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Transition metals in organic synthesis, highlights for the year 1997

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Contents

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1. General comments

This survey highlights the use of transition metals in organic synthesis for the year 1997. It offers a fairly comprehensive coverage of the literature, with extensive citations, but only unusual or significant transformations are presented in equation form. This format requires a little more effort from the user, and a lot less effort from the author, and allows thorough coverage in a minimum format.

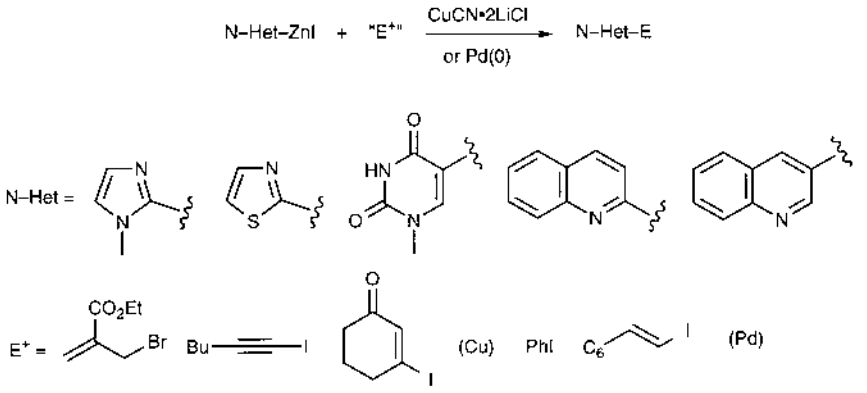
2. Carbon–carbon bond-forming reactions

2.1. Alkylations

2.1.1. Alkylation of organic halides, tosylates, triflates, acetates and epoxides

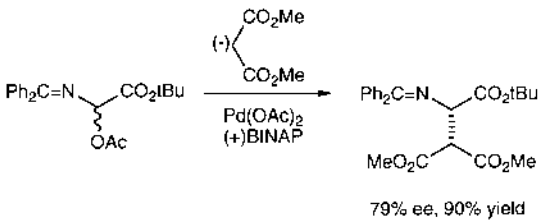
Palladium(II)(dppp) complexes catalyzed the alkylation of p-bromo-aryl triflates by Grignard reagents exclusively at the triflate, while Pd(II)(MeOMOP) complexes catalyzed coupling at the bromide [1,2]. Nickel(II)(dppf) complexes catalyzed the alkylation of aryl chlorides by aryl boronic acids [3,4], and the alkylation of alkenyl selenides by Grignard reagents [5]. Copper(I) iodide catalyzed the alkylation of alkenyl tellurides by Grignard reagents [6]. The copper complex systems (CuBr–PhSLi–LiBr–THF) catalyzed the alkylation of tosylates by Grignard reagents [7]

Nitrogen-containing heteroarylzinc iodides coupled to a wide range of organic halides in the presence of CuCN·2LiCl or palladium(0) [8]:



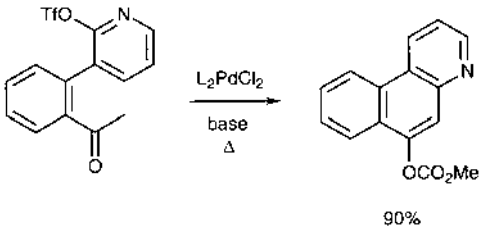
(1)

In a careful study of oxidative addition to Pd(0) complexes, it was shown that $Pd(diphos)_2$ does not react with iodobenzene, that Pd(diphos)(dba) is less reactive than Pd(diphos), and that the order of reactivity is DIOP > dppf > BINAP [9]. Dendritic diphosphine Pd, Ni, and Rh complexes have been made [10]. Palladium(0) complexes catalyzed the alkylation of vinyl chlorides with malonate anions [11], of sterically hindered penta-substituted aryl iodides with lithium acetylides [12], of *o*-lithiated amides with aryl, alkenyl and alkynyl halides [13], and of α-acetoxy amines of amino acids by malonates [14]:



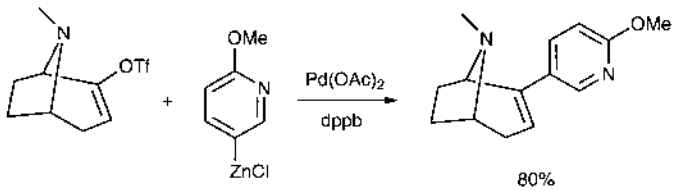
(2)

Palladium(0) complexes catalyzed the α-arylation of enolates by aryl bromides [15] and triflates [16], both intramolecularly (Eq. (3)) [15,16] and intermolecularly [17,18]:



(3)

Palladium(0)-catalyzed oxidative addition/transmetallation from zinc continues to be developed. Arylzinc bromides coupled to aryl iodides in the presence of $Pd(dba)_2$ using *p*-fluorinated phosphine ligands in a biphasic $PhCH_3/C_8F_{17}Br$ system [19]. Palladium(0) catalyzed the coupling of aryl or benzyl zinc bromides to polymer-bound functionalized iodobenzoate esters and iodobenzamides [20]. Indole-2- and -3-zinc chlorides coupled to 2-halopyridines under palladium(0) catalysis [21]. Polythiophenes were made by palladium(0)-catalyzed coupling of 2,5-bis(chlorozinc)thiophenes with 2-bromo-thiophenes [22]. Interesting synthetic applications are shown in Eq. (4) [23] and Eq. (5) [24]:



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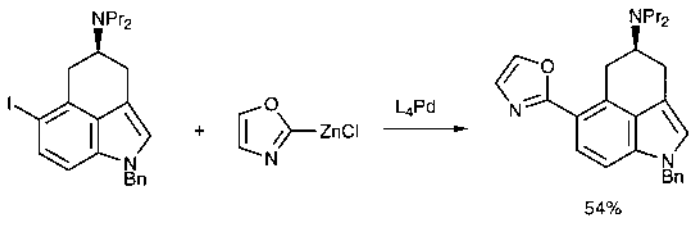
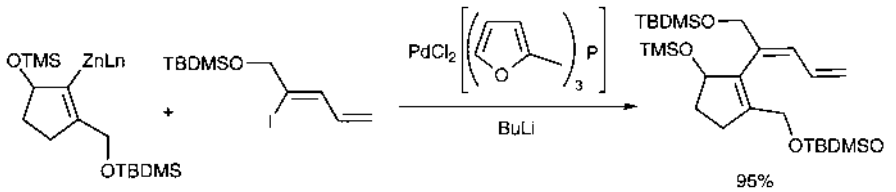


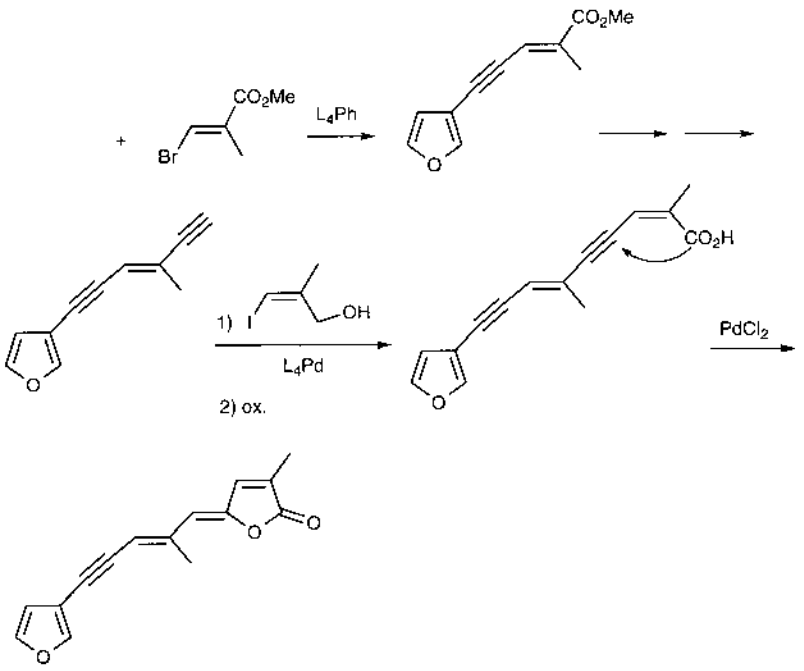
(5)

Fluorinated alkenylzinc halides coupled to aryl and fluoroaryl halides in the presence of palladium(0) catalysts [25], as did α-(iodozinc)cyclohexenones [26]. Trienes were synthesized using this type of coupling [27]:



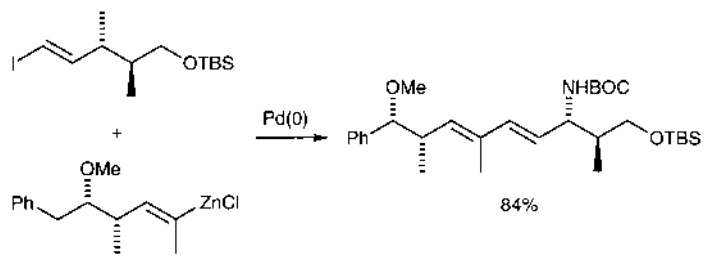
(6)

Palladium(0) catalyzed the coupling of aryl halides with alkynylzinc halides [28]. Freelingyne was synthesized using this methodology [Eq. (7)] [29]. Palladium(0) catalyzed the coupling of $CH_2(ZnI)_2$ with propargyl or allylic halides and electrophiles such as allylic halides or acid halides to give RCH_2E compounds [30]:



(7)

Brominated calixarenes were arylated by lithium–halogen exchange, treatment with zinc chloride, and finally palladium-catalyzed coupling with aryl halides [31]. Arylzinc halides (from lithium halogen exchange/$ZnCl_2$) coupled to alkenyl triflates in the presence of palladium(0) catalyst [32]. α-Lithiation of vinyl sulfones [33] or pyrazoles [34] followed by treatment with zinc halide and aryl halides in the presence of palladium catalysts led to arylation. Hydrozirconation of alkynes followed by treatment with zinc chloride produced alkenylzinc halides which underwent palladium(0) catalyzed coupling to alkenyl halides [35,36]:



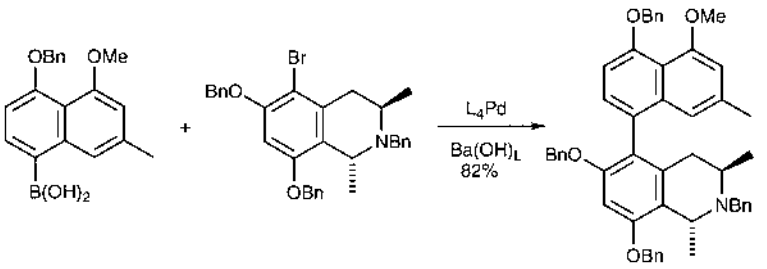
(8)

Alkenylzinc halides for use in palladium-catalyzed coupling processes were produced by the reaction of alkenyl iodides with zinc dust, and were coupled to a

range of electrophiles [37]. Rieke zinc inserted into 3-iodothiophene, and the resulting compound underwent palladium-catalyzed coupling with aryl halides [38]. Rieke manganese underwent similar processes. 2-Iodomethylglycine was coupled to 2-bromopyridine by treatment with activated zinc, followed by palladium(0) catalysts [39]. Functionalized aromatic oligomers containing up to 16 aryl groups were synthesized using palladium(0)-catalyzed couplings of benzylzinc halides to aryl halides [40]. Chloropurines were alkylated by alanes in the presence of palladium(0) catalysts [41].

Palladium(0)-catalyzed oxidative addition/transmetallation from boron (Suzuki coupling) continued being developed for organic synthesis. The compounds $ArBF_3^- K^+$ are very stable, easy to make and coupled better than arylboronic acids in Suzuki coupling [42]. Tetraphenylborates transferred all four phenyl groups [43]. Efficient Suzuki coupling in water has been developed [44]. Cesium fluoride promoted Suzuki coupling of electron deficient aryl chlorides [45]. The Suzuki coupling for the synthesis of unsymmetric, hindered biaryls has been optimized [46]. Platinum catalyzed the bis-boronation of alkynes. The resulting 1,2-bis-boronated alkenes underwent stepwise Suzuki coupling, the second to polymer-bound aryl iodides in a resin-capture step [47]. Suzuki coupling to resin-bound iodoanilides [48] and tetrazinc containing aryl bromides was efficient [49]. Polystyrene-bound palladium complexes catalyzed Suzuki coupling [50].

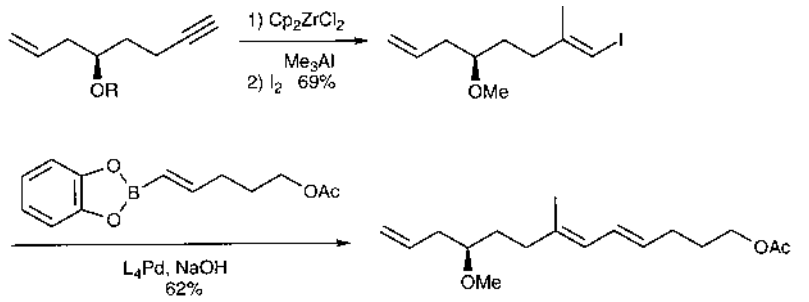
Suzuki coupling was used to arylate and alkenylate 2- and 3-boronated furans [51], 2-boronated indoles [52,53], 2-bromoindoles with arylboronic acids [54], 2-iodobenzimidazoles with arylboronic acids [55], 3-bromoquinolines and quinolones with arylboronic acids [56], 5-bromoquinolines with arylboronic acids [57] and 7-bromoisoxindoles with arylboronic acids [58]. Palladium(0) complexes catalyzed the arylation of dibromo binaphthols by arylboronic acids [59], the coupling of p-boronated phenylalanine imines with aryl halides [60], of 2-bromo-1,4,5-trimethoxynaphthol with o-boronated phenyl carbamates [61], of 2,4,6-trisubstituted aryl bromides with 3,5-bis-benzyloxy-4-boronated t-butylbenzoates [62] and of brominated calixarenes with arylboronic acids [63]. Suzuki coupling figured extensively in the synthesis of Michellamine A and C [64]:



(9)

The enol triflates from 3-alkoxycyclohexenones [65] and 3-hydroxypyrroles [66] were arylated by arylboronic acids. Alkenyl boronates having an R′Se group in the

2-position underwent palladium-catalyzed alkylation by organic halides lacking β hydrogens [67]. Similarly, palladium(0) complexes catalyzed the coupling of alkenyl boronates with β-bromo alkenyl selenides [68], with β-bromoacetamidoacrylates [69], and with hexabromobenzene to hexaalkenylated benzene [70]. Synthetic applications are shown in Eq. (10) [71], Eq. (11) [72], Eq. (12) [73], and Eq. (13) [74]:



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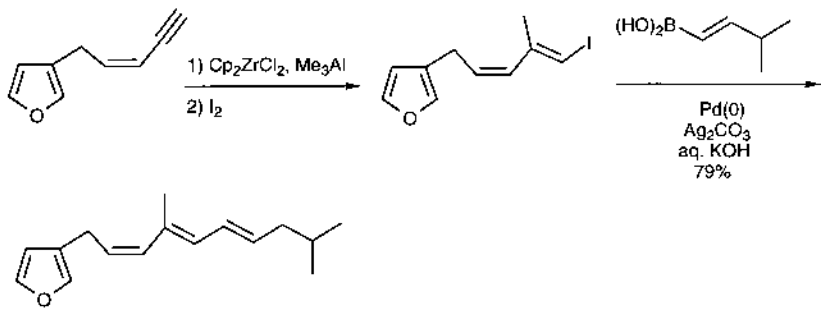

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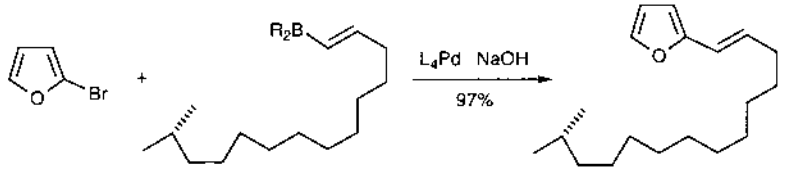

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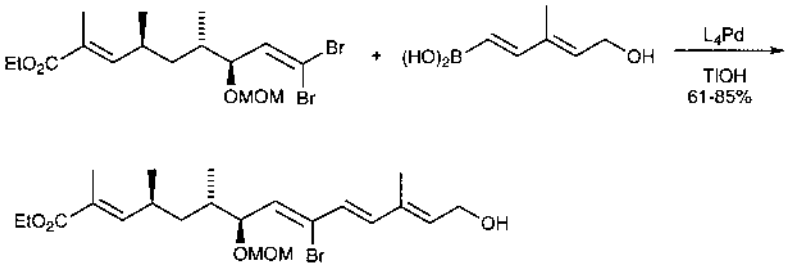
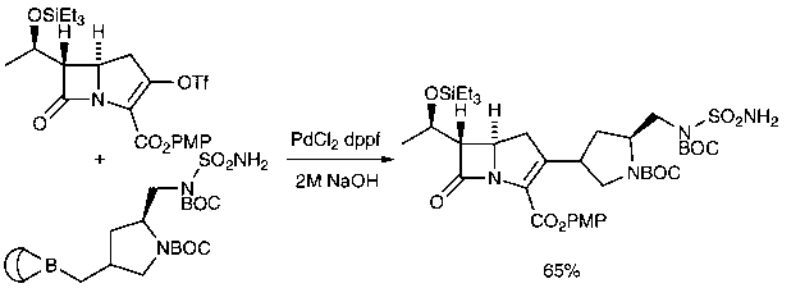


(13)

Cyclopropyl boronates coupled to cyclopropyl iodides under palladium catalysis [75]. 5-Methylene- [76] and 6-vinyl-glucosides [77] were hydroborated then arylated with aryl halides under Suzuki coupling conditions. Other synthetically significant coupling reactions of alkylboranes are seen in Eq. (14) [78], Eq. (15) [79], and Eq. (16) [80]:



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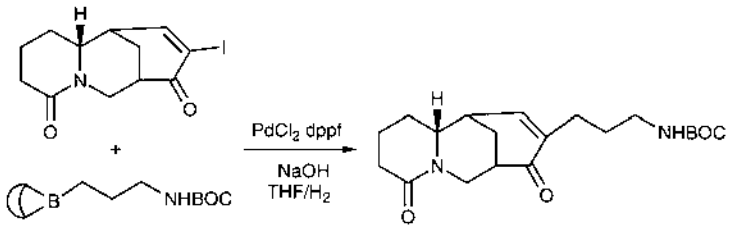

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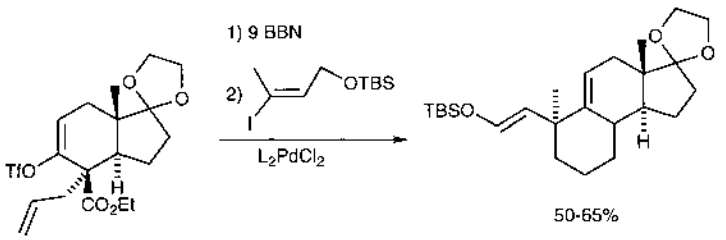


(16)

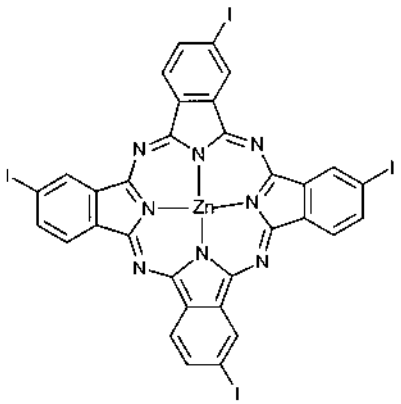
Suzuki coupling has become popular for the synthesis of polymers and materials. Functionalized AB type monomers [e.g. (bromo)aryl boronic acids] for Suzuki polymerization were synthesized [81]. Macrocyclic oligophenylenes were synthesized by Suzuki coupling of α-*m*-iodo-γ-(*p*-boronic acid)-hexaphenyls [82]. Oligophenyls were made by Suzuki coupling of *p*-dibromoarenes with p-(bis-boronic acid) benzenes [83]. Oligomers of molecular weight ≈ 8000 were synthesized by the Suzuki coupling of 9-substituted-2,7-dibromofluorenes with 9,9-disubstituted-2,7-(bis-boronic acid) fluorenes [84]. Covalently linked organic networks were synthesized by Suzuki coupling of (peri)-1-iodo-8-carbomethoxy naphthalenes with

aryl-bis-boronic acids [85]. Reproducible procedures for step-growth polymerization involving Suzuki coupling have been developed [86]. Chiral polybinaphthols were synthesized via Suzuki coupling [87,88]. Suzuki couplings of bis-*n*-hexyl-dibromoarene, bromoboronic acid arenes, and iodoarenes have been carried out [89].

Sodium hydroxide was a better promoter of the Hiyama coupling (palladium-catalyzed oxidative addition/transmetallation from silicon) than was fluoride [90]. Hiyama coupling was used to alkylate aryl halides with aliphatic groups transferred from $RSiF_3$ [91], and to synthesize biaryls from aryl halides and aryl silanes [92]. Copper iodide catalyzed similar coupling [93].

Stille coupling (palladium-catalyzed oxidative addition/transmetallation from tin) continued to be utilized extensively in synthesis. Full papers on the use of $(C_6F_{13}CH_2CH_2)_3SnR$ in the Stille reaction for ease in separation of tin residues [94], and the use of microwave irradiation to speed it up [95], have appeared. The reagents $RSnX(NTMS_2)_2TBAF$ have been used in Stille coupling [96], and the use of sulfonium salts (R_2S^+) as leaving groups in Stille coupling has been developed [97]. New phosphine/imine ligands have been developed for Stille coupling [98]. The ratio of ipso vs. cine substitution in the palladium-catalyzed reaction of aryl halides with α-tributylstannylstyrene was dependent on electron-withdrawing groups on the aryl halide [99]. Copper iodide/manganese(II) bromide catalyzed Stille type coupling reactions [100].

Alkenyl stannanes containing 3,4-diols [101] were subjected to a range of Stille coupling, as were chiral alkenyl- and alkynylsulfoxides [102], and 3-stannylsulfolenes [103]. Compound **1** underwent Heck, Stille and Suzuki couplings [104], while compound **2** was subjected to almost every palladium-catalyzed coupling known [105]. Electron-deficient alkenes underwent 1,2-dialkylation when subjected to allyl tin and allylic halides in the presence of palladium catalysts [106].



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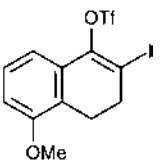
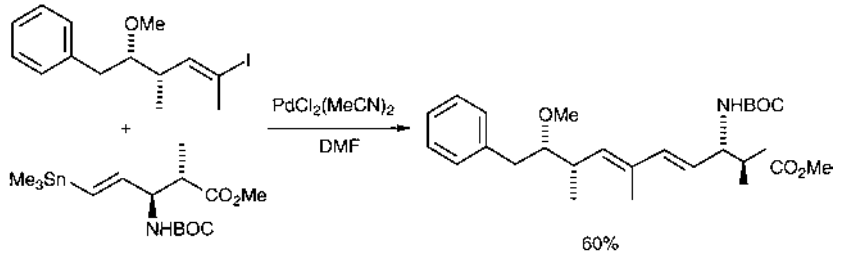


2

Palladium catalyzed the coupling of cyclic enol phosphonates with vinyl, α-alkoxyvinyl and β-alkoxyvinyl tin reagents [107]; of α-selenyl alkenylstannanes with alkenyl bromides [108]; and of pyrrolidinone enol triflates with alkenyl stannanes [109]. A tandem Stille coupling/Diels–Alder reaction was effected by Stille

coupling of vinyl tin reagents with alkenyl halides in the presence of fumarate esters [110]. Stille coupling was used to vinylate *iso*-penicillin analogs [111].

Highly functionalized polyenes were synthesized by alkenyl tin/alkenyl halide Stille couplings, Eq. (17) [112], Eq. (18) [113], Eq. (19) [114], Eq. (20) [115], and Eq. (21) [116,117]:



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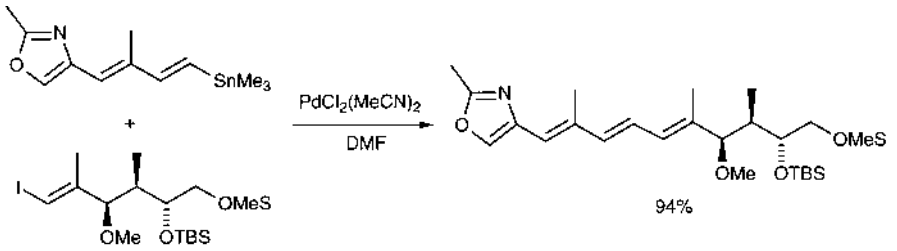

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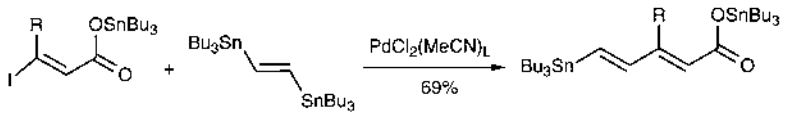

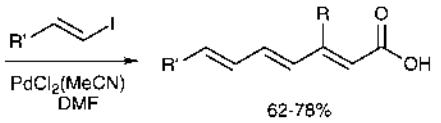

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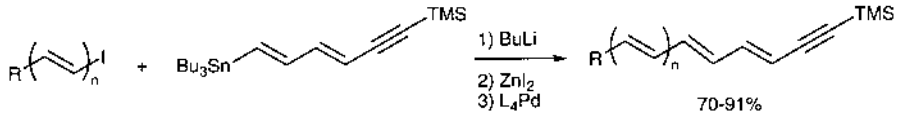

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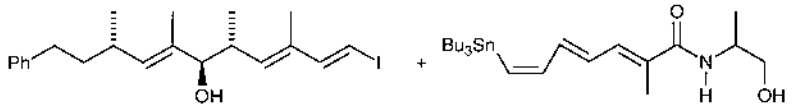
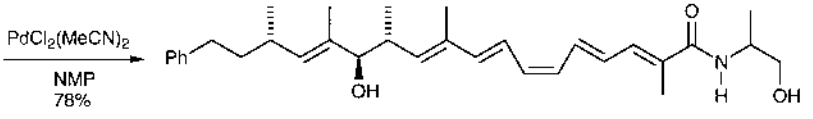
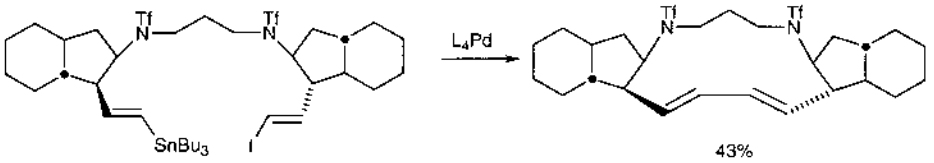




(21)

Intramolecular Stille couplings were used to make macrocycles, Eq. (22) [118], Eq. (23) [119], Eq. (24) [120], and Eq. (25) [121]:



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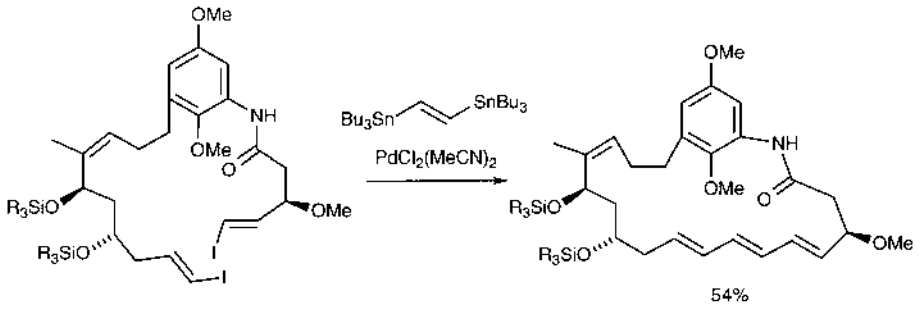

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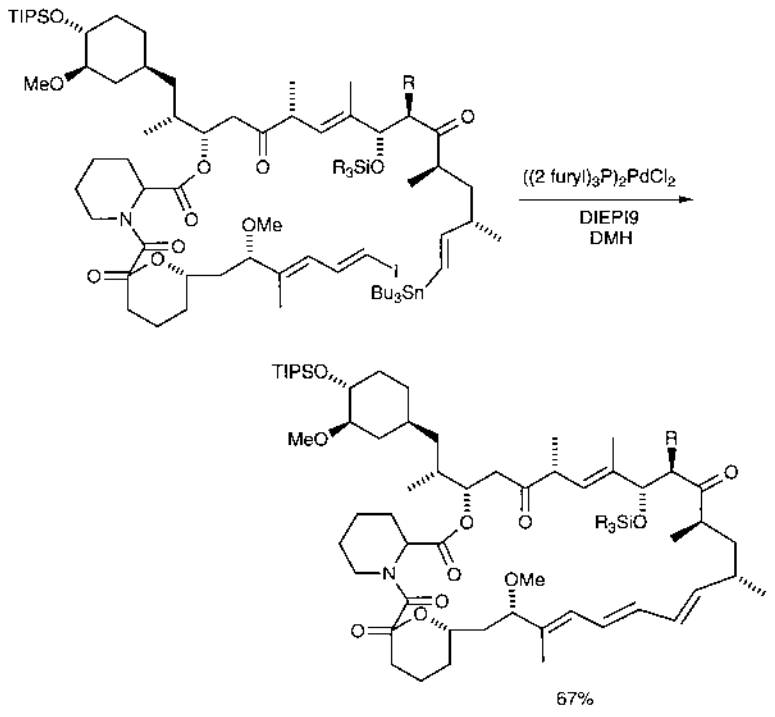

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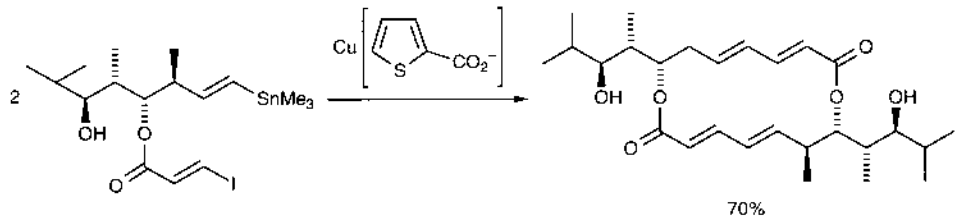
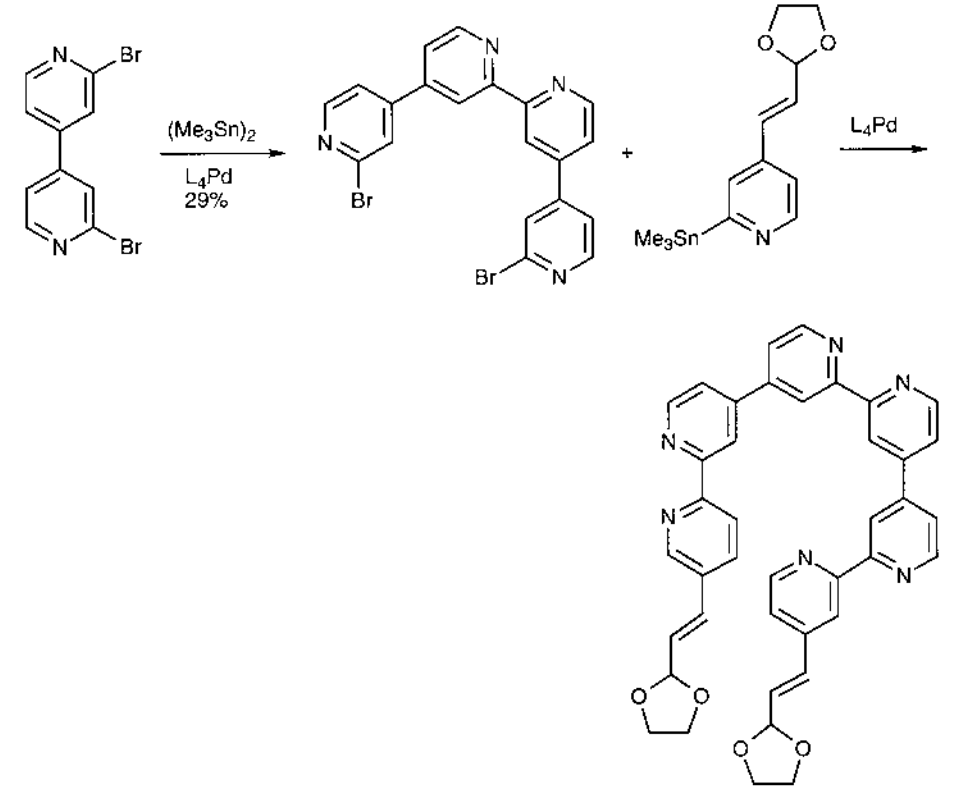


(25)

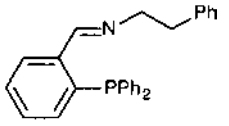
Palladium(0) complexes catalyzed the coupling between 5-bromo-benzofurans and polymethoxy arylstannanes [122]; 4-bromobenzyl ethers with 4-stannyl phenylacetic acid esters [123]; the enol triflate of 5,8-dimethoxy-7-nitro-4-quinolone with o-acetamido aryl stannanes [124]; dichloroaryl stannanes with 4-bromo-1-naphthols [125]; aryl halides with 3-stannyl pyrazines [126]; and aryl stannanes with o-triflyl [127], iodosylbenzyl [128], and bromonaphthoquinones [129]. An interesting helical structure was synthesized using Stille coupling [Eq. (26)] [130]. Nickel complexes catalyzed the Stille coupling of aryl chlorides [131]:



(26)

Palladium(0)/copper iodide catalyzed the Stille coupling of α-fluoro- [132] and α,β-difluoroalkenylstannanes with aryl halides [133]. p-Stannyl aryl alanines were alkenylated by alkenyl halides using Stille coupling [134]. Palladium(0) catalyzed the coupling of aryl iodides to α-stannyldiazoesters [135], and of allyl stannanes with functionalized aryl iodides in the synthesis of mycophenolic acid [136]. Indoles were synthesized by the Stille coupling of *o*-bromonitroarenes with alkenylstannanes followed by palladium catalyzed reductive cyclization of the *o*-nitrostyrene produced [137]. 1,2-Bis-iminoyl chlorides were alkylated by stannanes in the presence of palladium catalysts [138]. Functionalized, chiral 3-aminobut-1-en-4-ols were made by Stille coupling [139].

For the Stille coupling of alkynylstannanes with aryl iodides in the presence of dppp, the aryl iodide oxidatively adds to the palladium, while in the presence of **3**, the stannanes oxidatively add [140]. Synthetically interesting alkyne Stille couplings are shown in Eq. (27) [141], Eq. (28) [142], Eq. (29) [143], and Eq. (30) [144]:



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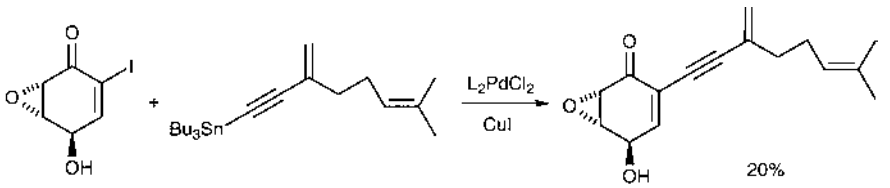
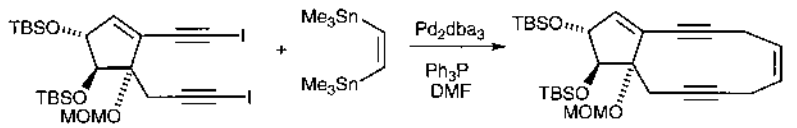
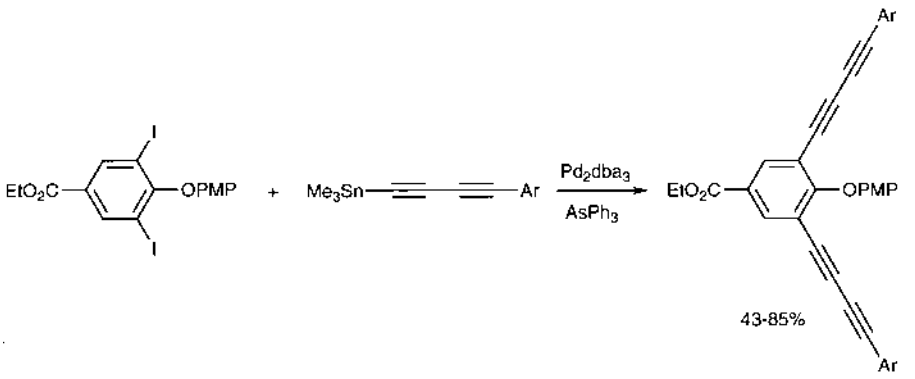


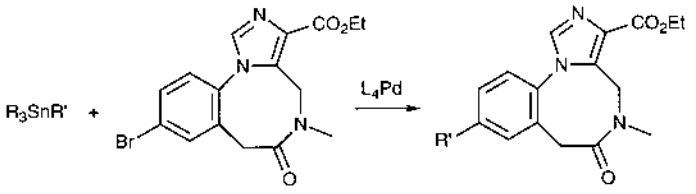
(27)



(28)

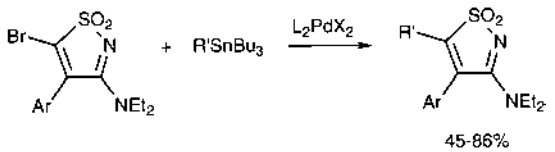


(29)

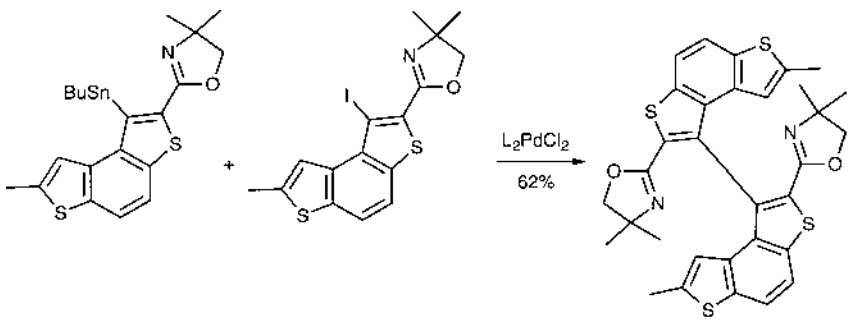


(30)

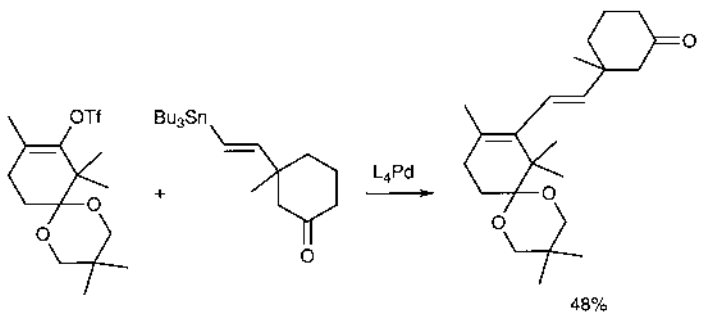
A wide array of heterocycles participated in Stille coupling. 3,4-Dibromopyrroles were diarylated by aryl stannanes in the presence of palladium(0) catalysts [145]. 4-Chloropurines were coupled to α-alkoxyvinyl stannanes [146], 2-stannyl pyrroles were coupled to iodobenzene [147], stannylpyridines were coupled to bromothiophenes [148], and 4-aryl pyrazoles were synthesized by Stille coupling [149]. 6-Chloropyrones were arylated by aryl tin reagents under palladium(0) catalysis [150]. α-Chloro-γ-pyrones were alkylated by organostannanes in the presence of palladium catalysts [151]. Kainic acids were homologated using Stille coupling [152]. Enol triflates of pyrimidines were coupled to alkenyl- and alkynylstannanes using Stille coupling [153]. Other useful couplings are shown in Eq. (31) [154], Eq. (32) [155], and Eq. (33) [156]:



(31)

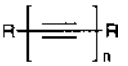


(32)



(33)

Stille coupling was used to make conducting polymetallorotaxanes containing polythiophene and polypyridine segments [157], mixed aryl–thiophene oligomers [158], alternating oligomers from functionalized 2,5-bis-stannylthiophenes, 1,6-dihaloarenes [159], and 2,5-dibromothiophenes with 2,5-bis-stannylthiophenes [160]. Polyacetylenes **4** and poly-1,6-bis-alkynyl benzenes were made via Stille coupling [161].



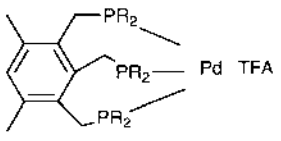
4

2.1.2. Alkylation of acid derivatives

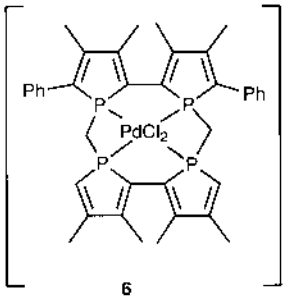
Treatment of 2,3-dibromothiophene with Rieke manganese followed by an acid halide resulted in acylation. This was done sequentially [162]. 3-Bromoindoles were acylated with acid halides by halogen metal exchange followed by transmetallation to zinc chloride [163]. Treatment of β-iodo ketones with zinc metal followed by palladium(0) and an acid chloride resulted in acylation [164]. α-Lithiated (Boc) pyrrolidine was acylated by acid chlorides in the presence of copper salts [165].

2.1.3. Alkylations of olefins

The Heck reaction continued to be developed and utilized for organic synthesis. A review entitled 'Palladacycles as reactive intermediates' (60 Refs.) contained substantial information concerning Heck reactions [166]. Heck reaction between aryl bromides and styrenes or enones proceeded in 20 min in the absence of solvent under microwave irradiation [167]. Complex **5** was a highly active Heck reaction catalyst with turnover numbers up to 528000 [168], while complex **6** was a very stable Heck catalyst, which remained active for long periods without precipitation of metallic palladium [169]. The effect of the counter ion on Heck aryl diazonium salt alkylation of olefins has been examined [170], as has Heck, Suzuki, and Sonigishara couplings of 3-iodothiophenes [171].



R = iPr, tBu **5**

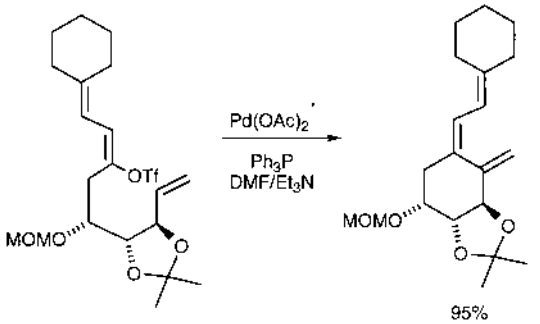


6

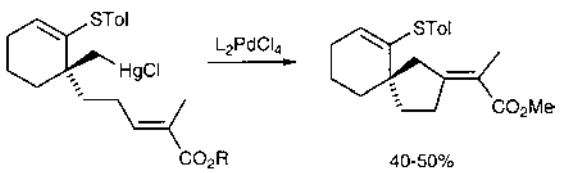
Palladium(0) supported on glass beads [172], palladium colloids in block copolymer micelles [173], palladium/copper-exchanged montmorillonite K^{10} clays [174], and palladium supported on poly-γ-mercaptopropylsiloxanes [175] were all effective catalysts for Heck arylation of olefins.

2-Iodo-5-chloropyridones were olefinated exclusively at the iodo position by *t*-butyl acrylates under palladium catalysis [176]. Iodonitrobenzopyrans arylate acrylates under Heck conditions [177]. Treatment of silylenol ethers with $C_4F_9SO_2F$/TBAF, followed by an electron-deficient alkene and a palladium catalyst, resulted in alkylation of the olefin by the enol ether [178]. Cinnamonitriles were arylated by substituted aryl iodides and substituted cinnanonitriles were arylated by iodobenzene under Heck conditions [179]. 4-(*p*-Iodophenyl)-β-lactams were olefinated by electron-deficient alkenes in the presence of palladium catalyst [180], and kainic acid analogs were arylated under similar conditions [181]. Zinc porphyrins bearing a bromo group on one of the pyrroles underwent Heck coupling three times with 1,3,5-trivinyl benzene and twice with 1,4-divinyl benzene [182]. Palladium(0) catalyzed the coupling of malononitriles with enynes to give alkylated allenes [183]. Nickel(0) catalyzed the arylation of electron-deficient alkenes by aryl iodides [184].

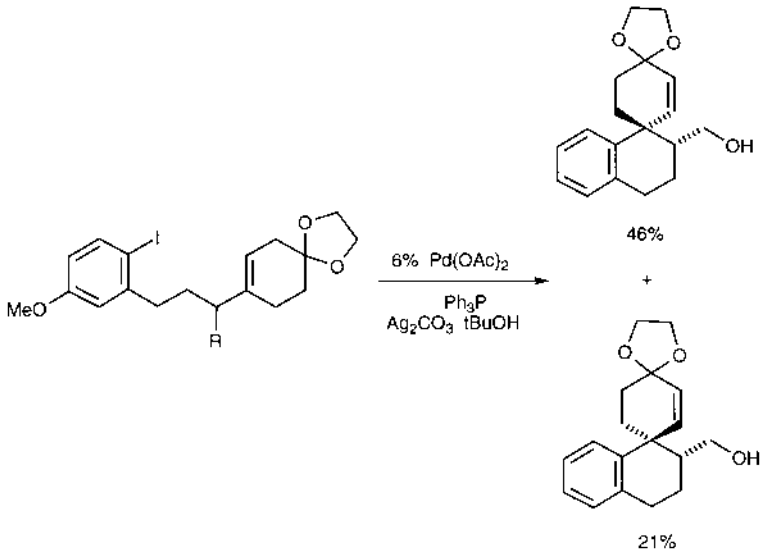
Heck type alkylation of olefins has been used in an intramolecular sense to make carbocycles [Eq. (34)] [185], [Eq. (35)] [186], [Eq. (36)] [187], macrocycles [Eq. (37)] [188], oxygen heterocycles [Eq. (38)] [189], [Eq. (39)] [190] and alkaloids [Eq. (40)] [191], [Eq. (41)] [192], [Eq. (42)] [193], [Eq. (43)] [194], [Eq. (44)] [195]:



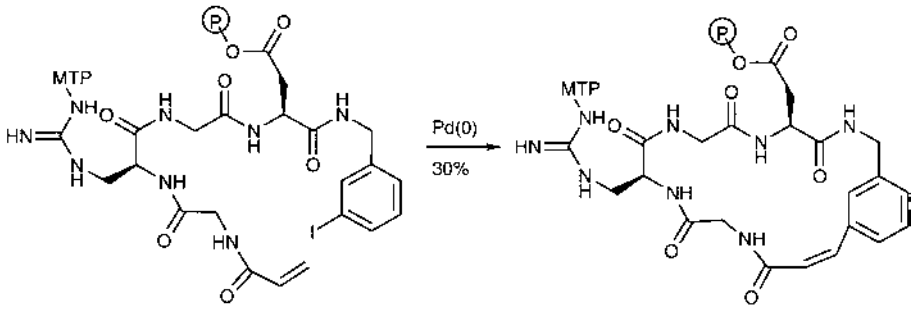
(34)



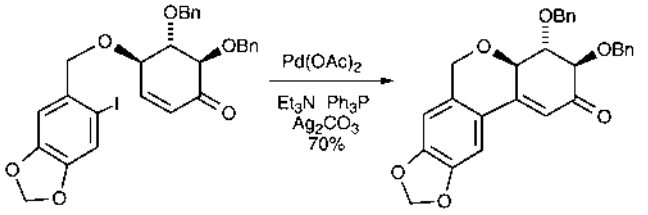
(35)


MeO
I
R
6% Pd(OAc)2
Ph3P
Ag2CO3 tBuOH
OH
46%
+
OH
21%

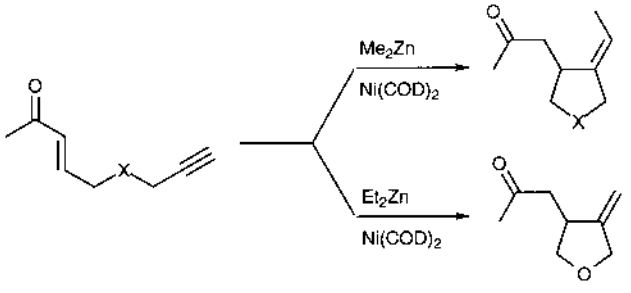
(36)


MTP
HN
NH
Pd(0)
30%
MTP
HN
NH

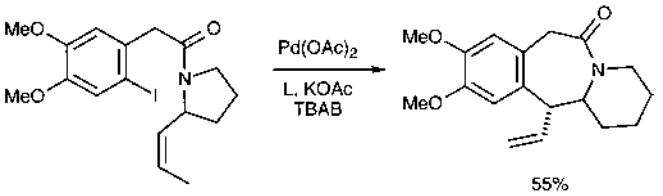
(37)


OBn
OBn
I
Pd(OAc)2
Et3N Ph3P
Ag2CO3
70%
OBn
OBn

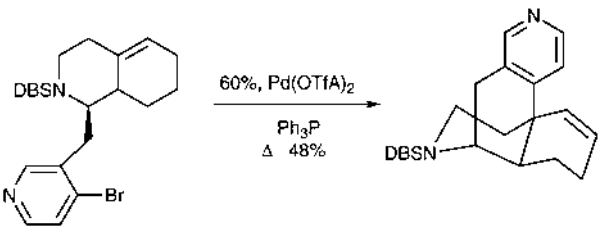
(38)


X
Me2Zn
Ni(COD)2
Et2Zn
Ni(COD)2
70-80%

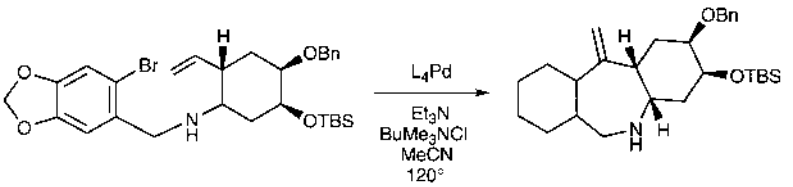
(39)



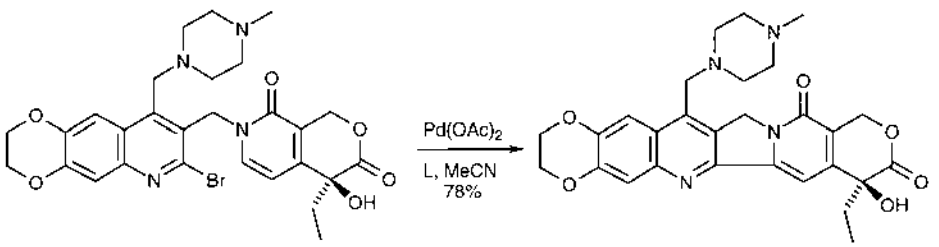
(40)



(41)

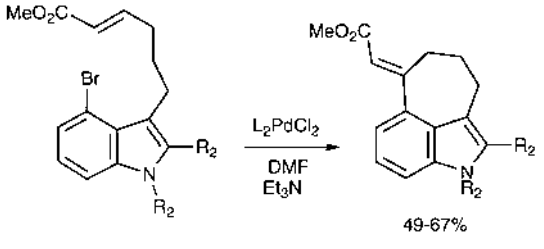


(42)

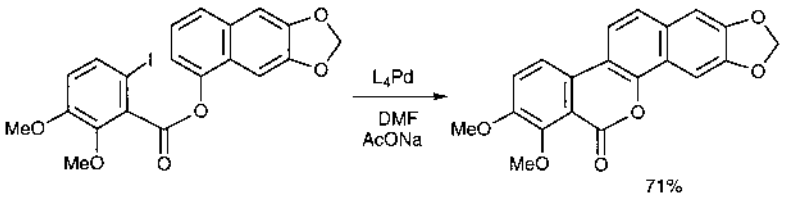


(43)

Heck olefin insertion into aromatic rings has been achieved with thiophenes bearing electron-withdrawing groups [196] as well as phenolic esters [197]:

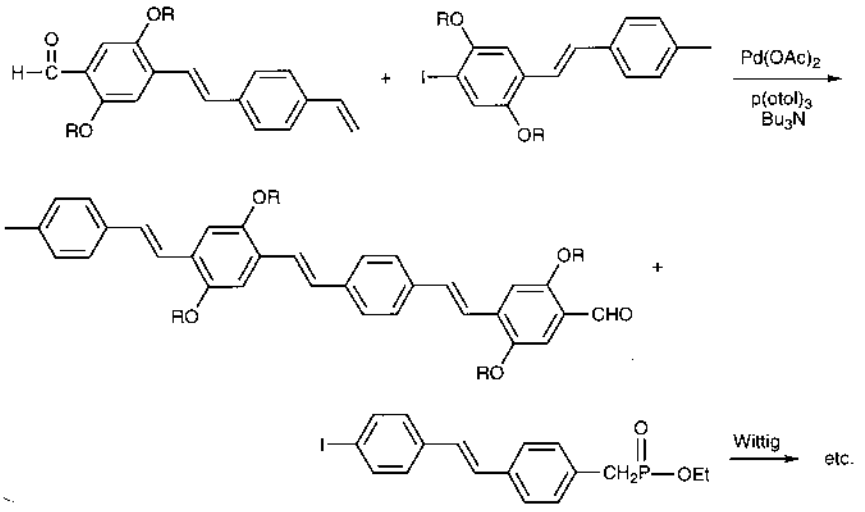


(44)

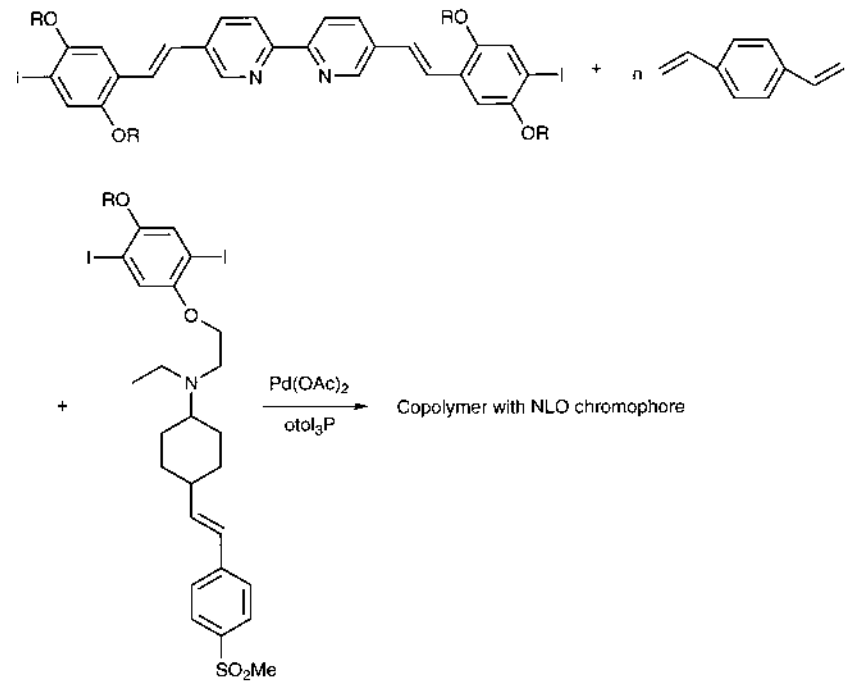


(45)

Heck arylation of 2,5-divinyl pyridine with 1,4-bis long-chain alkyl-2,5-diiodoarenes led to poly[pyridylvinylene phenylene vinylene] [198]. An orthogonal approach to polyvinylene dendrimers involved Heck arylation of 3,5-divinylbenzaldehyde with 3,5-bis-styryl bromobenzene [199]. Other uses in polymer synthesis are seen in Eq. (46) [200] and Eq. (47) [201]:

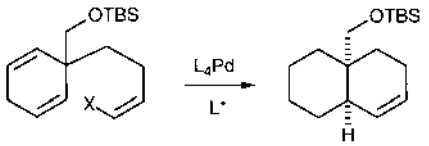


(46)

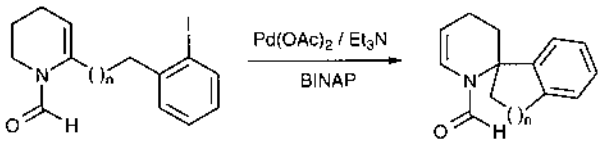


(47)

The asymmetric Heck reaction has been reviewed (88 Refs.) [202]. Asymmetric arylation of dihydrofurans and pyranes by aryl and vinyl triflates has been achieved [203,204]. Other examples are seen in Eq. (48) [205] and Eq. (49) [206]:



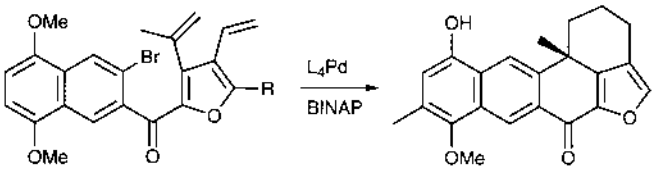
90% 82% ee (48)



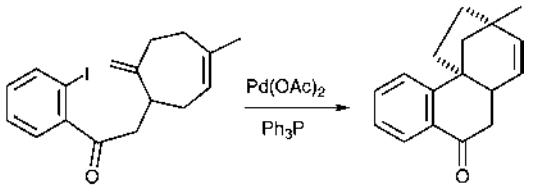
good ee when low conversion

n = 1 74%
n = 2 67% (49)

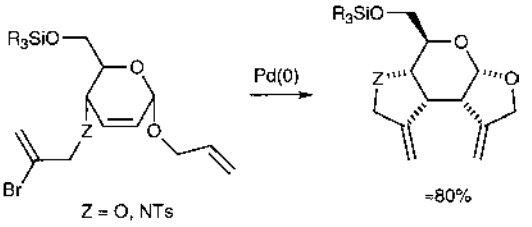
Cascade Heck sequences have been used to generate polycyclic compounds [Eq. (50)] [207], [Eq. (51)] [208], [Eq. (52)] [209], [Eq. (53)] [210]. Sometimes these take a strange course [Eq. (54)] [211], [Eq. (55)] [212], and can be truncated by nucleophilic attack [Eq. (56)] [213]:



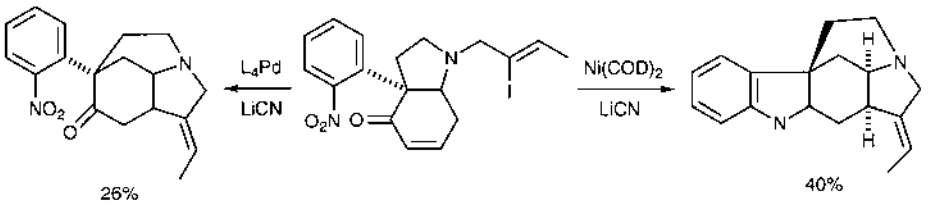
66% ee 82% yield (50)



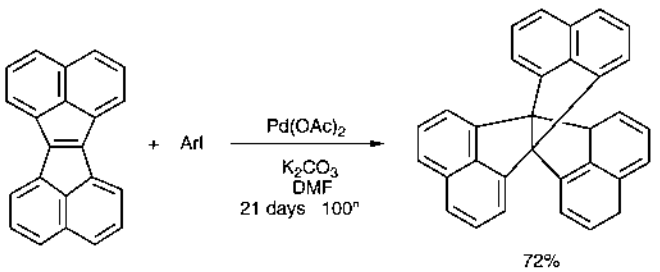
85% (51)


R3SiO
Pd(0)
Z = O, NTs
=80%

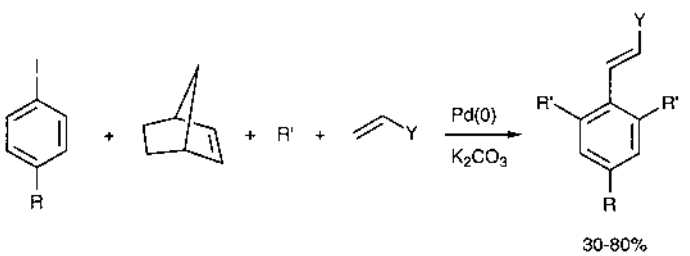
(52)


L4Pd
LiCN
Ni(COD)2
LiCN
26%
40%

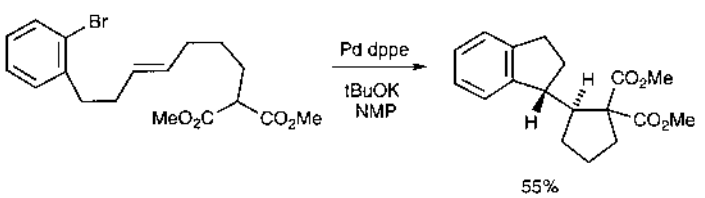
(53)


ArI
Pd(OAc)2
K2CO3
DMF
21 days 100°
72%

(54)

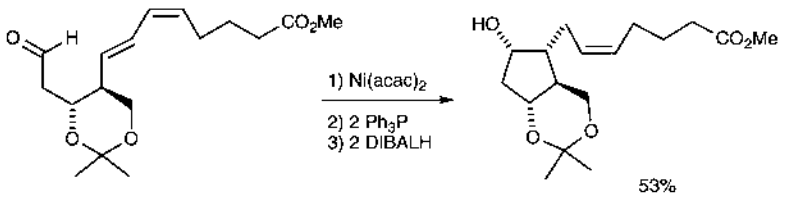

Pd(0)
K2CO3
30-80%

(55)

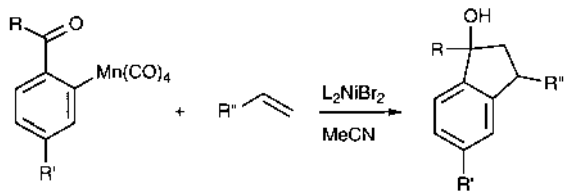

Pd dppe
tBuOK
NMP
55%

(56)

Rhodium(I) complexes catalyzed the β-alkylation of enones by alkylboronic acids [214], the addition of aldehydes to alkenes [215], and the asymmetric intramolecular hydroacylation of alkenes [216]. Nickel(0) complexes also catalyzed the hydroacylation of dienes [Eq. (57)] [217]. Ruthenium(II) complexes catalyzed the *ortho* alkenylation of aryl ketones [218], while Bu_3MnLi cyclized *o*-iodoallylphenolic ethers [219]. Nickel(II) salts catalyzed the olefination of *o*-manganated aryl ketones [Eq. (58)] [220]:



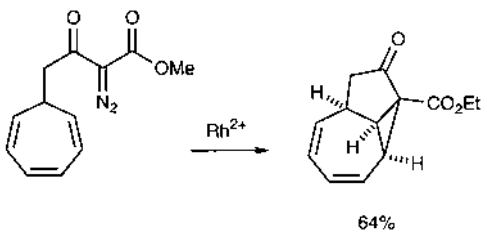
(57)



(58)

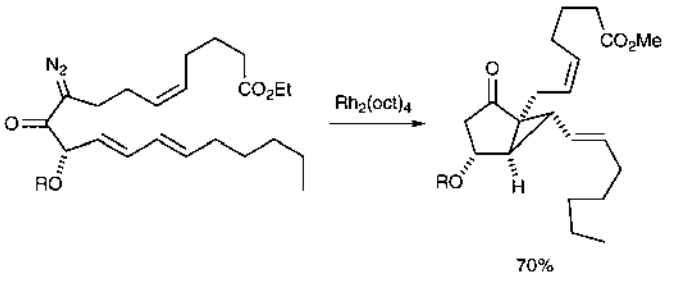
2.1.4. Metal-catalyzed diazodecomposition and other cyclopropanations

Rhodium(I) complexes catalyzed the intermolecular cyclopropanation of styrenes by diazoesters [221]. Macrocycles of 9–20 members were synthesized by metal-catalyzed cyclopropanation of alkenes by diazoketones [222]. Smaller rings could also be made this way [223], Eq. (59) [224,225]. Diazofluorene cyclopropanated alkenes in the presence of $Cr(CO)_5 \cdot THF$ [226]. The chromium carbene intermediate was detected by ^{13}C-NMR spectroscopy:

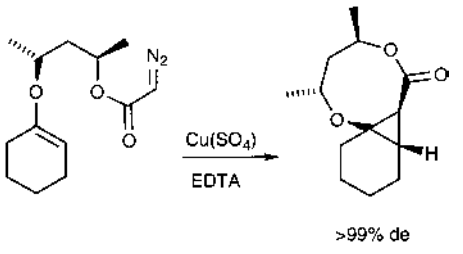


(59)

Reviews entitled 'Recent advances in stereoselective synthesis involving diazocarbonyl intermediates' (54 Refs.) [227] and 'Catalytic enantioselective cyclopropanations of olefins using carbenoid chemistry' (94 Refs.) [228] have appeared. Comparisons of enantiocontrol in the intramolecular cyclopropanations of alkenes by diazoesters with copper(I), rhodium(II) and ruthenium(III) catalysts have been made [229]. Copper triflate in the presence of chiral concave 1,10-phenanthrolines [230] and rhodium(II)–3(S)-phthalimido-2-piperidinonate complexes [231] catalyzed olefin cyclopropanation by diazoesters with high ee, while Pfaltz type copper catalysts gave no asymmetric induction in the cyclopropanation of acetamidoacrylates [232]. Diazoamides of Oppolzer's chiral sultam cyclopropanated alkenes with fair de in the presence of rhodium(II) catalyst [233]. Chiral centers resident in the substrate could also lead to high diastereoselectivity [Eq. (60)] [234], [Eq. (61)] [235]:

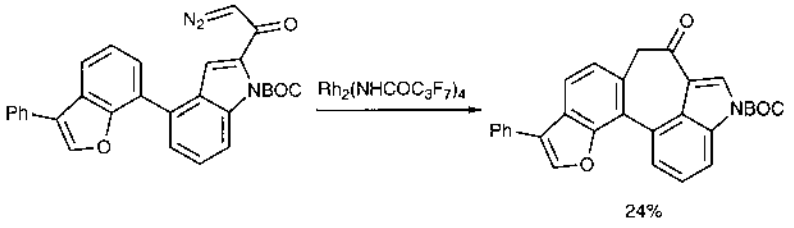


(60)



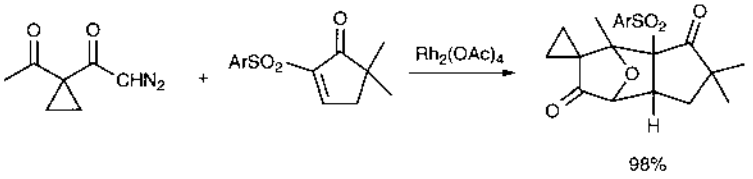
(61)

Rhodium(II) acetate catalyzed the formation of five-membered rings by C–H insertion processes [236]. Asymmetry could be induced with chiral rhodium(II) complexes in intermolecular C–H insertions into cycloalkenes [237]. Arene C–H insertion was used to synthesize polycyclic compounds [238]:

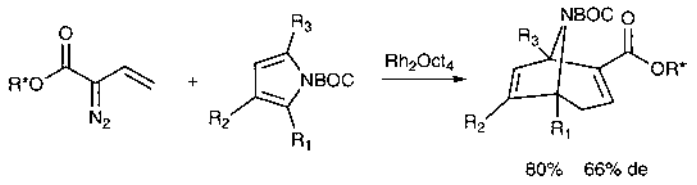


(62)

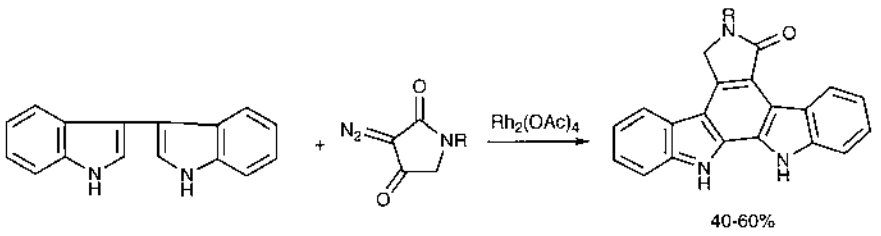
Rhodium(II) acetate catalyzed the cyclopropanation of enones by diazo esters with the presence of chiral thioethers via sulfur ylide chemistry [239]. Rhodium-catalyzed ylide chemistry was used with synthesis of illubin [Eq. (63)] [240,241]. Other synthetically interesting ylide reactions are shown in Eq. (64) [242], Eq. (65) [243], Eq. (66) [244] and Eq. (67) [245]. The complex $CpRuL_2$ coupled diazoketones to give 1,2-diketoalkenes [246], while $ReOCl_3L_2$ catalyzed the coupling of aldehydes to α-diazoesters to give acrylic esters [247]. Cobalt alkyne complexes were converted to cobalt diene complexes by reactions with diazoketones [Eq. (68)] [248]:



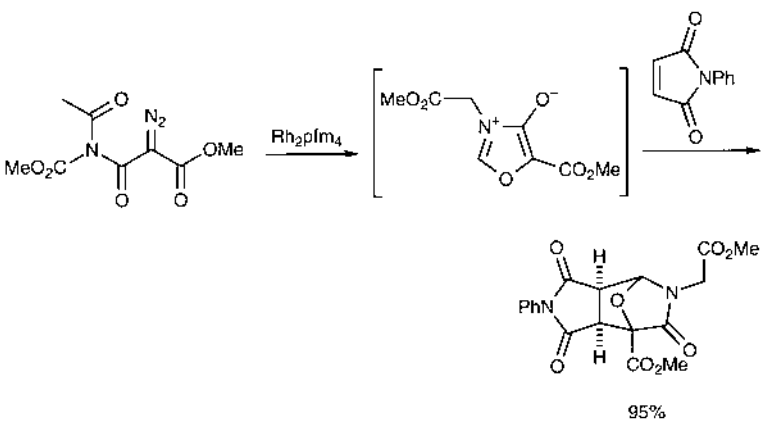
(63)



(64)



(65)



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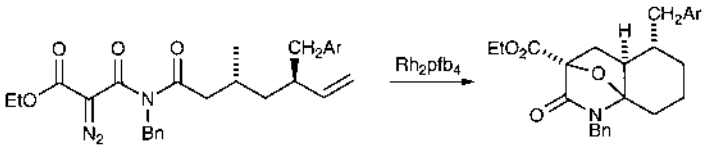
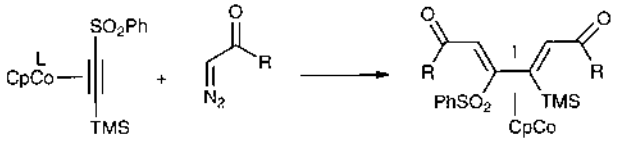


(67)

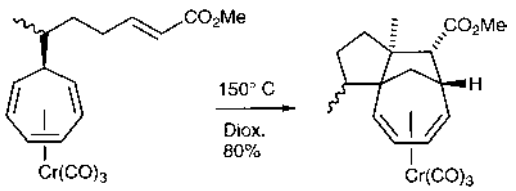


(68)

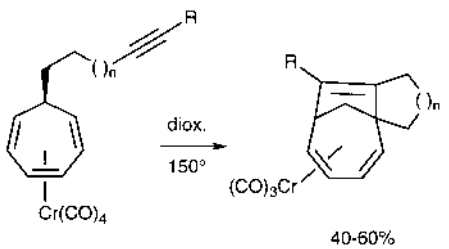
Rhodium(II) acetate catalyzed Si–H, OH, NH, and SH insertion reactions of α-diazoesters [249] and insertion into the amide NH bond of a polymer-bound α-amino amide [250].

2.1.5. Cycloadditions

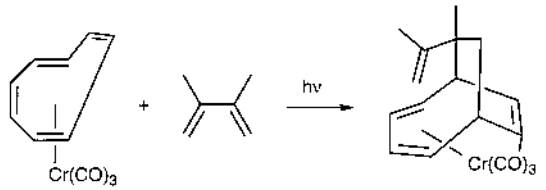
Metal-assisted cycloaddition reactions in organotransition metal chemistry have been reviewed (918 references) [251], as has molybdenum-catalyzed and mediated cycloaddition reactions (22 references) [252]. Nickel(0) complexes catalyzed the [2 + 2 + 2] cycloaddition between norbornadiene and electron-poor alkenes [253]. [6 + 2] Cycloaddition of alkenes [Eq. (69)] [254] and alkynes [Eq. (70)] [255] to chromium cycloheptatriene complexes has been developed. These complexes catalyzed the [4 + 6] cycloaddition of dienes to cycloheptatrienes [256]. Substituted cyclooctatetraenes were synthesized by the [6 + 2] cycloaddition of alkynes to chromium tricarbonyl complexes of cyclic eight-membered triene sulfone, followed by extrusion of sulfur dioxide [257]. Bridged bicyclic systems were synthesized by [6 + 2] cycloadditions [Eq. (71)] [258]:



(69)

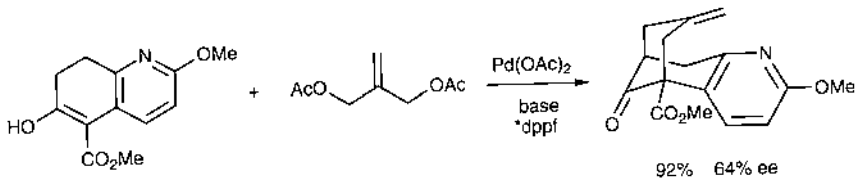


(70)

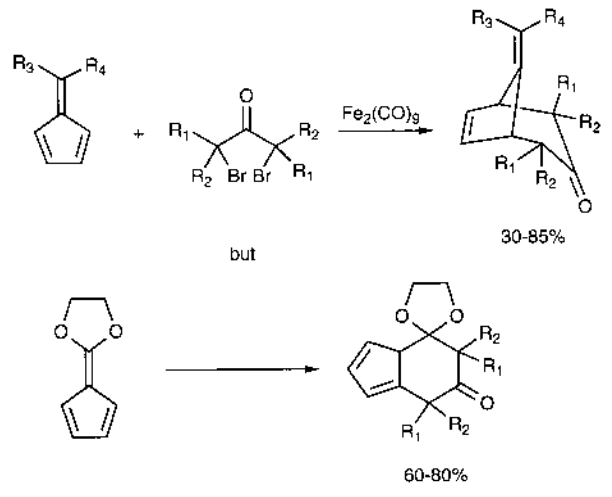


(71)

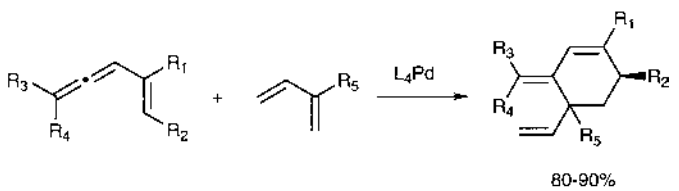
Nickel(0) complexes catalyzed [4 + 4] cycloaddition of linked bis-1,3-dienes to give cycloocta-1,5-dienes [259]. Palladium complexes catalyzed the 3 + 3-trimethylenemethane cycloaddition shown in Eq. (72) [260]. Oxallyliron species cycloadded to fulvenes in two ways [Eq. (73)] [261]. Another unusual cycloaddition is shown in Eq. (74) [262]:



(72)



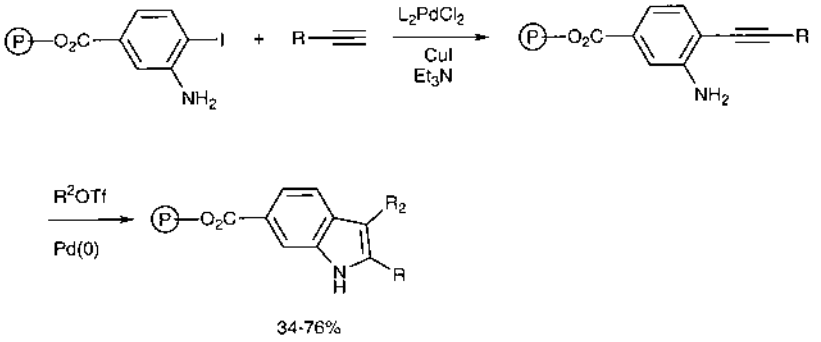
(73)



(74)

2.1.6. Alkylation of alkynes

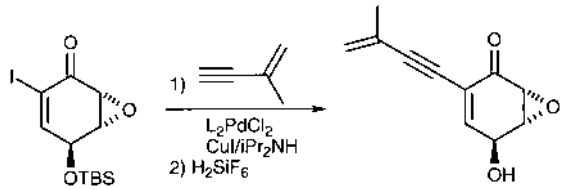
Palladium/copper catalysts (Sonigishara coupling) couple alkynes to polymer-supported *o*-iodoanilines to give indoles after triflate insertion chemistry [Eq. (75)] [263]. The same Pd(0)/Cu(I)/amine catalyst systems coupled terminal alkynes to *o*-iodobenzamides [264], *o*-iodo-phenol THP ethers [265], bromoperylenes [266,267], and 1,2-diiodoarenes (stepwise) [268]. 4-Iodophenylalanine was coupled with acetylene and the resulting phenylacetylene was bis- or tris-coupled to *p*-di-iodobenzene, or 1,3,5-tri-iodo-benzene [269]. Aminal protected propargyl aldehydes were coupled to aryl and alkenyl halides using palladium(0)/copper(I) catalysts [270]:



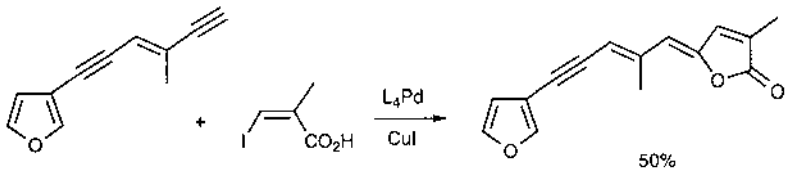
(75)

This coupling chemistry was used in the context of amino acid synthesis by coupling *p*-iodo or *o*-triflyl phenylalanines with long-chain alkynes carrying functionality at the ω-position [271], by arylating propargyl (bis) lactim ethers [272], and by arylating homopropargyl aminoketones [273].

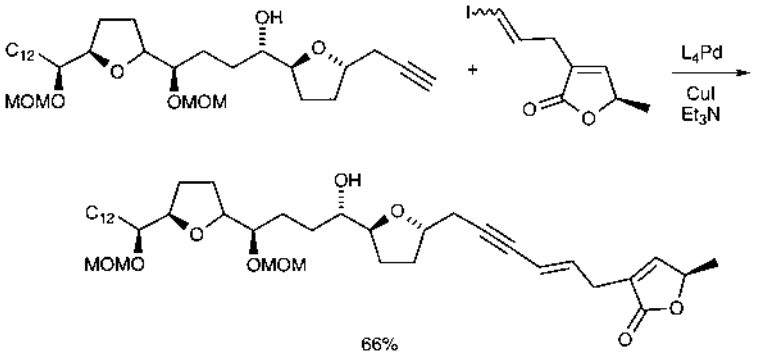
Palladium/copper systems catalyzed the coupling of iodoenynes to terminal alkynes to give endiynes [274], and were used in several total syntheses, Eq. (76) [275], Eq. (77) [276], and Eq. (78) [277]:



(76)

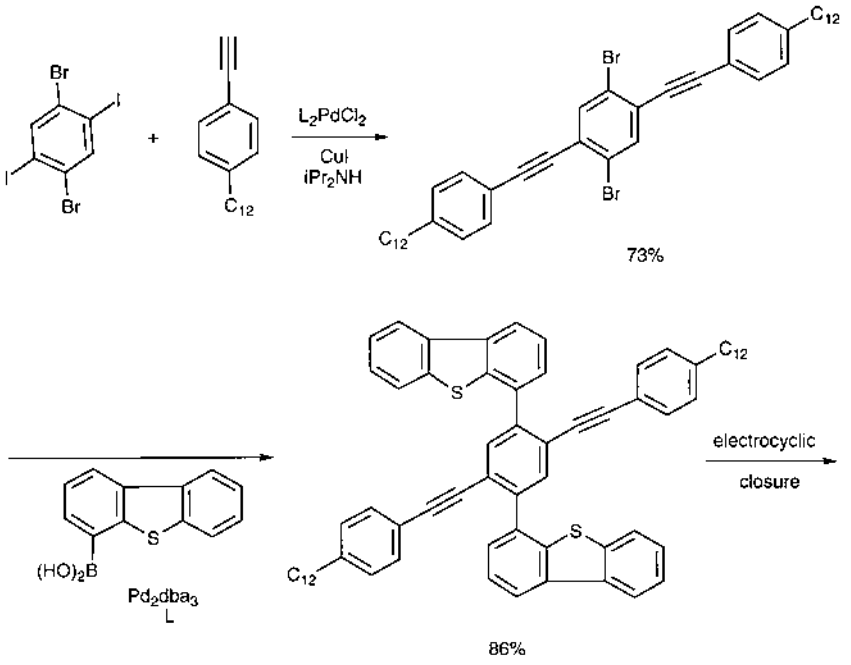


(77)



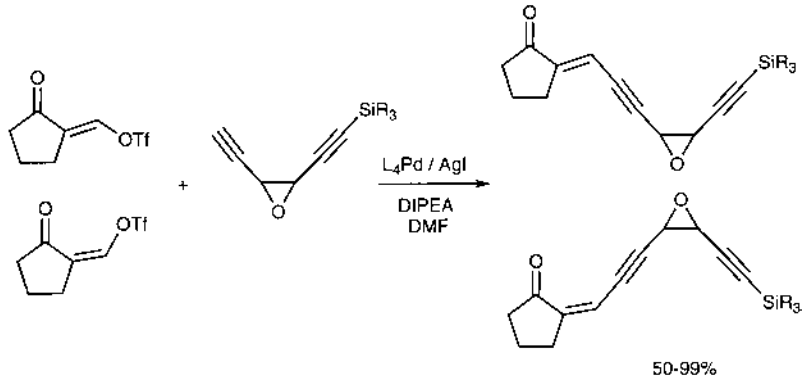
(78)

Heteroaryl halides (furyl, benzofuryl, thiophenyl, pyrrolidinolyl, quinolinolyl) couple to alkynylzinc halides in the presence of palladium catalysts [278]. Palladium/copper systems coupled terminal alkynes to 4-iodoindoles [279], iodopurines [280], iodonucleosides [281], 4,7-dibromo-1,10-phenanthrolines [282], and 2-iodofurans [283]. Sonigishara coupling was also used in the context of materials synthesis to prepare molecular wires consisting of 16 2-ethynylthiophene units [284], photorefractive materials by coupling 4-iodocarbazoles to 4,7-bis-ethynylcarbazoles [285], polypyrroles by the coupling of 2-iodopyrroles with aminoalkynes [286], as well as fused polycyclic aromatics [287]:

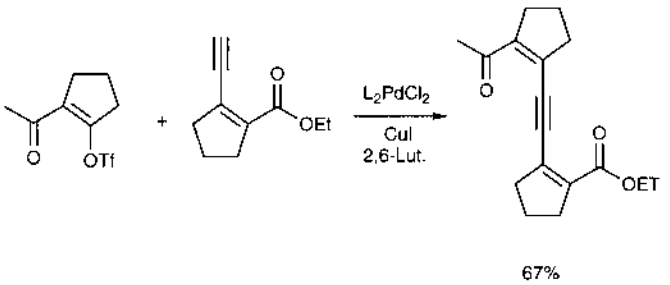


(79)

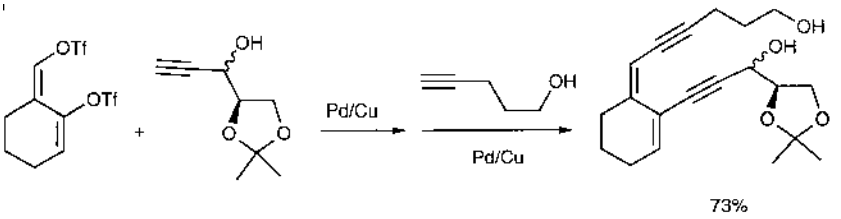
Aryl triflates were coupled to trimethylsilyl alkynes by palladium copper catalysts [288]. Useful enol triflate/alkyne coupling processes are seen in Eq. (80) [289], Eq. (81) [290], Eq. (82) [291], Eq. (83) [292], Eq. (84) [293], Eq. (85) [294], Eq. (86) [295], and Eq. (87) [296]:



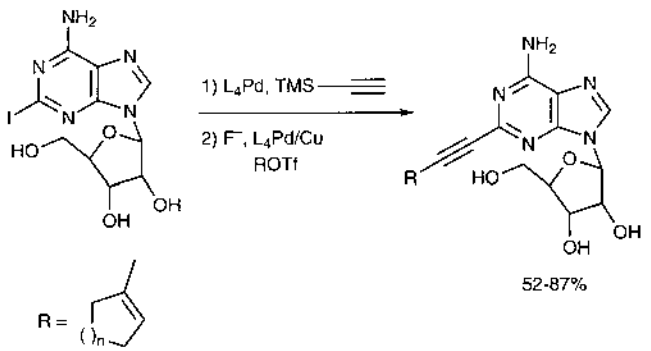
(80)



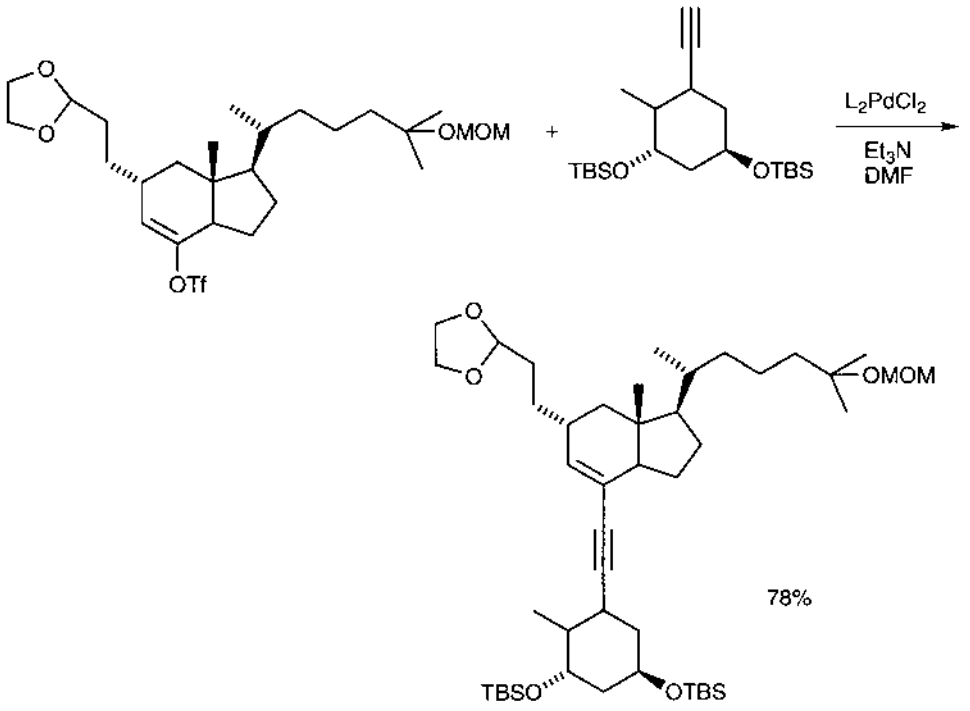
(81)



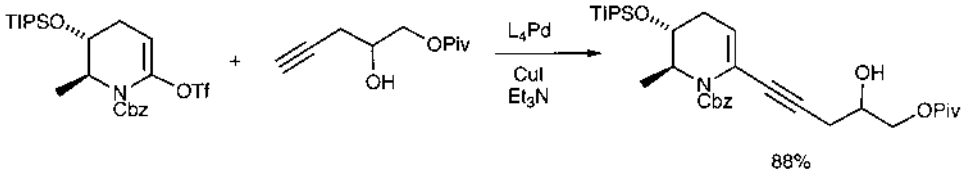
(82)



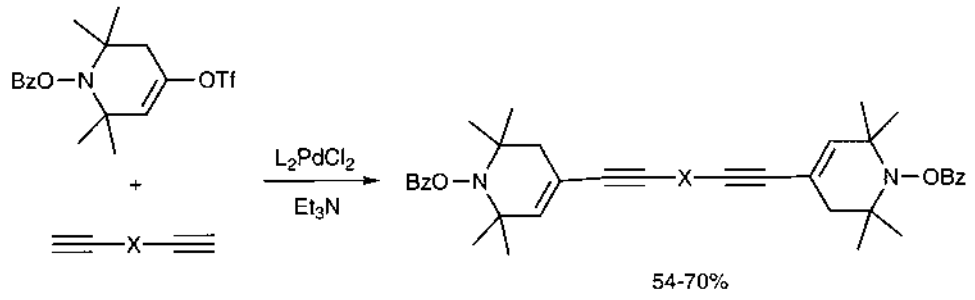
(83)


OMOM
OTf
TBSO
OTBS
L_2PdCl_2
Et_3N
DMF
78%

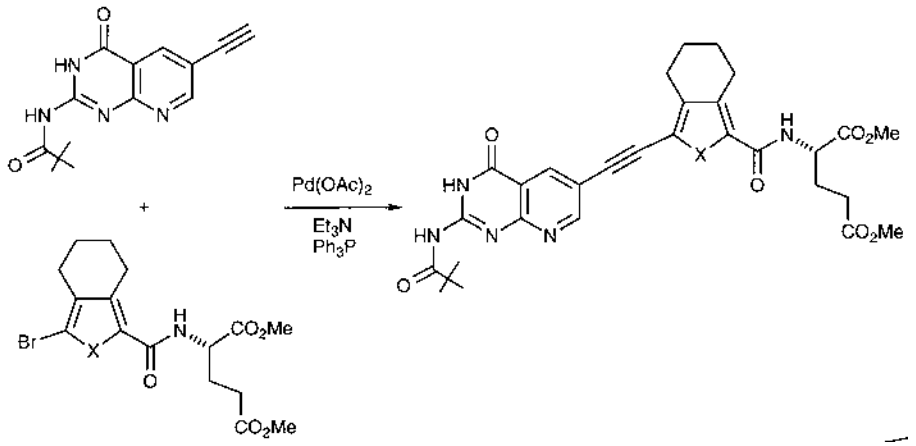
(84)


TIPSO
OTf
N
Cbz
OPiv
OH
L_4Pd
CuI
Et_3N
88%

(85)

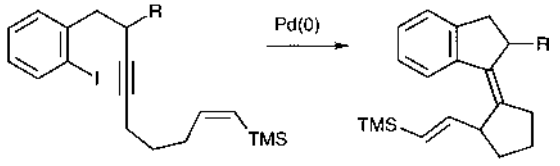

BzO–N
OTf
X
L_2PdCl_2
Et_3N
N–OBz
54-70%

(86)

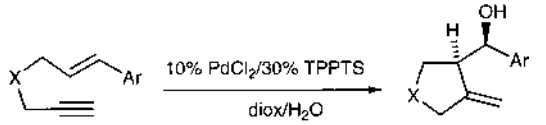


(87)

Alkynes were arylated by oxidative addition/alkyne insertion processes, to give styrenes [297] or allenes [298]. Propiolic amides were β-arylated to give cinnamides [299]. Intramolecular versions were efficient, Eq. (88) [300] and Eq. (89) [301]:

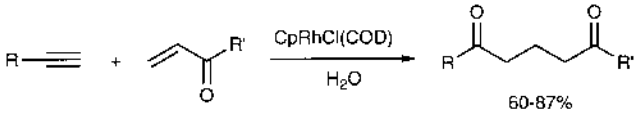


(88)



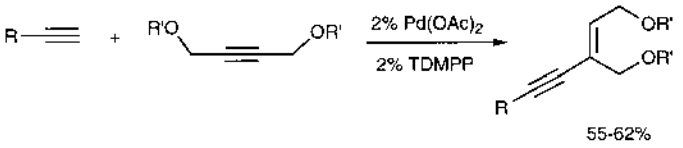
(89)

Alkynes were alkylated by malonates in the presence of molybdenum(0) catalysts [302], as were allenes in the presence of palladium catalysts (intramolecularly to make 9-, 10-, 16-, and 17-membered lactones) [303]. 1-(*p*-Bromophenyl)allenes were oligomerized, with incorporation of malonate, then treated with malonic ester and a palladium(0) catalyst, by alkylation at the central carbon and coupling of the terminal carbon with the bromobenzene group [304].



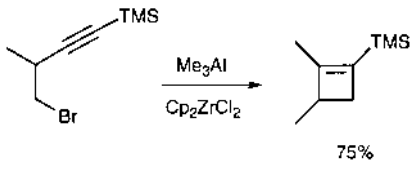
R = HO(CH2)3, NC(CH2)3, RO2C(CH2)3, etc.

(90)

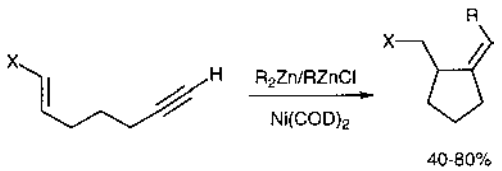


(91)

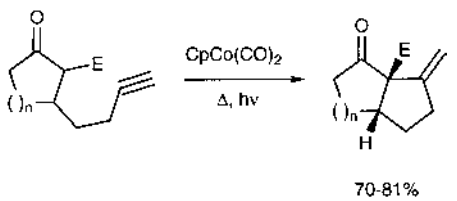
Zirconocene dichloride catalyzed the carboalumination of alkynes [308]. Homopropargyl bromides cyclized when carboaluminated [Eq. (92)] [309]. Low valent titanium complexes coupled alkynes with allylic halides and electrophiles to give substituted 1,4-dienes [310]. MAO coupled allylzirconium species with 2-position terminal alkynes to give 1,4-dienes [311]. Other unusual alkyne alkylations are seen in Eq. (93) [312], Eq. (94) [313], and Eq. (95) [314]:



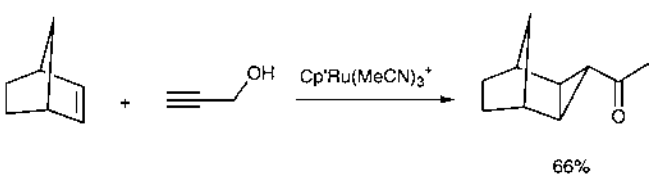
(92)



(93)



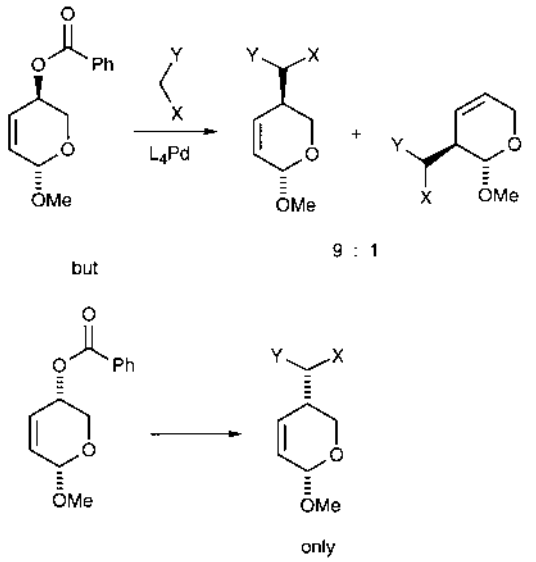
(94)



(95)

2.1.7. Alkylation of allyl, propargyl, and allenyl systems

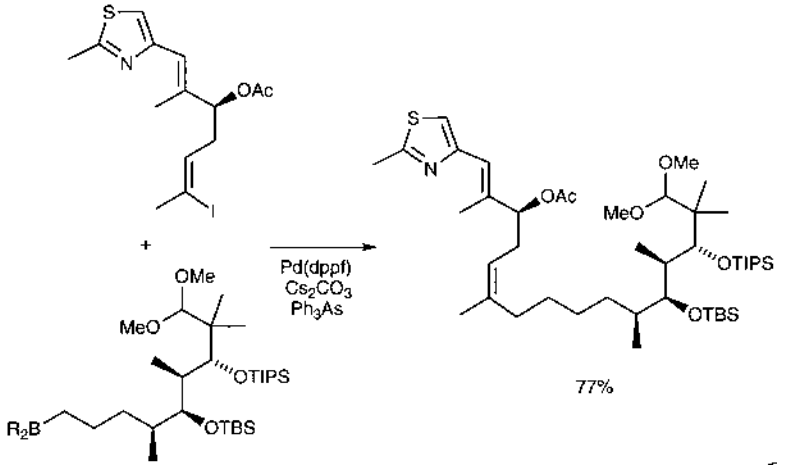
Water-soluble, polymer-bound, recoverable palladium catalysts for allylic alkylation reactions have been developed [315,316]. NMR evidence for olefin complexation in palladium-catalyzed allylic alkylation has been obtained [317]. Electronics have been shown to control the regioselectivity in palladium-catalyzed allylic alkylations of cyclic systems [318]:



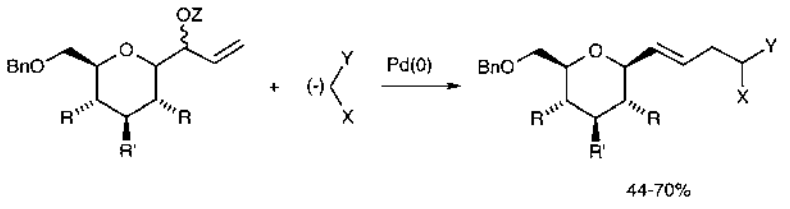
(96)

Palladium(0) catalyzed the allylic alkylation of allyl alcohols by initial treatment with oxalyl chloride [319] or under Mitsunobu conditions [320]. Polymer-bound palladium catalysts alkylated allylic alcohols by dialkylaryliodonium salts to give β-alkyl allyl alcohols [321]. Allyl sulfilimines [322] and allyl phenyl ethers [323] were good substrates for palladium-catalyzed allylic alkylation. Palladium(0) complexes catalyzed the ring-opening alkylation of methylene cyclopropanes by stabilized carbanions [324].

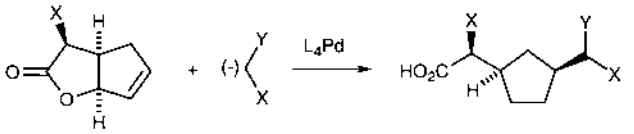
Palladium(0) catalyzed the allylic alkylation of γ-acetoxyvinyl phosphonates by glycine imine enolates [325]. Allyl carbonates and epoxides were similarly reactive toward masked glycine enolates [326]. Allyl acetates were stable to Suzuki coupling conditions [Eq. (97)] [327]. Other useful allylic alkylations are seen in Eq. (98) [328] and Eq. (99) [329]:



(97)

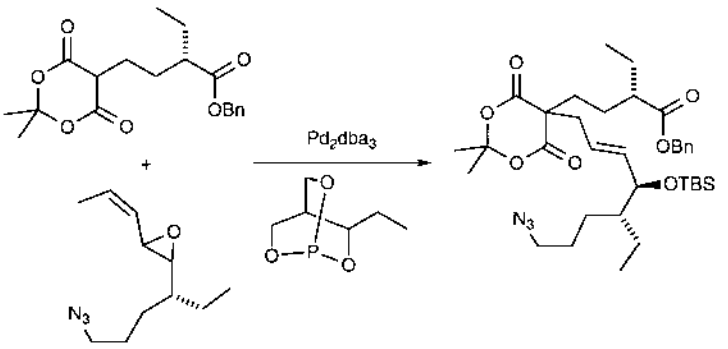


(98)

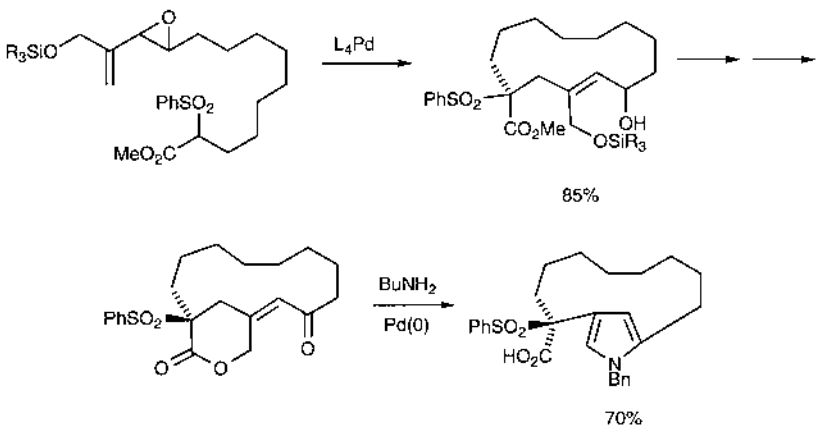


(99)

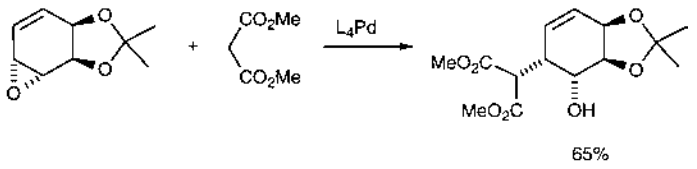
Palladium-catalyzed alkylations of allyl epoxides are shown in Eq. (100) [330], Eq. (101) [331], and Eq. (102) [332]:



(100)

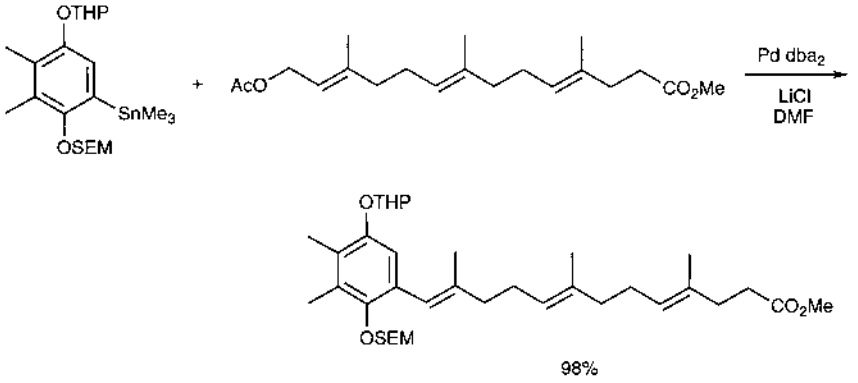


(101)

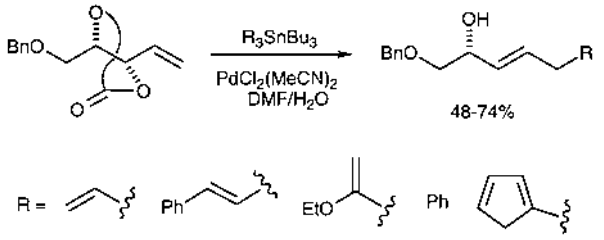


(102)

Allylic acetates and carbonates were alkylated by arylstannanes [Eq. (103)] [333], allylstannanes [334], alkenylstannanes [Eq. (104)] [335], and alkenylsilanes [336] in the presence of palladium(0) catalysts:

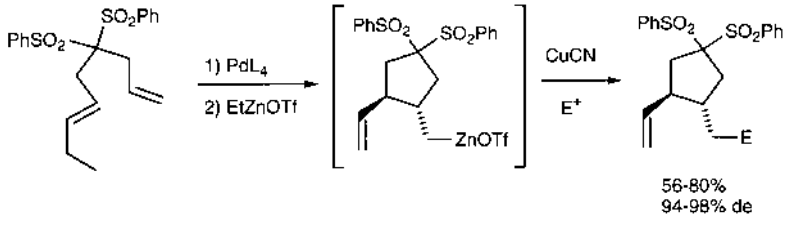


(103)

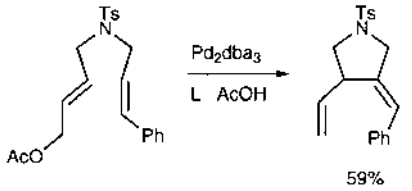


(104)

Palladium(0) catalyzed allyl acetate cascade processes, Eq. (105) [337] and Eq. (106) [338]:

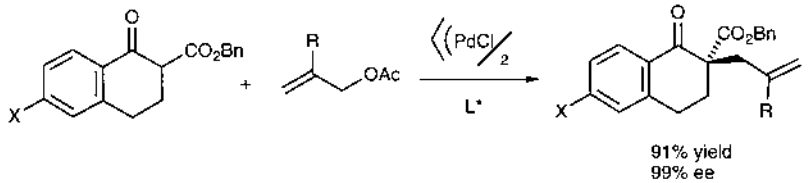


(105)

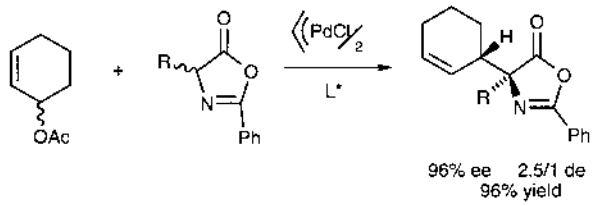


(106)

Palladium-catalyzed asymmetric allylic substitutions have been reviewed [339]. Work continued unabated to develop asymmetric allylic alkylation of 1,3-diphenylallyl systems, a transformation that can already be carried out with very high ee. Chiral amidines gave 91–95% ee [340]. Palladium-catalyzed allylic alkylation could be directed to the benzylic position of cinnamyl acetals, with high ee by using methyl dialkylmalonates and (R) MeOMOP ligands [341] or by blocking terminal attack with two removable silyl groups [342]. Cis-1,4-bis-benzoylcyclohept-2-ene was asymmetrically allylically alkylated by stabilized carbanions with modest ee in the presence of palladium(0) catalysts and BINAP [343]. The 1,3-diphenylallyl system was alkylated by acetamidomalonates with high ee in the presence of palladium(0) and bridged bicyclic phosphines [344]. Synthetically useful allylic alkylations are seen in Eq. (107) [345], Eq. (108) [346], and Eq. (109) [347]:



(107)



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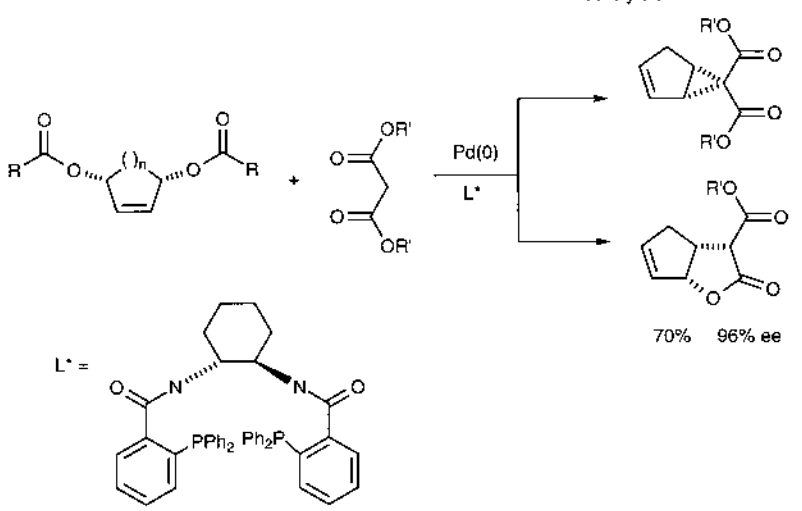
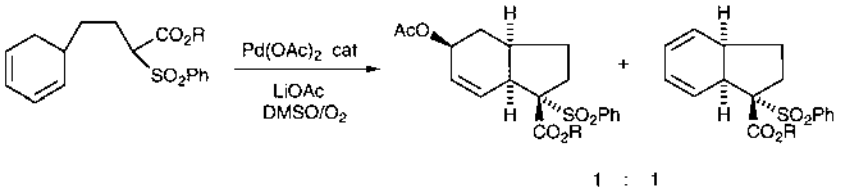
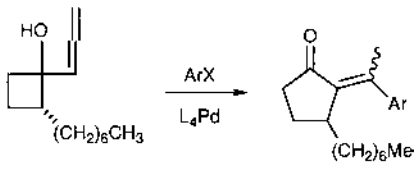


(109)

Cyclohexadienes with pendent nucleophiles cyclized in the presence of palladium acetate [Eq. (110)] [348]. Allenic cyclobutanols ring-expanded when treated with aryl halides and palladium(0) catalyst [Eq. (111)] [349]:

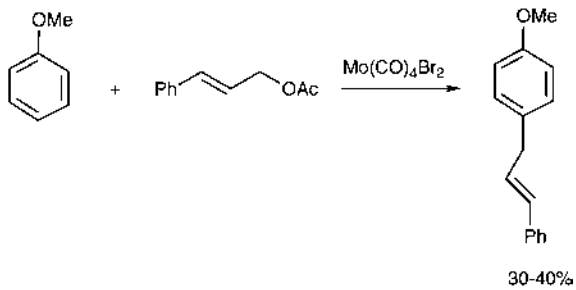


(110)



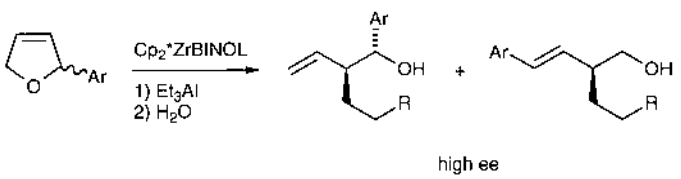
(111)

Nickel(0) dppb complexes catalyzed the alkylation of allyl amines by β-dicarbonyl compounds [350]. Molybdenum carbonyls catalyzed the alkylation of 3-acetoxycyclohexenes by stabilized carbanions [351]. Molybdenum(II) carbonyls catalyzed the alkylation of allyl acetates by electron-rich arenes [Eq. (112)] [352,353]. Iridium(I) complexes catalyzed the allylic alkylation of allyl acetates by a range of nucleophiles at the *more* hindered position, complementing the regiochemistry observed with palladium [354]. In the presence of chiral oxazolidine–phosphine ligands, high enantiomeric excesses were observed [355]:

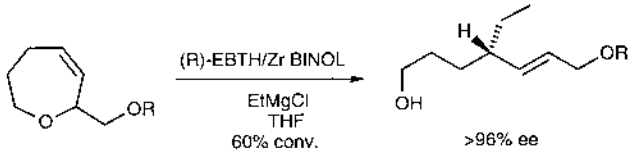


(112)

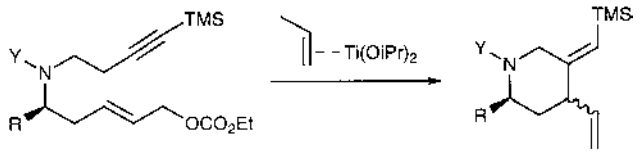
Cyclic allylic ethers were alkylatively opened [Eq. (113)] [356] and kinetically resolved [Eq. (114)] [357] by chiral zirconium arene complexes. Low-valent titanium complexes cyclized alkyne-containing allyl carbonates [Eq. (115)] [358]:



(113)

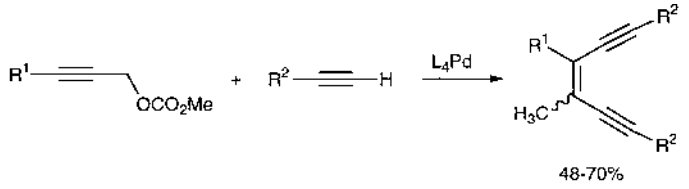


(114)

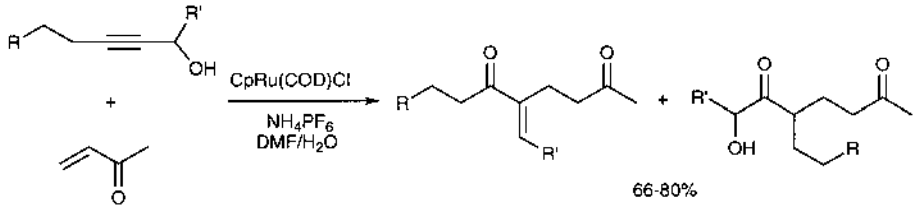


(115)

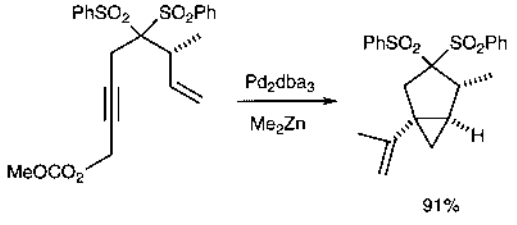
Chiral propargyl carbonates were S_N2' alkylated by phenylzinc bromide in the presence of copper/palladium catalysts to give optically active allenes [359,360]. Diethylzirconocene ethylated propargyl silyl ethers to give allenes [361]. Palladium(0) complexes catalyzed the alkylation of propargyl carbonates with terminal alkynes to give enediynes [Eq. (116)] [362], while ruthenium(II) complexes catalyzed the alkylation of propargyl alcohols by enones [Eq. (117)] [363]. Propargyl systems underwent palladium-catalyzed cascade cyclizations [Eq. (118)] [364]:



(116)

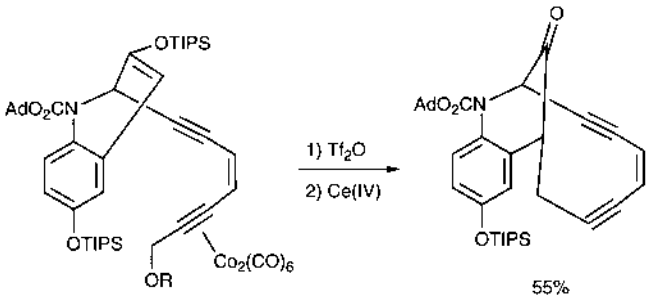


(117)

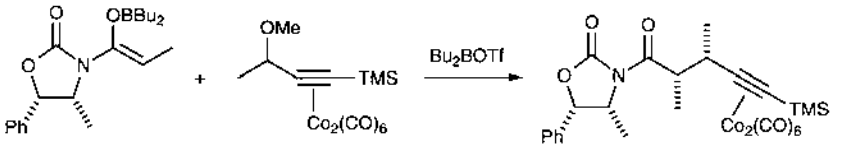


(118)

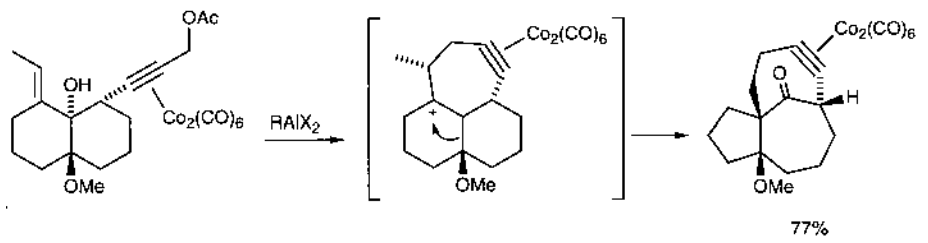
The full paper on the synthesis of (±) calicheamicinone, involving lots of cobalt-stabilized propargyl cation chemistry, has appeared [365], as have other examples of the use of cobalt–propargyl cations in synthesis, Eq. (119) [366], Eq. (120) [367,368], and Eq. (121) [369]. Cobalt also permitted propargyl radicals to be generated and used in synthesis [Eq. (122)] [370]:



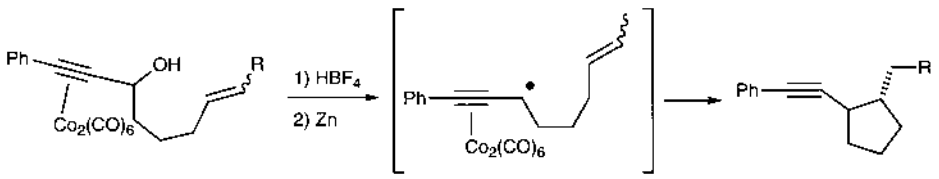
(119)



(120)



(121)

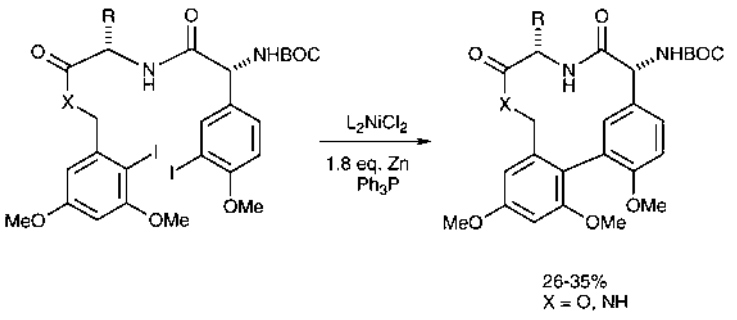


(122)

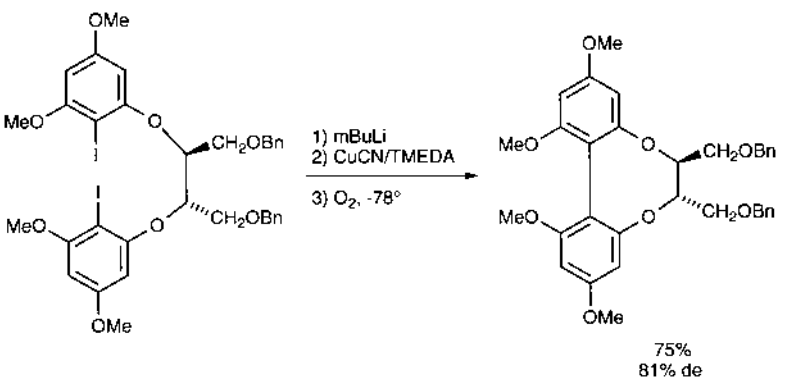
2.1.8. Coupling reactions

Palladium(0) complexes dimerized indole-2-boronic acids to give 2,2′-bis indoles [371]. Palladium acetate in the presence of air and base coupled arylboronic acids to biaryls [372], while palladium(0) in the presence of $PhCHBrCHBrCO_2Et$ as an oxidant coupled aryl stannanes, aryl boronates and aryl silanes to biaryls [373]. π-Allylpalladium halides catalyzed the coupling of aryl and alkynyl stannanes in the presence of phosphine–imine ligands [374]. Palladium(II) salts in the presence of oxygen coupled alkenyl stannanes to dienes [375], while palladium or nickel catalysts in the presence of reducing agents coupled aryl triflates to biaryls [376]. Palladium(II)/copper(II) systems catalyzed the coupling of conjugated dienyl, trienyl and tetraenyl silanes [377]. 2-Bromofurans were coupled by treatment with hexamethyl ditin in the presence of palladium(0) catalysts [378]. Biaryls were made by treatment of aryl halides with $(RO)_2BB(OR)_2$ in the presence of palladium catalysts [379]. Indoles were coupled at the 2-position by treatment with a three-fold excess of palladium(II) trifluoroacetate [380].

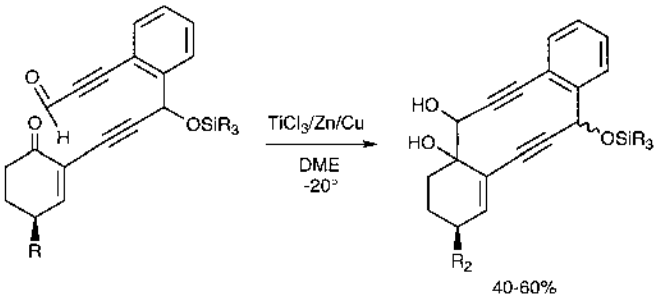
Nickel(II) phosphine complexes catalyzed the coupling of aryl iodides in the presence of zinc metal [Eq. (123)] [381]. Aryl halides were cross-coupled to α-chloroesters bearing chiral alkoxy groups under similar conditions with up to 96% ee [382]. Copper iodide coupled alkenylzirconocene halides [383] and alkenyl stannanes [384] to give 1,3-dienes. Chiral binaphthols were made by copper(I) coupling of chiral diol-linked naphthols [385] [Eq. (124)] [386]. Diynes were made by McMurry coupling [Eq. (125)] [387]:



(123)



(124)

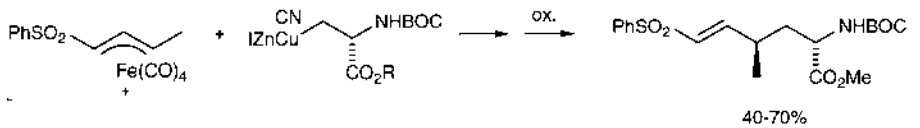


(125)

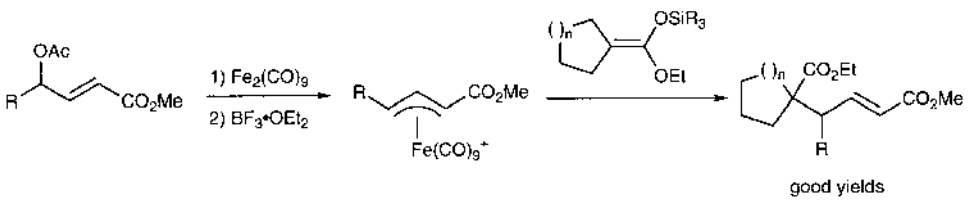
2.1.9. Alkylation of π-allyl complexes

The ^{13}C-NMR spectra of unsymmetrically substituted 1,3-diaryl-π-allylpalladium complexes showed δ ^{13}C correlated to a Hammett σ plot, and that the terminal carbon with the lower field ^{13}C chemical shift was the site of nucleophilic attack [388]. π-2-Chloroallylpalladium diphos cation complexes reacted twice with malonate, first at the central carbon, then at a terminal position [389]. Oppolzer coupling (intramolecular insertion of an alkene into a π-allyl–palladium system) occurred from the cationic π-allyl–π-olefin complex. Excess diphos inhibited the reaction by displacement of the alkene [390].

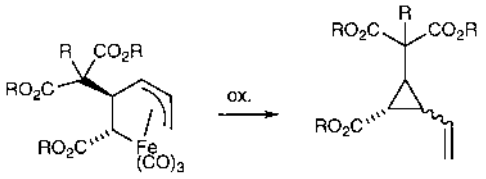
The synthesis of highly enantio-enriched compounds via iron-mediated allylic substitutions has been reviewed (52 Refs.) [391]. Nucleophilic attack on diastereoisomers 1-acyl–π-allyl iron nitrosyl dicarbonyl complexes went with very high de [392]. Cationic π-allyliron complexes underwent nucleophilic attack by functionalized organocuprates [Eq. (126)] [393] and ketene silylacetals [Eq. (127)] [394]. Oxidation of σ-alkyl–π-allyliron complexes produced cyclopropanes [Eq. (128)] [395]. π-Allylironcarbene complexes were alkylated by malonates [Eq. (129)] [396]. π-Allylmanganese tricarbonyl complexes allylated carbanions, including those from dithianes [397]. Isoprene underwent reaction with Cp_2TiCl_2/^{i}PrMgCl to give the π-1,2-dimethylallyltitanium complex, which allylated aldehydes [398]:



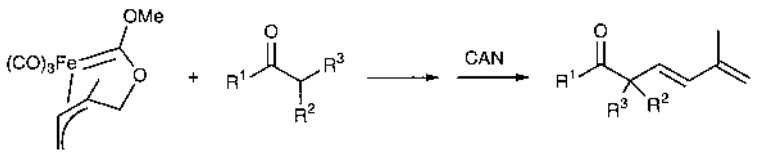
(126)



(127)



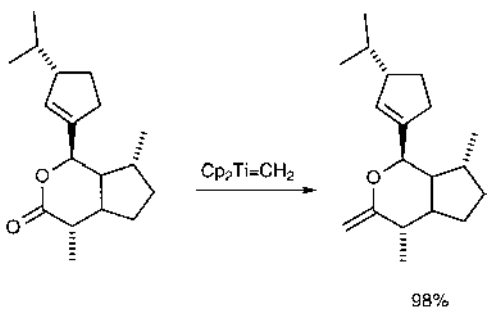
(128)



(129)

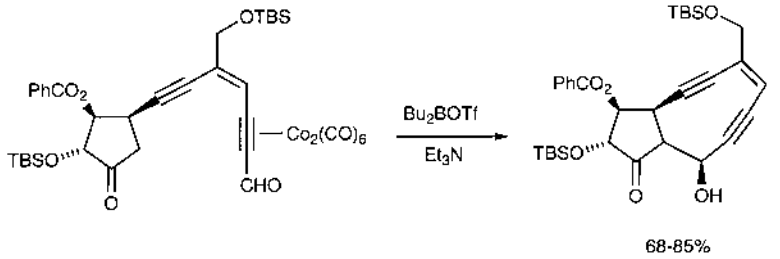
2.1.10. Alkylation of carbonyl compounds

Cobalt(0) phosphine complexes alkylated ketones with enolates generated from α-haloketones [399]. Treatment of α-haloketones with Bu_3MnLi followed by aldehydes resulted in ketone–aldehyde aldol chemistry [400]. Allylmanganese complexes allylated aldehydes [401]. Cp_2TiMe_2 methylenated conjugated esters to give 2-alkoxydienes [402]. Tebbe's reagent methylenated lactones [Eq. (130)] [403] and 2 halo and α-thio ketones [404]:

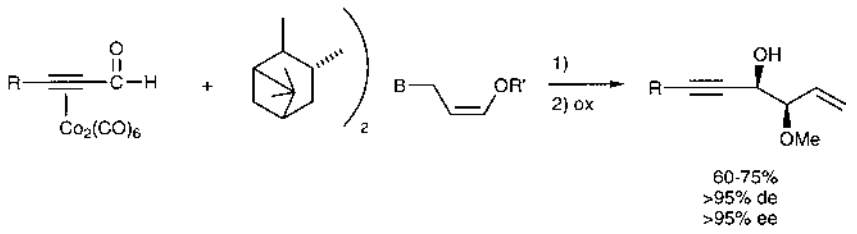


(130)

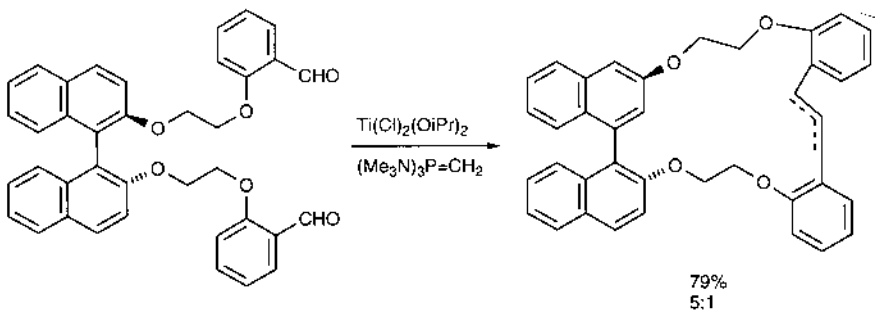
Cobalt-complexed propargyl aldehydes were alkylated by boron enolates [Eq. (131)] [405] and chiral allylboranes [Eq. (132)] [406]. The γ-anion of (propenyl)(amino)chromium carbene complexes alkylated aldehydes [407]. Other interesting alkylations of carbonyls are seen in Eq. (133) [408], Eq. (134) [409], Eq. (135) [410], and Eq. (136) [411]:



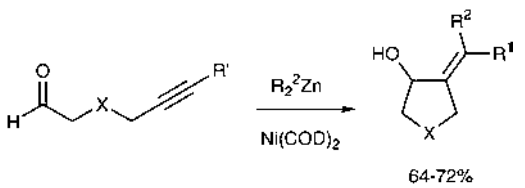
(131)



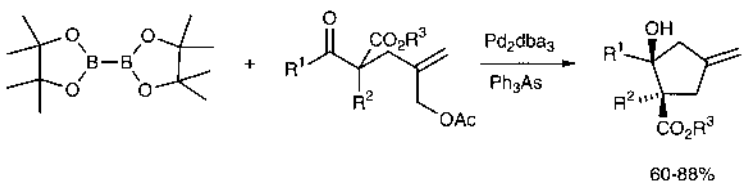
(132)



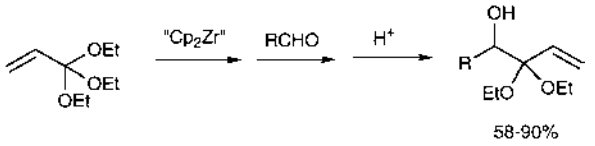
(133)



(134)



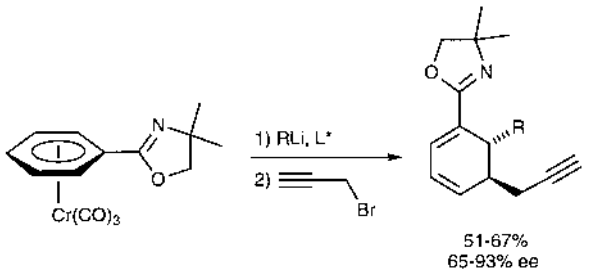
(135)



(136)

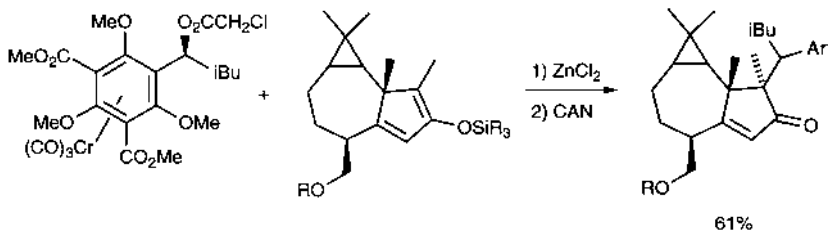
2.1.11. Alkylation of aromatic compounds

Chlorobenzenechromium tricarbonyl was alkylated by copper complexes of α-lithiohydrazones [412]. Chromium complexes of phenyloxazoline were asymmetrically lithiated in the presence of (1*S*)(2*S*)-1,2-dimethoxybibenzyl, and either oxidized with Ph_3C^+ to give chiral arene complexes (up to 96% ee) [413] or alkylated with propargyl bromide [Eq. (137)] [414]:



(137)

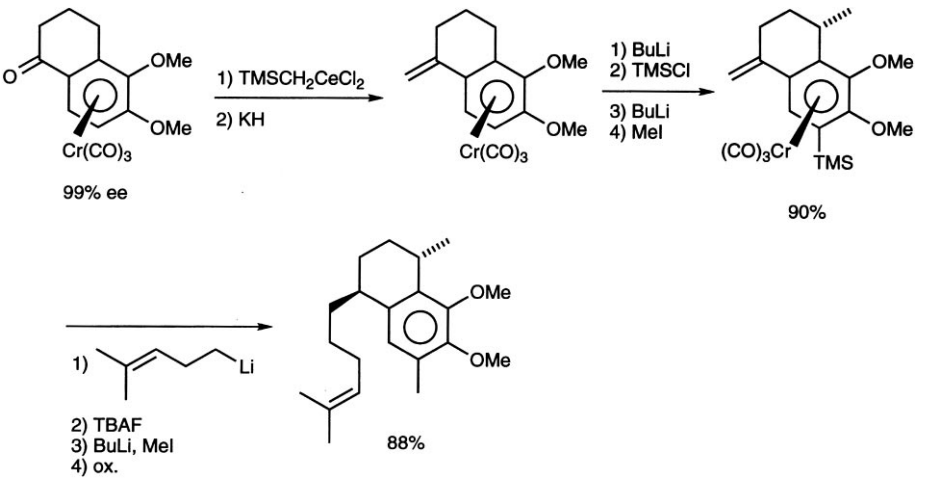
Chromium-complexed benzyl alcohols were converted to benzyl chlorides by treatment with HCl, aminated with aminonitriles, and the nitrile α-position cyclized onto the arene in modest yield [415]. Chromium-complexed benzyl thioethers were benzylically deprotonated with a chiral base, then alkylated with organic halides in good yield and 63–91% ee [416]. Benzylic alkylation is shown in Eq. (138) [417]:



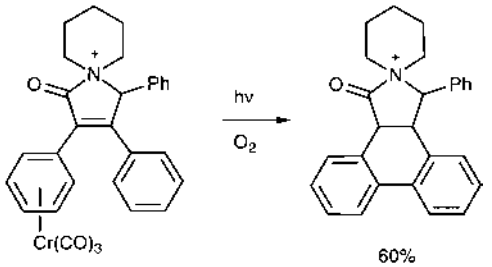
(138)

The ring site of deprotonation of chromium-complexed 1-trimethylsilyl-2,6-dimethoxybenzene depended on conditions [418]. Complexation of chromium to a chiral benzaldehyde ketal followed by lithiation/reaction with an electrophile, followed by hydrolysis gave optically active o-substituted chromium benzaldehyde complexes [419]. Monosubstituted chromium arene complexes were α-deprotonated by chiral bases, then reacted with electrophiles to produce chiral chromium arene complexes with good ee [420,421].

Chromium arene complexes were used in organic synthesis in methods resembling the extensive previous studies of Uemura [Eq. (139)] [422,423]. A strange arene coupling occurred upon irradiation of a chromium arene complex [Eq. (140)] [424]. Chiral chromium-complexed *o*-substituted benzaldehyde imines were coupled by samarium(II) iodide to give the complexed diaryl ethylenediamine in 40–80% yield and enantiomerically pure [425]:

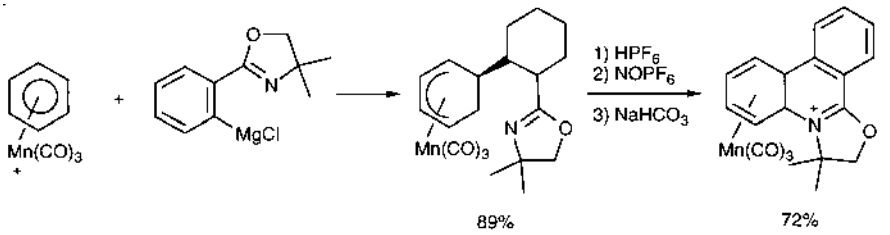


(139)



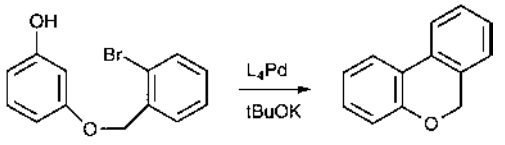
(140)

Cationic arene manganese tricarbonyl complexes (as well as cationic iron dienyl complexes) were alkylated by α-lithiated methyl Fischer carbene complexes [426]. Cationic manganese tricarbonyl complexes of A-ring methoxyaryl steroids were alkylated by Grignard reagents meta to the methoxy group [427]. Manganese arene complexes were alkylated by Grignard reagents from protected benzaldehydes [Eq. (141)] [428]. The cationic CpFe aniline complex was oxidized to the nitrobenzene complex and the nitro group underwent substitutions by stabilized carbanions [429]:

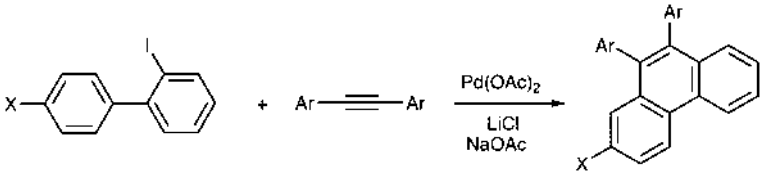


(141)

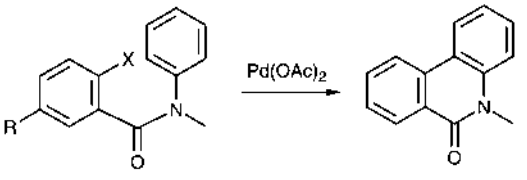
Palladium(II) catalyzed the arylation of 2-hydroxybiphenyls in the peri position of the nonhydroxylated ring by a peripalladation process [430]. Other palladium-assisted arylation of arenes are shown in Eq. (142) [431], Eq. (143) [432], Eq. (144) [433], and Eq. (145) [434]. Ruthenium(0) carbonyls catalyzed the *o*-alkylation of acetophenones by olefins [435]:



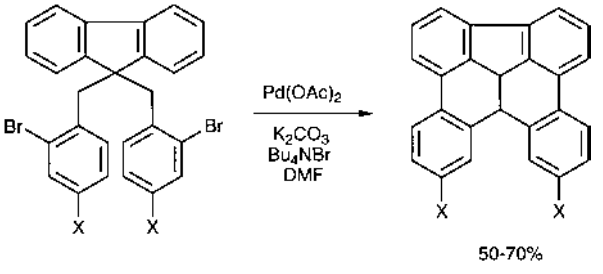
(142)



(143)

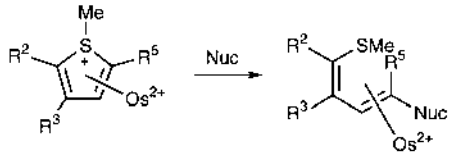


(144)



(145)

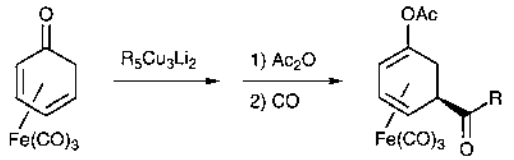
The activation of aromatic molecules with pentammineosmium(II) has been reviewed (92 references) [436]. Osmium(II)-complexed arenes were readily protonated and had pK_as of -6 to -8 [437]. Osmium(II)-complexed anisoles added, in a Michael sense, conjugated enones at the para position [438]. Osmium(II)-complexed thiophenes underwent nucleophilic ring-opening [439]:



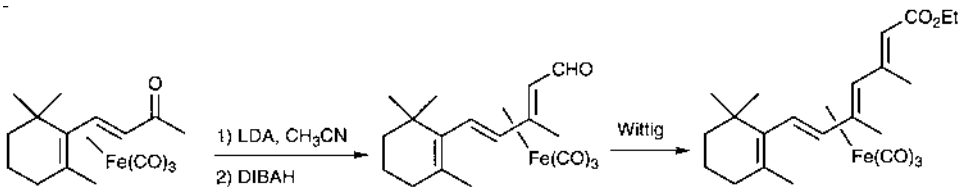
Nuc = H^-, CN^-, AcO^-, Py, N_3^-, PhO^-, PhS^- (146)

2.1.12. Alkylation of diene and dienyl complexes

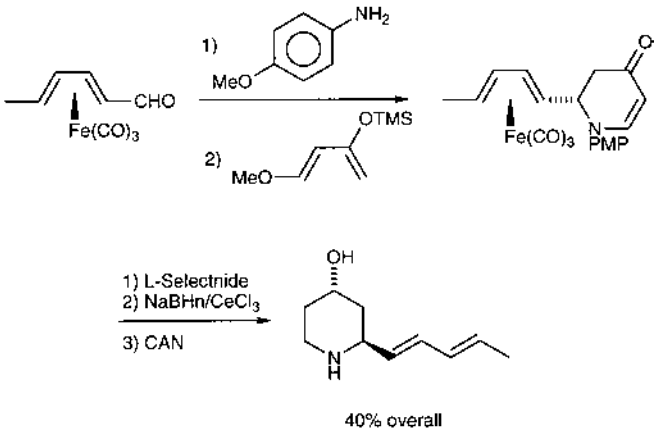
The iron tricarbonyl complex of cyclohexa-2,4-dienone (keto tautomer of phenol) underwent γ-alkylation by cuprates [Eq. (147)] [440]. The B-ring diene iron tricarbonyl complex of a steroid underwent specific hydride reduction at the A–B ring juncture terminus of the diene [441]. The aldol condensation of optically active trimethylsilylenol ethers of 1-acetyl-1,3-pentadieneiron tricarbonyl was used in the synthesis of streptenols C and D, 3,6-dideoxyhexoses, and 3-deoxypentoses [442]. The iron tricarbonyl complex of hexadienal was used in the stereocontrolled synthesis of 4,6-, 5,7-, and 6,8-diene-2-ols [443]. The optically active iron tricarbonyl complex of hexa-3,5-dienoic esters underwent alkylation α to the ester with good yield and up to 82% ee [444]. Related alkylation chemistry is shown in Eq. (148) [445] and Eq. (149) [446]. Iron vinylketene complexes inserted alkynes and carbon monoxide to produce iron diene-complexed cyclohexadienones [447].



68-97% (147)

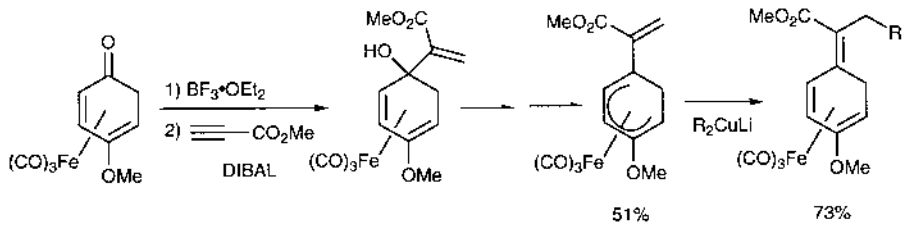


91% 56% (148)

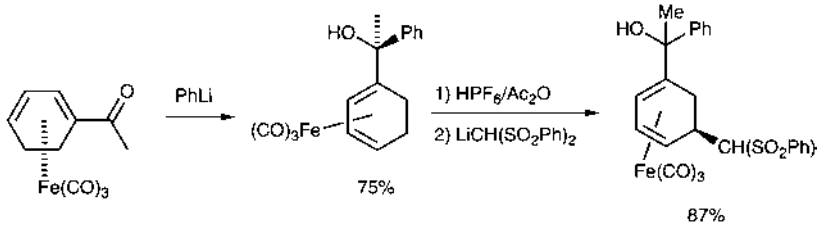


(149)

The regioselectivity for the alkylation of methoxycyclohexadienyl iron complexes could be altered by tethering the iron to a pendant phosphine [448]. This same complex was alkylated para to the methoxy group by the enolate of hexa-2,4-dienyl malonate [449]. Carbazoles were once again synthesized by the reaction between cyclohexadienyliron tricarbonyl complexes and *p*-anisidine [450]. Synthetically interesting applications of iron diene/dienyl complexes are shown in Eq. (150) [451] and Eq. (151) [452]. Acyclic hexadienyliron tricarbonyl complexes underwent reaction with nucleophiles to give mixtures of E and Z dienes [453].



(150)

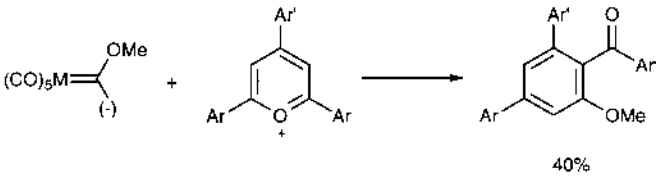


(151)

2.1.13. Metal carbene reactions

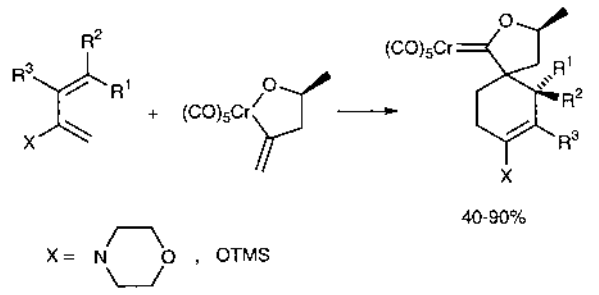
'Developing the physical organic chemistry of Fischer carbene complexes' is the title of a review (41 Refs.) [454]. The pK_a of the five-membered cyclic chromium alkoxy carbene complex is 100 times greater (less acidic) than the

(methyl)(methoxy)carbene complex [455]. Carbohydrate-containing chromium and tungsten Fischer carbene complexes were made by the reaction of the metal carbonyl dianion with carbohydrate acid chlorides [456] or by reaction of $Cr(CO)_5(OEt_2)$ with carbohydrate containing propargyl alcohols [457]. Methyl carbene complexes were α-alkylated with α-bromoamido acid esters [458]. The α-carbanion of (methyl)(methoxy) carbene complexes converted pyrylium salts to arenes [Eq. (152)] [459]. Treatment of the lithium salt of the anionic acyl metal pentacarbonyl complex with $BF_3 \cdot OEt$ produced acyl radicals which could be trapped [460]. Thermal decomposition of o-acylcarbene complexes produced enol esters [461]:

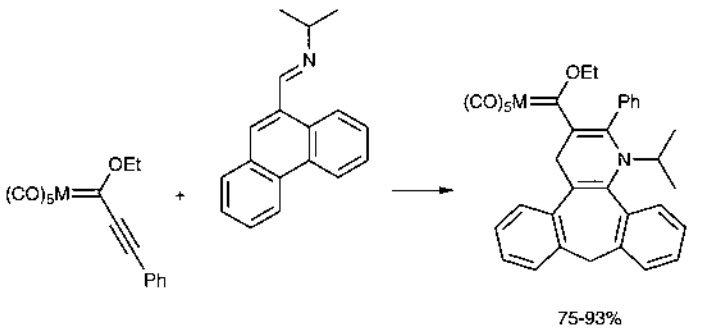


(152)

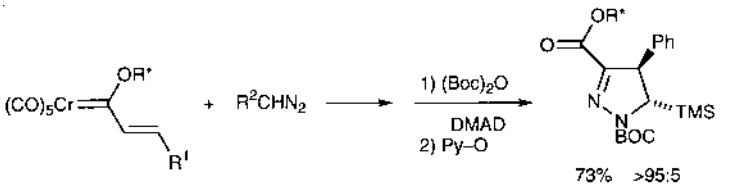
Cycloaddition reactions of α,β-unsaturated carbene complexes are shown in Eq. (153) [462], Eq. (154) [463], Eq. (155) [464], Eq. (156) [465], and Eq. (157) [466]:



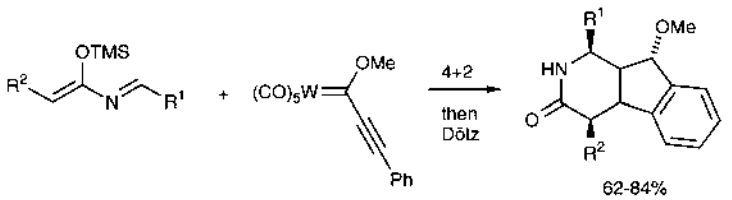
(153)



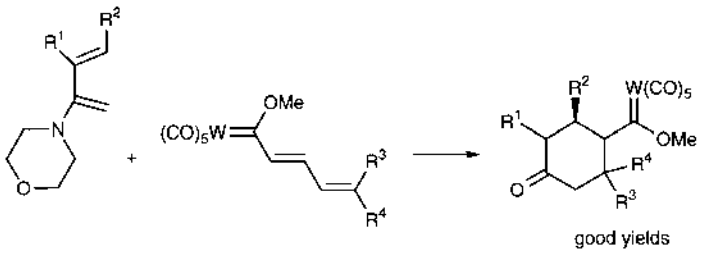
(154)



(155)

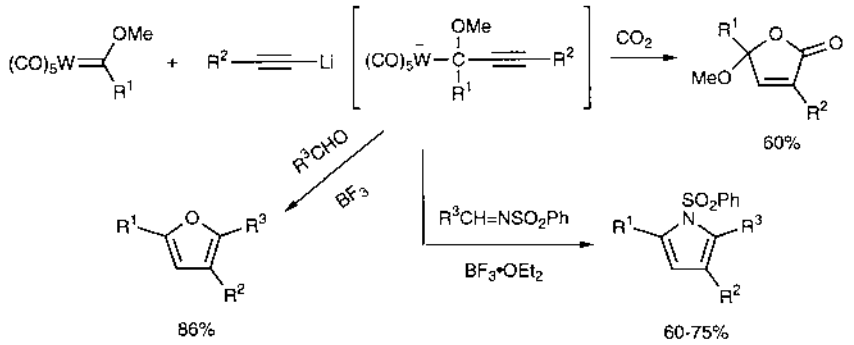


(156)



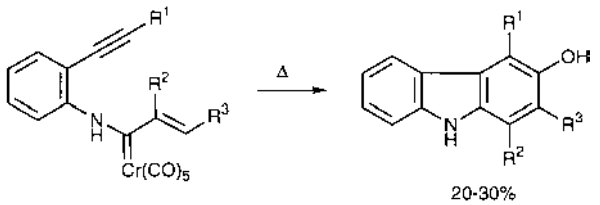
(157)

Reaction of alkoxycarbene complexes with vinyl stannanes or germanes gave γ-alkoxyallyl stannanes or germanes [467], while lithium acetylides followed by tosyl isocyanate produced unsaturated five-membered lactams [468]. Lithium acetylides attacked the carbene carbon to give anionic metal complexes which did further useful chemistry [Eq. (158)] [469]. Carbenoid complexes of electron-deficient transition metals and their use in synthesis have been reviewed (436 Refs.) [470]:

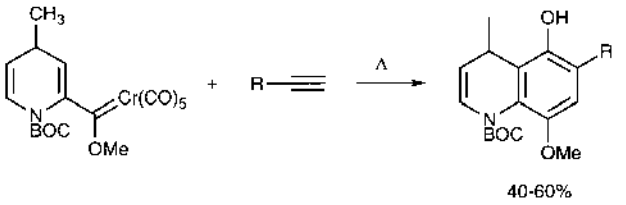


(158)

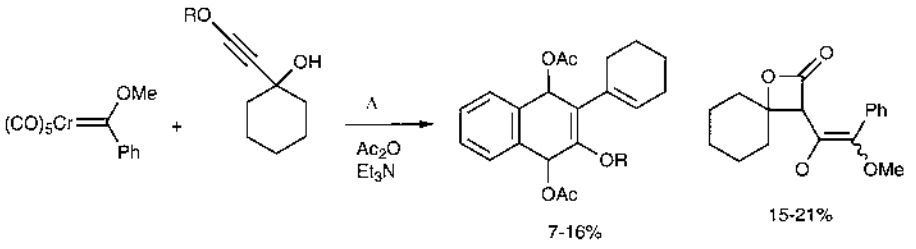
Dötz benzannulations were used to make a number of useful aromatics, Eq. (159) [471], Eq. (160) [472], Eq. (161) [473], Eq. (162) [474], Eq. (163) [475], Eq. (164) [476], Eq. (154) [477], and Eq. (166) [478]:



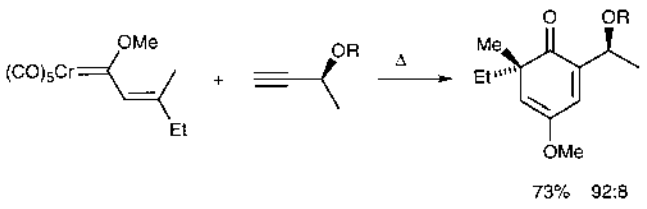
(159)



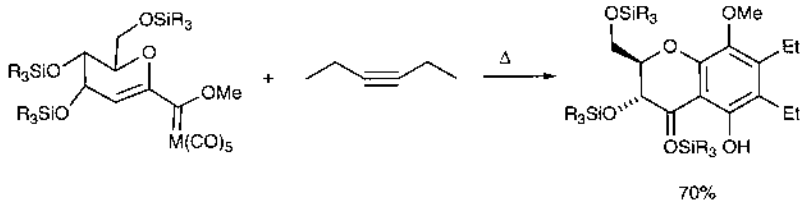
(160)



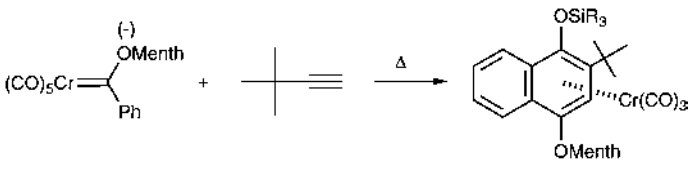
(161)



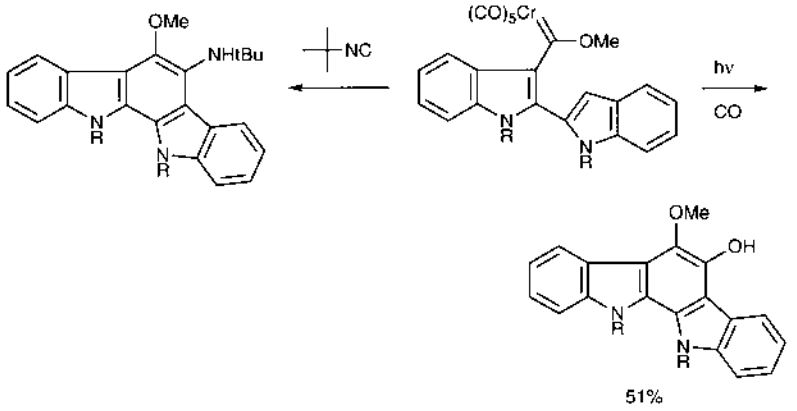
(162)



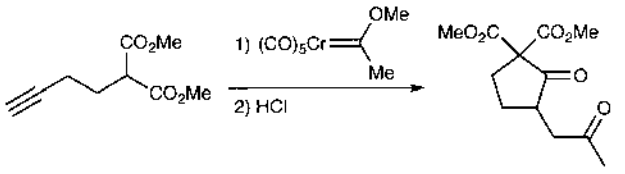
(163)



(164)

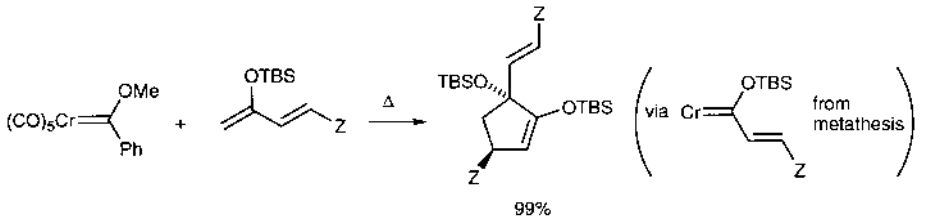


(165)



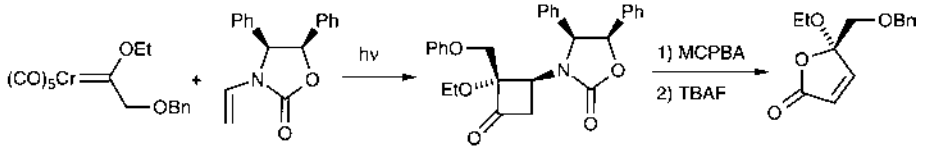
(166)

Chromium acetoxycarbenes cyclopropanated Danishefsky's diene at the silylenol ether double bond [479], while alkoxycarbene complexes metathesized and formed cyclopentanes [Eq. (167)] [480]. Chromium cyclopropyl carbene complexes reacted with alkynes to give cyclopentenones [481]:

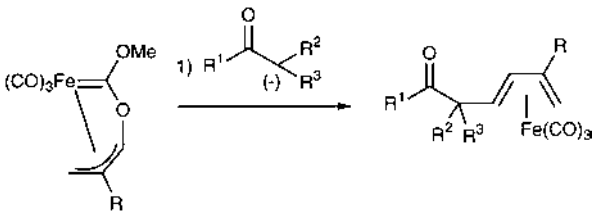


(167)

The use of chromium carbene complex photochemistry in organic synthesis has been reviewed (81 Refs.) [482]. Although these photoreactions appear to proceed through metal-bound ketenes, no evidence for this species was found in matrix isolation studies [483]. The process was used to make optically active butenolides [Eq. (168)] [484], and α-aminoacid activated esters [485]. Iron carbene complexes were alkylated by enolates [Eq. (169)] [486] and inserted into CH bonds [487]:



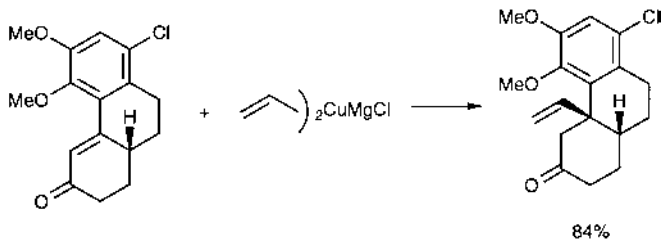
(168)



(169)

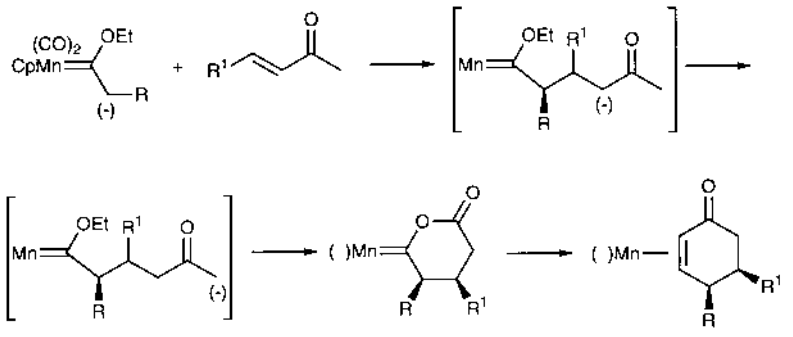
2.2. Conjugate addition

Copper acetylides in the presence of trimethylsilyl iodide added 1,4 to enones to give β-acetylenic silylenol ethers [488]. A number of chiral ligands were used to induce asymmetry into the copper-catalyzed conjugate addition of Grignard reagents to enones [489,490]. Similar studies involving conjugate addition of dialkylzincs have been carried out [491]. Diarylcuprates added anti to 4-substituted α,β-unsaturated five-membered lactams [492]. A useful copper-assisted conjugate addition is shown in Eq. (170) [493]:



(170)

Nickel complexes catalyzed the 1,4-addition of Ph_3ZnLi to chiral α,β-unsaturated sulfoxides with good yield and high ee [494]. Chiral rhodium(I) complexes catalyzed the Michael addition of cyanoacetates to enones with 50–70% ee [495]. Cyclohexenones were synthesized utilizing conjugate addition of manganese carbene α-anions [496]:

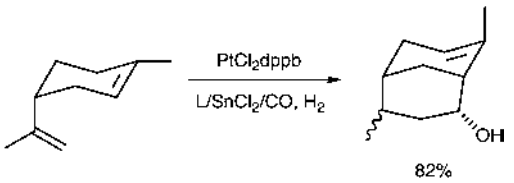


(171)

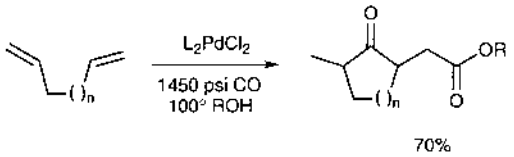
2.3. Acylation reactions excluding most hydroformylation

2.3.1. Carbonylation of alkenes and arenes

A review dealing with olefin carbonylation catalysis with cationic complexes (68 Refs.) [497] has appeared. The full scope and mechanism of rhodium-catalyzed silylformylation has appeared [498]. Studies on substrate-directed hydroformylation of alkenes have appeared [499,500]. Platinum [Eq. (172)] [501] and palladium [Eq. (173)] [502] catalyzed intramolecular carbonylation/olefin insertion processes:

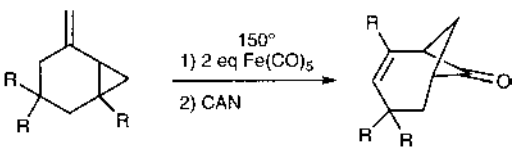


(172)

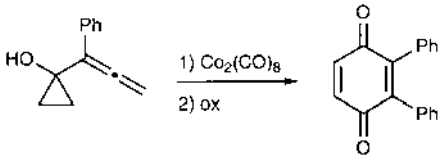


(173)

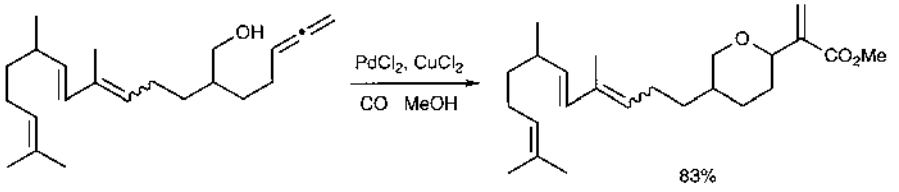
Iron carbonyl effected carbonylative rearrangements [Eq. (174)] [503]. Cobalt carbonyl converted cyclopropylallenes to quinones [Eq. (175)] [504]. Palladium catalyzed allene alkoxycarbonylation [Eq. (176)] [505] and allene cascade processes [Eq. (177)] [506]. Chiral rhodium complexes cyclocarbonylated 1,2,4-trienes [Eq. (178)] [507]. π-Allylrhodium complexes catalyzed living copolymerization of arylallenes with carbon monoxide [508]:



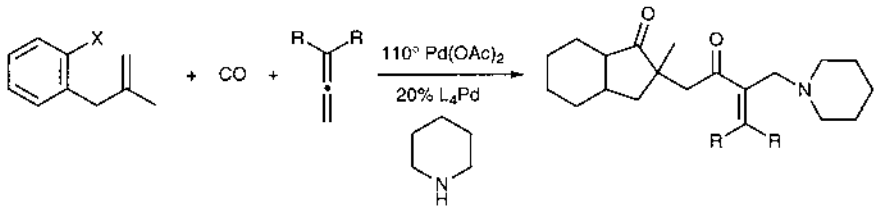
(174)



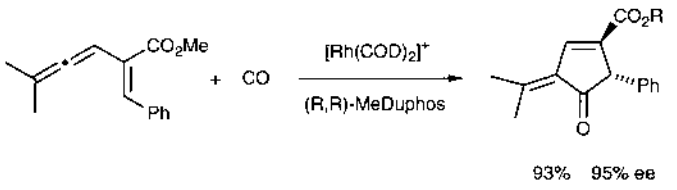
(175)



(176)



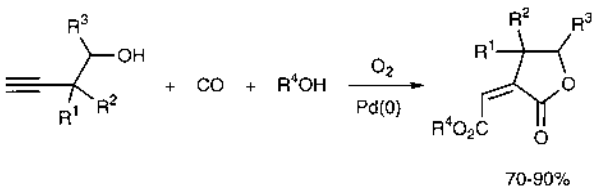
(177)



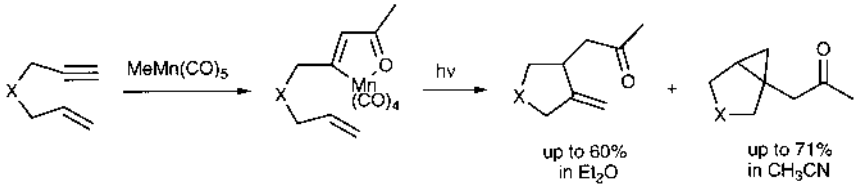
(178)

2.3.2. Carbonylation of alkynes (including the Pauson–Khand reaction)

Cyclobutenones were prepared by the reaction of alkynes with iron carbonyl under reducing conditions [509]. Zirconocene converted alkynes, ethyl Grignard reagent and carbon monoxide into cyclopentenones [510]. Propargyl and homopropargyl alcohols were converted to α-methylene lactones by palladium catalysts [Eq. (179)] [511]. Platinum(0) complexes catalyzed the conversion of terminal alkynes to α,β-unsaturated thio esters in the presence of carbon monoxide and thiophenol [512]. Enynes were cyclocarbonylated by manganese carbonyl [Eq. (180)] [513]:

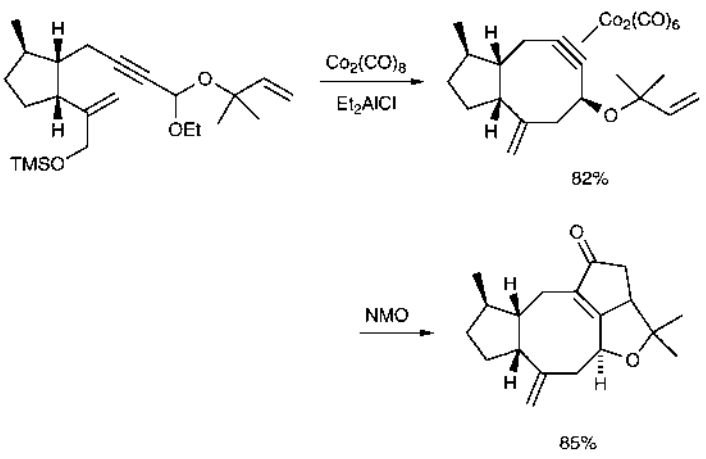


(179)

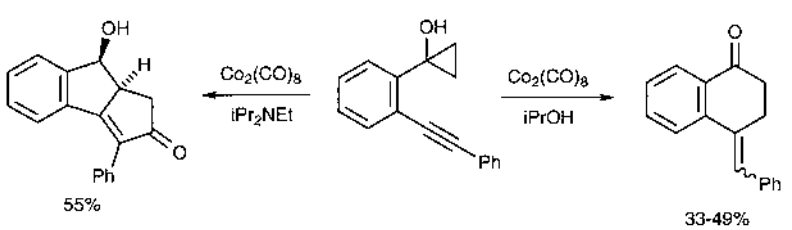


(180)

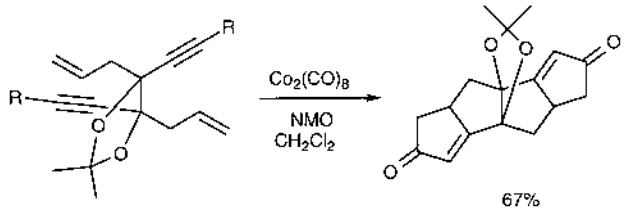
The Pauson–Khand reaction could be carried out catalytically [2% $Co_2(CO)_8$] in supercritical carbon dioxide [514]. Primary amines as well as ammonia under biphasic conditions enhanced the Pauson–Khand reaction [515], as did the use of polymer supports [516]. The $Co_2(CO)_6$ fragment was decomplexed from alkynes by treatment with TBAF in THF for 3 h at $-10°C$ [517]. Carrying out the Pauson–Khand reaction in the presence of trifluoroacetic acid led to the production of cyclopentanones rather than cyclopentenones [518]. Ligand (RS–) directed Pauson–Khand reactions have been developed [519,520]. The Pauson–Khand reaction between norbornadiene and propiolic amides of Oppolzer's chiral sultam proceeded in high yield and with greater than 800:1 de [521]. Synthetic applications of the Pauson–Khand reactions are seen in Eq. (181) [522], Eq. (182) [523], Eq. (183) [524], Eq. (184) [525], and Eq. (185) [526]:



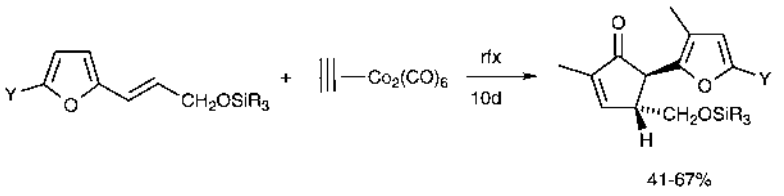
(181)



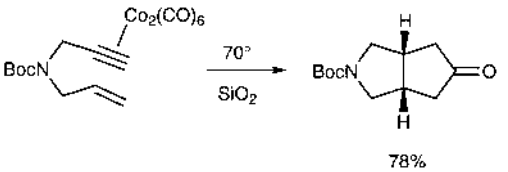
(182)



(183)

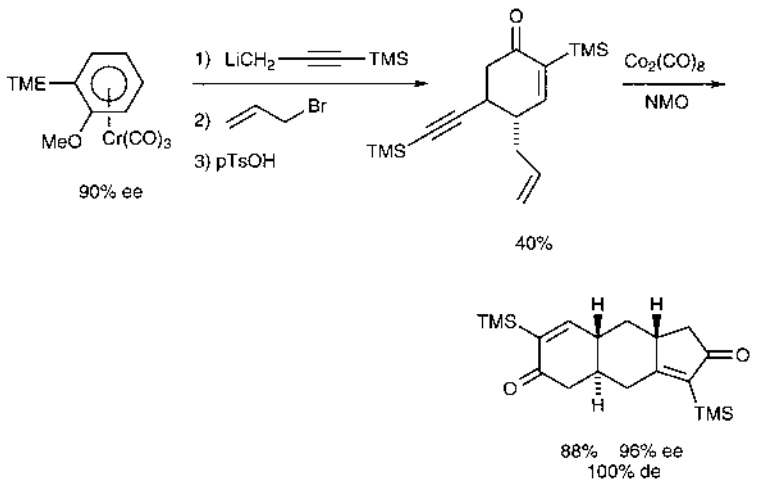


(184)

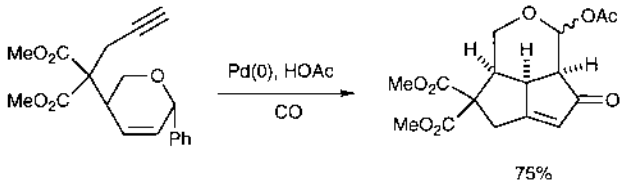


(185)

The Pauson–Khand reaction took place readily with alkynyl Fisher carbene complexes [527]. A nice synthetic application involving both chromium and cobalt chemistry is shown in Eq. (186) [528]. The complex $Ru_3(CO)_{12}$ promoted intramolecular Pauson–Khand reactions [529,530], as did palladium(0) complexes [Eq. (187)] [531]:



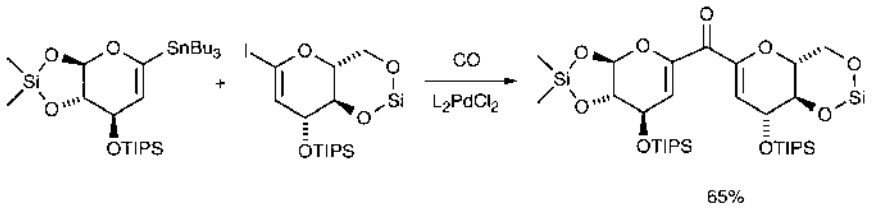
(186)



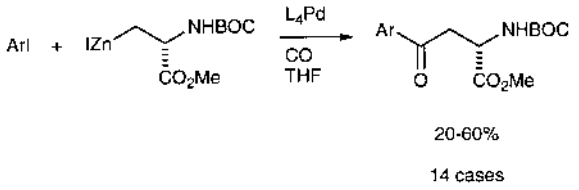
(187)

2.3.3. Carbonylations of halides and triflates

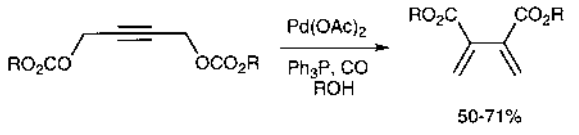
Palladium(0) complexes catalyzed the carbonylative coupling of aryl iodides with terminal alkynes to give alkynyl ketones [532], the carbonylation of allyl phosphonates to give β,γ-unsaturated esters [533], the carbonylative coupling of halides and triflates with stannanes to give ketones [534] [Eq. (188)] [535], and the carbonylative coupling of aryl iodides with functionalized organozinc halides [Eq. (189)] [536]. Palladium(0) also catalyzed the carbonylation of vinyl triflates [537,538], and the carbonylation of propargyl mesylates to give allenic esters [539] or diene diesters [Eq. (190)] [540]:



(188)



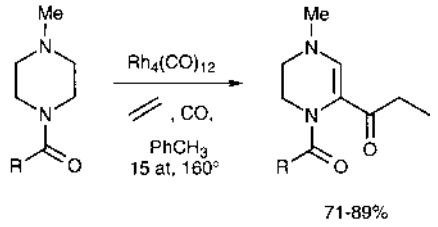
(189)



(190)

2.3.4. Carbonylation of nitrogen compounds

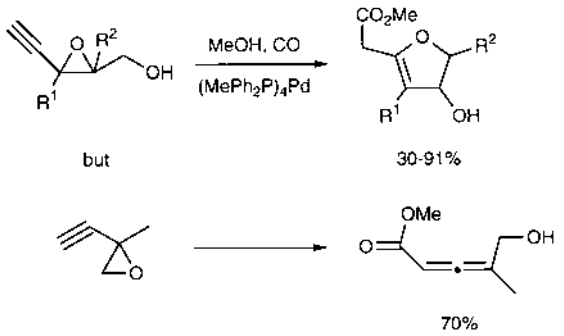
Nitroarenes were reductively carbonylated to N-phenyl carbamates using palladium(0) catalysts [541]. An unusual carbonylation is seen in Eq. (191) [542]:



(191)

2.3.5. Carbonylation of oxygen compounds

Allylic alcohols were converted to β,γ-unsaturated esters by reaction with phenols, and carbon monoxide in the presence of palladium(0) catalysts [543]. Propargyl epoxides were carbonylated under similar conditions [544]:



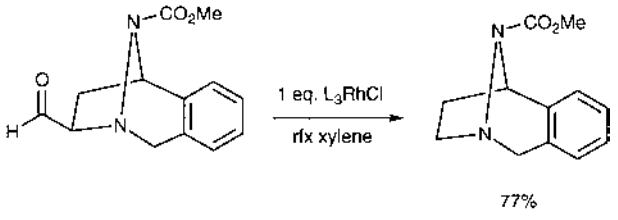
(192)

2.3.6. Miscellaneous carbonylations

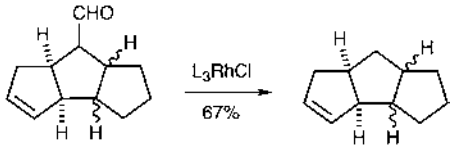
Higher order cyanocuprates underwent carbonylative coupling to give α-hydroxyketones [545]. Allylstannanes underwent reaction with carbon dioxide in the presence of palladium(0) complexes to produce allylcarboxylates [546].

2.3.7. Decarbonylations

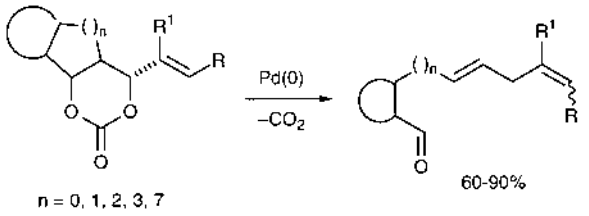
Useful decarbonylations are seen in Eq. (193) [547], Eq. (194) [548], and Eq. (195) [549]:



(193)



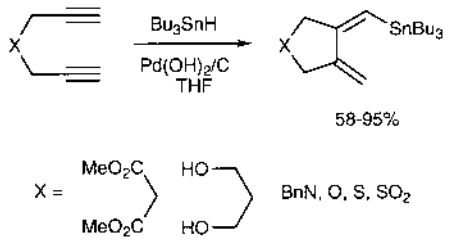
(194)



(195)

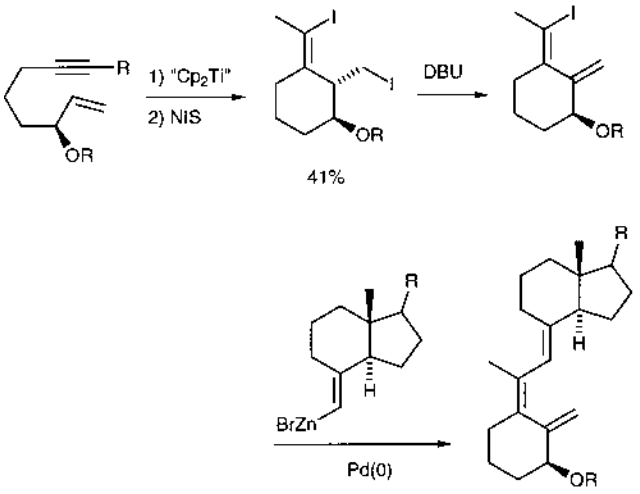
2.4. Oligomerization (including cyclotrimerization and metathesis polymerization)

Bridging rhodium(I) carbene complexes catalyzed the tail-to-tail dimerization of ethyl acrylate [550]. Diethyl zirconocene converted bis-alkynyl silanes into 1,3-dienes by dimerization [551]. Palladacyclic bis-imine complexes catalyzed the alkylative linear dimerization of dimethylacetylene dicarboxylate [552]. Imines were reductively dimerized to 1,2-diamines to (^{i}PrO)$_2$Ti(propene) [553]. Diynes were cyclodimerized by tin hydrides in the presence of palladium hydroxide on carbon [554]:

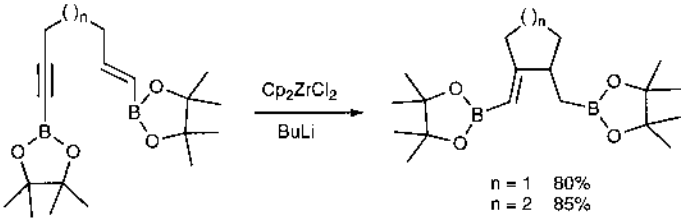


(196)

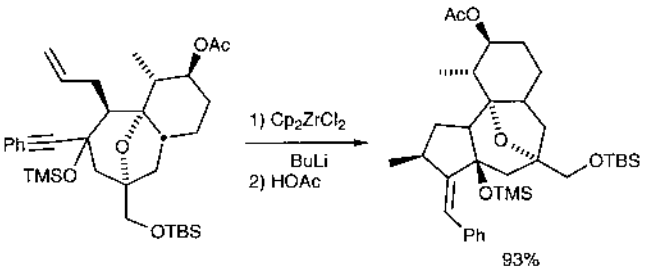
Zirconium Ziegler Natta catalysts cyclodimerized 1,5-dienes to methylenecyclopentanes and 1,6-dienes to methylenecyclohexanes [555]. The complex (^{i}PrO)$_2$Ti(propene) cyclodimerized yneallenes to 1-methylene-2-vinylcyclopentenes [556], and dienes or enynes having heteroatoms such as nitrogen, oxygen and silicon in the backbone to titanacyclopentanes or -pentenes which were further elaborated [557]. This reductive dimerization proved useful in synthesis, Eq. (197) [558], Eq. (198) [559], and Eq. (199) [560]:



(197)



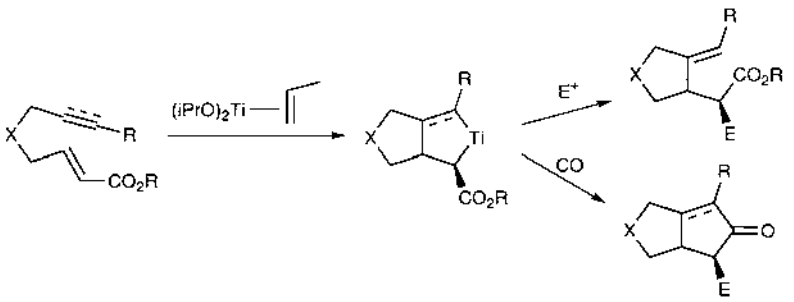
(198)



(199)

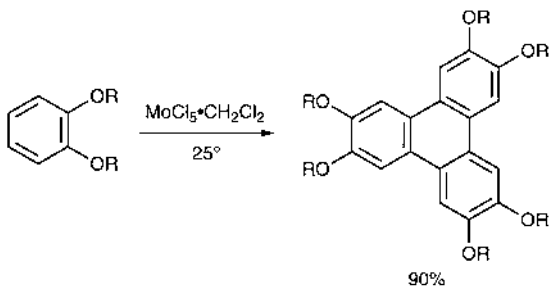
Alkynes underwent reaction with Cp_2Zr(ethylene) to give zirconacyclopentenes, which underwent further reactions with electrophiles [561]. Cp_2ZrBu_2 cyclized 1,6-heptadienes to give zirconacyclopentanes which coupled to propargyl chlorides [562]. The same complex cyclized enynes to zirconacyclopentenes which were subsequently cleaved by electrophiles [563]. A related example is seen in Eq. (200)

[564]. Iron pentacarbonyl cyclocarbonylated 1,8-diynes to produce iron-complexed cyclopentadienones [565]:

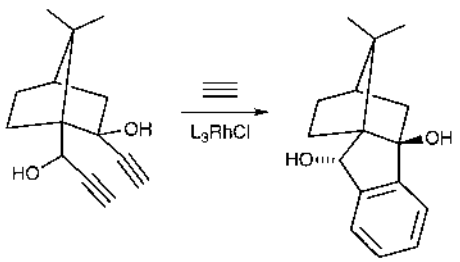


(200)

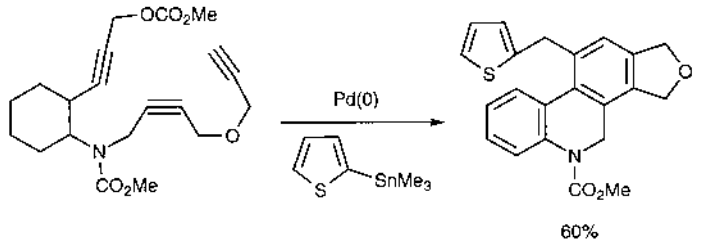
Molybdenum(V) chloride cyclotrimerized electron-rich arenes [Eq. (201)] [566]. Rhodium(I) complexes reacted with diynes to give rhodacyclopentadienes, which upon treatment with a third alkyne, gave arenes by a cocyclotrimerization [567,568], Eq. (202) [569,570]. Tetrahydroisoquinolines were prepared by the nickel(0)-catalyzed cocyclotrimerization of 4-aza-1,7-octadiynes with alkynes [571]. Palladium(0) catalyzed the cyclotrimerization of triynes [Eq. (203)] [572], while the Grubb's ruthenium metathesis catalyst cyclized triynes to arenes by a metathesis route [573]:



(201)

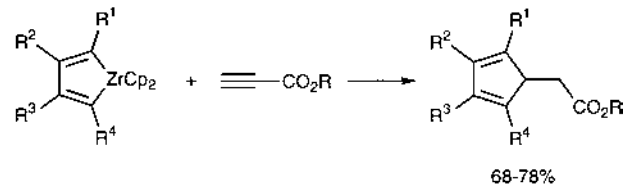


(202)

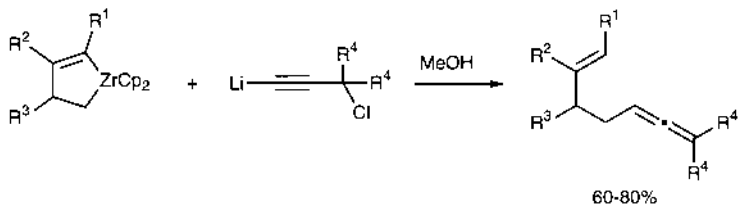


(203)

Dialkoxytitanacyclopentadienes catalyzed the cocyclotrimerization of alkynes with alkenes to give cyclohexadienes [574], and the cyclotrimerization of t-butylethyne to give 1,3,5-tri-t-butylbenzene [575]. Zirconacyclopentadienes (from alkyne dimerization) reacted with ethyl propiolate to give cyclopentadienes [Eq. (204)] [576] and with 1,4- or 1,2-dichlobutenes to give vinyl cyclohexadienes [577]. Zirconacyclopentenes coupled with lithiated propargyl chlorides to give ene allenes [Eq. (205)] [578]:

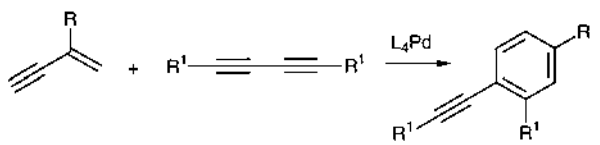


(204)



(205)

Palladium(0) complexes catalyzed the cyclotrimerization of 1,3-diynes [579] and the cocyclotrimerization of 1,3-diynes with enynes [580,581] to give substituted arenes [Eq. (206)]. Diphenylacetylene cocyclotrimerized with β-bromostyrene to give tetraphenylfulvenes [582]. Nickel(II) acac in the presence of trimethylaluminum cocyclotrimerized cyclohexenone with alkynes to give decalinones [583]:



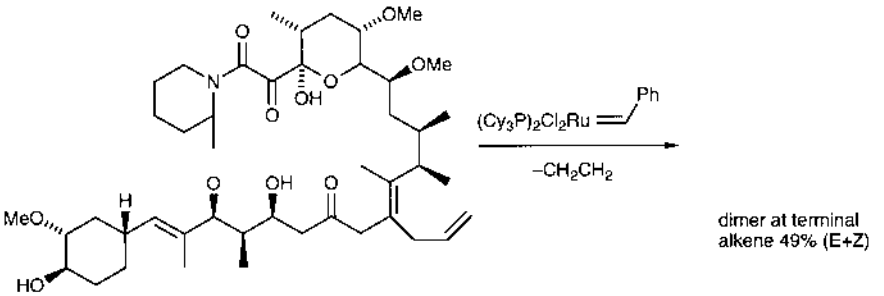
(206)

Nickel(II) salts oligomerized phenyl isocyanide to polyimines [584]. Rhodium(I) complexes [585] and arylmetal tricarbonyl complexes [586] catalyzed the polymerization of phenylacetylene to polymers of Mn 15000–120000. 1,3- and 1,4-bis propynylbenzenes bearing long-chain aliphatic side chains were metathesis oligomerized [587]. Allyl ethers were oligomerized by treatment with (methyl)phenylsilane and dicobalt octacarbonyl [588]. In the presence of ethylene glycol diallylether cross-linked networks were produced. Palladium(II) complexes catalyzed the alternating copolymerization of allenes with carbon monoxide [589], while π-allylnickel trifluoroacetate made living polymers from allenes [590]. Nickel(II) dppp complexes catalyzed the oligomerization of 2-bromo-3-*n*-hexyl-5-lithiothiophene [591], and 1,4′-dibromobipyridines connected through a silicon backbone [592]. Nickel(0) complexes catalyzed the cooligomerization of diynes with maleic anhydride [593].

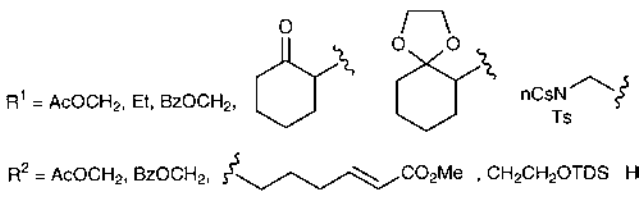
2.5. Rearrangements

2.5.1. Metathesis

Olefin metathesis in organic chemistry has been reviewed (213 references) [594]. New syntheses of the Grubb's $Ru{=}CH{=}CPh_2$ catalyst [595] and the $Ru{=}CPh_2$ catalyst [596] have been developed. The mechanism and activity of these catalysts have been studied [597]. Imines were metathesized by a molybdenum bis-nitrene complex [598]. Schrock's Mo metathesis catalyst was used to metathesize trimethyl allylstannane with alkenes to make homologated allylstannanes [599]. Glycine having an α terminal butenyl group was metathesized with terminal alkenes to increase the side chain length [600]. Metathesis was used to dimerize a macrocycle [Eq. (207)] [601]. Functionalized alkynes were cross-metathesized with ethene to give butadienes [Eq. (208)] [602]:

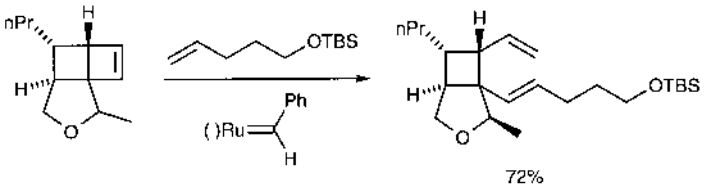


(207)

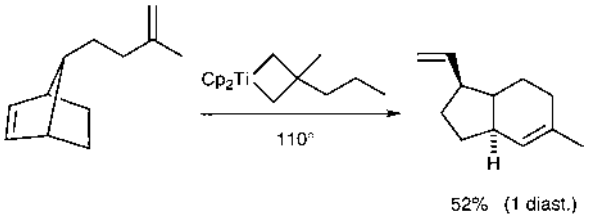


(208)

The mechanism and selectivity of ring-opening metathesis using Grubb's catalysts have been studied [603] and an intermediate ruthenium carbene–olefin complex has been trapped [604]. The process has been used to functionalize cyclobutenes [Eq. (209)] [605], to ring-open cross-metathesize polymer-bound norbornenes with styrenes [606], and to ring-open metathesize a number of [2.2.1] bicyclic olefins [607]. Titanacyclobutanes ring-open–ring-closed metathesized bridged bicyclic alkenes [Eq. (210)] [608]:

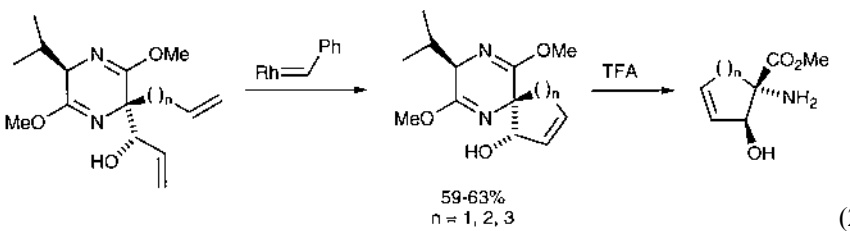


(209)



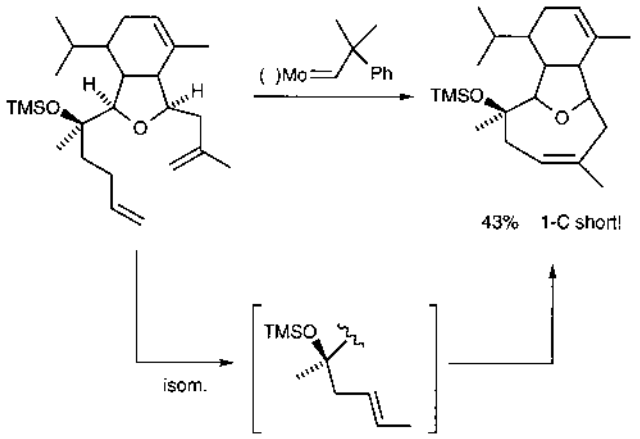
(210)

The effect of olefin substitution on ring-closing diene metathesis for the formation of five-, six-, and seven-membered rings has been studied [609]. The Grubb's ruthenium catalyst was best for systems with functionalized allyl groups, while the Schrock Mo catalyst was best for sterically-hindered or electron-poor alkenes. Bicyclic β-lactams were made by ring-closing metathesis of *N*-propargyl-4-allyl- and *N*-allyl-4-propargyl-β-azetidinones (six- and seven-membered rings) [610,611]. 2-Alkenyl-*N*- (unsaturated acyl group) pyrrolidinones were ring-closing metathesized to make 5,5- and 5,6-bicyclic lactams [612], while *N*-pentenyl-5-propargyl pyrrolidinones metathesized to give bicyclic aminodienes [613]. *N*,2-allyl cyclic ureas ring-closed to give six-membered fused ureas [614], and polymer-bound. *N*,*N*-bis-alkenylamines and amides ring-closing metathesized to tetrahydro pyridines, pyridones, or seven-membered lactams bearing an α-ester group [615]. Geminally disubstituted bis-lactim ethers metathesized to give optically active amines after hydrolysis [616–618]:

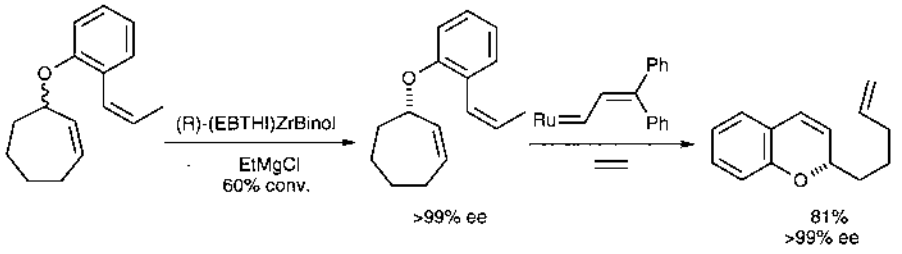


(211)

Schrock's metathesis catalysis was used to ring-close diallyl disulfides, diallylethers, -amines, and -thioethers [619] and to ring-close allyl-*o*-homoallylsiloxanes [620,621]. Dihydropyrans having a 2-furyl group were synthesized by ring-closing metathesis [622]. Polymer-bound unsaturated cinnamyl ethers were metathesized with cleavage from the polymer in the olefin metathesis step to give dihydropyrans [623]. Ring-closing metathesis was used to make six-, seven-, eight- and nine-membered cyclic ethers having chiral ethoxy substituents [624], eight- and nine-membered cyclic ethers fused to chiral 1,3-dioxanes [625,626] and dihydropyrans fused to sugars [627]. Interesting applications are seen in Eq. (212) [628] and Eq. (213) [629]:

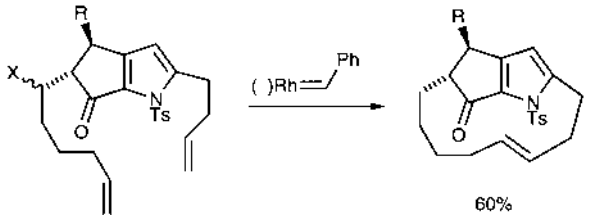


(212)



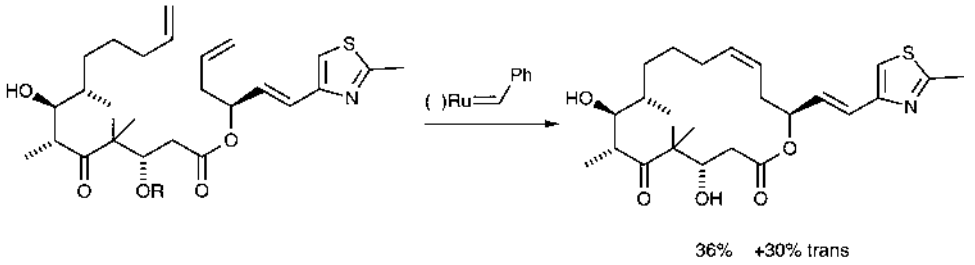
(213)

Having a tributylstannyl group α to the oxygen of bis-alkenylethers gave a conformational bias to favor ring-closing metathesis to form six-, seven- and eight-membered cyclic ethers [630]. High dilution with the Grubb's metathesis catalyst was used to synthesize the first ten-membered ring by ring-closing metathesis [631]. Medium-ring and macrocyclic ethers were made by ring-closing metathesis [632]. More functionalized medium rings could be made in this way [633]:



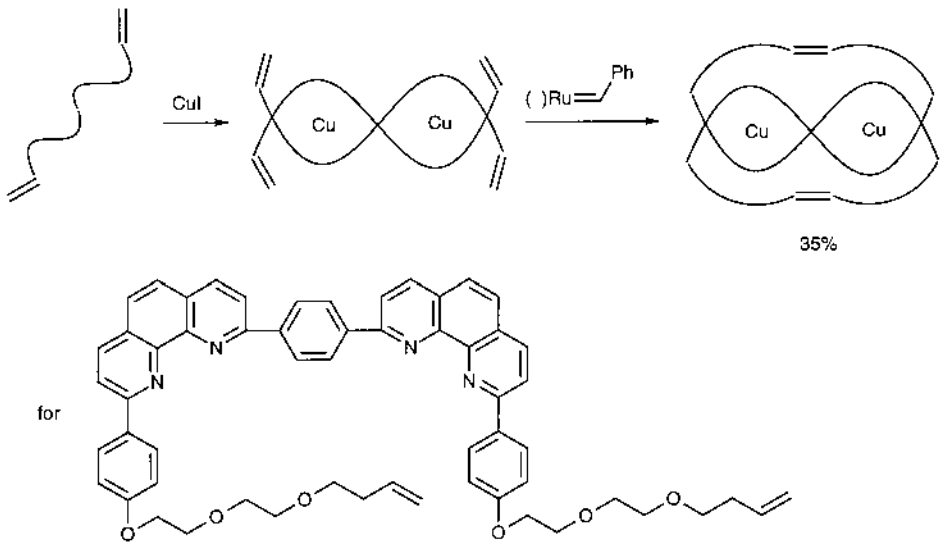
(214)

Macrocyclic esters [634,635], amides [636] and polyethyleneglycol cyclic ethers [637,638] were made by ring-closing metathesis. In the former case, lithium perchlorate complexation was used to pre-organize the alkene ends for metathesis [637]. A macrocyclic polyamide was made by ring-closing metathesis on a polystyrene support [639]. Ring-closing metathesis was used by almost everybody to synthesize epothilones [Eq. (215)] [640–646]. Using polymer suports, a combinatorial library of 60 epothilones was synthesized [640]:



(215)

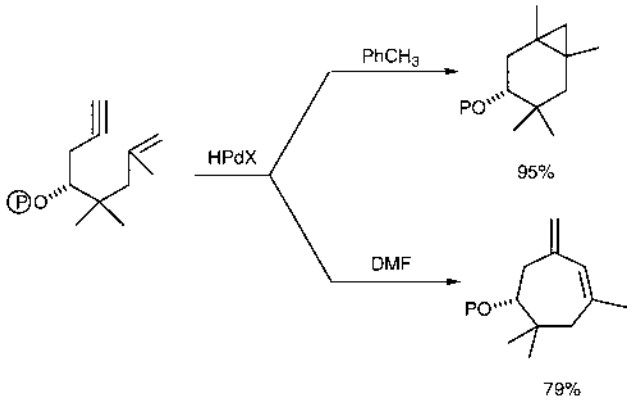
Ring-closing metathesis was used to make catenanes [647] [Eq. (216)] [648]:



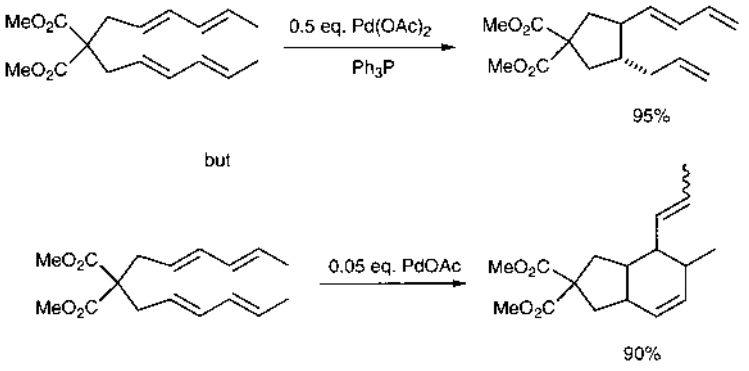
(216)

2.5.2. Olefin isomerization including cycloisomerization

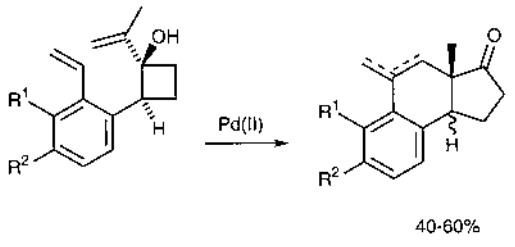
1-Ene-6-heptynes were cyclized to methylenecyclopentanes by palladium(0) catalysts and alkylstannanes [649]. The course of palladium-catalyzed cycloisomerization was dependent upon the solvent [Eq. (217)] [650]. Useful cycloisomerizations are seen in Eq. (218) [651], Eq. (219) [652], and Eq. (220) [653]:



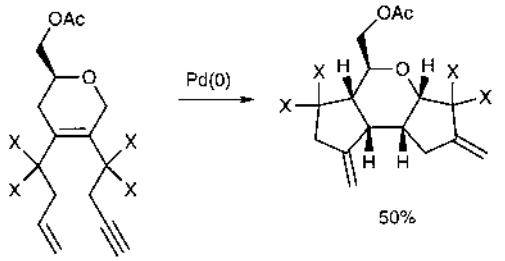
(217)



(218)



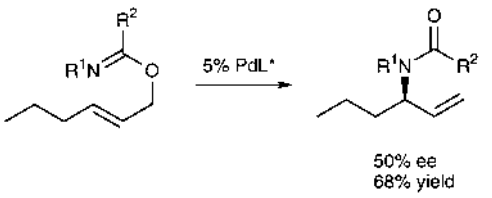
(219)



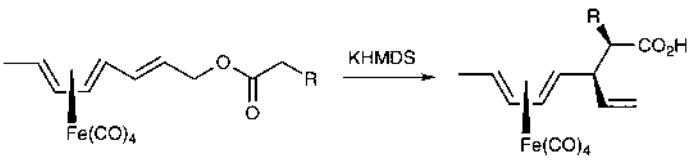
(220)

2.5.3. Rearrangements of allylic and propargylic compounds

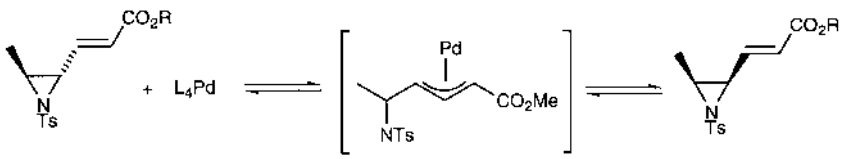
π-Allylruthenium phosphine complexes catalyzed the rearrangement of propargyl alcohols to α,β-unsaturated aldehydes (propargylic transposition) [654]. The complex $ReO_3(OSiR_3)$ catalyzed the allylic transposition of allyl alcohols [655]. Palladium(II) complexes catalyzed the rearrangement of *o*-allylthiocarbamates to *s*-allylthiocarbamates [656] and *o*-allyl imidates to allyl amides [Eq. (221)] [657,658]. Base catalyzed the allylic transposition of allyl esters on iron diene complexes [Eq. (222)] [659]. The palladium-catalyzed rearrangement of allyl aziridines was studied in detail [Eq. (223)] [660,661]. Both palladium(0) and nickel(0) complexes catalyzed allyl group scrambling of allyl malonates, via π-allyl complexes produced by the *reverse* of metal-catalyzed allylic alkylation. This process could lead to racemization in asymmetric allylic alkylation [662]. An interesting propargyl rearrangement is seen in Eq. (224) [663]:



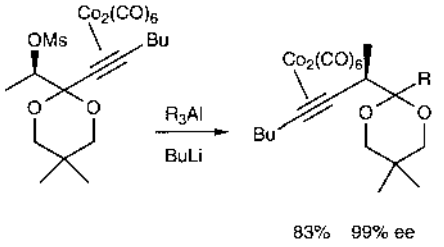
(221)



(222)



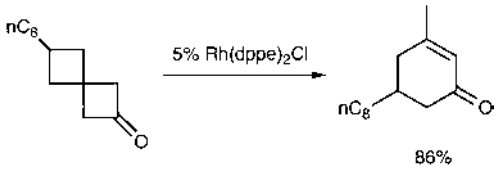
(223)



83% 99% ee (224)

2.5.4. Skeletal rearrangements

Palladium acetate catalyzed the rearrangement of styrene epoxides to benzyl ketones [664]. Rhodium(I) complexes catalyzed the rearrangement of strain spirocyclic ketones [665]:



86% (225)

3. Functional group preparation

3.1. Halides

Hydrazones were converted to geminal dibromides by copper(I) bromide/lithium-*t*-butoxide [666]. Palladium(II) catalyzed the 1,2-dibromination of terminal allenes [667]. Internal alkenes were dichlorinated and epoxides were converted to chlorohydrins by permanganate/TMSCl [668]. Nickel(II) acac catalyzed the arylzincation of alkynes. Cleavage with I_2 gave the alkenyl iodide [669].

3.2. Amides, nitriles, azides, imines

Palladium complexes catalyzed the reaction of primary amines with isonitriles to give carbodiimides [670]. Copper catalyzed the N-alkylation of mesyl imines by Grignard reagents [671].

3.3. Amines, alcohols

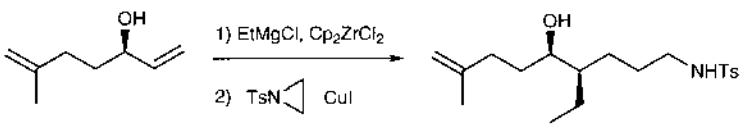
A paper dealing with Pd-catalyzed removal of allyl protecting groups from alcohols has appeared [672]. Palladium(0)/sulfinic acid systems were used to remove alloc protecting groups from alcohols, acids, carbamates, and amines [673]. N-ter-

minal alloc protecting groups were removed by $Pd(OAc)_2$/TPPTS/Et_2NH/aq. MeCN at 25°C [674]. Automated palladium(0) catalyzed allyl protecting group cleaving in solid phase cyclic peptide synthesis has been developed [675].

Palladium-catalyzed amination of aryl halides and triflates has become a growth industry and has been reviewed [676]. The secret seemed to be the use of bases such as t-butoxide [677] or cesium carbonate (for aryl triflates) [678] and chelating diphosphines. Aryl triflates were efficiently aminated by primary and secondary amines [679–681], and chiral primary amines aminated aryl halides without racemization [682]. Acetophenone imine could act as an NH_3 surrogate in the palladium-catalyzed amination of aryl halides [683] and new catalyst systems for various aryl substrates have been developed [684]. Poly(anilines) [685], including dendrimers [686] and poly(imino-1,3-phenylenes) [687], have been made this way. Nickel(0) [688] as well as various palladium(0) complexes [689–691] catalyzed the amination of aryl chlorides.

Salen manganese nitrides converted olefins into amino alcohols [692] and glycals into amino sugars [693]. Cationic Cp iron fluorobenzene complexes were aminated at the aryl fluoride by α-amino acids in low yield [694]. Cationic $Mn(CO)_3$ chlorobenzene complexes were aminated by *C*-2 symmetric amines (Cl substitution) then ring alkylated with hard nucleophiles with good de [695].

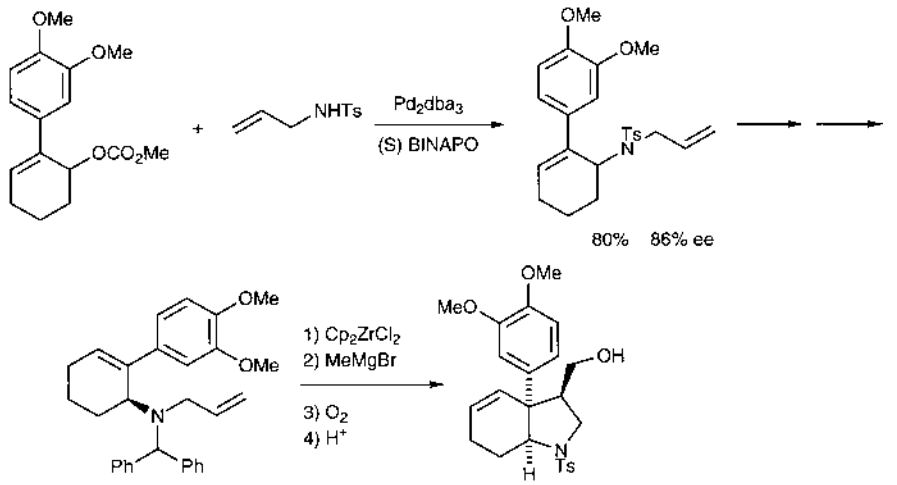
Imines were reduced to amines by butylmagnesium chloride in the presence of Cp_2TiCl_2 [696]. Palladium(0) complexes catalyzed the terminal amination of allenes to give allyl amines [697]. Palladium(I) catalyzed the rearrangement of o-allylimidates to allyl amines [698]. Ruthenium complexes catalyzed the addition of carboxylic acids to alkynes to give enol carboxylates which underwent reaction with amines to give enamines [699]. Ruthenium TMP catalyzed the insertion of diazoesters into amine nH groups to give α-amino esters [700]. An interesting synthesis of an amine is seen in Eq. (226) [701]:



42% (226)

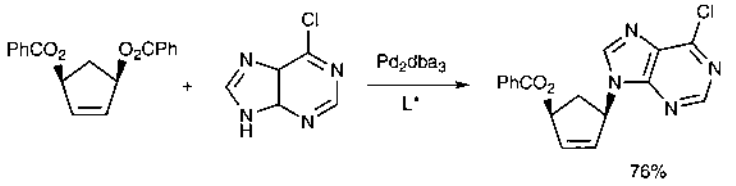
Palladium(0) catalyzed the amination of benzylic acetates on the naphthalene ring system [702] and the amination of allyl chloride by TMS_2N^- [703]. Palladium-catalyzed amination of allyl acetates followed by ozonolysis of the olefin provided a general approach to α-amino acids [704]. Benz-fused thioureas and thiocarbamates were N-allylated using palladium(0) catalysts [705].

Palladium-catalyzed asymmetric allylic amination continued to be studied in excruciating detail, usually on symmetric 1,3-disubstituted allyl systems which can already be aminated in $>99\%$ ee [706–708]. A nice synthetic application is seen in Eq. (227) [709]:

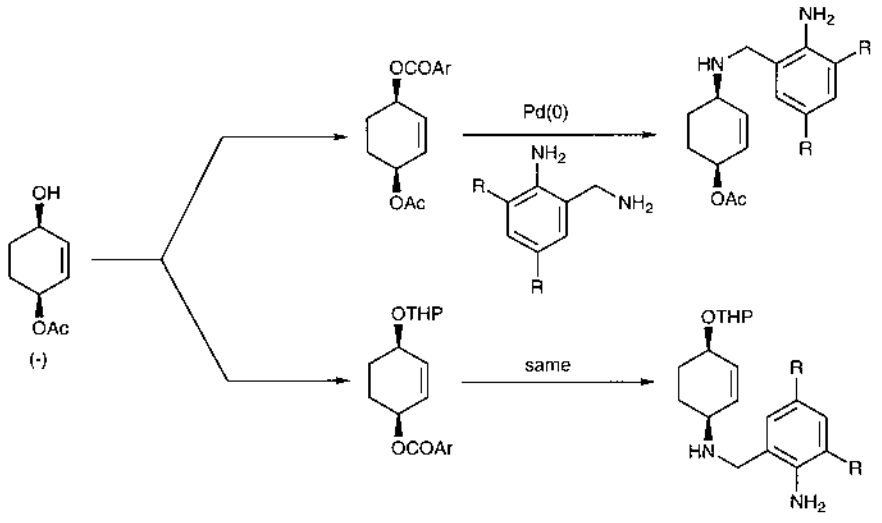


(227)

The use of palladium-catalyzed allylic amination in the synthesis of carbocyclic nucleoside analogs has been developed further [Eq. (228)] [710–713]. By use of appropriate sequences, cyclohex-2-ene-1,4-diol could be aminated at either allylic position [Eq. (229)] [714]:

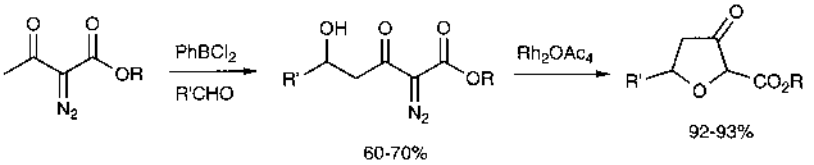


(228)

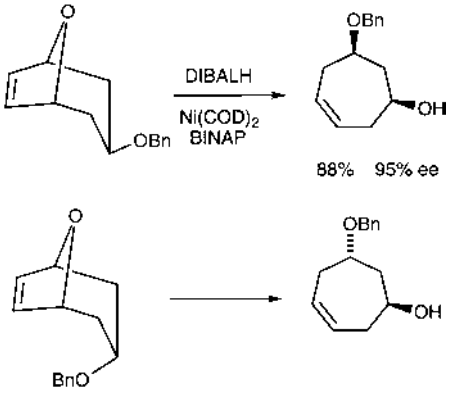


(229)

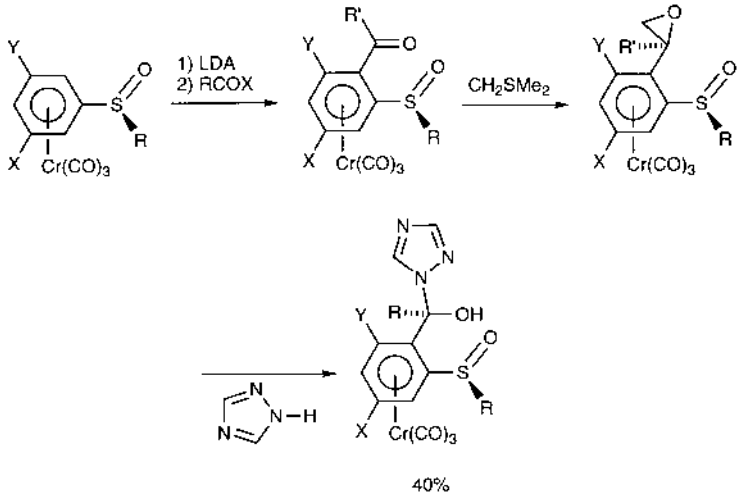
Lactones were reduced to lactols by silanes in the presence of Cp_2TiF_2 [715]. Diazoacetacetates alkylated aldehydes, then underwent OH insertion in the presence of rhodium(II) catalysts [Eq. (230)] [716]. Nickel(0) complexes catalyzed the reductive ring-opening of bicyclic ethers [717], [Eq. (231)] [718]. Unsaturated chromium carbene complexes were converted to 1,3-diols by treatment with $BH_3 \cdot SMe_2$ followed by oxidation [719]. Chromium-complexed styrene oxides were asymetrically ring-opened [Eq. (232)] [720]:



(230)



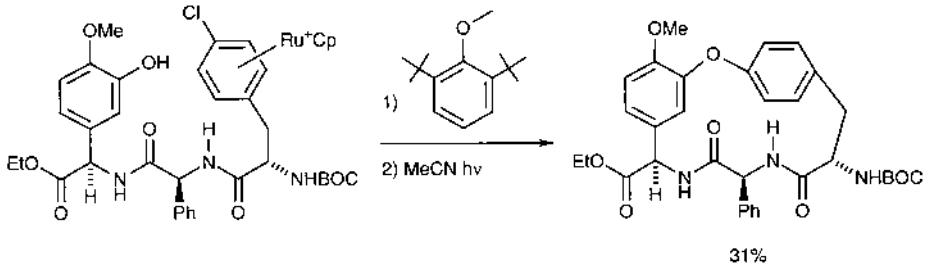
(231)



(232)

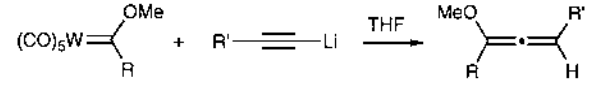
3.4. Ethers, esters, acids

Palladium(0) and nickel(0) complexes catalyzed the coupling of aryl halides with alkoxides to give ethers [721–723]. Direct observation of C–O reductive elimination to form ethers in this process has been made [724]. Palladium(0) catalyzed the β-alkoxylation of α,β-dibromoacrylates by tin alkoxides [725]. Diarylethers related to vancomycin were made by attack of phenoxides on cationic ruthenium chloroarene complexes [726] [Eq. (233)] [727]. Copper triflate catalyzed the cross-coupling of aryl halides with substituted phenols to give unsymmetrical diaryl ethers [728]:



(233)

Palladium acetate/titanium isopropoxide catalyzed the reaction of allyl alcohols with phenols to give aryl allyl ethers [729], while palladium(0) catalyzed the bis-alkoxylation of 2,3-dibromopropene by phenoxides [730]. Palladium(II) salts catalyzed the addition of alkoxides to allyl alcohols to give β-alkoxyketones via nucleophilic attack/β-elimination into the OH position to give enol ethers [731]. Rhodium(II) catalyzed the OH insertion of diazoesters into polyunsaturated fatty alcohols to give polyunsaturated poly ethers [732]. Other interesting ether-forming reactions are shown in Eq. (234) [733], Eq. (235) [734] and Eq. (236) [735]:



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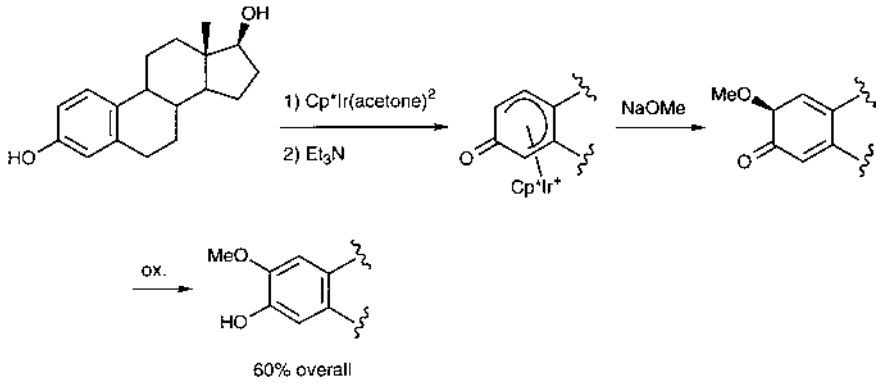
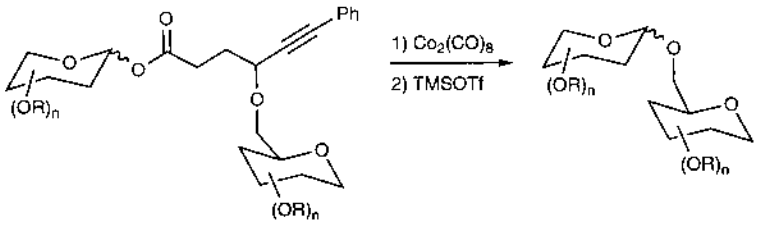
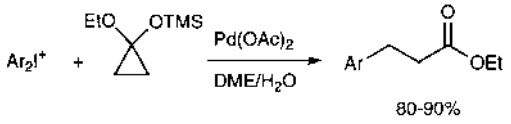


(235)



(236)

Chiral copper(I) bisoxazoline complexes catalyzed the allylic oxidation of cycloalkenes by peracids to give allyl esters with 80% ee [736]. Palladium(II) salts catalyzed the arylation of several substrates by diaryliodonium salts [Eq. (237)] [737]. Ruthenium complexes catalyzed the reaction of propargyl alcohols with carboxylic acids to give esters [Eq. (238)] [738]. Palladium(II)/copper(I) Wacker systems catalyzed the aminocarbonylation of alkenes [Eq. (239)] [739]. Palladium(0) complexes in the presence of morpholine deprotected allyl esters [Eq. (240)] [740] and catalyzed the reaction in Eq. (241) [741]:



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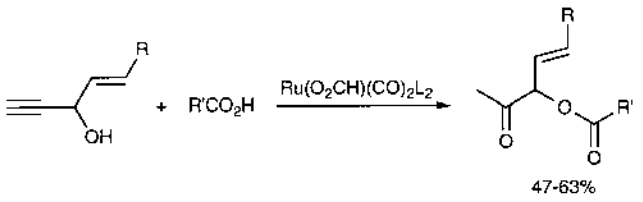

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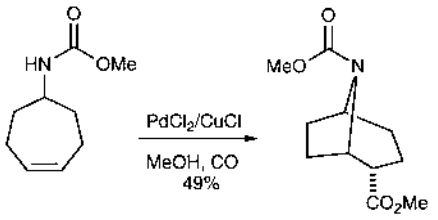

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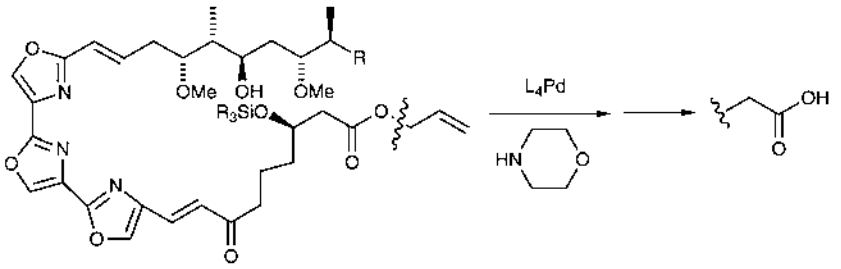
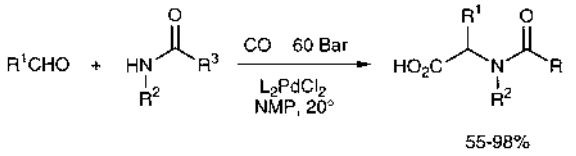


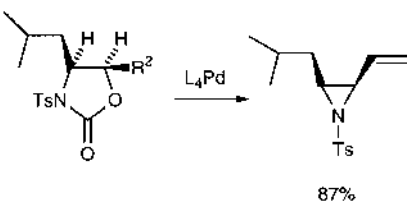
(240)



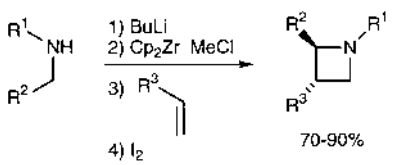
(241)

3.5. Heterocycles

Aziridines were made by the manganese-catalyzed decomposition of diazoesters with imines [742] and by the palladium-catalyzed decarboxylation of oxazolidinones [Eq. (242)] [743]. β-Lactams were made by rhodium-catalyzed C–H insertion processes [744] and azetidines were made by zirconium-assisted reaction of amines with alkenes [Eq. (243)] [745]:

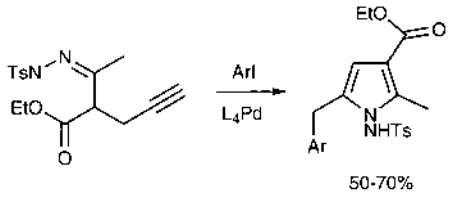


(242)

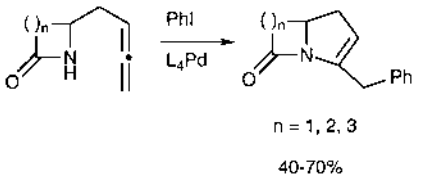


(243)

Five-membered nitrogen heterocycles were made as in Eq. (244) [746], Eq. (245) [747], Eq. (246) [748], Eq. (247) [749], Eq. (248) [750], and Eq. (249) [751]:



(244)



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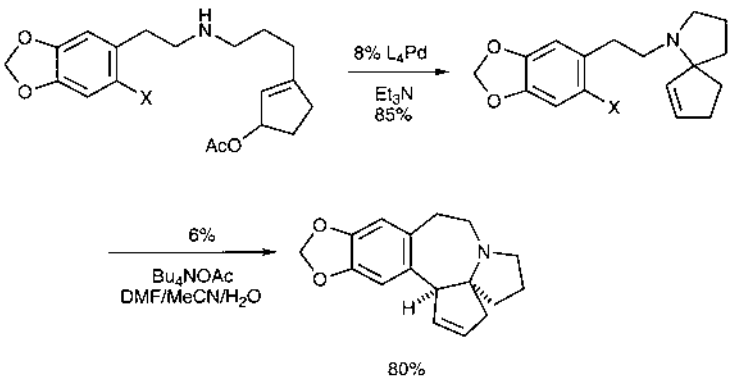
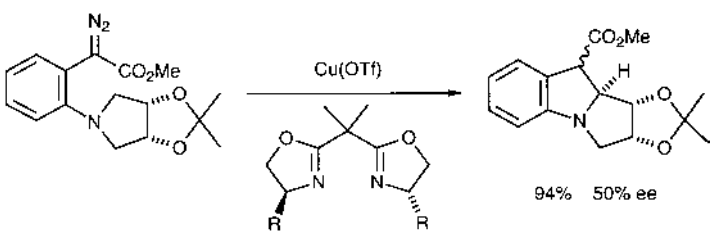
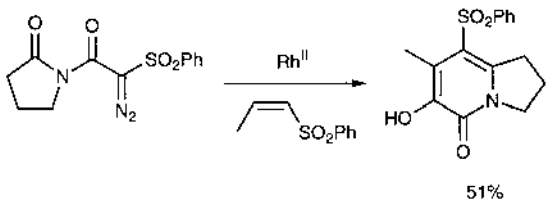


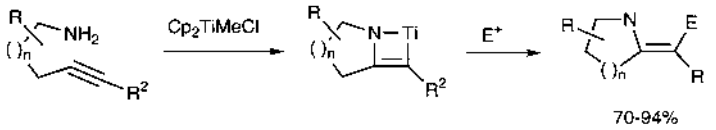
(246)



(247)



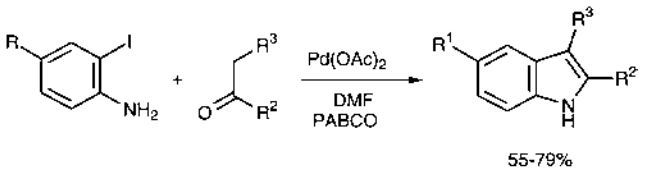
(248)



(249)

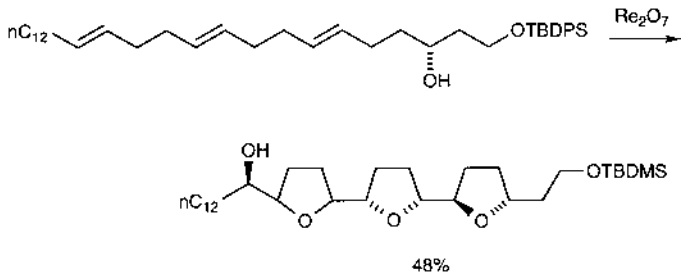
Addition of halide salts dramatically increased the ee of the palladium(0)-catalyzed cyclization of o-triflyl (but not bromo) N-acryloyl anilines to oxindoles, arguing that *neutral* organopalladium intermediates were required for high ee [752]. β-Cp iron acroleins cyclized to five-membered unsaturated lactams when treated with primary amines [753]. Titanium isopropoxide/i-propylmagnesium chloride converted alkynes, imines and carbon dioxide into unsaturated five-membered lactams [754].

Indoles were synthesized by the molybdenum carbonyl-catalyzed cyclization of 2-ethynylanilines [755], the palladium(0)-catalyzed reaction of alkynes with polymer-bound 2-iodoanilines [756], the palladium-copper-catalyzed reaction of 8-iodoquinoline with propargyl alcohol [757], the palladium-catalyzed reaction of polymer-bound 2-iodoaniline with alkynes, followed by cyclization [758], the palladium-catalyzed reaction of aryl iodides with 2-ethynyl anilines [759], and the palladium-catalyzed reaction between 2-iodoanilines and ketones [Eq. (250)] [760]. Intramolecular Heck cyclization of polymer-bound N-allyl-2-iodo-anilines was used in a combinatorial synthesis of indoles [761]. 2-Allyl anilines were cyclized to indoles in the presence of one equivalent of palladium(II) chloride [762], and 2-alkenyl anilines were cyclized to indoles by rhodium-catalyzed hydroformylation [763]. Carbazole natural products continued being made from the reaction of anilines with cyclohexadienyliron complexes [764]:

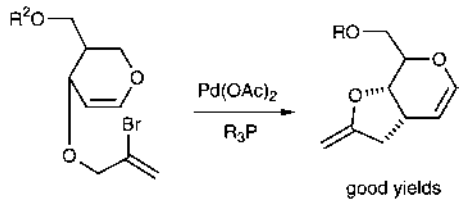


(250)

Tetrahydrofurans were made by rhenium oxidation of polyenes [Eq. (251)] [765], the molybdenum-catalyzed cyclization of allylphenols (aryl CH activation) [766,767], palladium(0) oxidative addition/olefin insertion chemistry [Eq. (252)] [768], the palladium-catalyzed reaction between allyl alcohols, aryl iodides and methylene malonates [769], the palladium-catalyzed cyclization of acetylacetonyl allyl carbonates [Eq. (253)] [770], and the palladium(II)-catalyzed cyclization of 2-allyl phenols in the presence of optically active ligands (high ee) [771]. They were also made by rhodium-catalyzed CH insertion reactions [Eq. (254)] [772], [Eq. (255)] [773], and by the thermal reaction of chromium carbene complexes [Eq. (256)] [774]:



(251)



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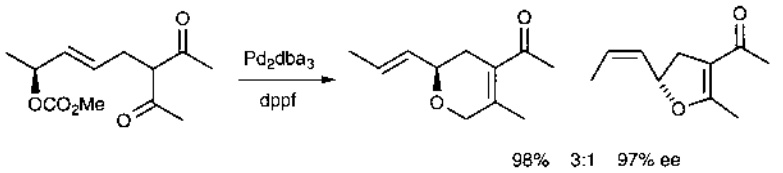
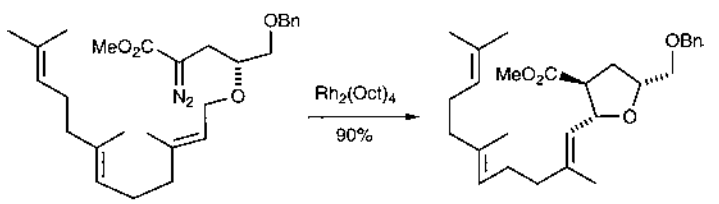
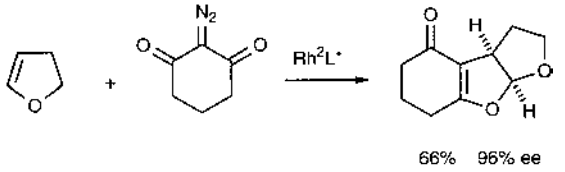


(253)



(254)



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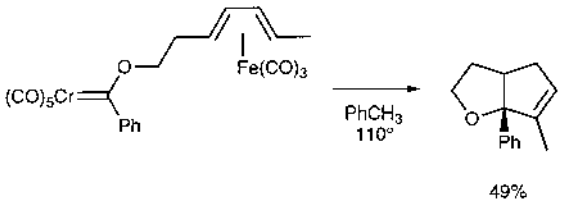
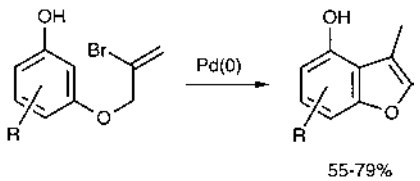
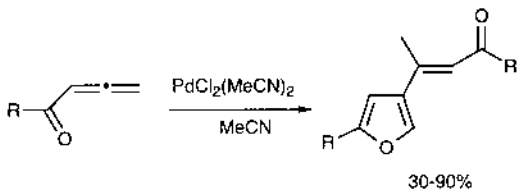


(256)

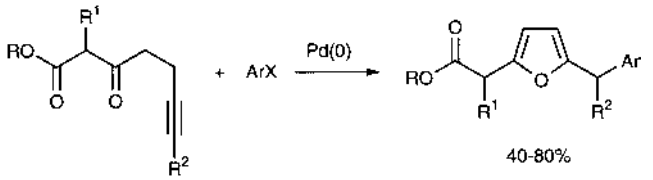
Benzofurans were synthesized by palladium(0)-catalyzed reaction of 2-iodophenols with alkynes in solution [775–778] and on solid supports [779], the palladium(0)-catalyzed cyclization of 3-hydroxy-O-2-bromopropenyl phenols [Eq. (257)] [780] and the palladium(II)-catalyzed dimerization of allenylketones [Eq. (258)] [781,782]. Palladium(0) complexes catalyzed the inter- [783] and the intramolecular reactions of β-dicarbonyl enols with unsaturates [Eq. (259)] [784]. The regiospecific synthesis of 3,4-disubstituted furans and thiophenes has been reviewed (35 Refs.) [785]. Furans were also made by rhodium-catalyzed diazo decomposition chemistry [Eq. (260)] [786], [Eq. (261)] [787,788]:



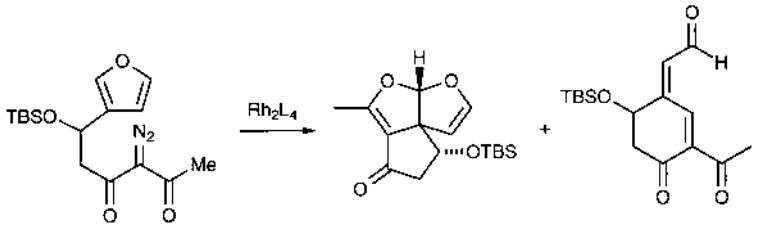
(257)



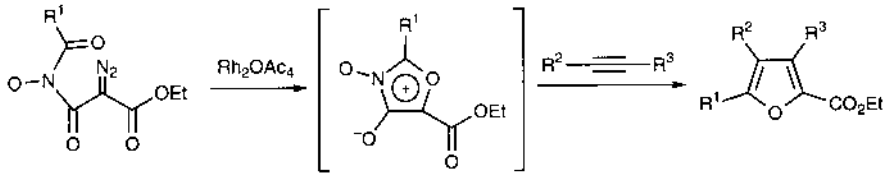
(258)



(259)

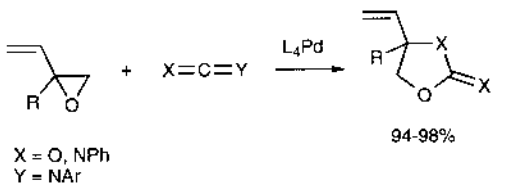


(260)

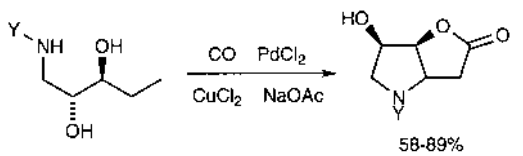


(261)

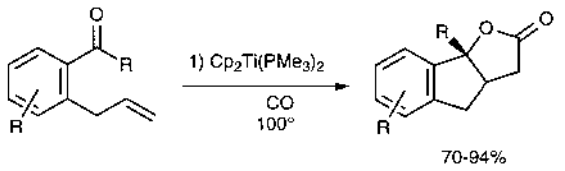
Butyrolactones were synthesized as in Eq. (262) [789], Eq. (263) [790], Eq. (264) [791], Eq. (265) [792], Eq. (266) [793], and Eq. (267) [794]:



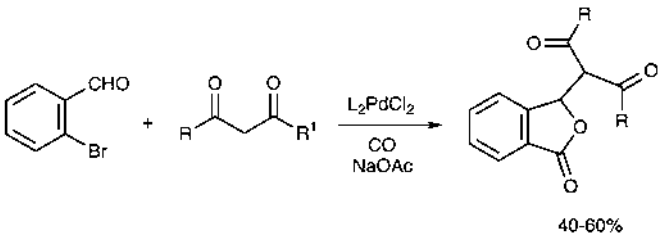
(262)



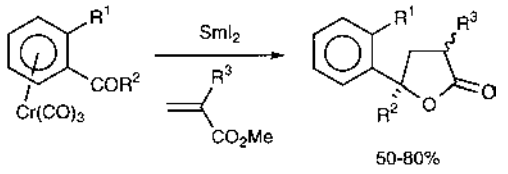
(263)



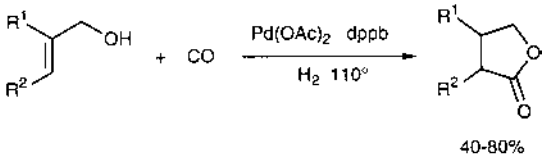
(264)



(265)



(266)

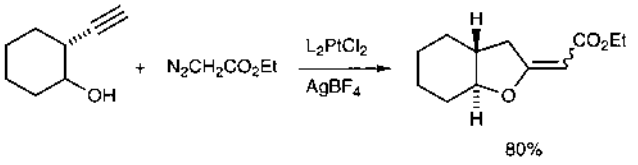


(267)

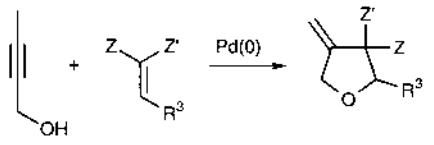
Regio- and stereoselective syntheses of γ-alkylidene butenolides and related compounds have been reviewed (111 references) [795]. Butenolides were prepared by the palladium(0)-catalyzed cyclocarbonylation of propargyl alcohols [796], [Eq. (268)] [797], the palladium(0)-catalyzed reaction of β-bromoacrylic acids with alkynes [798], and the palladium-catalyzed carbonylation of 2-iodo-4-hydroxy-1-alkenes [799]. Treatment of alkynes with Grignard reagents and iron pentacarbonyl produced butenolides [800], as did the reaction between alkynes and chromium (alkoxy)(amino)carbene complexes [801]:

(268)

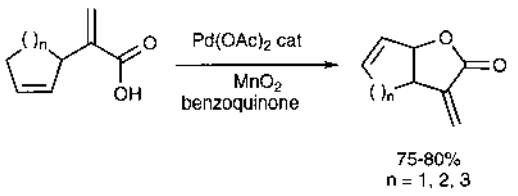
Preparation of α-methylene lactones by chloropalladation of *o*-allyl propiolates [Eq. (269)] [802] has been reviewed [803]. α-Methylene lactones [Eq. (270)] [804] and β-methylene tetrahydrofurans [805] were available from tungsten π-allyl chemistry. Other routes to five-membered oxygen heterocycles are shown in Eq. (271) [806], Eq. (272) [807], Eq. (273) [808], Eq. (274) [809], Eq. (275) [810], and Eq. (276) [811]:



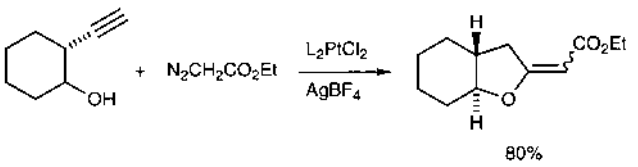
(269)



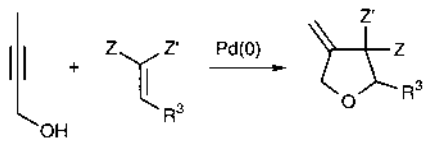
(270)



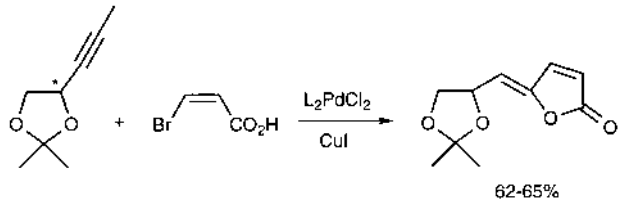
(271)



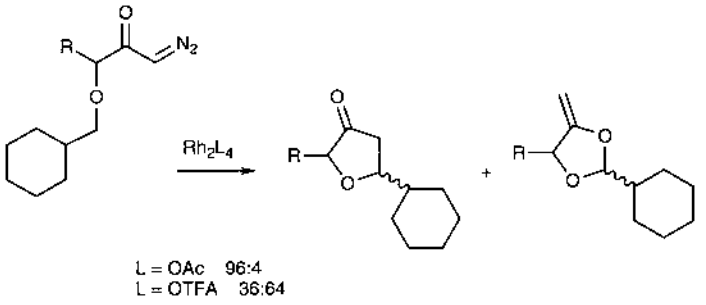
(272)



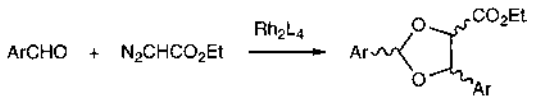
(273)



(274)

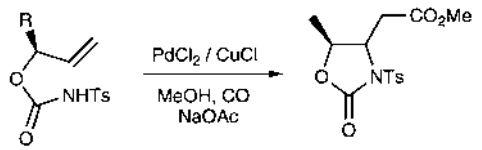


(275)

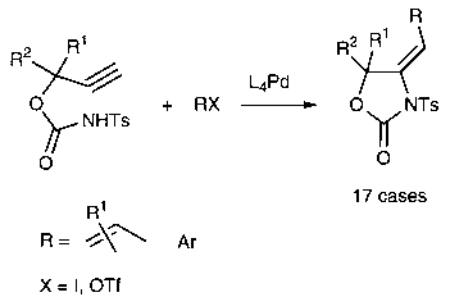


(276)

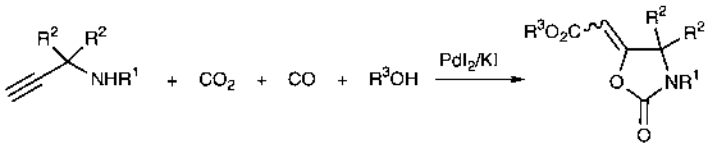
Oxazolidinones were made by the palladium(II)-catalyzed cyclocarbonylation of o-allylcarbamates [Eq. (277)] [812], the alkylative cyclization of o-propargyl carbamates [Eq. (278)] [813,814], and the intermolecular carboxylation of propargyl amines [Eq. (279)] [815]. Propargyl hydrazines cyclized to 3-arylpyrazoles when treated with aryl halides and palladium catalysts [816]. Propargyl N-hydroxyureas cyclized to N-acylamino isoxazoles in the presence of palladium(II) catalysts [817]:



(277)

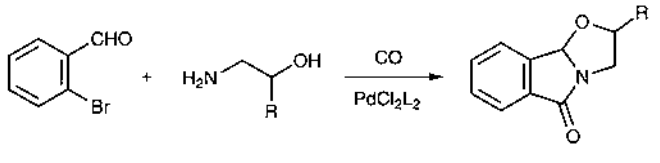


(278)

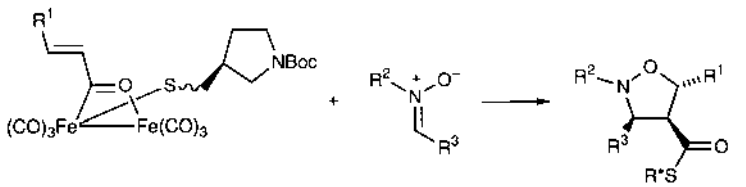


(279)

2-Bromobenzaldehyde and amino alcohols were carbonylatively coupled to give N-acylisoxazoles in the presence of palladium catalysts [Eq. (280)] [818]. Palladium(II) catalyzed the reaction between oximes and N-methyl maleimide (nitrone 1,3-dipolar cycloaddition) to give isoxazoles [819,820]. Chiral isoxazoles were available by nitrone cycloaddition to iron enone complexes [Eq. (280)] [821]. Oxindoles were synthesized by palladium(0)-catalyzed cyclization of polymer-bound 2-iodo-N-acryloylaniline [822]:

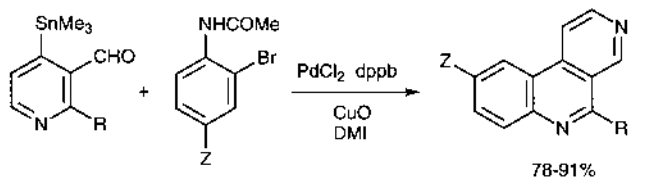


60-90% (280)

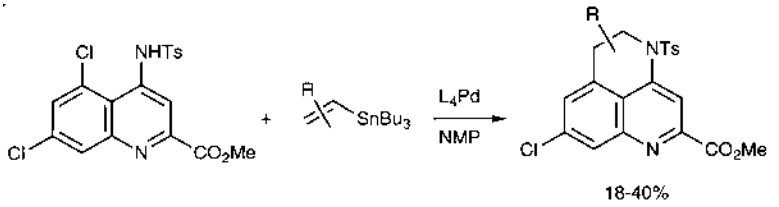


60-96% 10:1 to >25:1 (281)

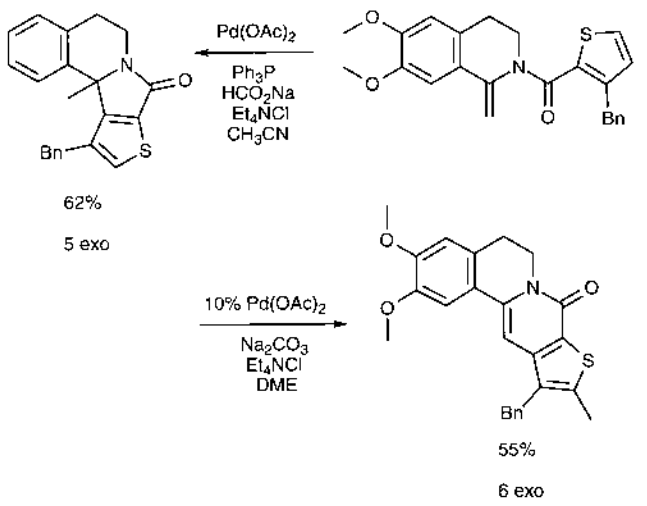
Palladium(0) complexes catalyzed the reaction of 2-iodo acetanilides with methyl acrylate to give dihydroisoquionolones [823], and the reaction of 2-iodoanilines with acrylates to give dihydroquinolines [824]. Palladium(II) complexes catalyzed the cyclization of allyl ethers [825] and allyl alcohols [826] having amino groups five carbons away from the allyl terminus to piperidines. Palladium(0) catalyzed the heterocyclizations in Eq. (282) [827], Eq. (283) [828], and Eq. (284) [829]:



78-91% (282)

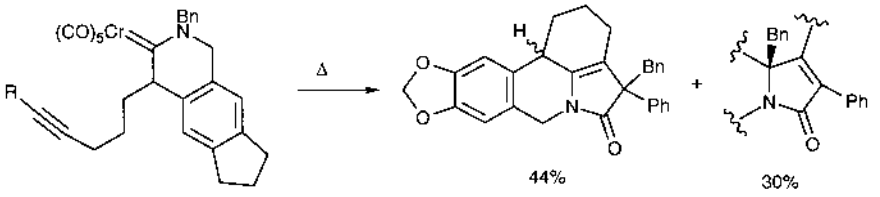


(283)

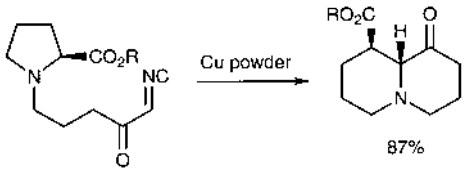


(284)

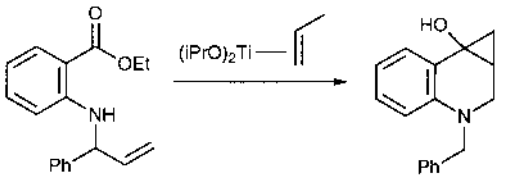
Polycyclic heterocycles were prepared by thermal reactions of chromium carbene complexes [Eq. (285)] [830]. Other heterocyclizations are seen in Eq. (286) [831], Eq. (287) [832], Eq. (288) [833], and Eq. (289) [834]:



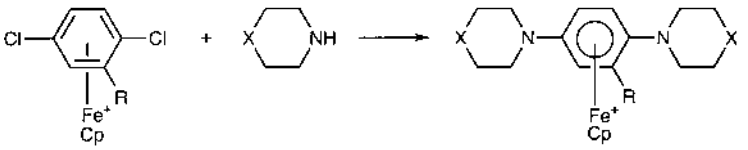
(285)



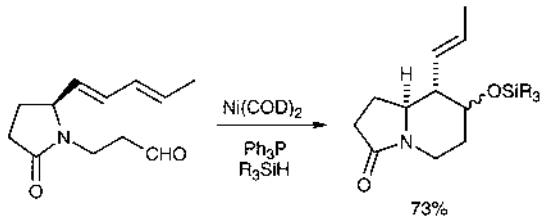
(286)



(287)

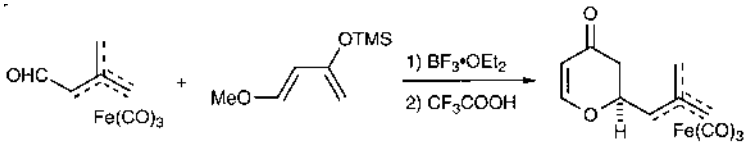


(288)

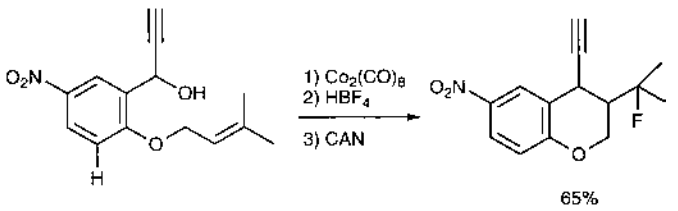


(289)

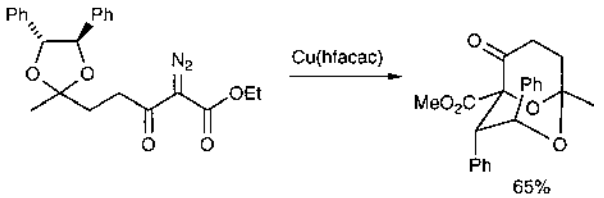
Palladium(0) catalyzed the reaction between norbornene and 2-iodo-phenol to give the γ-pyrone [835]. Palladium(0) catalyzed the cyclization of γ-hydroxy allylepoxides to pyranes (allylic alkoxylation) [836] and o-hydroxy allylic carbonates to benzopyrans [837]. Palladium(I) complexes catalyzed the cyclization of homopropargyl alcohols to dihydropyrans [838]. Iron formyltrimethylenemethane complexes formed dihydro-γ-pyrones by cycloaddition to Danishefsky's diene [Eq. (290)] [839]. Cobalt-stabilized propargyl cations figured in the formation of tetrahydropyrans [Eq. (291)] [840]. Dibenzopyrans were synthesized by palladium(II)-catalyzed cyclization of 2-hydroxy-2-ethenyl biphenyls [841]. Tungsten carbonyl cyclized 5-hydroxyalkynes to dihydropyrans [842]. Pyrans were synthesized by the cyclization of o-hydroxybenzaldehydes with the methylene group of chromium-complexed 2-methylene indolines [843]. Ruthenium(II) complexes catalyzed the addition of allyl alcohols to propargyl alcohols to give six-membered cyclic hemiketals [844]. Oxygen heterocycles were prepared by copper [Eq. (292)] [845] and rhodium-catalyzed diazodecomposition [846]:



(290)

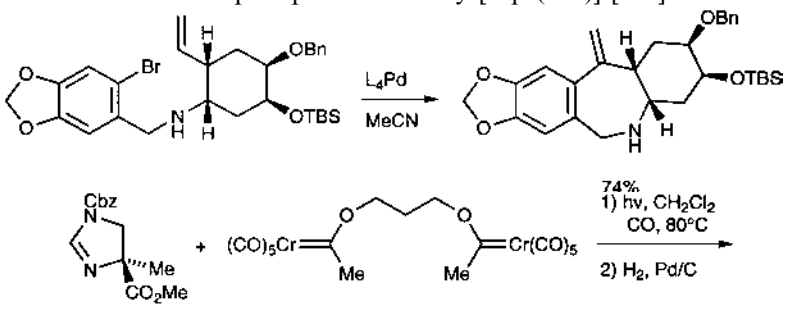


(291)

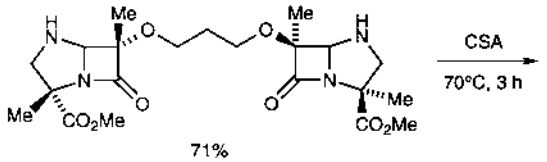


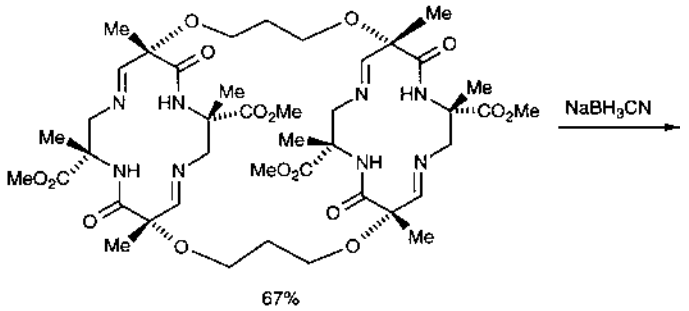
(292)

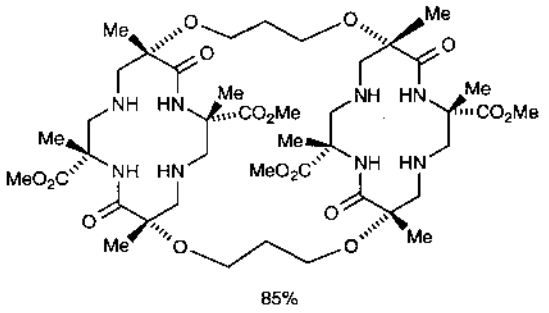
C–N bond formation via transition metal catalysts [palladium(II)-catalyzed olefin aminocarbonylation] has been reviewed (36 Refs.) [847]. Seven- [Eq. (293)] [848], eight-, and nine-membered nitrogen heterocycles [849] were made by palladium(0)-catalyzed oxidative addition/insertion chemistry. Bis-cyclams were made via chromium carbene complex photochemistry [Eq. (294)] [850]:



(293)

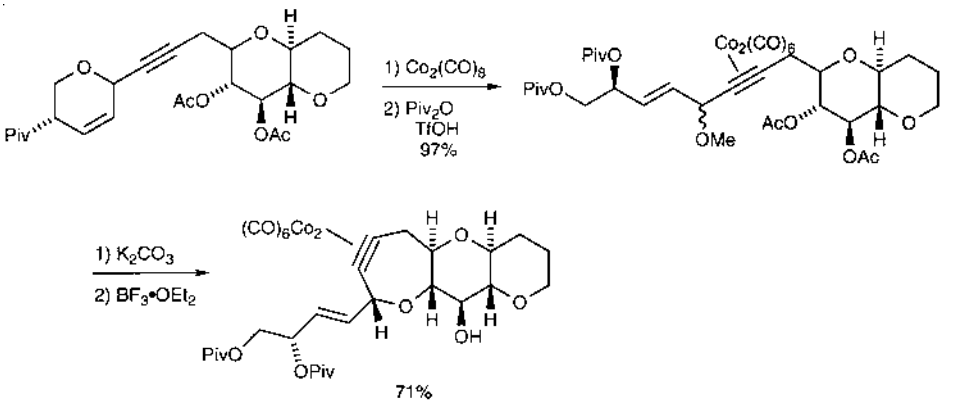




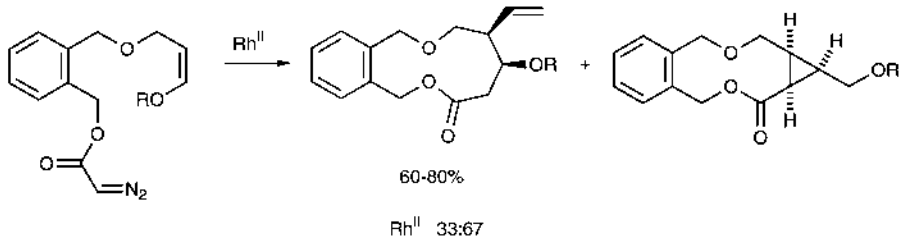


(294)

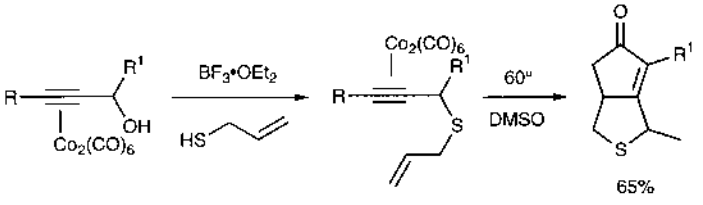
Cobalt carbonyl provided the heterocyclization seen in Eq. (255) [851]. Macrocyclic diethers were prepared by rhodium(II)-catalyzed diazodecomposition [Eq. (296)] [852]. Sulfur heterocycles were prepared by Pauson–Khand chemistry [Eq. (297)] [853]. Silicon-containing heterocycles were prepared by rhodium-catalyzed alkene silyl formation [854], palladium-catalyzed reactions between alkynes and silacyclopropanes to give silacyclopentadienes [855] and palladium-catalyzed addition of disilanes to alkenes [856]:



(295)



(296)

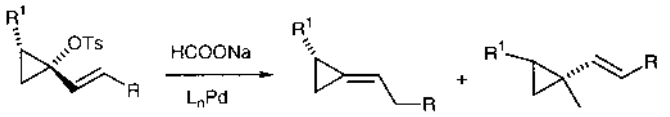


(297)

3.6. Alkenes, alkanes

Chromium-complexed benzylphosphonates underwent Wittig olefination reaction with ketones [857]. The complex $Cp_2Ti(CH{=}CH_2)(Me)$ converted ketones to allenes [858] and $Cp_2Ti[P(OEt)_3]_2$ olefinated ketones aldehydes and esters with dithianes of unsaturated aldehydes to give dienes [859]. Cp_2ZrBu_2 deoxygenated allyl epoxides to dienes [860]. The mechanism of the palladium(0)-catalyzed elimination of allyl carbonates to give dienes was shown to be a base-promoted anti-elimination in cyclic systems [861].

Treatment of propargyl phosphonates with samarium iodide and a palladium(0) catalyst in the presence of a chiral proton source gave optically active allene with $>95\%$ ee [862,863]. Palladium(0) complexes catalyzed the reduction of cepham alkenyl chlorides or triflates to a mixture of the alkene and alkane [864], and the reduction of (cyclopropyl)(allyl)tosylates [Eq. (298)] [865]. Vinycyclopropanes were cleaved by Pd/C hydrogenolysis [866]. Aryl mesylates were reduced to arenes by zinc metal in methanol in the presence of nickel(II) phosphine catalysts [867] and aryl bromides were reduced to arenes by Grignard reagents in the presence of zirconocene dichloride [868]. C-Linked glycopeptides were made by asymmetric reduction [Rh(I)duphos] of the corresponding dehydroamino acid [869]. Alkynes attached to iron–diene complexes were semi-hydrogenated to the cis alkene, without overreduction [870]:

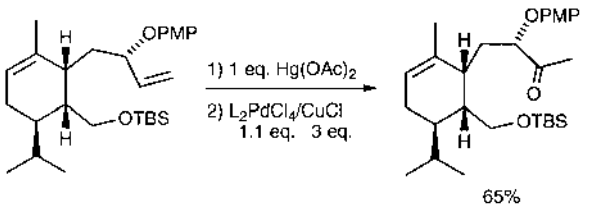


L = dppe 81% 1:1
Ph_3P 80% 100:0

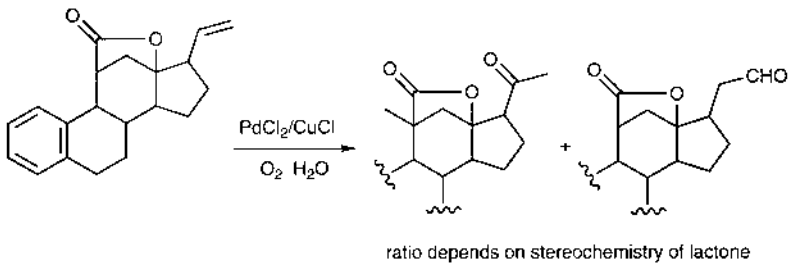
(298)

3.7. Ketones, aldehydes

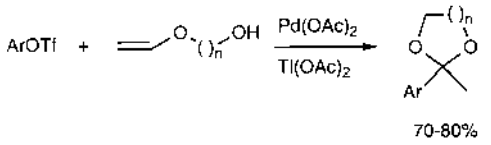
Molybdenum hexacarbonyl catalyzed the hydrolysis of oximes to ketones [871]. The system $^{n}Pr_4N^{+}RuO_4$/4 Å mol sieve catalyzed the air oxidation of 1°, 2°, allylic, benzylic and α-keto alcohols to carbonyl groups [872]. Nickel(II) phosphine complexes catalyzed the reduction of acid chlorides to aldehydes by tributyltin hydride [873]. The cluster $Pd_{561}Phen_{60}OAc_{180}$ was an effective catalyst for the oxidation of primary allylic alcohols to aldehydes [874]. Wacker chemistry oxidized alkenes to ketones, Eq. (299) [875] and Eq. (300) [876]. Palladium(0) complexes catalyzed the rearrangement of (allyl)(silyl) epoxides to α-silyl-β,γ-unsaturated aldehydes [877]. Rhodium(II) complexes catalyzed the addition of aldehydes to alkenes to give ketones (hydroacylation) [878]. Chiral palladium BINAP complexes catalyzed the stereoselective deuteration of silylenol ethers to give α-deuteroketones [879]. Other carbonyl-forming reactions are shown in Eq. (301) [880] and Eq. (302) [881]:



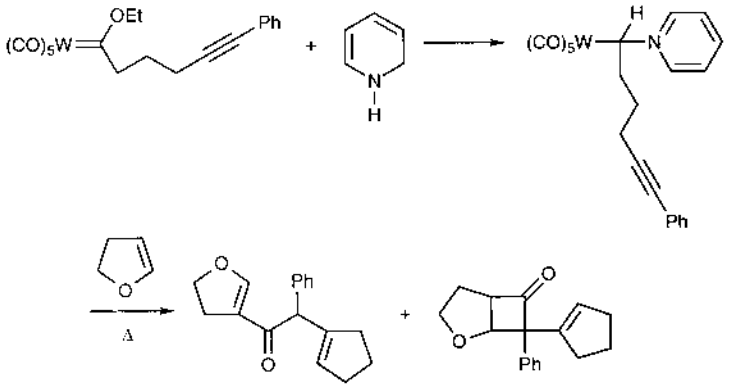
(299)



(300)



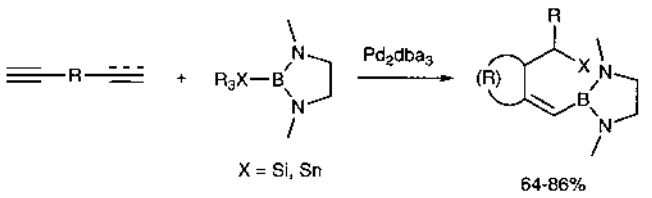
(301)



(302)

3.8. Organoboranes, silanes, germanes and stannanes

Palladium(0) catalyzed the reaction of tetraalkoxyl diborons with aryl triflates [882] and aryl halides [883], including polymer-supported iodobenzamides [884], to give aryl boronate, the addition of tetraalkoxydiborane to alkenes [885] and alkynes [886] to give 1,2-bis boronates, and platinum(0) catalyzed the 1,4-addition of tetraalkoxy diborons to enones to give β-boronated enol ethers [887]. Palladium(0) catalyzed the addition of silylamino boranes [888] and stannyl aminoboranes [889] to enynes:



(303)

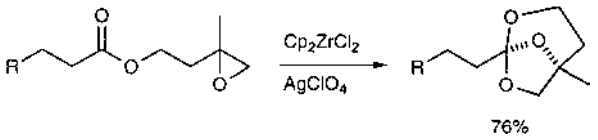
Chiral rhodium(II) complexes catalyzed the Si–H insertion reaction of diazoesters to produce α-silyl esters with high ee [890]. The de values were lower when achiral catalysts and chiral diazoesters were used [891]. The mechanism of the hydrosilylation of alkenes by cationic palladium(II) phenanthroline complexes has been studied [892]. Palladium(0) catalyzed the conversion of aryl iodides to aryl silanes with triethoxysilanes [893]. Manganese(II) chloride catalyzed the addition of $R_3SiMgCl$ to terminal alkynes to give terminal alkenyl silanes [894]. Silacyclopropanes were converted to silacyclopropenes and silacyclopentadienes by palladium(II)-catalyzed reaction with alkynes [895]. Bromopyridines, bipyridines and terpyridines were stannylated by palladium-catalyzed reaction with hexamethylditin [896]. Palladium(II) catalyzed the radical hydrostannylation of alkynes by tributyl stannane [897].

3.9. Organophosphorus and sulfur compounds

Nickel(II) chloride catalyzed the reaction of *p*-bromoacetophenone with triethylphosphate to give the aryl diethylphosphonate [898], and the reaction of aryl bromides or triflates with chlorodiphenylphosphine to give triarylphosphines [899]. Palladium(0) catalyzed the addition of disulfides to alkynes to give 1,2-dithioalkenes [900]. Palladium(II) complexes catalyzed the conversion of terminal alkenes, sulfur dioxide and hydrogen to terminal sulfinic acids, and their subsequent 1,4-addition to enones [901].

3.10. Miscellaneous

Eq. (304) [902]:



(304)

4. Reviews

The following reviews have appeared.

- Asymmetric ylide reactions: epoxidation, cyclopropanation, aziridination, olefination and rearrangement (> 182 Refs.) [903].
- Transition metals in organic synthesis 1995 (1039 Refs.) [904].
- Alkenylidenes in organic synthesis [905].
- Selective transformations of alkynes with ruthenium catalysts (43 Refs.) [906].
- Hydroboration catalyzed by transition metals (132 Refs.) [907].
- Tandem reactions on a zirconocene template (10 Refs.) [908].
- Regio- and stereoselective syntheses with organocopper reagents (115 Refs.) [909].
- Asymmetric transfer hydrogenation by chiral ruthenium complexes (39 Refs.) [910].
- New organometallic reagents using highly reactive metals (54 Refs.) [911].
- Recent advances in C–F bond activations (83 Refs.) [912].

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