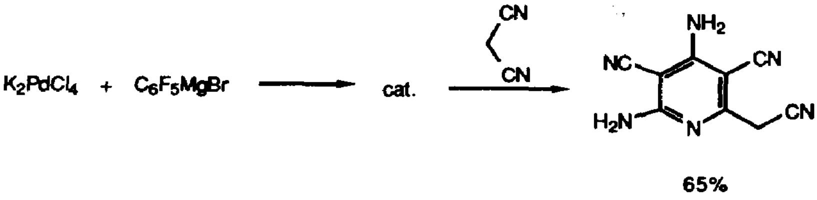

Br
NHTs
Pd(II) cat.
BQ
Br
N
Ts
1) Hg(II)
2) I2
Br
I
N
Ts
Pd(OAc)2
Et3N
NHAc
CO2Me
Ac
HN
CO2Me
Br
N
Ts
Pd(0)
HO
OH
Ac
HN
CO2Me
N
Ts
Pd(II)
Ac
N
CO2Me
N
Ts
Ac
N
CO2Me
N
H
12% overall from
Br
NO2

(556)

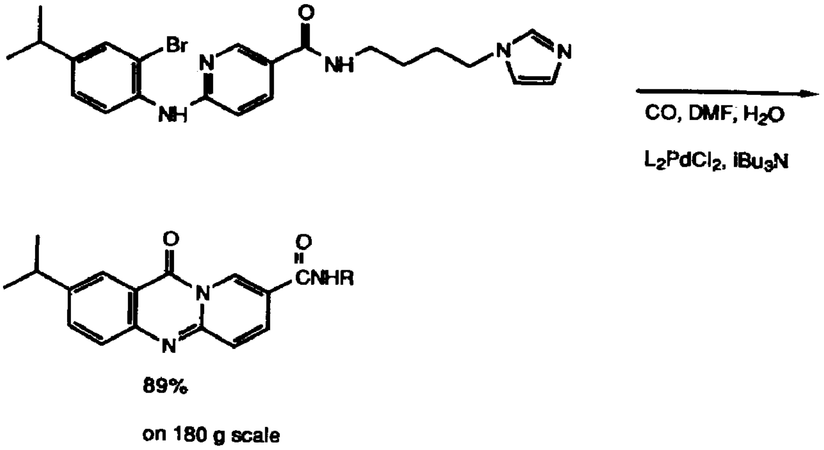

Br
I
N
Ts
Cp2Zr
OEt
Cl
Ni(acac)2
Br
OEt
N
Ts
Br
OEt
OEt
N
Ts
OH
Pd(0)
OH
OEt
OEt
N
Ts
pTsNH2
TsOH rfx PhH
Ts
N
N
Ts
H
N
N
H

Malononitrile was cyclotrimerized by palladium/perfluoroarene complexes (equation 557) [722]. Palladium catalyzed cyclocarbonylation to give tricyclic pyridones went in excellent yield on 180 g scale (equation 558) [723]. Alkynes inserted into cyclopalladated 1-naphthyl amines (equation 559) [724]. Chiral ferrocenyl amine was used as a chiral auxilliary for the synthesis of the lactam C Corynoline (equation 560) [725].

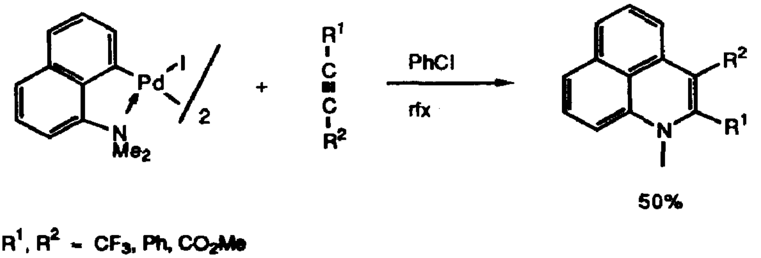
(557)

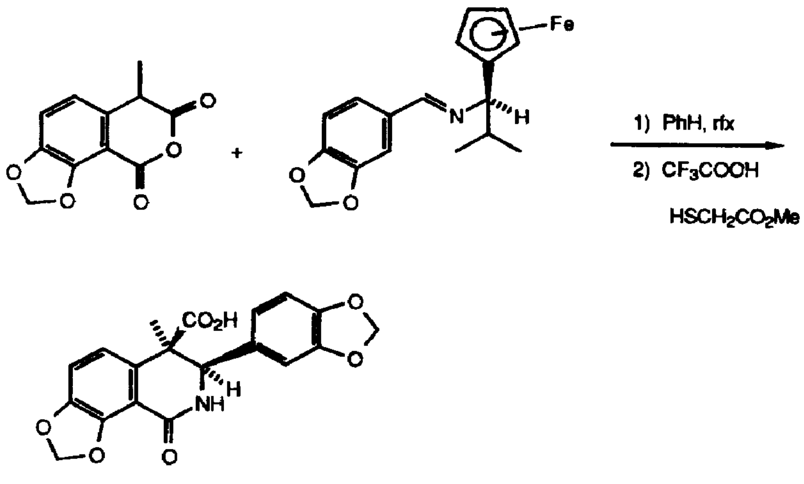


(558)



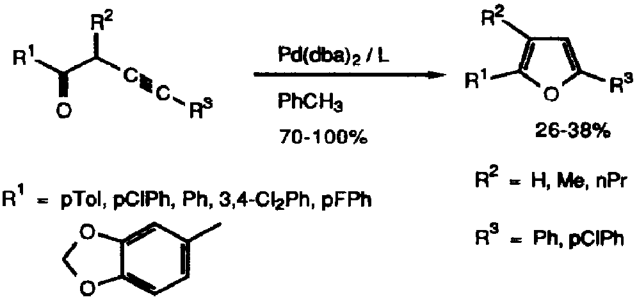
(559)



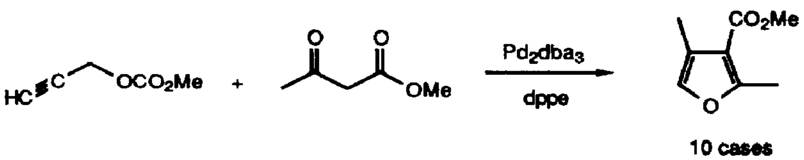


(560)

Acetylenic ketones were cyclized to furans by palladium(0) catalysts (equation 561) [726]. β-Keto esters condensed with propargyl alcohol esters to give furans in the presence of similar catalysts (equation 562) [727]. γ-Hydroxy allenes were cyclized to furans by palladium(II) catalysts (equation 563) [728]. Cyclopropenyl ketones and esters were carbonylated to pyrones and furans by rhodium(I) catalysts (equation 564) [729]. Cyclopropenones were converted to furans by reaction with trimethylsilyl cyanide (equation 565) [730].



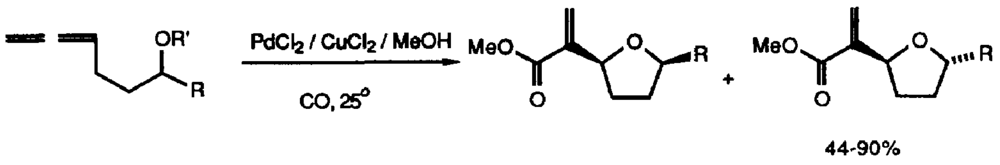
(561)



10 cases

(562)

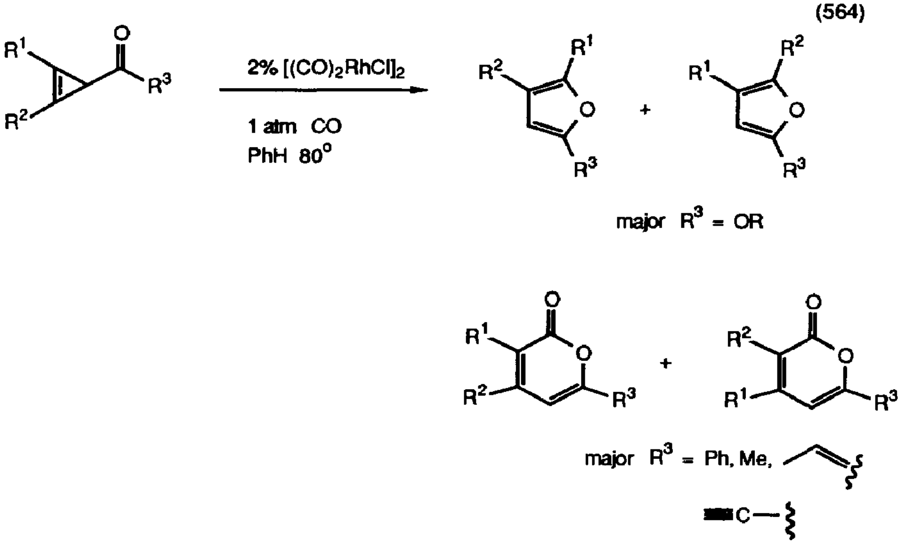
(563)

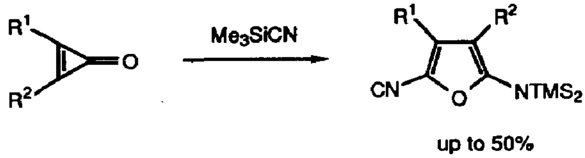


R = H, Me, $CH_3COCH_2$, $CH_2CO_2tBu$

$R^1$ = H, $Me_2SitBu$

(564)

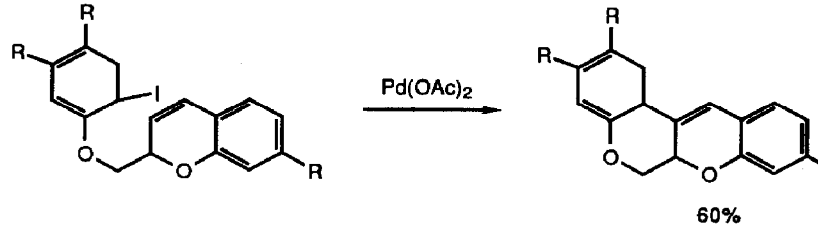




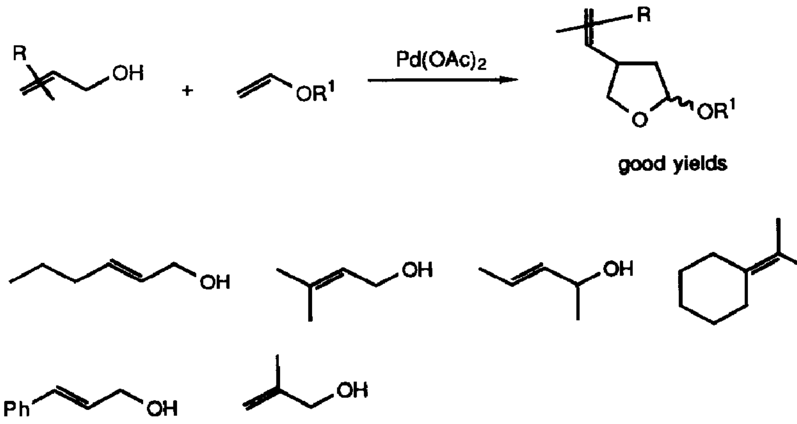
(565)

$R^1, R^2$ = Ph, $-(CH_2)_5-$

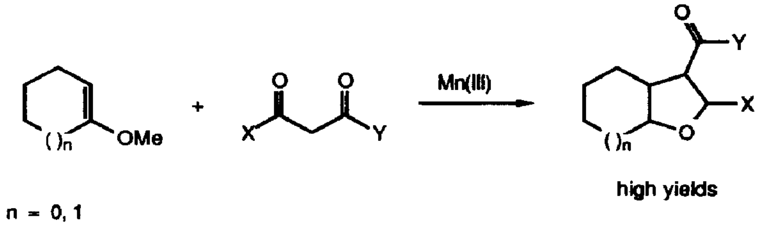
Mundaserone was synthesized by palladium catalyzed intramolecular arylation to form a pyran ring (equation 566) [731]. Palladium acetate catalyzed the reaction between enol ethers and allyl alcohols to give tetrahydrofurans (equation 567) [732]. Manganese(III) acetate promoted the reaction of enol ethers with β-dicarbonyl compounds to give tetrahydrofurans (equation 568) [733]. Dihydrobenzofurans were prepared by a cobalt catalyzed radical cyclization of o-bromo-O-alkylphenols (equation 569) [734].



(566)

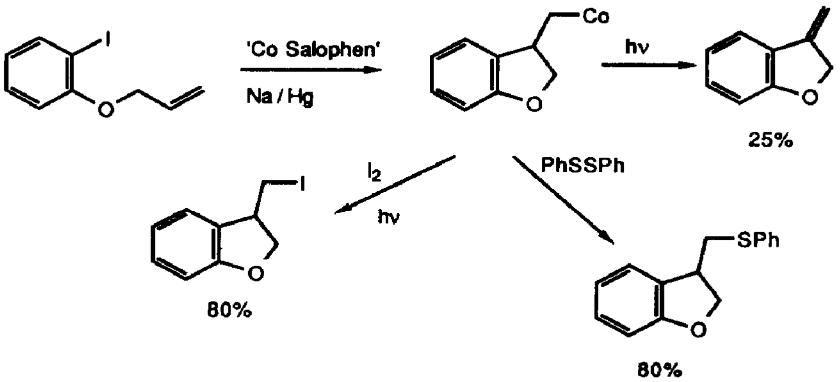


(567)



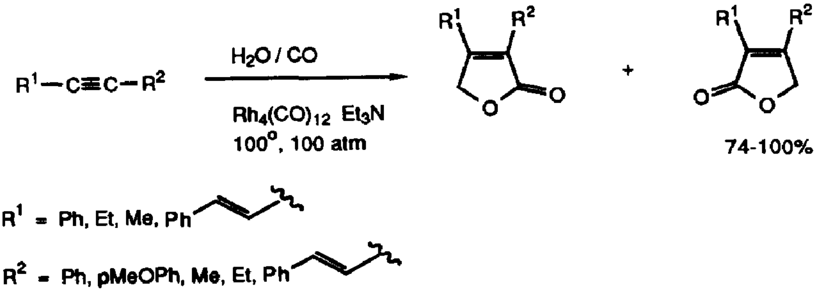
(568)

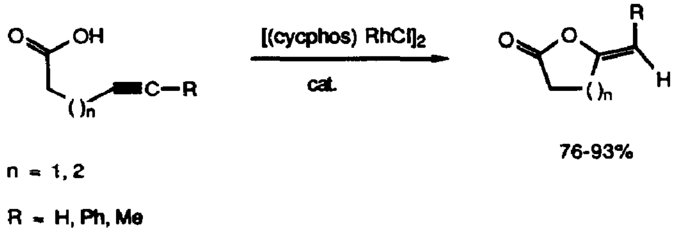
(569)



A review dealing with ruthenium catalyzed stereoselective lactone synthesis (14 references) has appeared) [735]. Alkynes were carbonylated to butenolides by rhodium carbonyl catalysts (equation 570) [736]. Rhodium(I) complexes cyclized ω-acetylenic acids to lactones (equation 571) [737]. Macrocyclic acetylenic lactones were prepared using cobalt-stabilized propargyl cation chemistry (equation 572) [738]. Nickel(0) complexes converted a 6-membered cyclic anhydride to a 5-member cyclic anhydride (equation 573) [739]. Ruthenium complexes catalyzed the reductive cyclization of butadiene-2,3-carboxylates to butyrolactones (equation 574) [740].

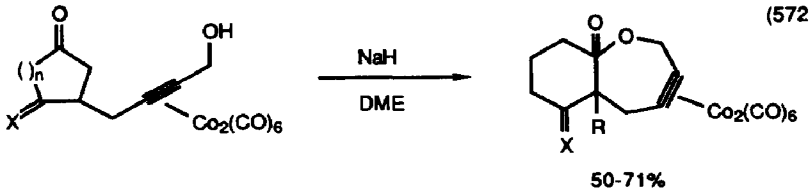
(570)

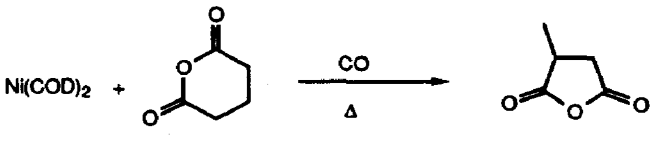




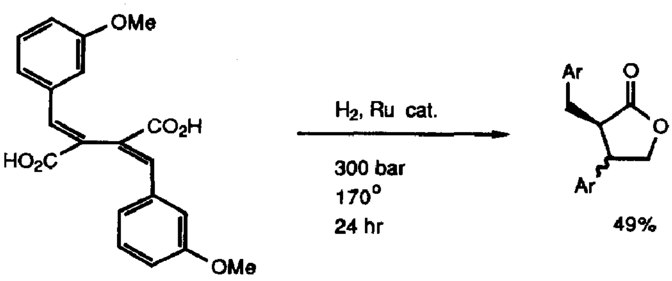
(571)

(572)



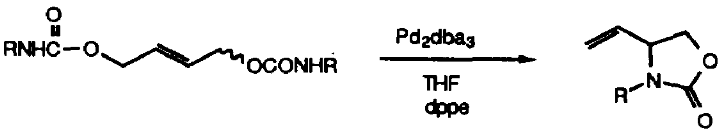


(573)



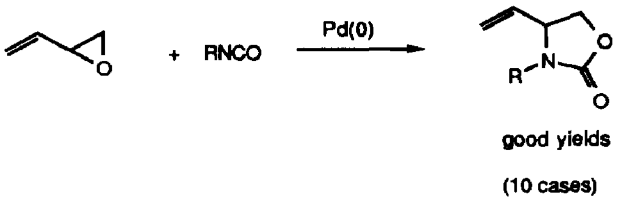
(574)

Palladium catalysis was used in several different systems to make cyclic carbamates from butadiene derivatives (equation 575) [741], (equation 576) [742], (equation 577) [743]. Chiral oxazolines were made in high yield with high diastereoselectivity by the gold(I) catalyzed reaction of aldehydes with isocyanoacetic esters (equation 578) [744]. Cobalt carbonyl converted N-acyl diazirines to oxazolines (equation 579) [745]. A variety of heterocycles were prepared as in equation 580 [746]. Palladium(0) catalyzed the reaction thioamides with iodoanilines to give benzothiazolines (equation 581) [747]. Iron pentacarbonyl converted ketenimines to lactams (equation 582) [748]. Rhodium(I) complexes catalyzed the reaction o-phenylene diamines with aliphatic aldehydes to give benzimidazoles [749].

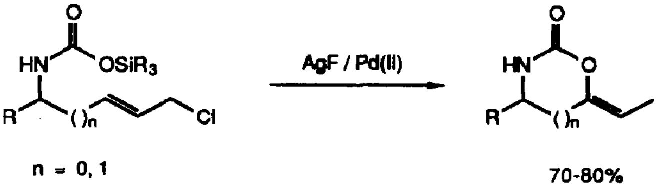


(575)

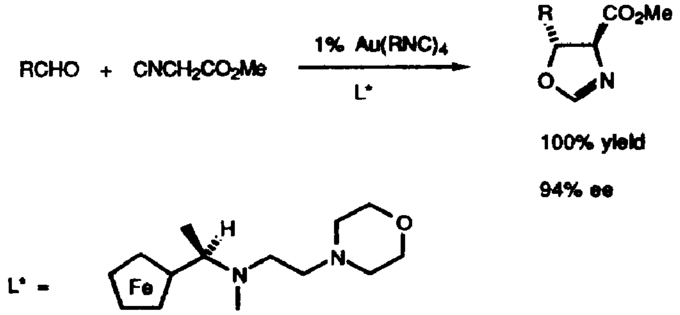
R = Ph, pMeOPh, pClPh, $PhCH_2$, iPr, Me, PhCO



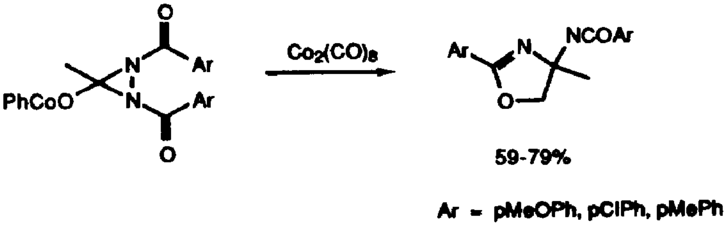
(576)



(577)

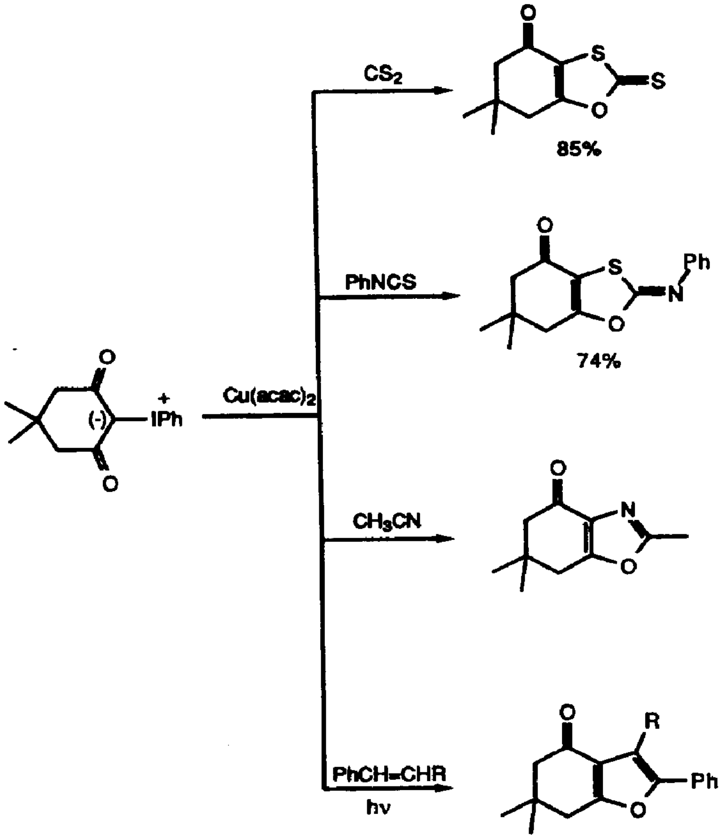

RCHO + CNCH$_2$CO$_2$Me
1% Au(RNC)$_4$
L*
R
CO$_2$Me
O
N
100% yield
94% ee
L* =
Fe
H
N
N
O

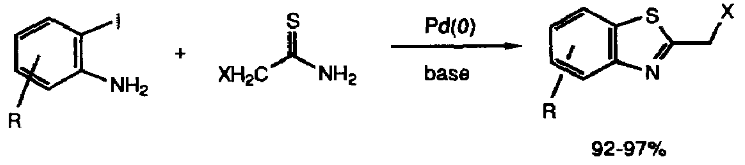
(578)


O
N
Ar
PhCoO
N
Ar
O
Co$_2$(CO)$_8$
Ar
N
NCOAr
O
59-79%
Ar = pMeOPh, pClPh, pMePh

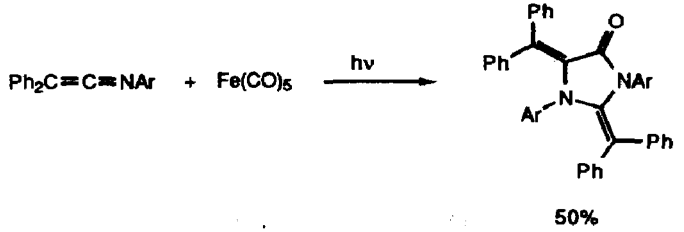
(579)

(580)


O
(-)
+
IPh
O
Cu(acac)$_2$
CS$_2$
S
S
O
85%
PhNCS
S
Ph
N
O
74%
CH$_3$CN
N
O
PhCH=CHR
hν
R
Ph
O

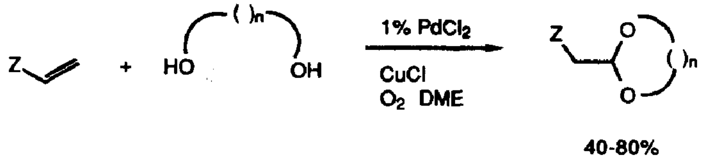


(581)



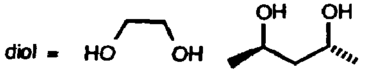
(582)

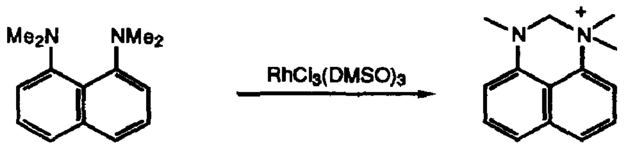
A number of unusual, metal catalyzed, heterocycle-forming reactions have been developed (equation 583) [749], (equation 584) [750], (equation 585) [751], (equation 586) [752], (equation 587) [753], (equation 588) [754].



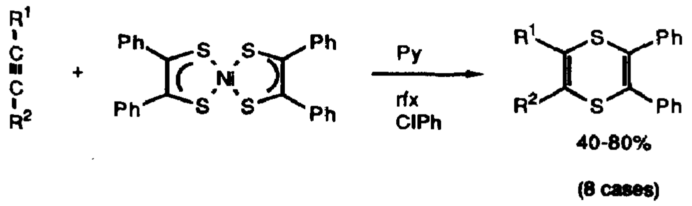
(583)

Z = $CO_2Me$, CN, COMe, Ph, ptoly



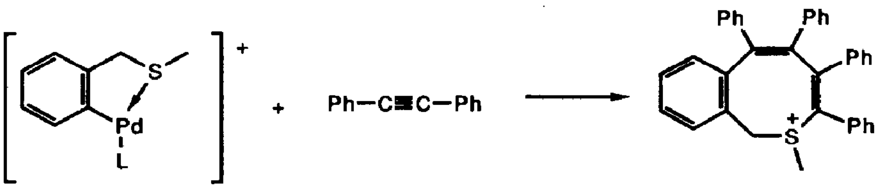


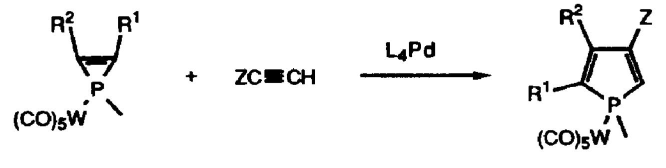
(584)



(585)

(586)

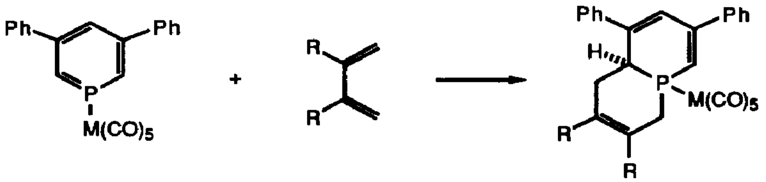




(587)

$R^1 = R^2 = Ph, Et, CO_2Et$ 35-85%

$Z = Ph, EtO, CO_2Et$



(588)

## F. Alkanes, Alkenes

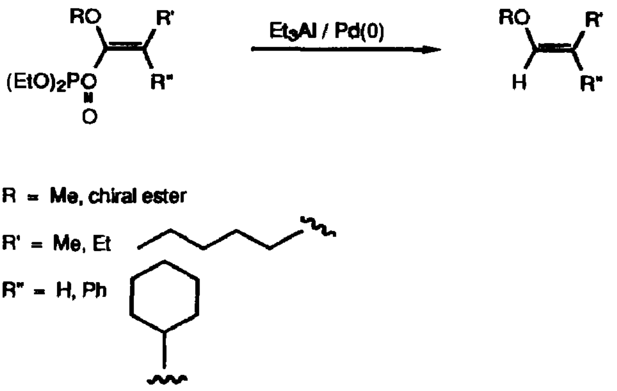
A wide variety of aryl triflates were reduced to arenes using palladium(0)/hydride reducing systems (equation 589) [755]. Enol phosphates were reduced to alkenes by palladium(0)/triethylaluminum (equation 590) 756]. Allylsulfones were reduced by borohydride in the presence of palladium catalysts (equation 591) [757]. Reduction of α-haloketones to ketones by phenyl silane was catalyzed by molybdenum hexacarbonyl (equation 592) [758]. Propargyl halides, mesylates and phosphonates were reduced to allenes by hydrides and palladium catalysts (equation 593) [759].

(589)

$$\mathrm{ArOTf} \xrightarrow[\text{or } \mathrm{Et_3NH_2CO_2}\text{-}\ \mathrm{L_2Pd(OAc)_2}]{\mathrm{NaBH_4\ /\ Pd(0)}} \mathrm{ArH}$$

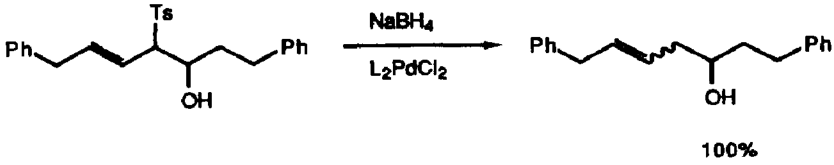
50-90%

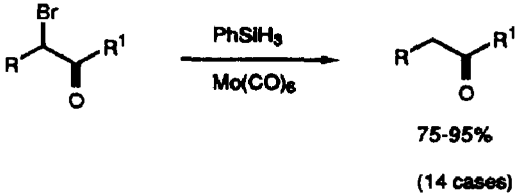
$Ar = Ph, pCNPh, pMeOPh, pCO_2MePh$
1-Naph, etc.



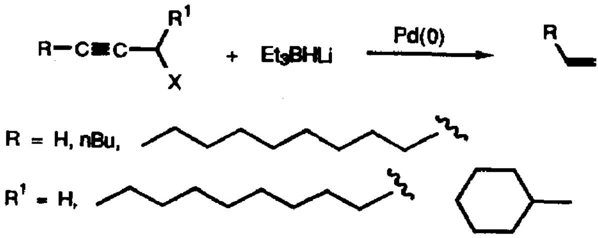
(590)

(591)





(592)

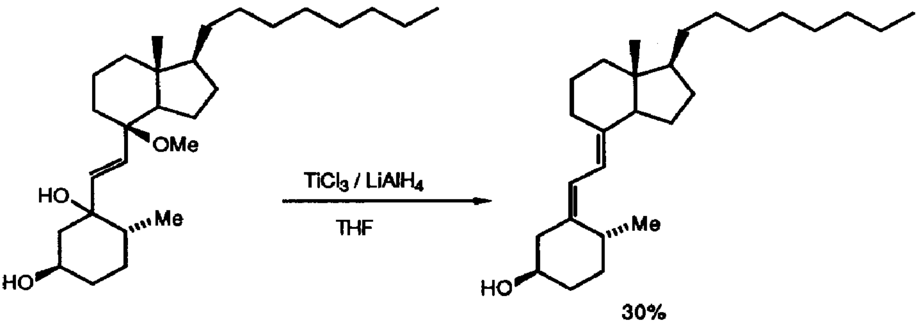


(593)

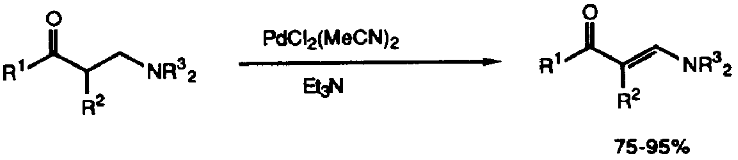
Molybdenum hexacarbonyl catalyzed the dehydrochlorination of compounds containing the trichloromethyl group to give gem-dichloroalkenes [760]. Low valent titanium converted allylic alcohols to dienes (equation 594)

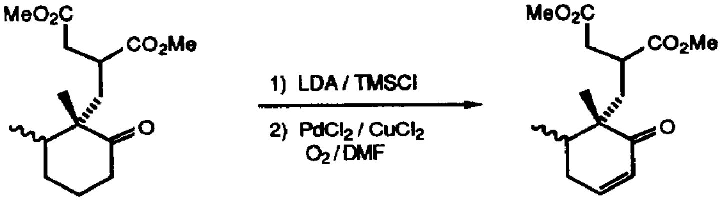
[761]. Ketones were oxidized to conjugated enones by palladium(II) compounds (equation 595) [762] (equation 596) [763]. Dithioketals were converted to olefins by reaction with Grignard reagents and nickel catalysts (equation 597) [764].

(594)

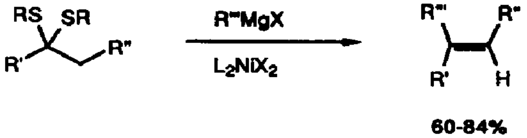


(595)





(596)



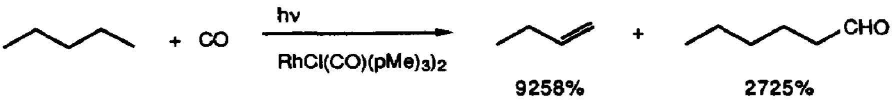
(597)

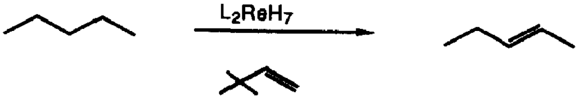
R' = Ph; R" = Ph, Me' R''' = Me, Et, nPr, iPr, nBu, Ph

Alkanes were oxidized to alkenes by rhodium (equation 598) [765], rhenium (equation 599) [766], and iridium catalysts (equation 600) [767]. Copper(I) salts catalyzed the air oxidation of indolines to indoles [768].

Allyl benzyl carbamates were cleaved by palladium(0)/tin hydride catalysts (equation 601) [769]. Benzocyclobutanes were prepared from arenes via zirconium chemistry (equation 602) [770].

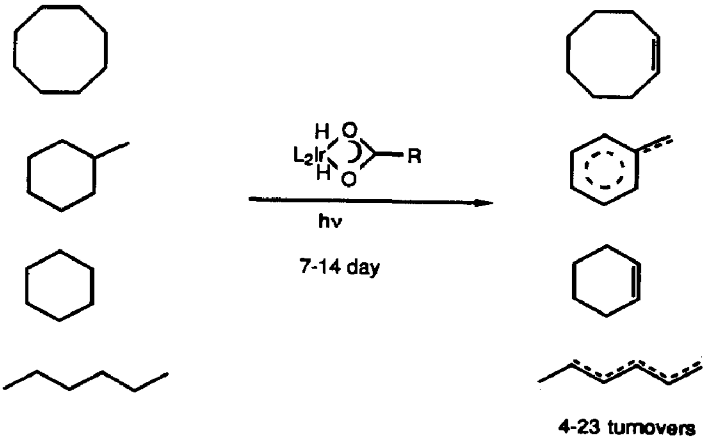
(598)



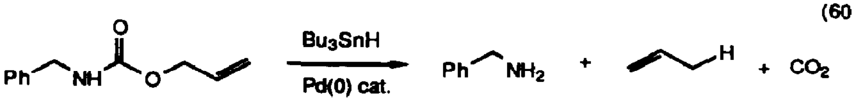


(599)

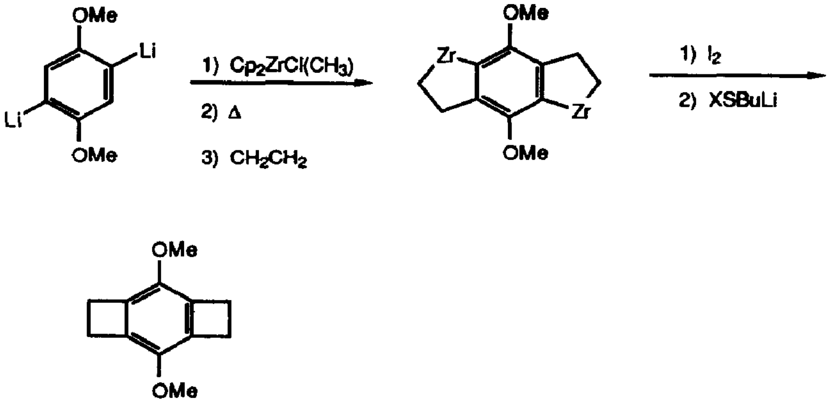
(600)



(601)



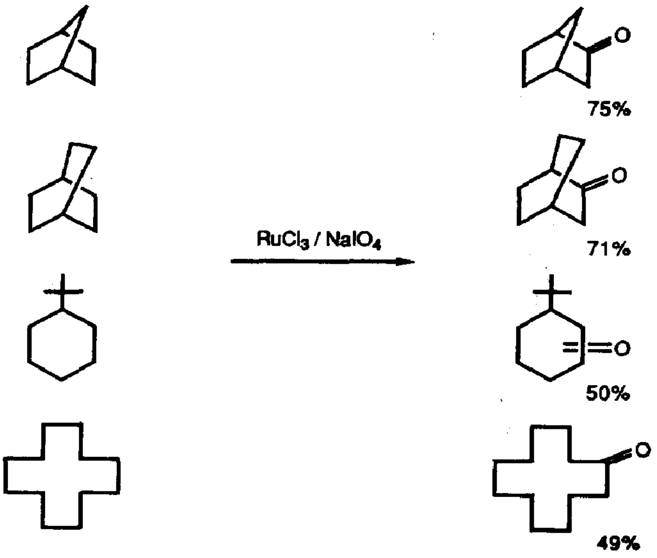
(602)



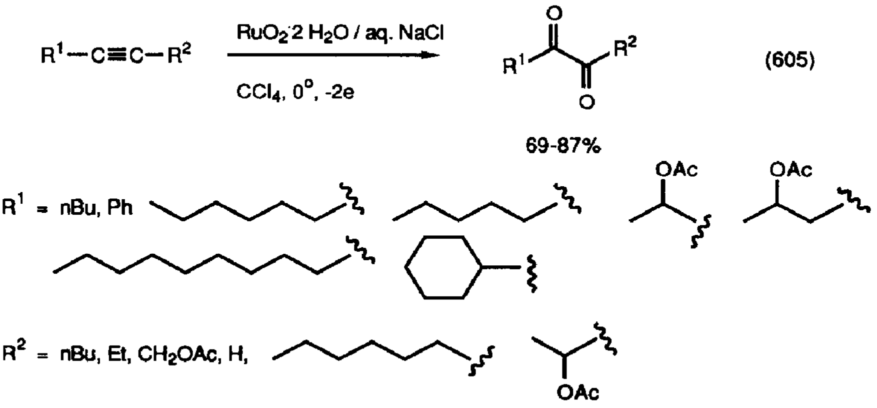
## G. Ketones, Aldehydes

Cycloalkanes were oxidized to ketones by ruthenium(III) chloride, sodium periodate (equation 603) [771]. Terminal olefins were oxidized to methyl ketones by palladium(II) acetate/oxygen [772], by $PdCl_2/CuCl_2$ in the presence of cyclodextrins [773][774], by oxygen in the presence of acetoxime/copper chloride/palladium chloride [775], and by palladium chloride, using electrochemical reoxidation through quinone intermediates [776]. Palladium(0) olefin complexes isomerized 2-buten-1,4-diol to the corresponding hydroxy aldehyde in good yield (equation 604) [777]. Alkynes were oxidized to α-diketones by ruthenium oxide (equation 605) [778], and were hydrated to ketones by palladium(II) catalysts with ultrasound (equation 606) [779]. Palladium clusters catalyzed the air oxidation of hydroquinones to quinones [780].

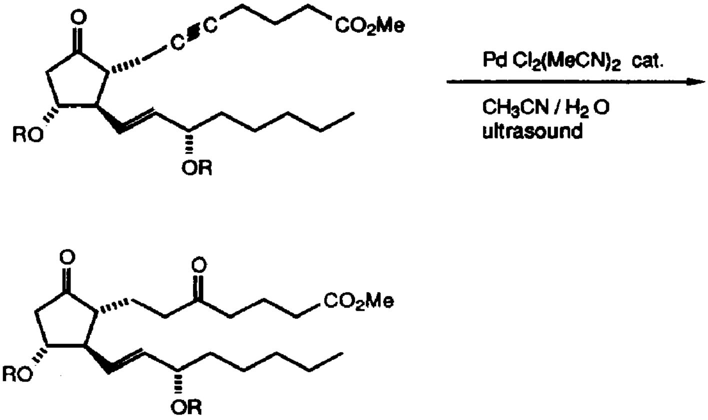
(603)



(604)

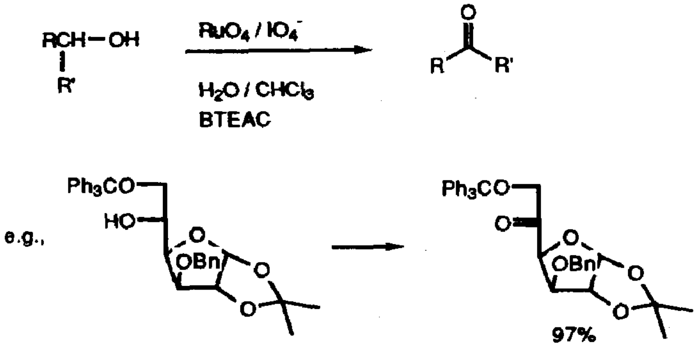


(605)

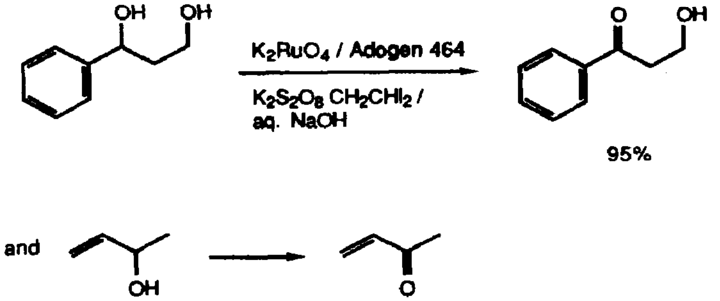


(606)

Ruthenium tetraoxide/periodate oxidized alcohols to ketones (equation 607) [781], while ruthenate peroxydisulfate took diols to ketoalcohols (equation 608) [782]. Amine oxides oxidized alcohols to ketones or aldehydes in the presence of ruthenate (equation 609) [783]. Molybdenum peroxo species performed similar oxidations (equation 610) [784]. In contrast $Mn_2O_7$ oxidized primary alcohols to carboxylic acids, but secondary alcohols were cleanly oxidized to ketones (equation 611) [785]. Allyl alcohols (equation 612) [786] were oxidized to conjugated enones by $Cp_2ZrH_2$, while steroidal alcohols were oxidized to $\Delta^{4,6}$-dien-3-ones by polymer anchored palladium chloride [787].

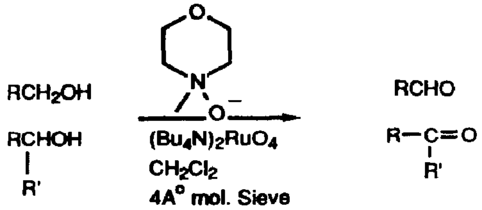


(607)



(608)

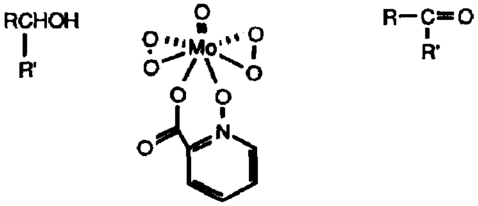
(609)



high yields

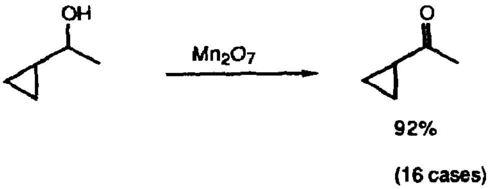
(20 cases)

(610)



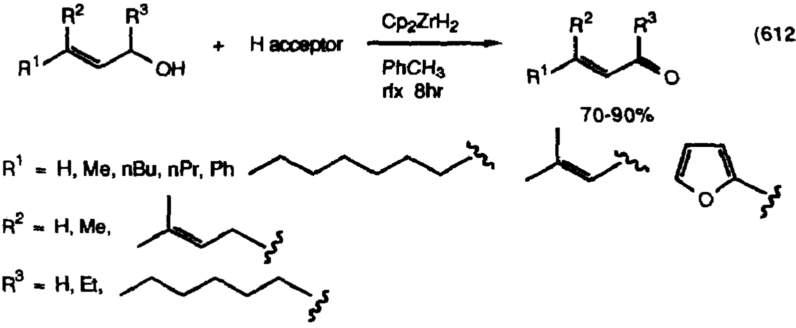
high yields

(12 cases)



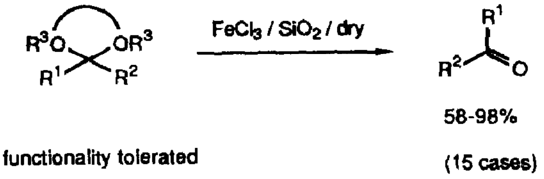
(611)

(olefins → diacids ethers → esters)

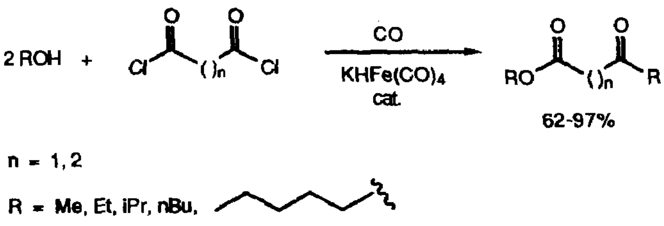


(612)

Acetals and ketals were cleaved by iron(III) chloride on dry silica gel (equation 613) [788]. Methyl acetate was reduced to acetaldehyde by hydrogen carbon monoxide and methyl iodide in the presence of palladium(II) acetate [789]. α,ω-Bis acid chlorides were converted to keto esters when treated with carbon monoxide and $KHFe(CO)_4$(equation 614) [790].

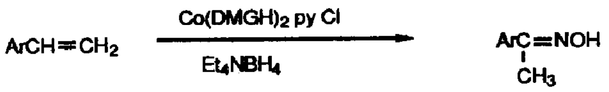


(613)



(614)

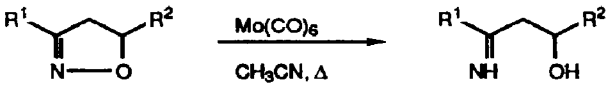
Styrenes were converted to oximes by reduction in the presence of cobalt dimethylglyoxime complexes (equation 615) [790]. Molybdenum carbonyl cleaved the N-O bond of cyclic oximes to give β-hydroxy imines (equation 616) [791]. Chromium-complexed toluenes were oximated by reaction with tBuONO (equation 617) [792]. O-Allyl oximes were readily converted to oximes by reaction with palladium(0) catalysts and ammonium formates [793]. Imines were made by two strange reactions (equation 618) [794], (equation 619) [795].



(615)

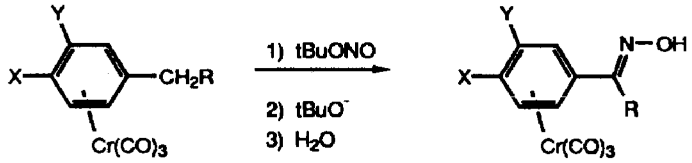
30-70%

Ar = Ph, pClPh, oClPh, oPrPh, oMePh, pMePh, pMeOPh, CH$_2$=CH-Ph



(616)

fair yield



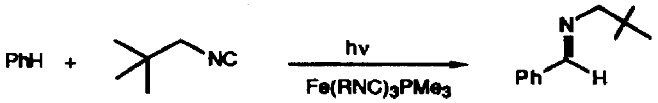
(617)

R = H, Me, Et, MeO

X = H, tBu, Me

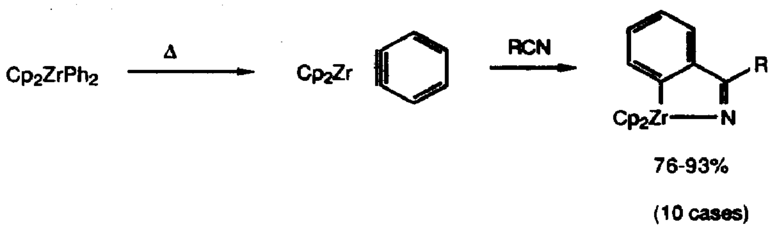
Y = MeO, H

65-80%



(618)

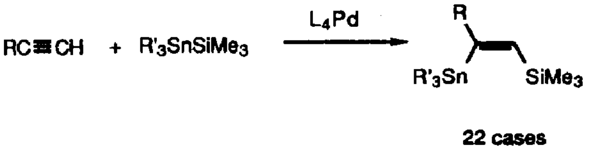
44% conversion
9 turns on cat.



(619)

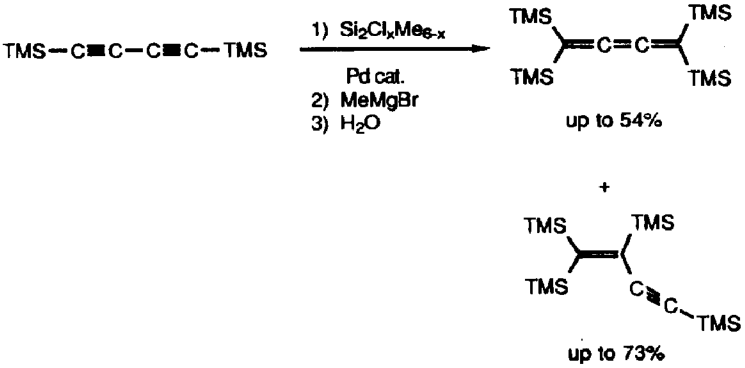
## H. Organosilanes

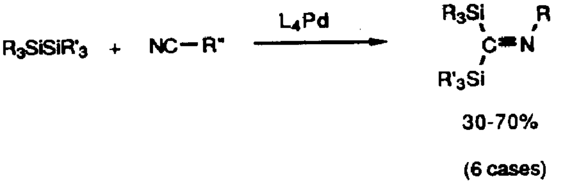
Transition metal complexes of polystryene-bound aminophosphine ligands functioned as hydrosilylation catalysts [796]. Alkynes were silylstannylated using palladium catalysts (equation 620) [797]. Palladium catalyzed silylation of trimethylsilyl diynes give cumulenes (equation 621) [798]. Isonitriles were bissilylated by disilanes with palladium(0) catalysts (equation 622) [799]. Vinyl halides (equation 623) [800], and acid halides (equation 624) [801] were converted to vinyl and acyl silanes using palladium(0) catalysts.



(620)

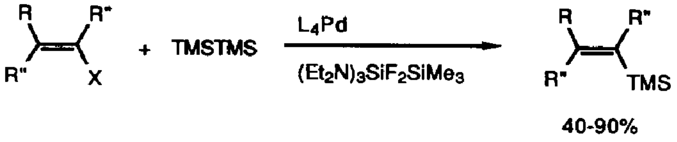
(621)





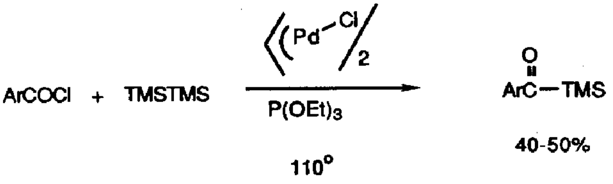
(622)

(623)



also ArI

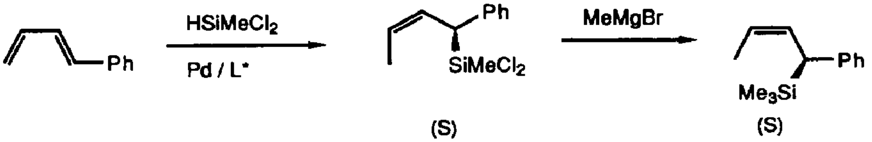
(624)



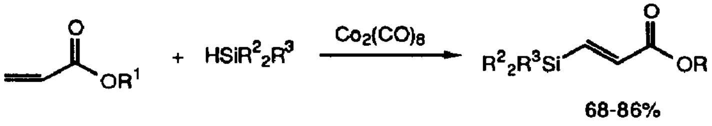
Ar = Ph, o-, m-, pMe, OMe, Cl, Bu, $CO_2Me$, $NO_2$, $CF_3$, CN

Dienes were hydrosilylated using chiral palladium catalysts (equation 625) [802]. Unsaturated esters were converted to β-silylenones using cobalt carbonyl as a catalyst (equation 626) [803]. Conjugate silylation was accomplished using organocopper chemistry (equation 627) [804]. Styrene was hydrosilylated using chiral ferrocene catalysts to give mixture of α- and β-trichlorosilane [805]. Rhodium(I) complexes catalyzed the conversion of conjugated esters to silyl ketene acetals (equation 628) [806]. Dienes were converted cyclic disilanes as in equation 629 [807].

(625)

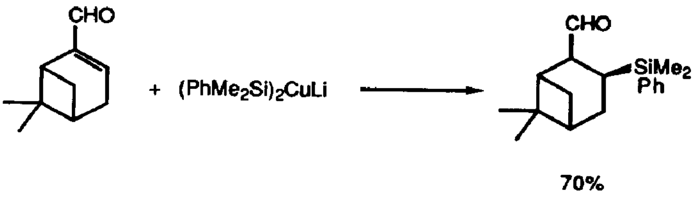


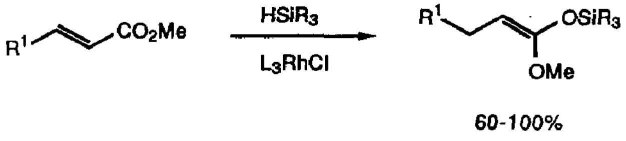
(626)



$R^1$ = Me, Et, nBu; $R^2$ = Et, Me, OEt; $R^3$ = Me, Ph, OEt

(627)



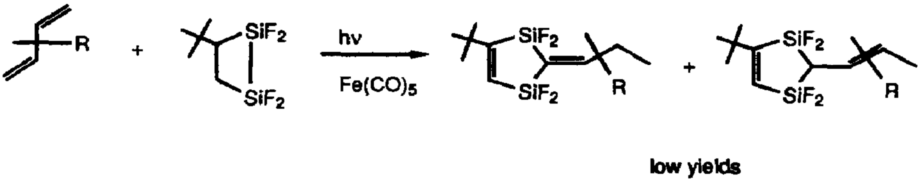


(628)

R1 = nBu, H, Me

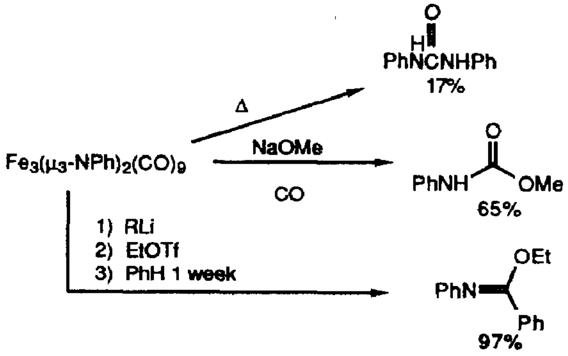
R = Me2tBu, Me2Ph, Ph3

(629)



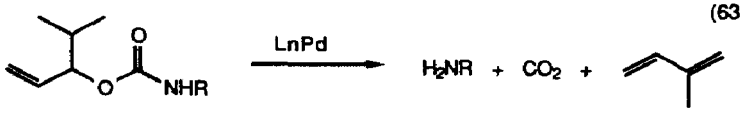
## I. Miscellaneous

Herein reside the difficult-to-classify- organometallic reactions of the year. They are somewhat randomly arranged. The literature reference is in brackets following the equation number. An improved method for the synthesis of $Cp_2Zr(H)Cl$ has been published [808].



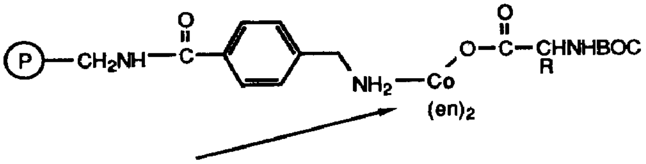
(630) [809]

(631) [810]



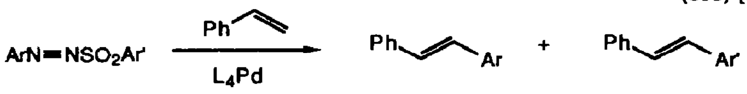
(amino and protecting group)

(632) [811]



Spacer in solid phase polypeptide synthesis

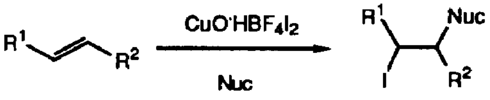
(633) [812]



up to 56%

up to 31%

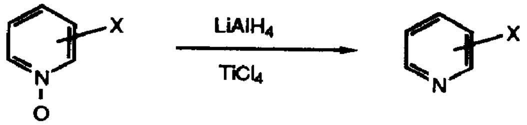
(634) [813]



60-90%

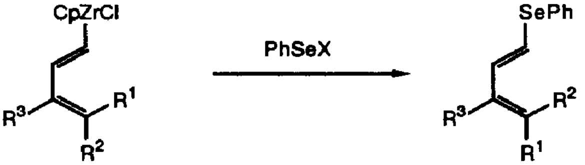
Nuc = $NO_2^-$, Tos$^-$, I$^-$, H$^-$, MeOPh, $H_2O$, MeO, AcO, Br

(635) [814]

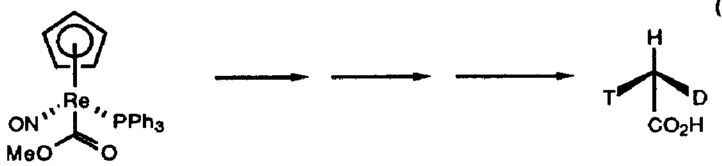


high yield

(636) [815]



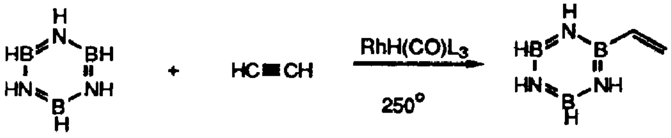
(637) [816]



S(+)

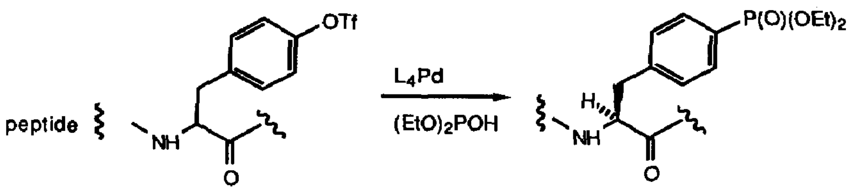
>90% ee

(638) [817]



71%

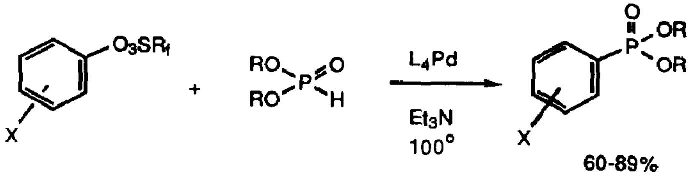
(639) [818]



80-90%

(20 cases)

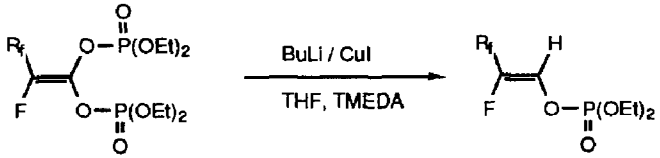
(640) [819]



60-89%

(641) [820]

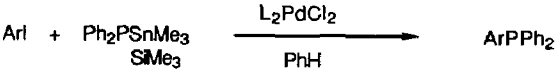
(642) [821]



62-81%

$R_f$ = $CF_3$, $CF_3CF_2$, $CF_3(CF_2)_2$, $CF_3(CF_2)_2$, $CHF_2(CF_2)_8$

(643) [822]

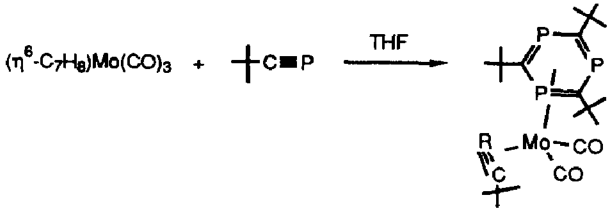


$$\mathrm{ArI} + \underset{\mathrm{SiMe_3}}{\mathrm{Ph_2PSnMe_3}} \xrightarrow[\mathrm{PhH}]{\mathrm{L_2PdCl_2}} \mathrm{ArPPh_2}$$

60-80%

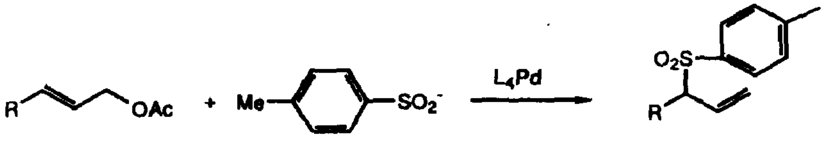
(16 cases)
all substituents

(644) [823]

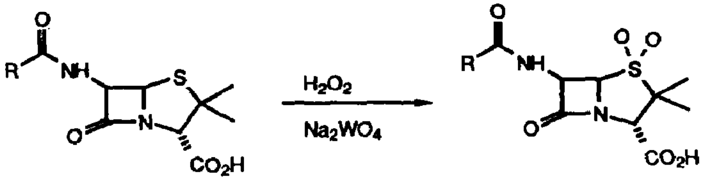


59%

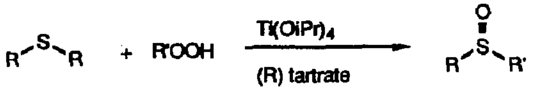
(645) [824]



(646) [825]



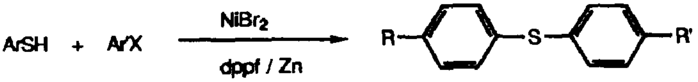
(647) [826]



90% yield

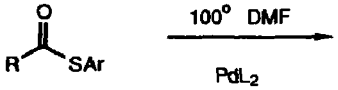
85-95% ee

(648) [827]



80-90%

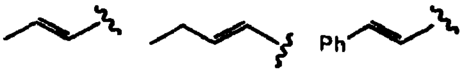
R = H, Me, MeO; R' = Me, $CH_3CO$, CN



RSAr (649) [828]

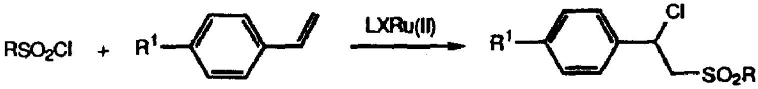
98-100%

R = Ph, pMePh,



Ar = Ph, pMePh

(650) [829]

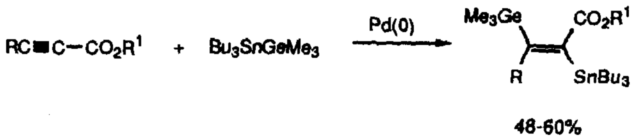


R = Me, Ar

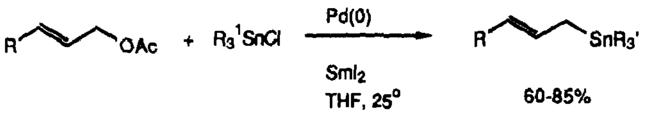
40-94%

up to 40% ee

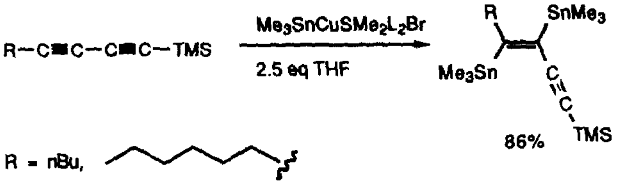
(651) [830]



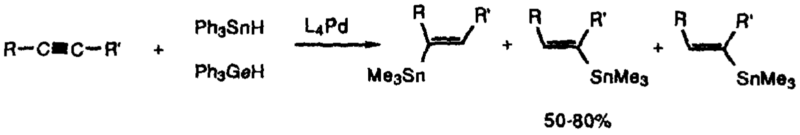
(652) [831]



(653) [832]



(654) [833]



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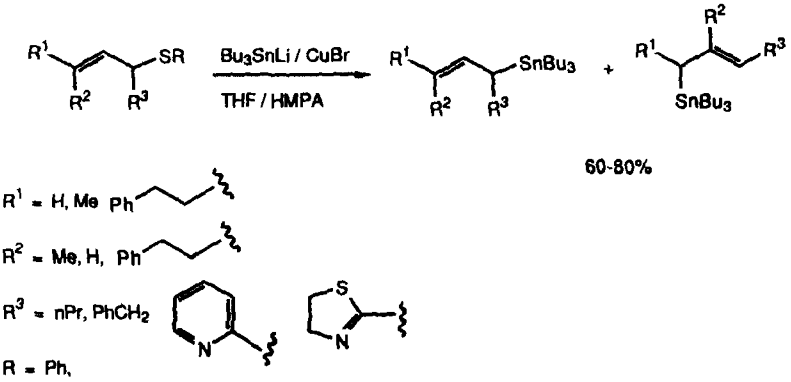

## IV. Reviews

The following review articles and dissertations have been published.

Experimental methods to control metal-induced and metal-catalyze organic synthesis (9 references) [835].

Transition metals and organic synthesis. Future survey on ruthenium as probe (56 references) [836].

New synthetic reactions catalyzed by palladium complexes [837].

Nickel in organic synthesis (24 references) [838].

Transition metals in organic synthesis. Annual survey covering the year 1985 (895 references) [839].

Transition metals in organic synthesis. Hydroformylation, reduction, and oxidation. Annual survey covering the year 1985 (616 references) [840].

Organic synthesis promoted by transition metal complexes I (35 references) [841].

Organic synthesis promoted by transition-metal complexes III (34 references) [842].

Application of organotitanium compounds in synthesis (29 references) [843].

Applications of higher-order mixed organocuprates to organic synthesis (98 references) [844].

Organic synthetic methods involving cobalt carbonyl complexes [845].

Developments in synthetic chemistry with nickel catalysts (8 references) [846].

Metals as catalysts (13 references) [847].

Fine organic synthesis by using the characteristics of palladium catalysts (23 references) [848].

Activation of alkane CH bonds by organometallics (46 references) [849].

The preparation and reaction of palladium(II)- and platinum(II)-carbon bonds [850].

Homogeneous catalysis by transition-metal complexes (335 references) [851].

Carbon-hydrogen bond activation (115 references) [852].

Homogeneous catalysis of organic reactions by complexes of metal ions (193 references) [853].

Homogeneous, palladium-catalyzed, carbon-carbon bond formation (29 references) [854].

Molybdenum-centered carbon-carbon bond formation reactions (19 references) [855].

Metal-mediated making and breaking of carbon-carbon bonds in aromatic hydrocarbons [856].

Carbon-hydrogen activation and homogeneous catalytic reactions (15 references) [857].

Phase-transfer reactions catalyzed by metal complexes (21 references) [858].

Triphase catalysis in organometallic anion chemistry [859].

Reductive behavior of low valent tungsten compounds and their applications in organic synthesis (18 references) [860].

Asymmetric synthesis using organometallic compounds. Its applications to the side chains of steroids (27 references) [861].

Stereochemical control in palladium templated reactions (17 references) [862].

Camphor derivatives as chiral auxilaries in asymmetric synthesis (83 references) [863].

Preparation of chiral ligands from amino acids: application to catalytic asymmetric syntheses (43 references) [864].

Planar chirality in organometallic complexes: applications in organic enantiomer synthesis [865].

Norbornadienes in the synthesis of polycyclic strained hydrocarbons with participation of metal complex catalysts (173 references) [866].

Study of organometallic complexes of rhodium fixed to oxide supports (38 references) [867].

New applications of tetracyanoethylene in organometallic chemistry (311 references) [868].

Application of metal vapor chemistry to catalytic chemistry (51 references) [869].

Preparation of highly reactive metal powders: some of their uses in organic and organometallic synthesis [870].

Homogeneous catalysis by metal clusters (116 references) [871].

Supported metal cluster catalysts for alkene conversion (20 references) [872].

Controlled carbometallation as a new tool for carbon-carbon bond formation and its application to cyclization (101 references) [873].

Diene, alkyne, alkene, and alkyl complexes of early transition metals: structures and synthetic applications to organic and polymer chemistry (131 references) [874].

Novel organic and macromolecular syntheses using metalloprophyrin as catalyst (10 references) [875].

Organoiron chemistry. Annual survey for the year 1985 (423 references) [876].

Organocopper-Lewis acid complex reagents. The past and present (67 references) [877].

Synthesis, characterization, catalytic, and redox chemistry of 8-halo-1-naphthyl manganese and iron porphyrins [878].

Copper-mediated oxygenations by iodosylbenzene [879].

Oxidations with metal compounds and peroxides (282 references) [880].

Ruthenium tetroxide oxidations (57 references) [881].

Transition metal ion catalyzed oxidation of a residual lignin-related compound by alkaline hydrogen peroxide [882].

Ruthenium and osmium complexes as organic oxidants (54 references) [883].

Polyoxometalates as homogeneous oxidatively resistant catalysts for difficult selective organic oxidations. Functionalization of alkanes [884].

Oxidations using palladium compounds (110 references) [885].

Utilization of $O_2$ for the specific oxidation of organic substrates with cobalt(II) catalysts (29 references) [886].

Oxidations by cobalt compounds (347 references) [887].

Oxidations of organic compounds catalyzed by copper- and cobalt-amine complexes (68 references) [888].

Oxidation of organic compound with nickel peroxide (97 references) [889].

The use of metal reagents in stereo- and regioselective, functionalization of conjugated dienes (29 references) [890].

Nucleophilic attack on $\eta^2$-acyls of zirconium. The synthesis and reactivity of zirconium ketone complexes [891].

Metallacycloalkanes and -alkenes (216 references) [892].

Olefin complexes of organometals as models for organometallic catalysts (36 references) [893].

Organocyanocobaltate(I) chemistry: avenues for exploration (50 references) [894].

Reactions with manganese(III) acetate (144 references) [895].

Reactions of unsaturated systems with chromium(VI) (389 references) [896].

Ene and retro-ene reactions in group 14 organometallic chemistry (126 references) [897].

Homogeneous catalysis...in water (29 references) [898].

Attach catalysts and reagents to polymers that dissolve...sometimes (17 references) [899].

Asymmetric synthesis. Kinetic amplification of enantiomeric excess [900].

The chemistry of the metal-carbon bond, Vol 4: The use of organometallic compounds in organic synthesis [901].

Activation of carbon-hydrogen bonds by metal atoms (30 references) [902].

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