

## Transition metals in organic synthesis: highlights for the year 1996

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**Keywords:** Organic synthesis; Transition metals

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## 1. General comments

This survey highlights the use of transition metals in organic synthesis for the year 1996. 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.

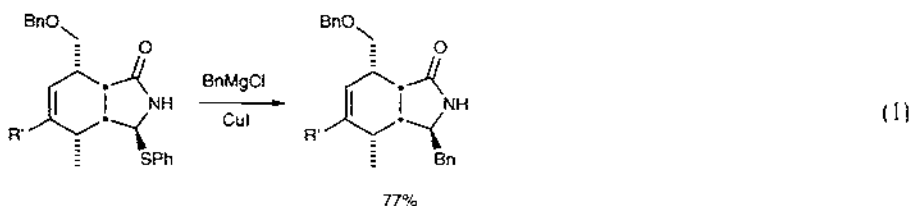
## 2. Carbon–carbon bond-forming reactions

### 2.1. Alkylations

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

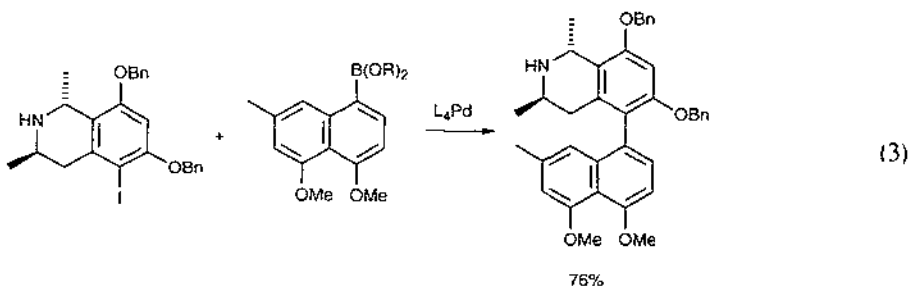
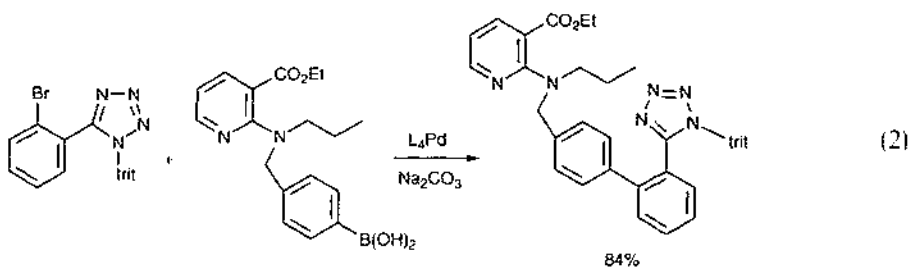
Iron(III) acetylacetonate catalyzed the alkylation of alkenyl halides by aliphatic organomanganese(II) halides with retention of geometry [1].  $\beta$ -Difluoroalkenyl cuprates alkylated a range of aliphatic, benzyl, and allyl halides,  $\alpha$ -haloesters, and acid halides [2]. Alkenyl triflates were alkylated by  $\alpha$ -lithiated *N*-Boc amines in the presence of copper(I) cyanide [3]. Organocuprates alkylated purine nucleosides [4].  $\alpha$ -Silylated bromoallenes were alkylated by organocuprates with clean inversion [5]. Copper(II) salts catalyzed the alkylation of benzyl iodides by heteroaromatic Grignard reagents [6]. Copper(I) bromide catalyzed the carbocupration of ethoxyacetylene by Grignard reagents. The resulting *trans*- $\beta$ -alkoxyvinyl cuprates were coupled to aryl halides using palladium(0) catalysts [7]. Copper(I) salts catalyzed the

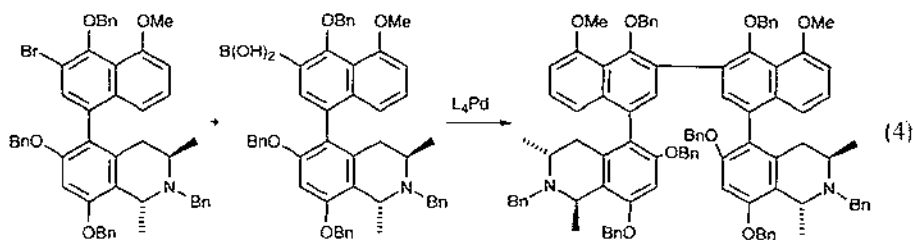
alkylation of thioaminals (Eq. (1)) [8]. Palladium catalyzed the peralkylation of hexaiodobenzene by the copper acetylide of propargyl alcohol THP ether [9].



Nickel(II) phosphine complexes catalyzed the alkylations of vinyl selenides by Grignard reagents [10,11]. New chiral ligands for the asymmetric coupling of benzyl Grignard reagents with halides involving binaphthyl amine-phosphines have been developed [12]. Nickel(0) catalysts were superior to palladium(0) catalysts for the alkylation of benzyl chlorides with vinyl alanes [13].

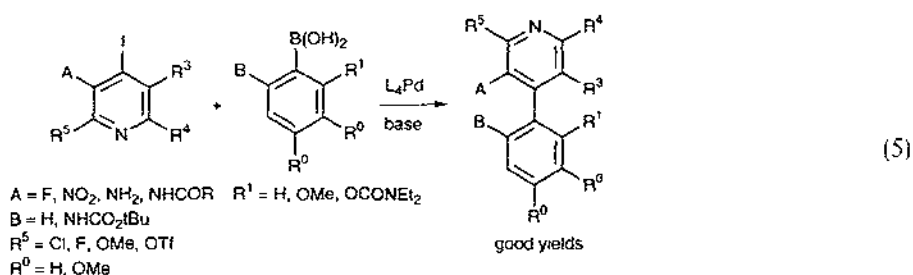
Palladium clusters and palladium-nickel clusters were used to catalyze Suzuki and Heck coupling reactions [14]. Triphenyls containing unsaturated side chains were prepared by palladium-catalyzed coupling of functionalized 1,4-dibromobenzenes with diene-containing arylboronic acids [15]. Arylboronic acids coupled to tetrazole-containing aryl halides (Eq. (2)) [16], *o*-bromonitrobenzene and *o*-bromoacetamide [17], 2,4,6-trialkoxyaryl bromides [18], and *o*-bromobenzenesulfonyl ureas [19] in the presence of palladium catalysts. This chemistry was used to make Korupensamine C (Eq. (3)) and ancastobrevin B [20] and *ent*-Korupensamine D [21,22], as well as the Michellamines (Eq. (4)) [23].



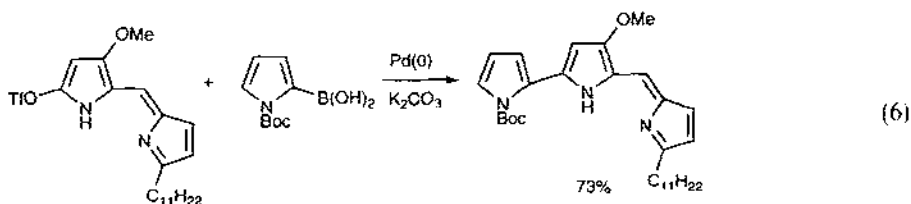


Suzuki coupling has been applied to solid phase synthesis for combinatorial chemistry, making biaryls on Wang silicon resins [24], biphenylcarboxylic acids on polystyrene [25], biaryl benzamides on PAM linked resins [26] and *p*-alkenylbenzamides [27].

Aryl boronic acids coupled to 2-halopyridines [28], 2-carboxy-3-triflylpyridines [29] and  $\alpha,\alpha'$ -dichlorophenanthroline [30] in the presence of palladium(0) catalysts. Highly substituted heterocycles on the route to streptonigrin and lavendamycin were prepared by Suzuki coupling (Eq. (5)) [31].

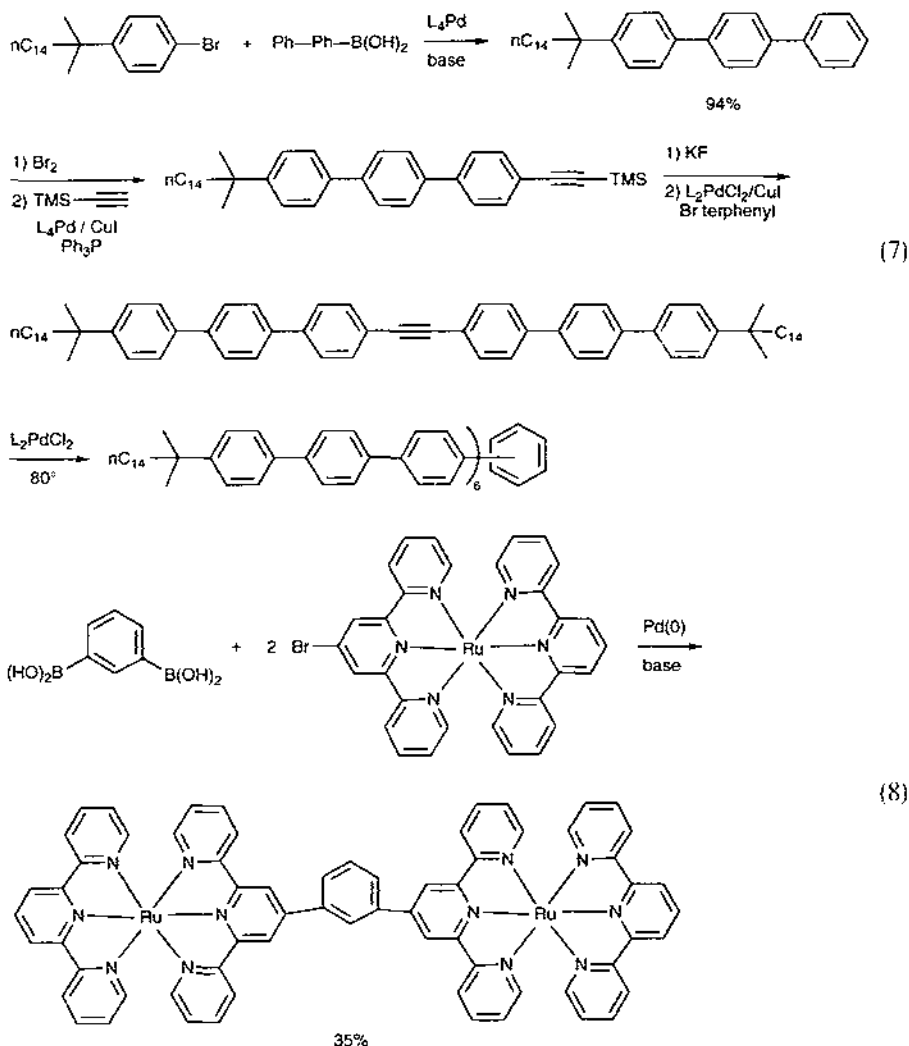


Palladium(0) catalyzed the coupling of 2-triflylindoles with heteroaryl boronic acids [32], 3-iodopyrimidoindoles with aryl boronic acids [33] and pyrrole triflates with pyrrole boronic acids (Eq. (6)) [34]. Mono, tetra and octabromo-tetraphenylporphyrins were perphenylated by arylboronic acids in the presence of palladium(0) catalysts [35].



The cationic Cp ruthenium complex of 1,4-dibromobenzene was diphenylated, to give complexed terphenyl, by phenylboronic acid and a palladium catalyst [36]. Arenes containing oligophenyl groups were synthesized using Suzuki coupling (Eq. (7)) [37]. The oligophenyl containing up to 15 phenyl groups in a row was

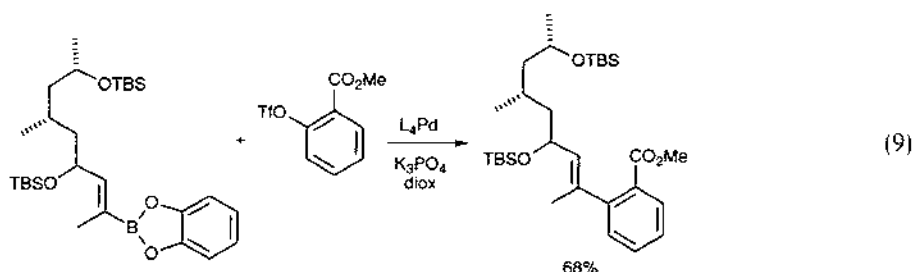
synthesized via Suzuki coupling [38]. Another unusual Suzuki coupling is shown in Eq. (8) [39].



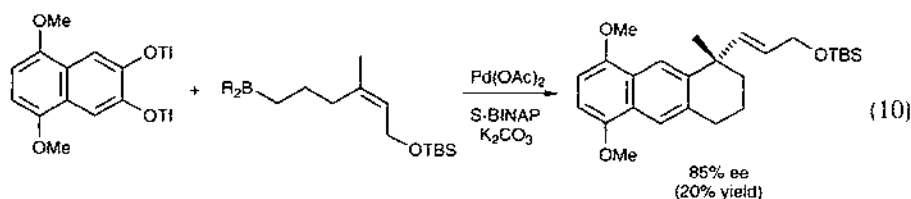
Palladium(0) catalyzed the coupling of arylboronic acids with aryl diazonium salts [40,41]. Iodides and triflates were tolerated in the diazonium species. Aryl iodonium salts also coupled [42]. Nickel(II) phosphine complexes were the preferred catalysts for coupling arylboronic acids with aryl sulfonates [43] and aryl chlorides [44]. Palladium catalysts failed in the latter case.

Alkenes having boronic ester groups at the 1 and 2 positions [45] and dienes having them at the 1 and 3 positions [46] were selectively arylated at the 1-position under Suzuki coupling conditions. Ketone hydrazones were converted to arylated

alkanes by a combined Shapiro reaction (3 equiv. BuLi followed by  $B(OiPr)_3$ ) followed by a Suzuki coupling [47]. Arylation of 4-bromo- or 4-triflylisocoumarins by arylboronic acids was efficient under Suzuki conditions [48]. Vinyl boronic acids coupled efficiently to aryl triflates under Suzuki conditions (Eq. (9)) [49].



Long chain alkylboranes coupled to aryl triflates under Suzuki conditions [50]. This process was used as part of a very nice Suzuki–Heck cascade (Eq. (10)) [51].



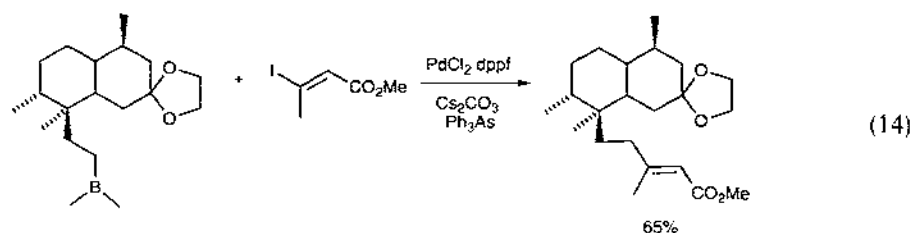
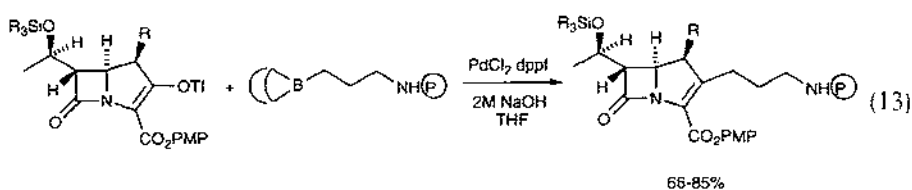
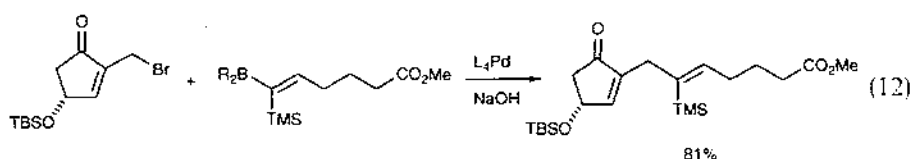
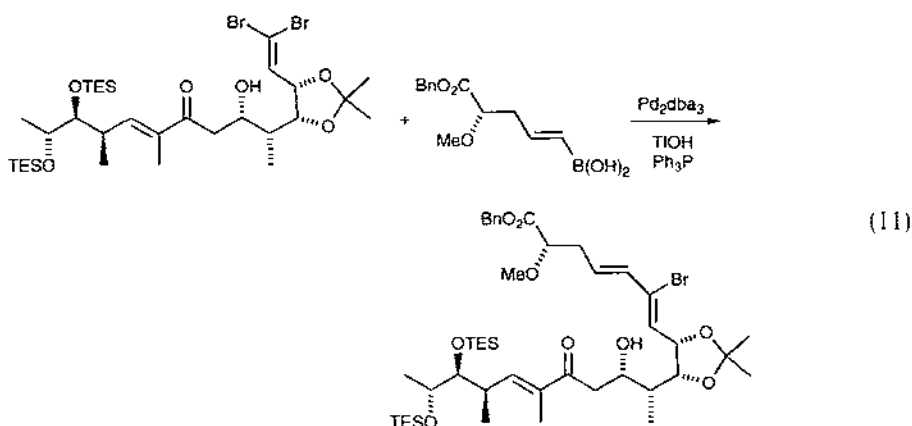
Suzuki coupling was used to connect arylboronic acids with vinyl halides [52], and vinylboronic acids with vinyl bromides to make dienes [53,54], (in both cases with extensive functionality present). With gem-dibromoalkenes, only one halide site underwent coupling (Eq. (11)) [55]. Butenolides having  $\beta$ -triflate leaving groups were efficiently coupled to alkenylboronates under palladium catalysis [56].

Other synthetically interesting couplings are shown in Eq. (12) [57], Eq. (13) [58] and Eq. (14) [59].

Palladium catalyzed the coupling of cyclopropylboronic acids with aryl halides [60], cyclopropyl iodides with alkenylboronic esters [61] and alkynylboronates with aryl halides [62].

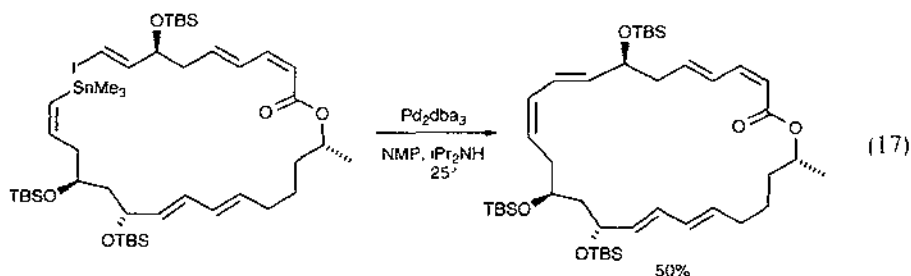
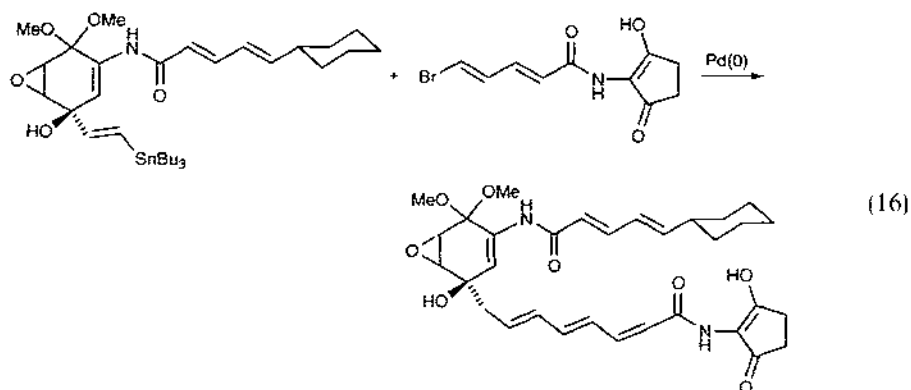
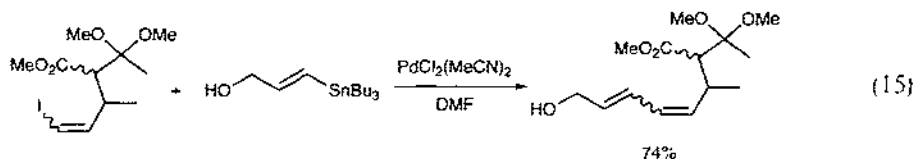
The coupling of halides to organosilanes in the presence of fluoride and palladium catalysts (Hiyama coupling) was rare this year. Alkenyl silanes were coupled to iodopyrimidines [63] and aryl chlorides [64] under Hiyama conditions.

In contrast the palladium-catalyzed coupling of stannanes and organic halides and triflates (Stille coupling) was a very active area. “New perspectives in the cross-coupling reactions of organostannanes” is the title of a review with 24 references [65]. By replacing the spectator alkyl groups on tin with  $CH_2CH_2C_6F_{13}$  groups, the tin residue from Stille coupling was easily removed by extraction [66]. Palladium(0) complexes catalyzed the coupling of aryl iodonium salts [67] and related hypervalent

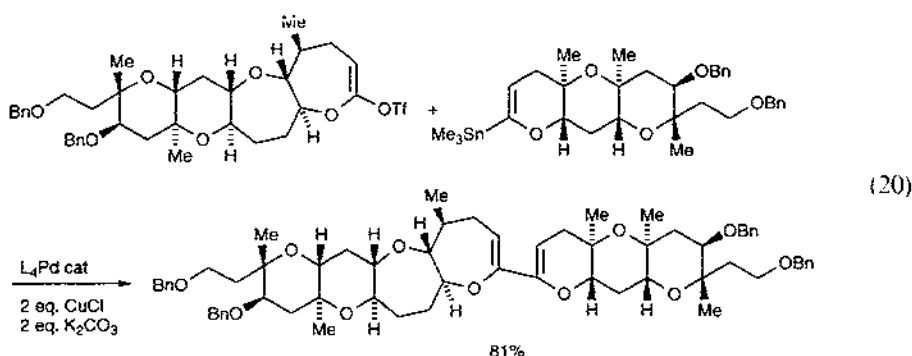
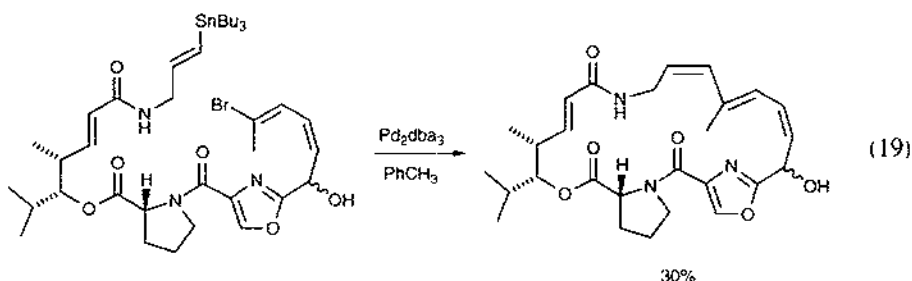
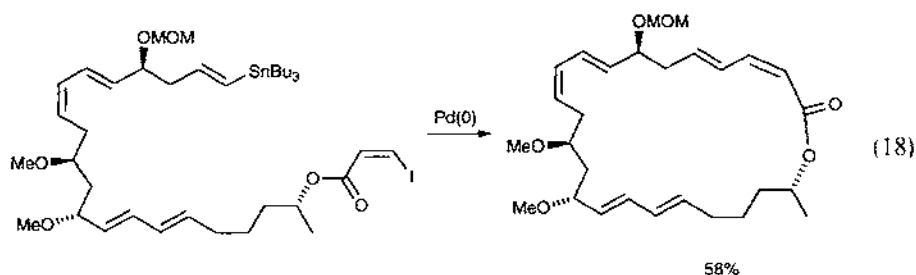


iodine compounds with organostannanes [68]. Steroids having 17-iodoalkenyl substituents coupled to vinyl tin reagents in the presence of dienophiles to give Diels Alder cycloadducts in one pot [69]. Stille coupling was used to prepare 1,3-dienes from cyclic vinyl triflates and vinyl stannanes [70], dienes containing polyhydroxy functionality from functionalized vinyl stannanes and functionalized vinyl iodides [71], and 1,3,5-trienes from vinyl stannanes and bromodienes [72].

Palladium(0) catalyzed the coupling of cyclic vinyl triflates with 2-stannyldioxenes [73], and the coupling of vinyl, allyl, and allenyl stannanes with cephalosporin vinyl triflates [74]. Other synthetically significant vinyl vinyl couplings are seen in Eq. (15)–Eq. (20) [75–80].



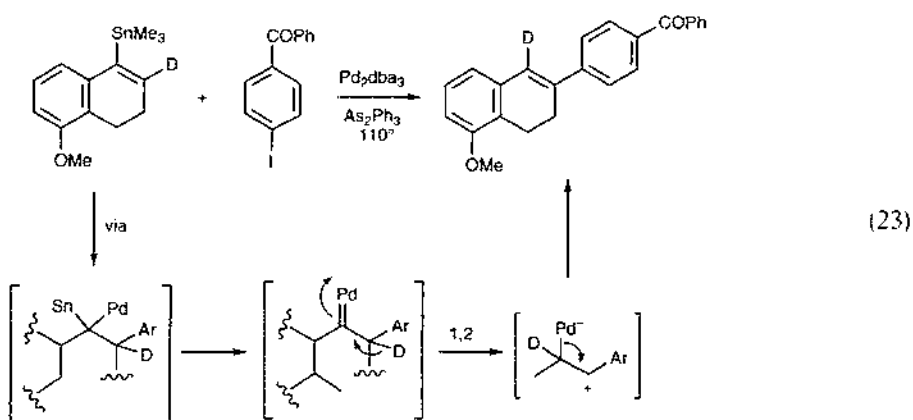
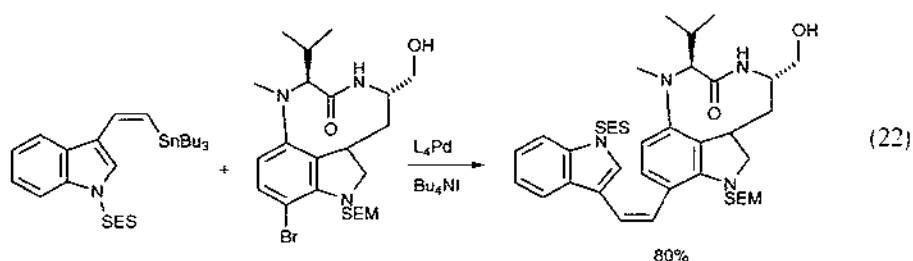
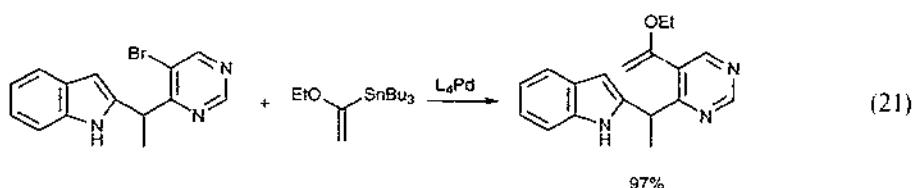
Palladium(0) complexes catalyzed the coupling of aryl stannanes and boronic acids with 2-iodocyclohexenones [81]. Both  $\alpha$ - and  $\beta$ -stannyl butenolides were reluctant to couple to aryl halides and only  $L_2PdCl_2$  worked [82]. 2-Bromonaphthoquinones were arylated by 2,4,6-trisubstituted aryl stannanes using palladium(0) catalysts [83]. 3-Stannylfurans alkylated enol triflates of cyclopentanes under palladium(0) catalysis [84], while 5-iodobenzofurans were alkenylated by propenyl tin reagents [85].  $\beta$ -Stannylenones were arylated by aryl halides [86] and  $\gamma$ -amino vinyl stannanes were coupled to aryl and alkenyl halides under palladium



catalysis [87]. The 17-enol triflates of steroids were coupled to 3-stannyl- $\alpha$ -pyrone [88], and the enol triflates of cyclic  $\beta$ -ketoesters were coupled to oxygenated arylstannanes [89] using Stille coupling. Other useful Stille couplings are shown in Eq. (21) [90] and Eq. (22) [91]. An unusual rearrangement observed during Stille coupling is shown in Eq. (23) [92].

Palladium(0) complexes catalyzed the coupling of stannylpyridines with bromopyridine *N*-oxides to form bipyridines and terpyridines [93]. 2,6-Oligopyridines were made in a similar manner [94]. The triflate of *N*-tosyl-4-hydroxyindole underwent Stille coupling with a wide range of organostannanes [95].

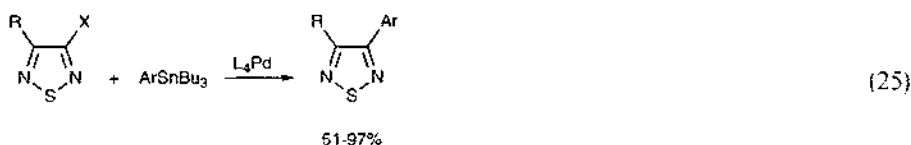
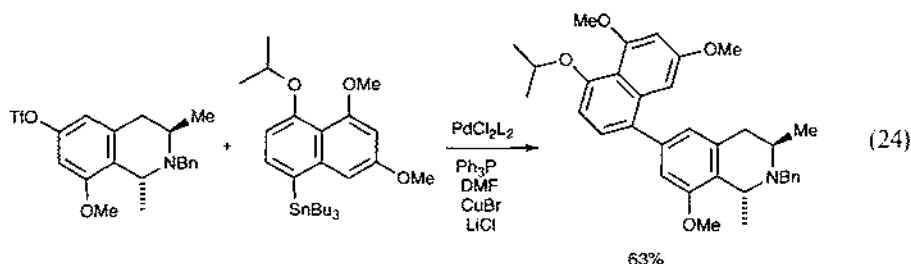
The Stille reaction was used to couple 3-stannylpyrroles to aryl and heteroaryl halides [96]. 4-stannylnitrobenzenes with 2-hydroxypyridine triflates [97],



2-stannylpyridines to substituted bromobenzaldehydes [98], and 3-stannylpyrroles, thiophenes, indoles and benzothiophenes to 2,3-dibromobenzoquinones [99]. Materials for crystal engineering of stacked aromatic columns were synthesized by palladium-catalyzed coupling of 5-stannyl-1,3-pyrazines with 9,10-dibromoanthracenes [100].

Iodopyrimidine nucleosides [101] and chloroadenines [102] were alkylated by a variety of stannanes under palladium catalysis. *p*-Bromobenzyl pyroglutamic esters were alkylated without loss of stereochemistry [103]. 3-Iodo-2-formyl indoles were coupled to stannylated cyclic unsaturated carbamates using Stille coupling [104]. Functionalized 3-stannylpyridines were coupled to functionalized 3-bromopyrroles [105], and 2-bromothiazoles [106] under Stille conditions. Brominated benzyl phosphonates were coupled to heteroaryl stannanes using palladium catalysis [107].

Other useful Stille couplings are seen in Eq. (24) [108] and Eq. (25) [109].



Palladium catalyzed the alkylation of 2-bromothiazolines [110] and optically active 2-bromooxazolines [111] by tin reagents. Pyrrolidinone triflates were also alkylated by a range of tin reagents under palladium catalysis [112,113]. Alkynyl stannanes were coupled to highly functionalized vinyl iodides [114], 3-iodo-2,2'-binaphthyl [115], 3-bromobutenolides [116], 3-iodothiophenes [117] and norbornadiene *bis* phenyliodonium salts [118] in the presence of palladium catalysts.

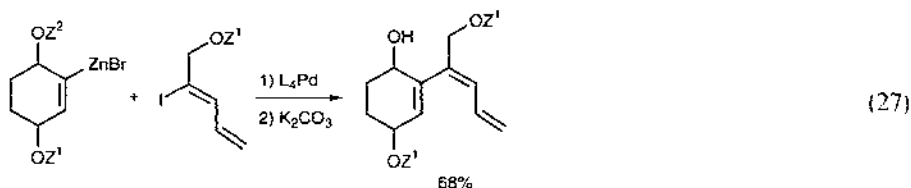
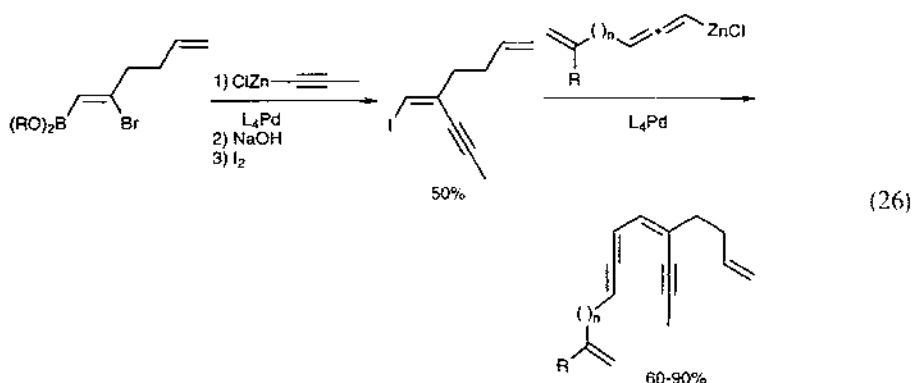
The iron tricarbonyl complex of 1-*O*-triflyl-1,3-butadiene was vinyllated and alkynylated by coupling with the appropriate tin reagent [119]. Penta-iodocyclopentadienyl mangananes tricarbonyl was coupled to cyclopentadienyl trimethyltin at all five positions under Stille coupling conditions [120]. The iodo position of a 1-butyl-2-iodoalkene coupled selectively to a range of organotin reagents under Stille coupling conditions [121]. Functionalized allyl bromides [122] and  $\text{sp}^3$  halides containing  $\beta$ -perfluoroalkyl groups [123] coupled cleanly to organotin reagents in the presence of palladium catalysts. Copper(I) iodide [124] and the copper(I) salt of thiophene-2-carboxylic acid [125] catalyzed the coupling of organostannanes and organic halides without the necessity of palladium cocatalysts.

Organozinc reagents also underwent efficient transmetalation to palladium and participate in a range of useful coupling reactions. Functionalized organozinc halides for the process were prepared by the nickel(II) catalyzed reaction of diethylzinc with primary halides or terminal alkenes [126] or by lithiation of functionalized aryl or alkenyl iodides at  $-90^\circ\text{C}$  followed by treatment with zinc bromide [127]. Arenes containing both a triflate and an iodide coupled with functionalized arylzinc halides exclusively at the iodide position in the presence of palladium catalysts [128]. Halobenzoic esters with additional electron-withdrawing groups were alkylated by organozinc halides in the presence of palladium/copper catalysts [129]. Polymer-bound biaryls were synthesized by the palladium-catalyzed coupling of polymer-

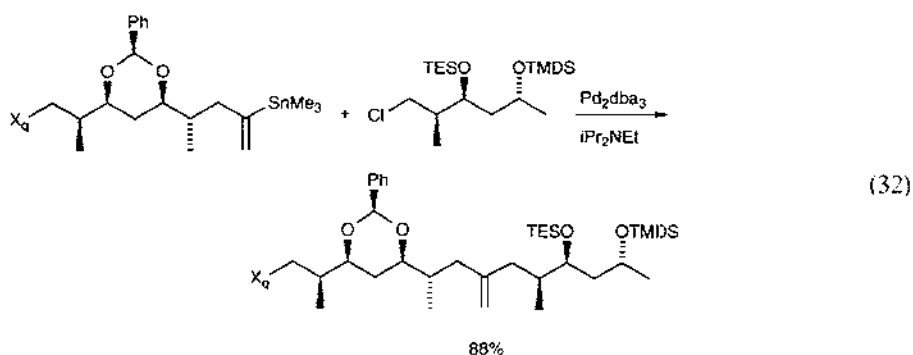
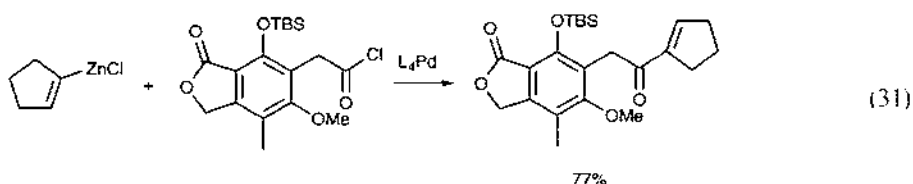
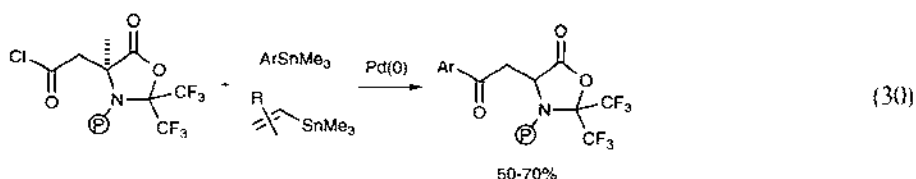
bound bromobenzoates with arylzinc halides [130].  $\alpha,\beta$ -Dibromoacrylates were alkylated exclusively at the  $\beta$  position by organozinc halides in the presence of palladium(0) catalysts [131].  $\beta$ -Iodoacrylic acid coupled to vinyl and alkynylzinc halides in the presence of palladium catalysis [132].

Indole zinc halides coupled to aryl and heteroaryl halides [133] and to 2-bromopyridine in the presence of palladium catalysis [134]. Bipyridyls were made by palladium-catalyzed coupling of bromopyridines with pyridinezinc halides [135]. Chloroadenine was converted to cyanoadenine by zinc cyanide in the presence of palladium catalysts [136]. Iodopurines and pyrimidines as well as their nucleosides were converted to their organozinc derivatives and coupled to aryl halides using a palladium catalyst [137]. Phenyl alanines containing a boronic acid group on the benzene ring were prepared by palladium-catalyzed coupling of the iodophenylboronic acid with alanine-zinc halide reagent [138]. Functionalized alkenyl iodides [139] and  $\alpha$ -bromo- $\beta$ -methoxyacrylates [140] were coupled to organozinc reagents using palladium catalysis.

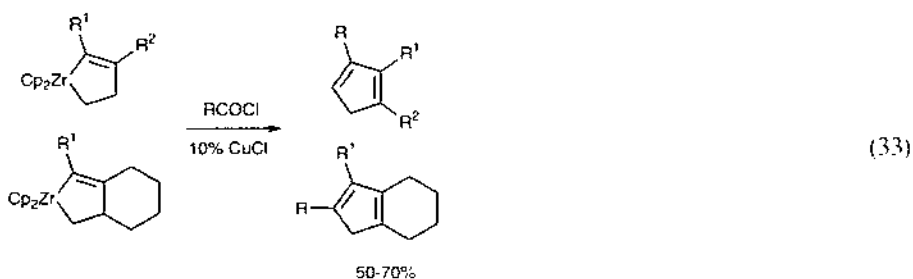
A variety of aryl halides were alkylated by oxazole-2-zinc chloride [141], and furan-, thiophene-, *N*-methylpyrrole- and selenophene-2-zinc chloride [142] in the presence of palladium catalysts. Interesting uses in synthesis are seen in Eq. (26) [143], Eq. (27) [144] and Eq. (28) [145].







species were acylated by methyl chloroformate [166] and the dimethyl chloroformamides [167].

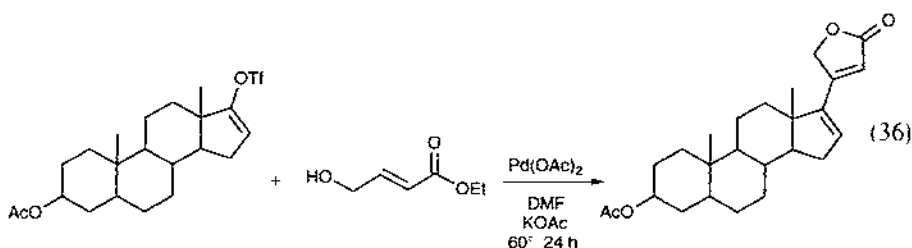
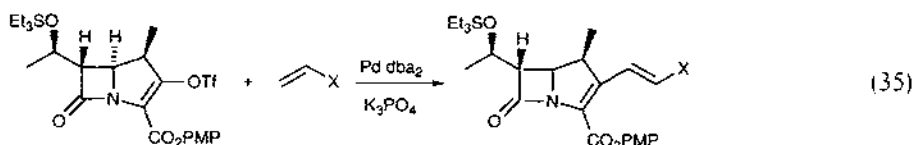
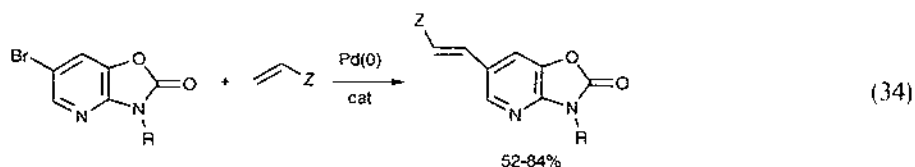


### 2.1.3. Alkylation of alkenes

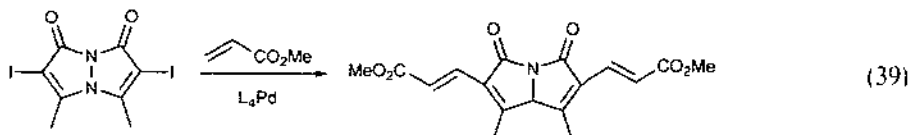
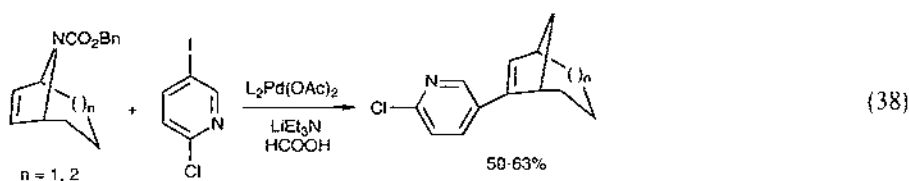
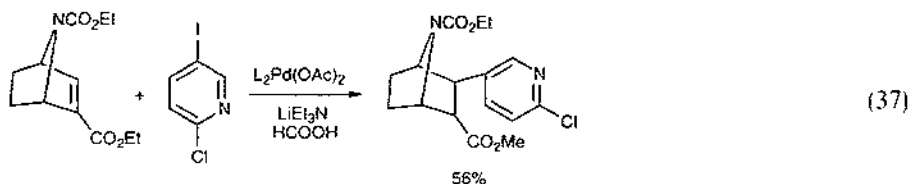
Palladium-catalyzed reactions of unsaturated triflates with alkenes and alkynes has been reviewed (32 references) [168], as has selectivities in carbametallation of

olefins (15 references) [169]. The electronic effects on insertion of *p*-substituted styrenes into palladium alkyl complexes has been studied [170]. Electron-withdrawing groups accelerate the process. The reactive intermediate in the palladium-catalyzed arylations of methyl acrylate has been characterized [171]. The palladiacycle prepared by heating palladium acetate with tri-(*o*-tolyl)phosphine had high turnover numbers, but low selectivity for the phenylation of methyl methacrylate [172]. The Heck reaction was catalyzed by palladium colloids in the presence of base [173]. Palladium(0) catalyzed the arylations of butyl acrylate by aryl halides in molten salts [174]. Tetraalkyl ammonium sulfates promote the Heck reaction and allow it to be run in water, water-organic or anhydrous solvents [175]. The Heck reaction between aryl halides and electron-poor alkenes was dramatically accelerated by microwave irradiation (2–6 min reaction times) [176].

Ethyl cinnamate, methyl acrylate [177,178] and styrene [179] were arylated by aryl halides in the presence of palladium(0) catalyst. 1,2-Dibromo-, 1,2,3-tribromobenzene and 1,2-dibromocyclohexene underwent Heck reaction with  $\alpha,\beta$ -unsaturated ketones at all halogen-bearing sites [180]. Heck arylation of 2-acetoxyprepone gave arylacetones [181]. 1-Methoxy-2-trifluoromethyl ethene underwent Heck arylation exclusively  $\alpha$  to the methoxy group [182]. Other synthetically useful Heck reactions are seen in Eq. (34) [183], Eq. (35) [184] and Eq. (36) [185].



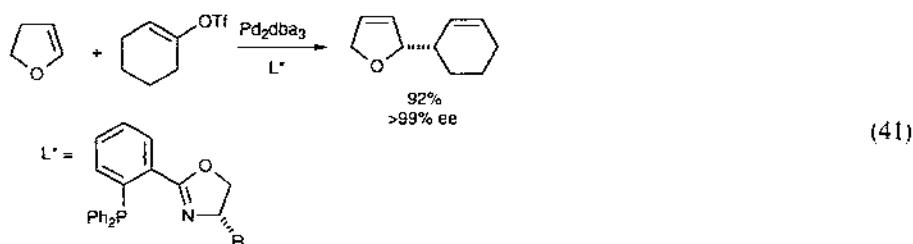
Alkenyl silanes underwent Heck arylation predominately at the nonsilyl site of the alkene [186]. Platinum complexes were inefficient catalysts for the Heck reaction [187].  $\gamma$ -Acyloxy unsaturated ketones [188],  $\gamma$ -carboethoxy acrylates [189], allyl amines [190] and optically active allylamines [191] all underwent efficient Heck arylation or alkenylation. Other interesting olefin alkylation reactions are seen in Eq. (37) [192], Eq. (38) [193], Eq. (39) [194] and Eq. (40) [195].



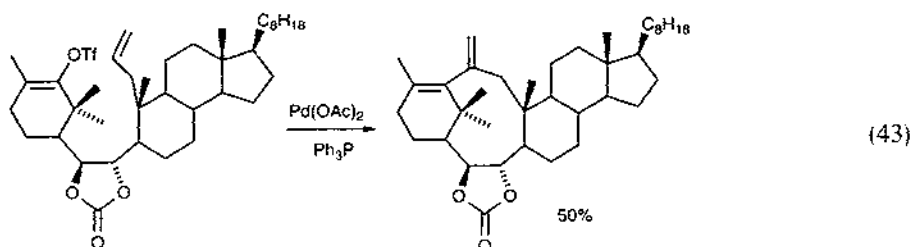
Ar = pMePh, pNO<sub>2</sub>Ph, pMeOPh, Ph  
R = H, Me

The SO<sub>2</sub> adduct of butadiene [196] and dihydrofurans [197] were arylated by aryl diazonium compounds using palladium catalysts. Dihydrofuran was alkylated by iodopurines using palladium(0) catalyst [198]. Using vinyl triflates and a chiral ligand, the reaction proceeded with high ee (Eq. (41)) [199]. In the arylation of dihydrofuran by iodobenzene in the presence of palladium catalysts, at 8 kbar pressure the turnover number was greater than 100 000 at 833 per hour, whereas at 10<sup>-3</sup> kbar it was 190 per hour [200]. In the arylation of dihydropyrroles, olefin

migration was suppressed by using additives (Eq. (42)) [201].



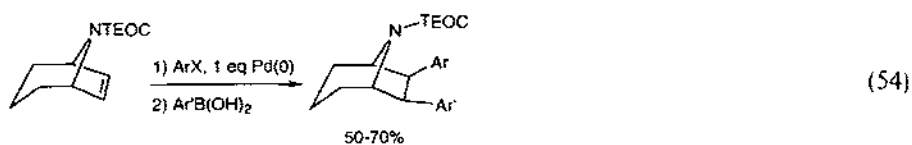
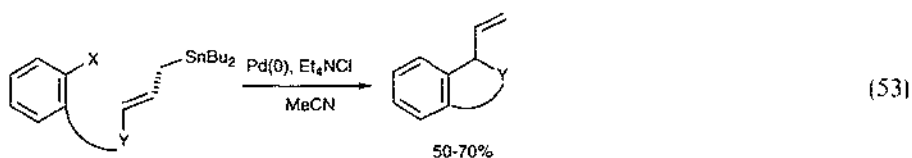
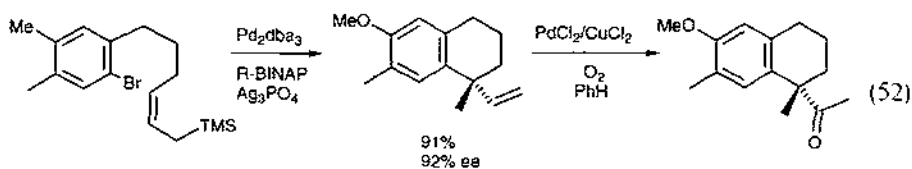
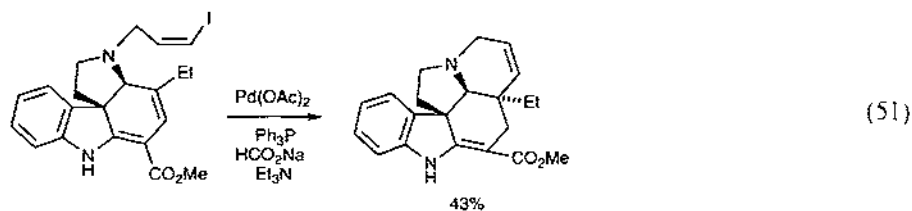
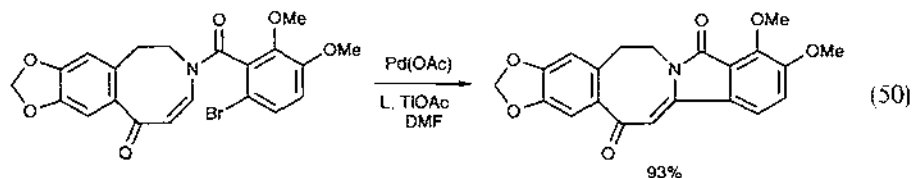
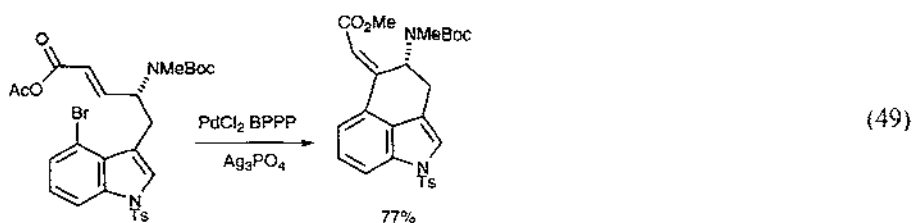
Intramolecular olefin insertion reactions continued to prove useful in organic synthesis. Cyclic carbopalladation has been reviewed (110 references) [202]. Under normal Heck conditions 5-*exo* closure is favored but in aqueous methanol with water-soluble ligands 6-*endo* can be achieved [203]. Intramolecular Heck reactions have proven very useful in the synthesis of carbocycles (Eq. (43) [204], Eq. (44) [205], Eq. (45) [206] and Eq. (46) [207]), as well as alkaloids (Eq. (47) [208], Eq. (48) [209], Eq. (49) [210], Eq. (50) [211] and Eq. (51) [212]). Using chiral ligands, asymmetry has been induced (Eq. (52)) [213]. A tin mediated variant is seen in Eq. (53) [214].

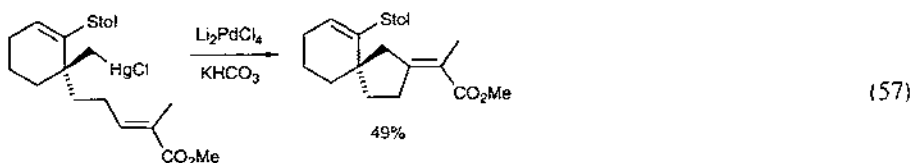
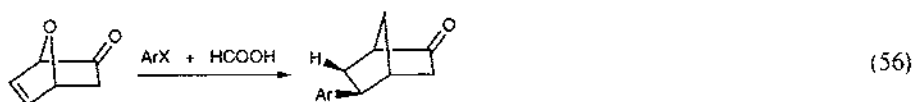
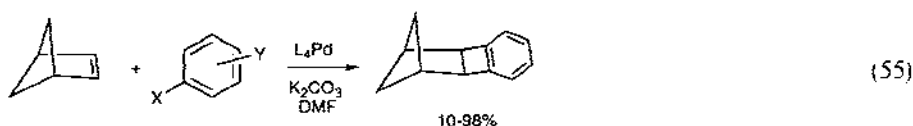


Palladium catalyzed the double alkylation of strained double bonds in bridged bicyclic compounds (Eq. (54) [215], Eq. (55) [216] and Eq. (56) [217]). Palladium(0) catalyzed the arylation of alkenes by diaryltellurium species [218] and by organomercuric halides (Eq. (57)) [219].

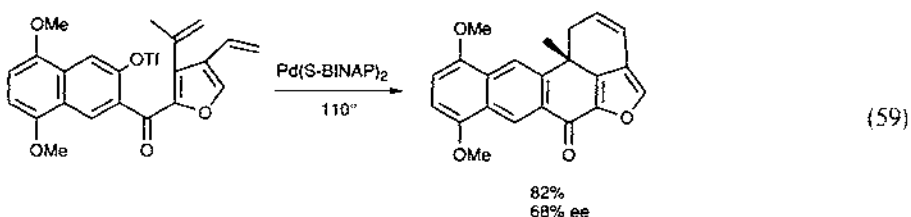
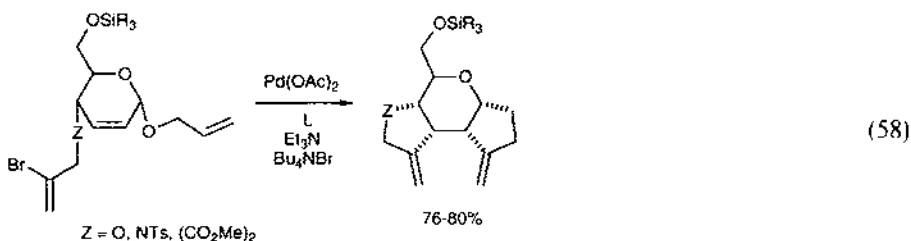
“The stereochemistry of palladium-catalyzed cyclization reactions—cascade reactions” was the title of a review with 198 references [220]. Several cascade reactions

(44)



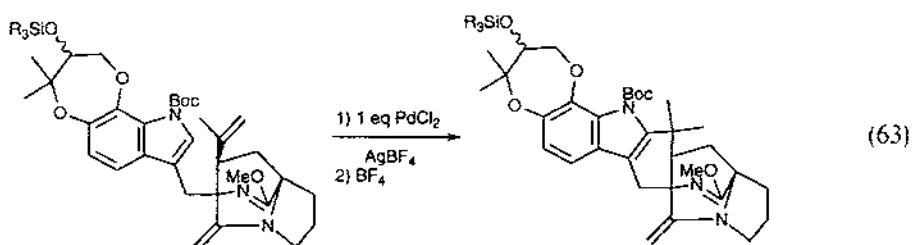
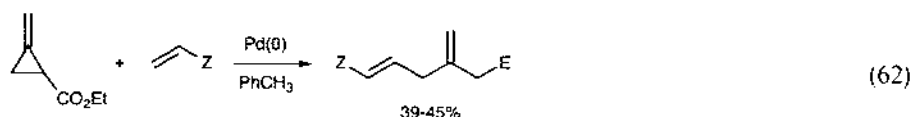
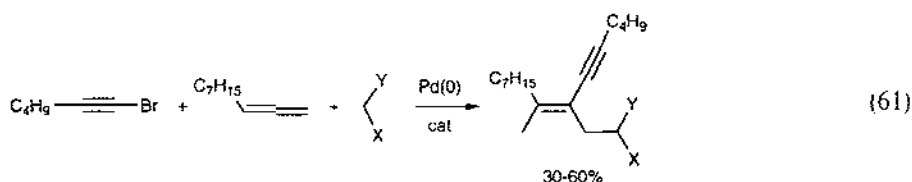
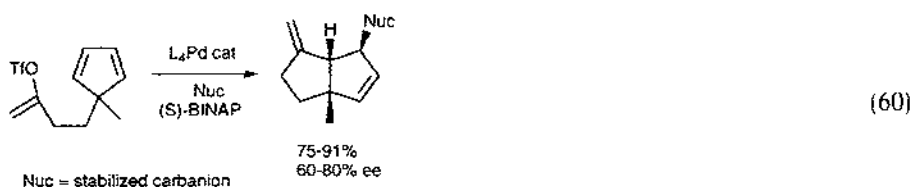


are shown in Eq. (58) [221], Eq. (59) [222], Eq. (60) [223] and Eq. (61) [224].



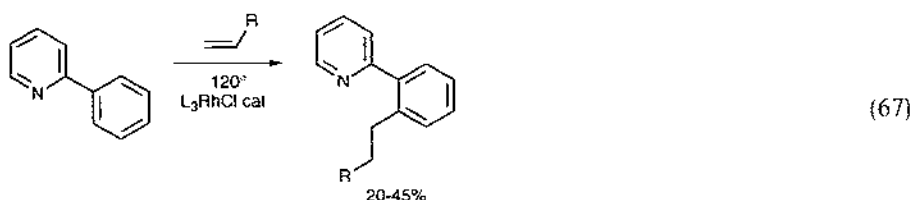
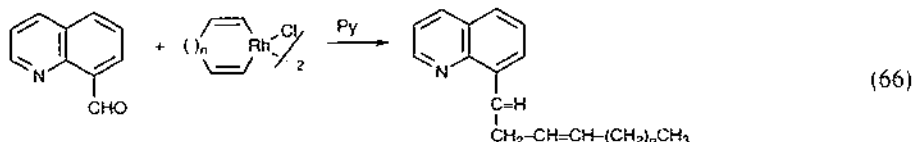
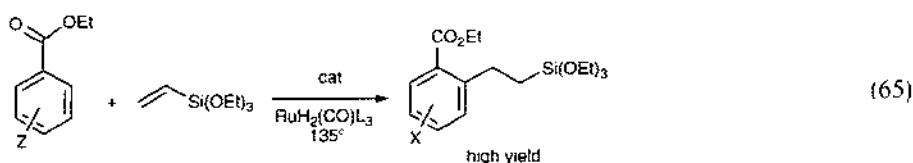
Palladium(0) complexes catalyzed the alkylation of alkenes by methylene cyclopropanes (Eq. (62)) [225]. Stoichiometric palladation of an indole by palladium(II) resulted in allylation of a pendant alkene (Eq. (63)) [226].

Nickel(acac) catalyzed the alkenylation of electron-poor alkenes by alkenylboranes [227] while nickel(0) complexes catalyzed intramolecular couplings similar to palladium (Eq. (64)) [228].

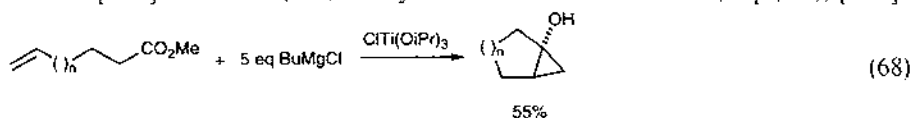


Ruthenium(II) complexes catalyzed the *o*-alkylation of aromatic carbonyl compounds by vinyl silanes via C–H activation (Eq. (65)) [229–231], while rhodium(I) hydrides catalyzed the alkylation of olefins by stabilized carbonium ions [232]. Rhodium(I) also catalyze alkylations of olefins by *o*-metallation processes (Eq. (66) [233] and Eq. (67) [234]).

Optically active zirconium complexes catalyzed the addition of diethyl magnesium



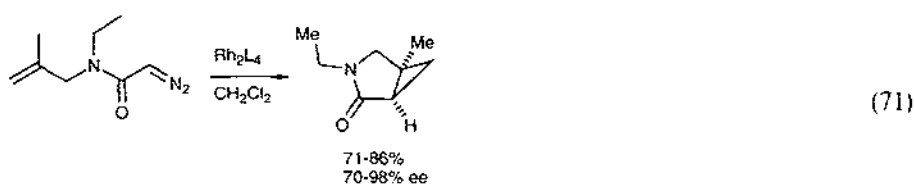
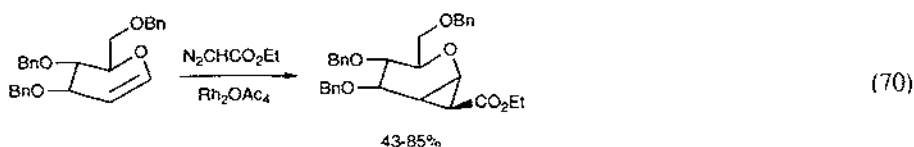
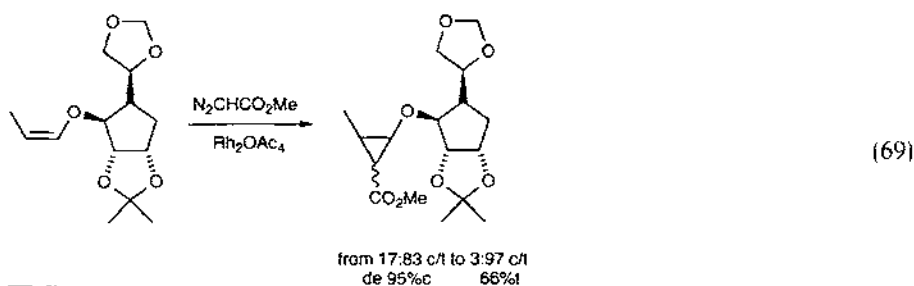
to alkenes [235], while corresponding titanium complexes did the same for triethylaluminum [236]. Titanium(IV) catalyzed the reaction shown in (Eq. (68)) [237].



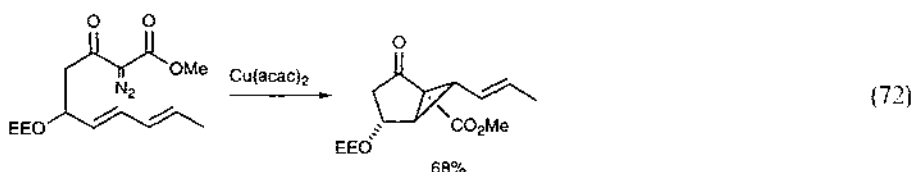
#### 2.1.4. Metal-catalyzed diazodecomposition and other cyclopropanations

Several reviews on various aspects of rhodium(II) catalyzed cyclopropanation of alkenes by diazo compounds have appeared [238–240] and new ligands for asymmetric cyclopropanation of alkenes continue to be developed [241–246]. Interesting applications are seen in Eq. (69) [247], Eq. (70) [248] and Eq. (71) [249].

Copper(I) triflates catalyzed the asymmetric cyclopropanation of styrene by diazoesters in the presence of chiral BINAP *bis* oxazoline ligands [250] including intramolecular processes [251] (Eq. (72) [252] and Eq. (73) [253]).  $\text{MeReO}_3$  catalyzed the entire range of diazoester reactions -- cyclopropanations, C–H, O–H, S–H, NH insertions, etc. [254]. Palladium catalyzed the cyclopropanation of vinylboronic esters by diazomethane [255] as well as the intramolecular process in Eq. (74) [256]. Osmium(II) cymene complexes catalyzed the cyclopropanation of styrenes



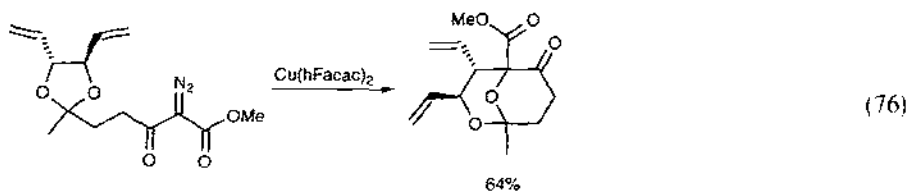
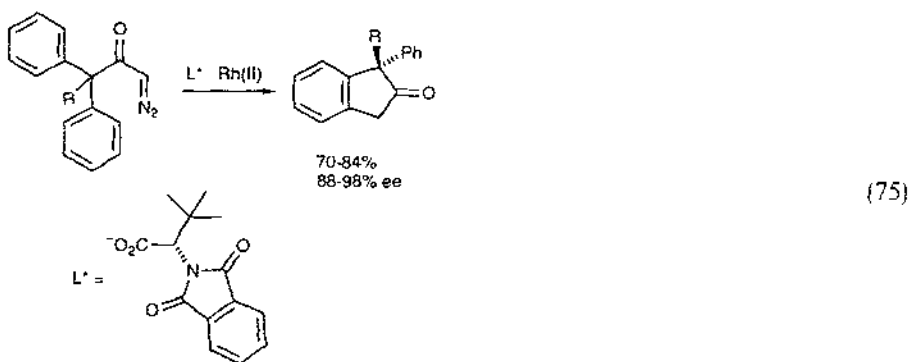
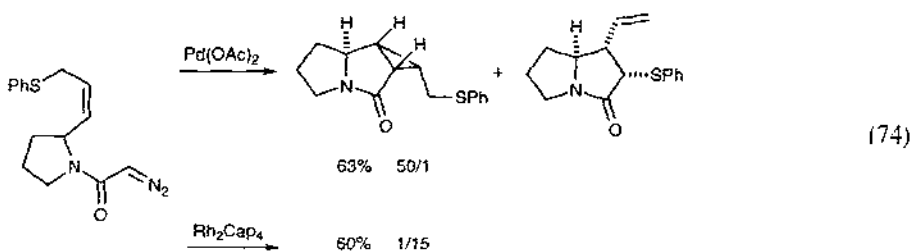
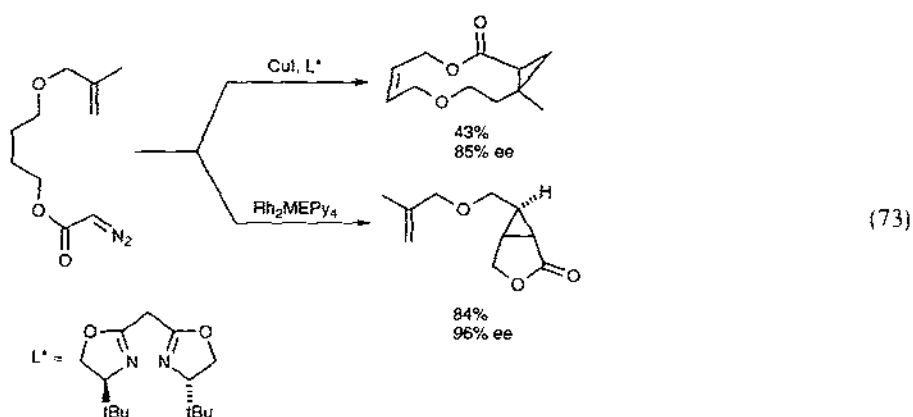
by diazoesters [257] while nickel(II) acetylacetonate catalyzed the cyclopropanations of alkenes by sulfone ylids [258].

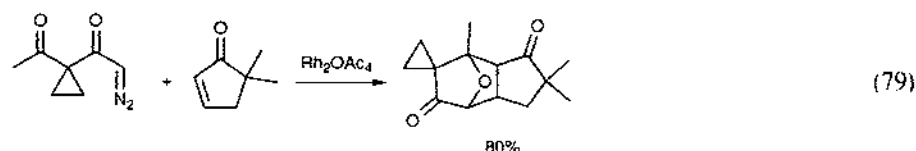
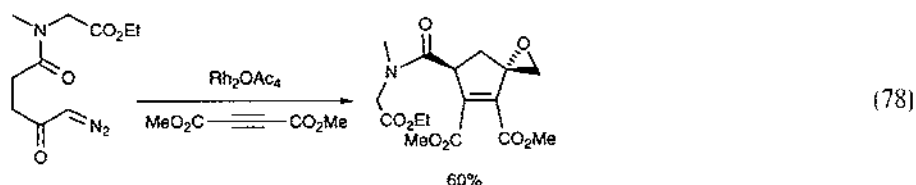
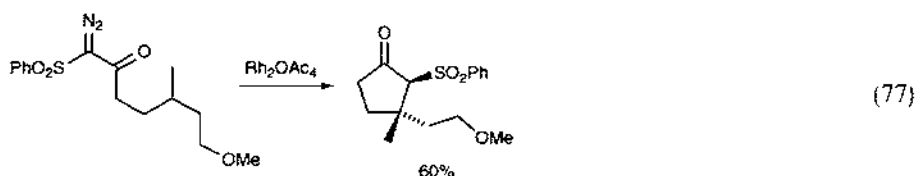


Transition metals also catalyze C–H insertion reactions of diazoalkenes. This has been reviewed [259]. The computational model ZINDO has been used to predict diastereoselectivity in Rh mediated CH insertion reactions [260]. Insertion into aryl CH bonds [261,262] is efficient and, with C-2 symmetrical diphenylmethyl diazocompounds high ee has been achieved (Eq. (75)) [263]. Insertion into aliphatic CH bonds was also efficient [264] (Eq. (76)) [265] and [266,267] (Eq. (77)).

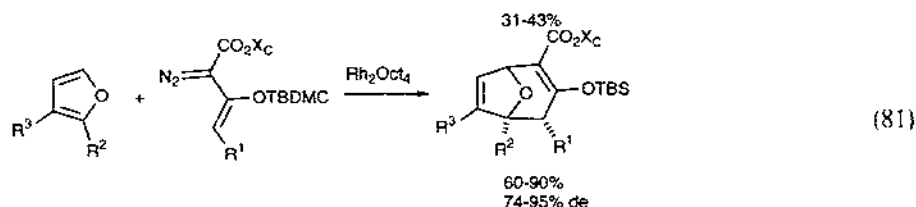
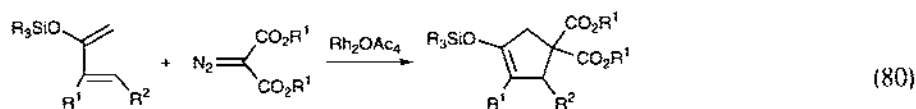
The effects of the ligand on rhodium(II) catalyzed cyclopropanation versus ylide chemistry has been studied [268]. Two interesting reactions proceeding via ylides are shown in Eq. (78) [269] and Eq. (79) [270].

Cascade processes of metallocarbenoids has been reviewed (320 references) [271].





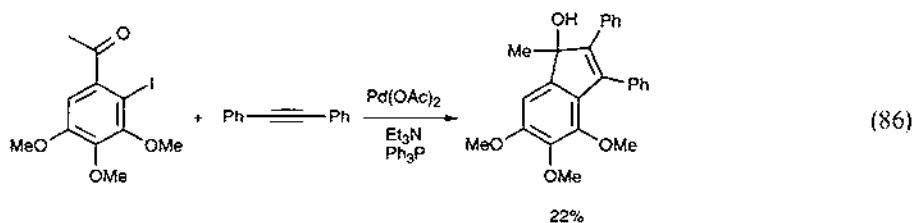
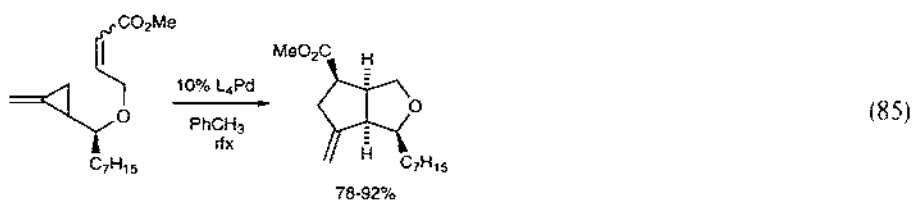
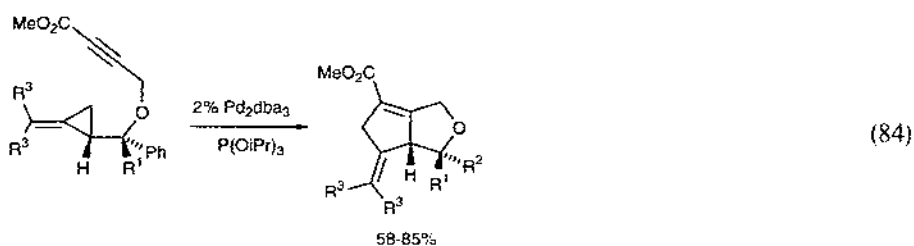
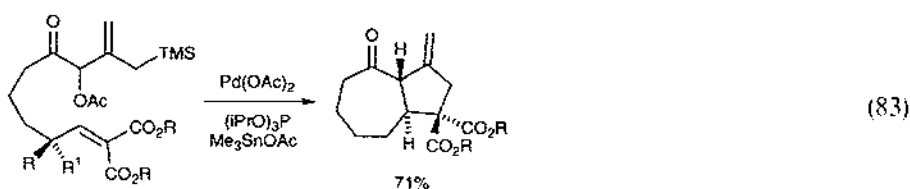
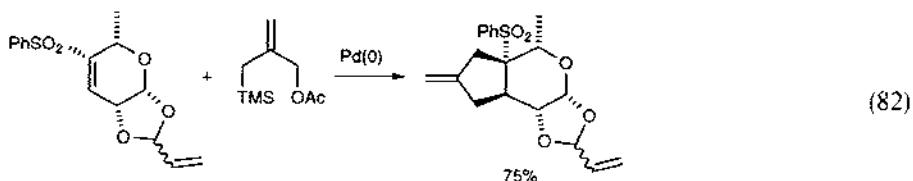
(Eq. (80) [272] and Eq. (81) [273]).



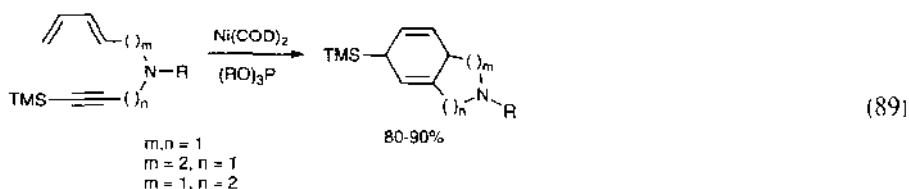
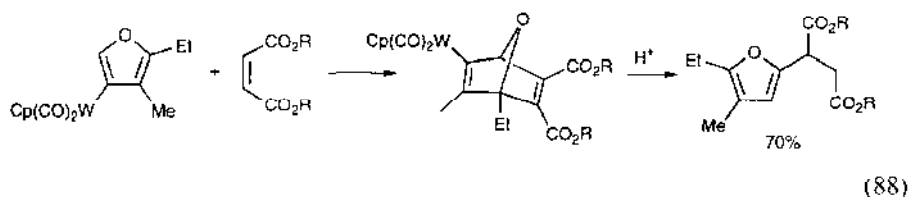
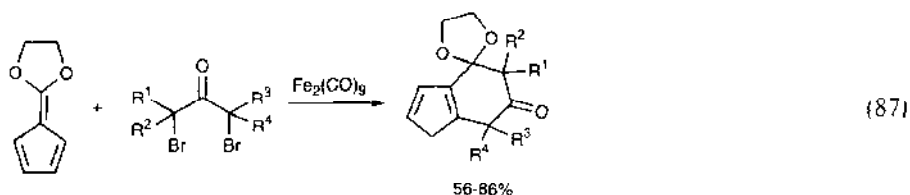
### 2.1.5. Cycloadditions

Transition metal mediated cycloaddition reactions have been reviewed (226 references) [274]. Palladium-catalyzed cycloadditions of trimethylenemethane moieties were efficient with dihydropyranes (Eq. (82)) [275,276] and intramolecularly (Eq. (83)) [277]. Palladium(0) complexes catalyzed the cycloaddition of methylene-cyclopropane-derived trimethylenemethanes to alkynes [278]. (Eq. (84)) [279] and

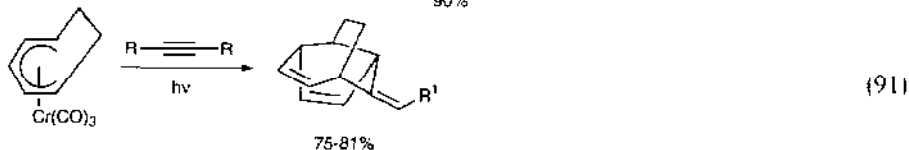
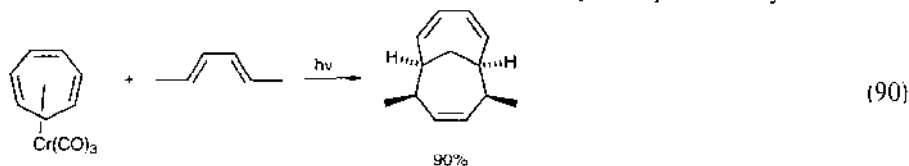
alkenes (Eq. (85)) [280]. Palladium(0) complexes catalyzed the cycloaddition of diphenylacetylene to *o*-iodoacetophenones (Eq. (86)) [281].

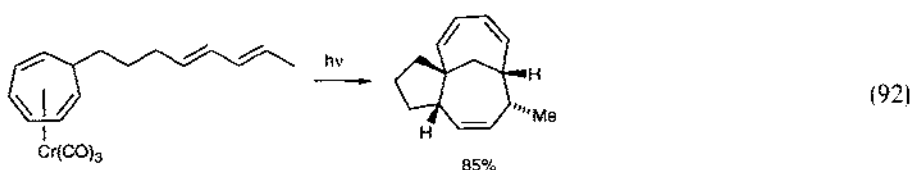


Iron carbonyl promoted an unusual 3+3 cycloaddition (Eq. (87)) [282]. Diels Alder reactions with iron tricarbonyl complexes of 1,4,6-hexatriene-3-ones went with high diastereoselectivity [283]. Similarly, Diels-Alder reaction with acrylic esters having a chromium-complexed arene pendent from the ester group proceeded with >95% *endo* selectivity versus 78:22 for the uncomplexed species [284]. A tungsten furan complex underwent cycloaddition with dienophiles (Eq. (88)) [285]. Nickel(0) complexes catalyzed the intramolecular 4+2 cycloaddition of a diene to an alkyne (Eq. (89)) [286].



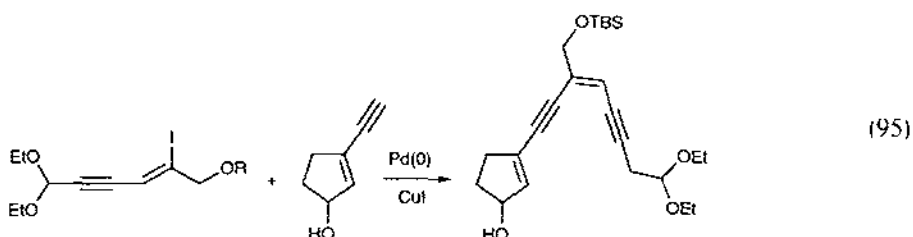
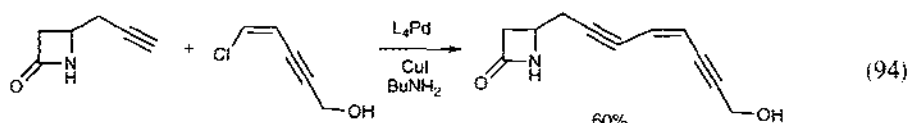
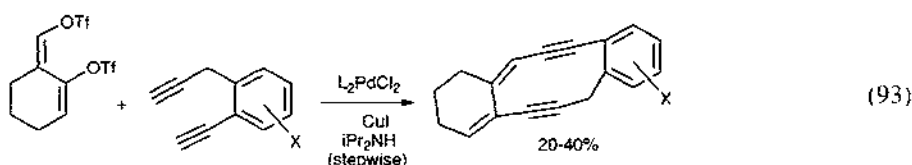
The [4+4] cycloaddition in natural product synthesis has been reviewed (126 references) [287]. Photochemical [6+4] cycloadditions [288], (Eq. (90)) [289], iterative [6+2] cycloadditions [290], (Eq. (91)) [291,292] and higher order cycloadditions (Eq. (92)) [293] all proceeded with chromium tricarbonyl complexes of cyclic trienes.



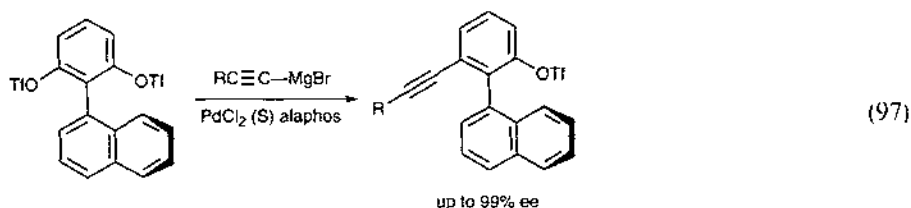
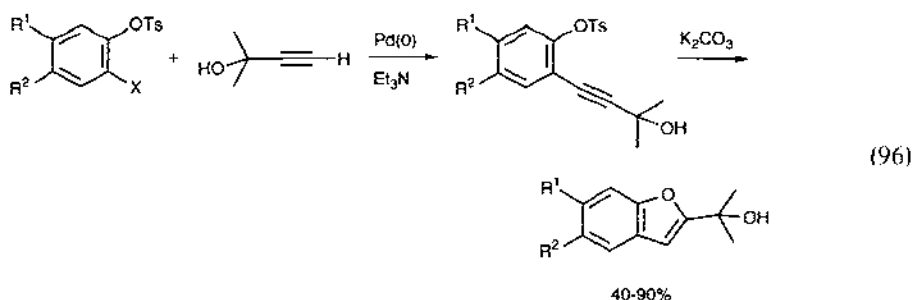


### 2.1.6. Alkylation of alkynes

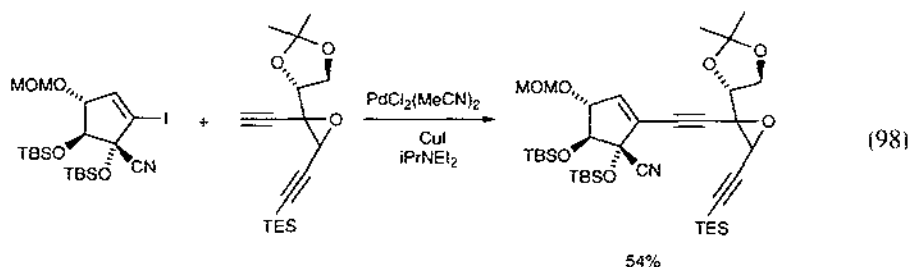
Much of the research involving alkyne alkylation is in the context of the synthesis of enediyne compounds and two reviews [294] (189 references) [295] have appeared on this subject. A full paper on the synthesis of Dynemicin A, involving palladium-catalyzed alkylation of alkynes, has appeared [296]. A number of studies involving the palladium-catalyzed alkylation of alkynes by the *bis*-triflate shown in (Eq. (93)) have appeared [297–300]. Other ene–diyne syntheses are shown in Eq. (94) [301] and Eq. (95) [302]. Conditions for the palladium-catalyzed coupling of terminal alkynes with aryl halides in the absence of copper [303] and under aqueous conditions [304] have been developed.



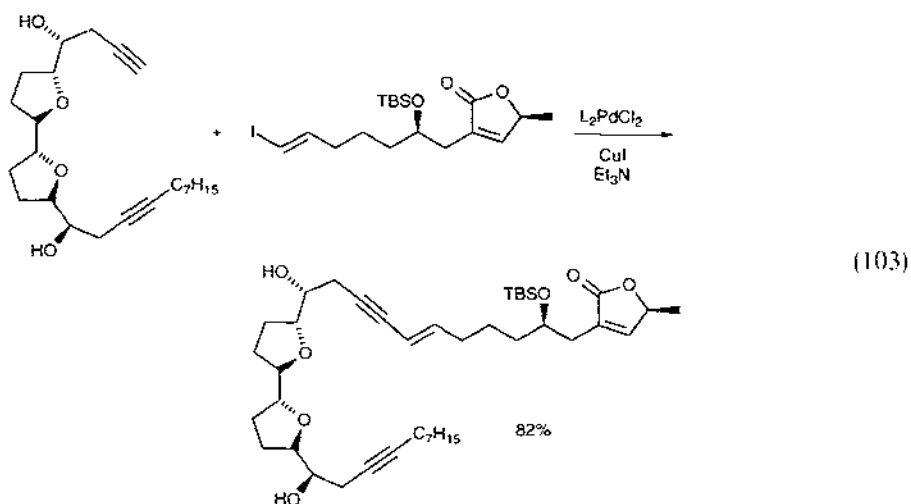
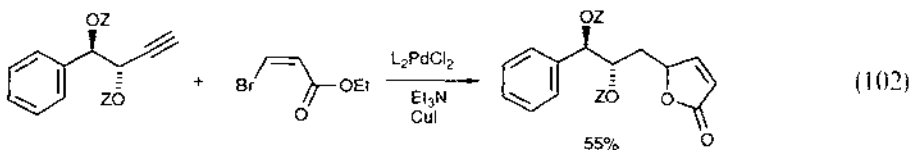
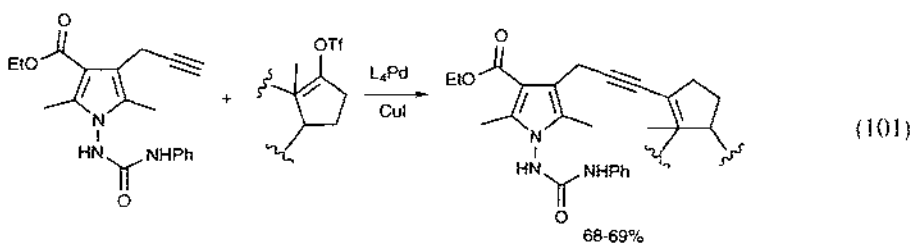
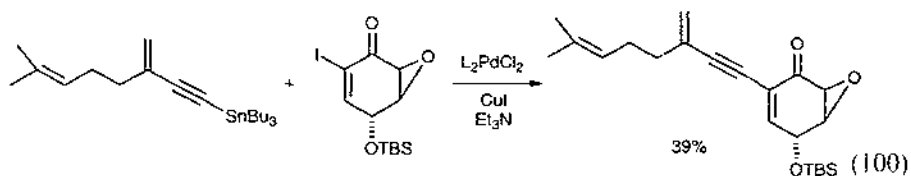
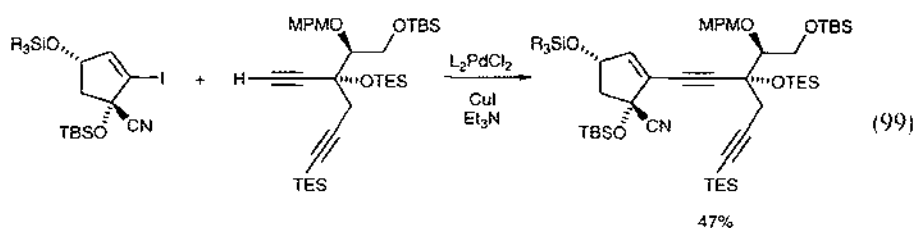
A palladium/copper catalyst was used to couple terminal alkynes to *o*-haloaniline amides [305,306], and dimethoxy iodoarenes [307], and 2-bromo-4,5-methylenedioxobenzaldehydes [308]. It has been used in the synthesis of benzofurans (Eq. (96)) [309] and optically active biaryl systems (Eq. (97)) [310].



Palladium/copper systems catalyzed a large number of alkyne/alkenyl halide coupling reactions. Propargyl alcohols were coupled to *cis* and *trans* 1,2-dichloroethylene [311],  $\alpha$ -bromocinnamaldehydes with acetylenic alcohols [312], chloroallyl amines with terminal alkynes [313], 4,7-diene-1-yne with functionalized alkenyl bromides [314] and propargyl alcohols with alkenyl iodides [315]. Propargyl and homopropargyl epoxides coupled to 4-*t*-butylcyclohexanone triflate [316] and highly functionalized alkenyl iodides (Eq. (98)) [317]. Other useful palladium/copper catalyzed alkylations of alkynes are seen in Eq. (99) [318], Eq. (100) [319,320] and Eq. (101) [321]. The reaction has been used to make butenolides (Eq. (102)) [322] and to incorporate acetylenic side chains into butenolides (Eq. (103)) [323,324].

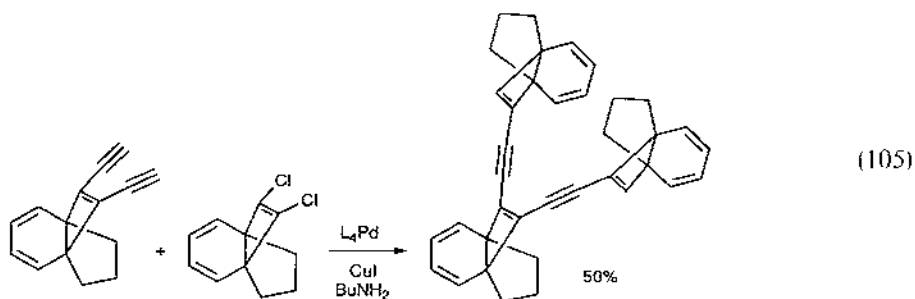
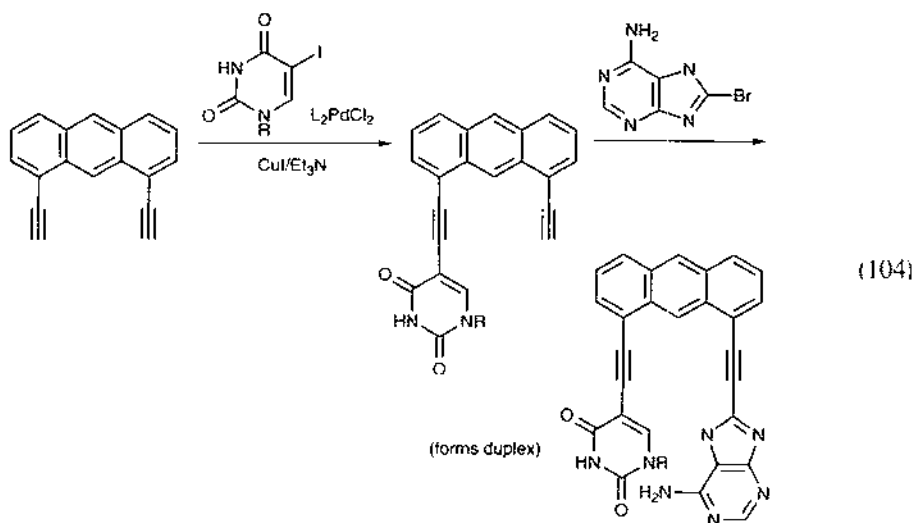


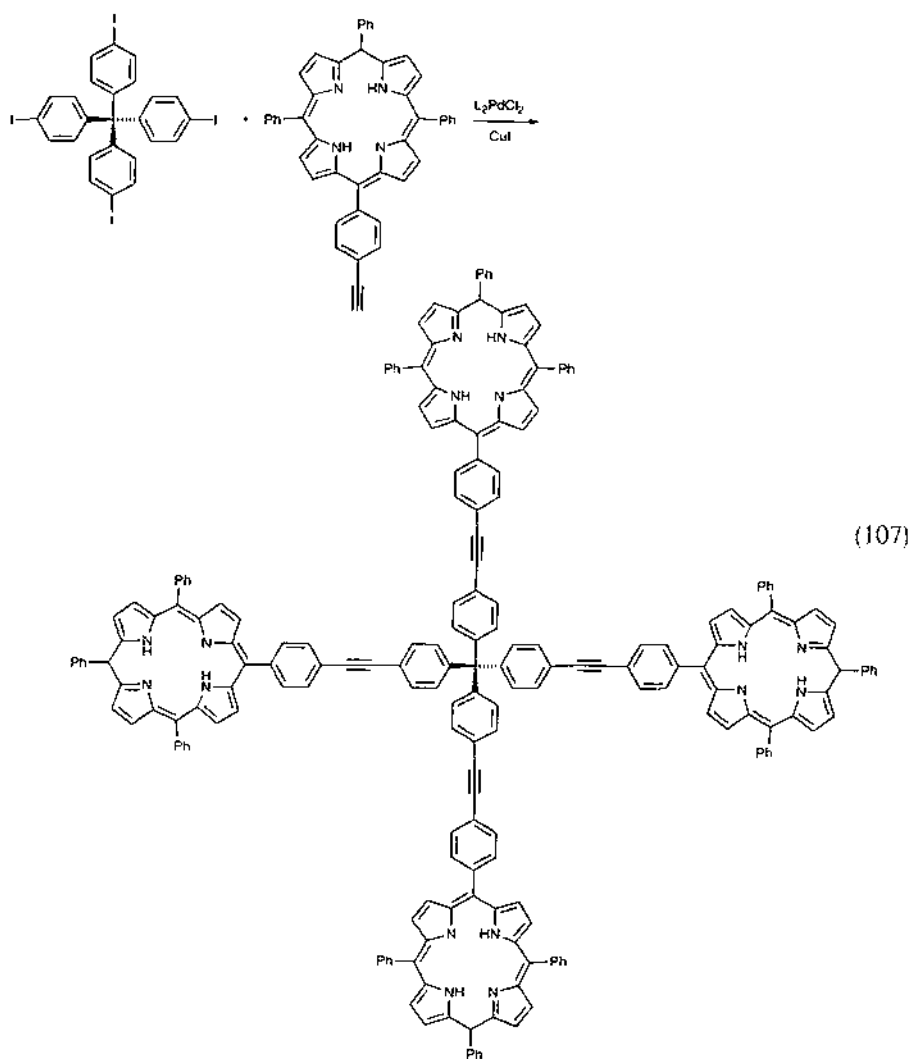
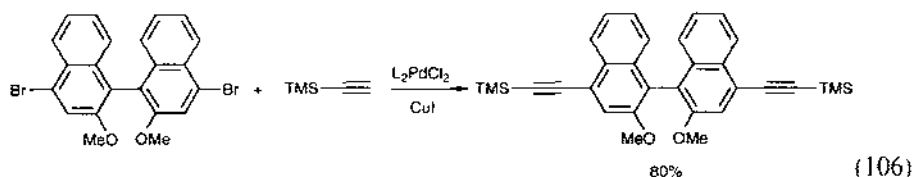
Palladium/copper systems were used to catalyze the coupling of alkynes with heteroaromatic halides including 2-chloropyridines [325], iodopurine [326], and pyrimidine nucleosides [327], and iodoimidazoles [328]. Phenyl acetylenes having long chain phenol ethers *para* were coupled to 2-iodo-6-bromopyridazines at the



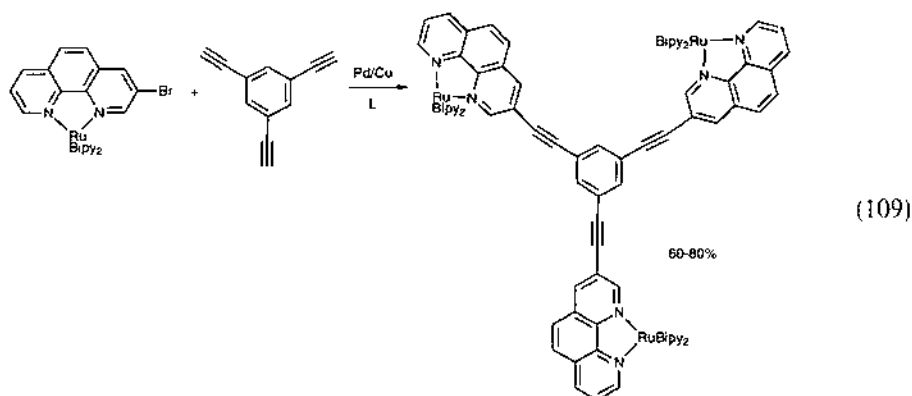
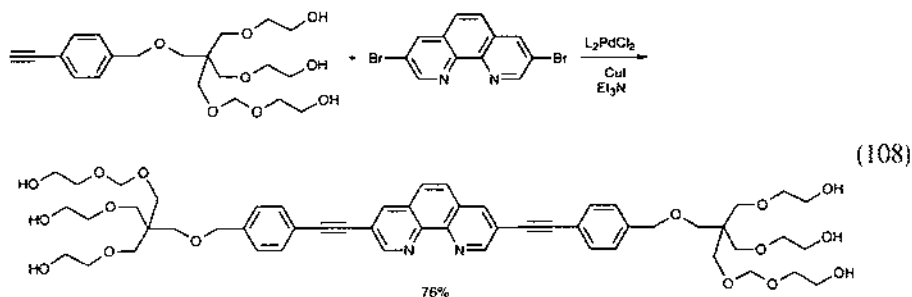
iodo position under palladium/copper catalyst and subsequently Suzuki coupled at the bromo position with similar aryl boronic acids [329]. Boc-protected *m*-iodoaniline coupled to trimethylsilyl acetylene using palladium catalysis [330]. Alkyne-functionalized oligopyridine building blocks were synthesized by palladium/copper catalyzed coupling of alkynes to bromopyridines [331].

Ortho diiodobenzene [332] and *o*-quinone *bis* triflates [333] were *bis*-alkynylated by terminal alkynes in the presence of palladium/copper catalysts as was 4,5-dibromotriazine [334]. Two procedures for the “orthogonal” coupling of alkynes to aryl halides have been developed [335,336]. Polybrominated 2,2'-*bis* thiophenes [337] and phenanthrenes containing long chain alkyl ethers for use as the tail in triphenylene discotic liquid crystals [338] were polyalkynylated by terminal alkynes and palladium/copper catalysts. Acetylene was *bis*-arylated under similar conditions [339]. Other multiple couplings are shown in Eq. (104) [340], Eq. (105) [341], Eq. (106) [342,343], Eq. (107) [344], Eq. (108) [345] and Eq. (109) [346].





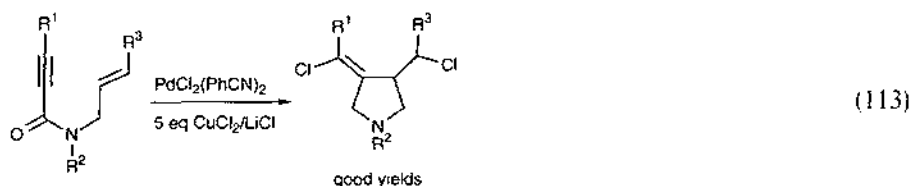
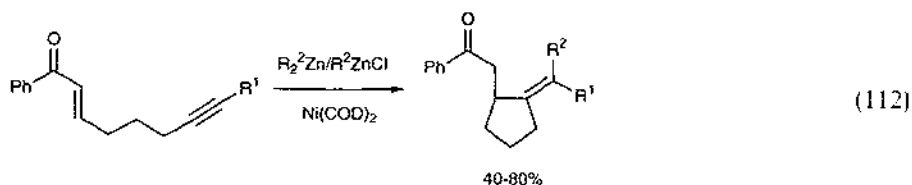
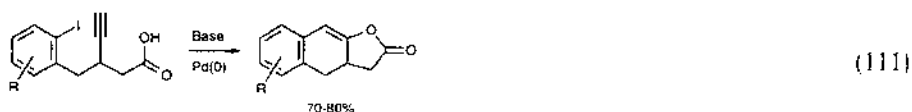
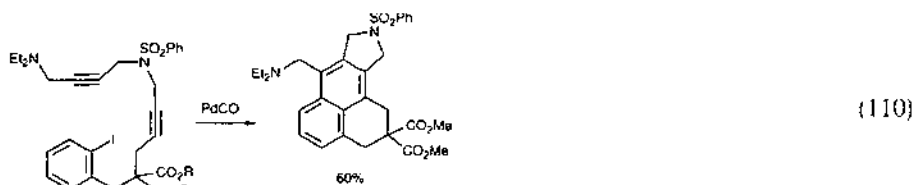
Linear amphipathic porphyrin dimers and trimers were synthesized by coupling porphyrins with pendant aryl halides with those having terminal alkyne groups [347]. A solid phase synthesis of phenylacetylene oligomers using palladium/copper catalyzed alkyne/alkenyl halide coupling has been developed [348]. This has also



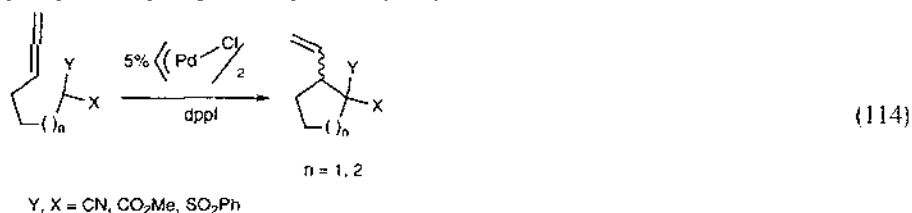
been accomplished in solution using alkyne/aryl iodide coupling [349]. Oligomeric optically active BINAPS connected by phenylacetylene linkages were made by palladium/copper catalyzed coupling of *p*-diiodobenzene with *bis* phenylacetylene BINAP [350], and 6,6'-dibromo BINAPs linked at the 2,2'-positions by a polyethylene glycol group, with 1,4-*bis* ethynyl-2,5-bis(C<sub>18</sub> ether) benzene [351]. Terminal alkynes were dimerized by reaction with allyl bromide and Pd<sub>2</sub>dba<sub>3</sub> in the presence of 50% NaOH [352]. Chromium tricarbonyl complexed chlorobenzene was coupled to trimethylsilylacetylene using palladium/copper catalysis [353]. A related catalysis coupled 1-bromo-2-trimethylsilyl ethyne to a terminal alkyne pendant from a cyclopropanated fullerene [354].

A full paper entitled "Palladium-catalyzed tandem cyclization anion capture — part 2. Cyclization onto alkynes and allenes with hydride capture" has appeared [355]. Related examples are seen in Eq. (110) [356] and Eq. (111) [357]. Nickel(0) complexes catalyzed the alkylative cyclization of alkynes onto enones (Eq. (112)) [358]. Palladium/copper catalyst systems cyclized enynes via chloropalladation (Eq. (113)) [359,360]. Iodoferrocene was coupled to phenylacetylene dimer using palladium/copper catalysts [361].

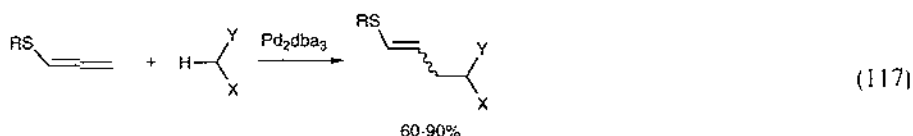
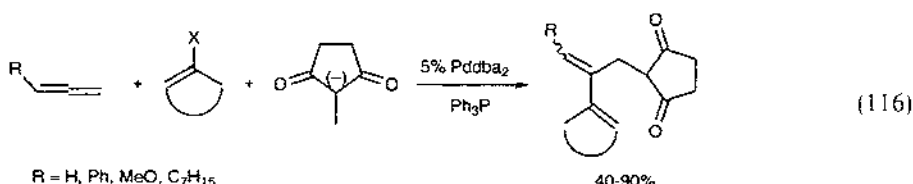
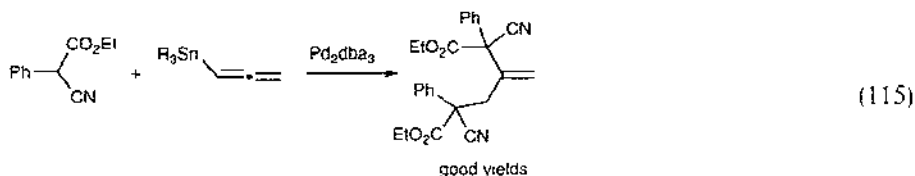
Alkynes were 1,2-*bis* arylated by aryl trifuryltins using palladium/copper catalysts [362]. Palladium(0) catalyzed the *cis*-addition of aryl cyanides to aryl ethenes placing the cyano group on the arene-bearing carbon [363]. Palladium(II) catalyzed the 1,2-addition of a halogen and an enone (Michael addition) across an alkyne [364].



A full paper on the mechanism of zirconium-catalyzed carboalumination of alkynes has appeared [365]. Manganese(II) iodide catalyzed the 1,2-addition of allyl Grignards to alkynes [366]. The resulting alkenylmagnesium halide was quenched with electrophiles. Palladium-catalyzed hydrocarbonation of allenes has been reviewed [367]. Variations of this process are seen in Eq. (114) [368]. Eq. (115) [369]. Eq. (116) [370] and Eq. (117) [371].



Palladium catalyzed the arylation of terminal alkynes by aryl iodonium salts [372], terminal propargyl alcohols by heteroaromatic halides [373] and by  $\alpha$ -bromo- $\beta$ -(phenyltellurium) styrene at the bromo position [374]. Copper iodide in pyrrolidones couple terminal alkynes with alkynyl iodides [375]. Palladium catalysts were required for alkynyl bromides.

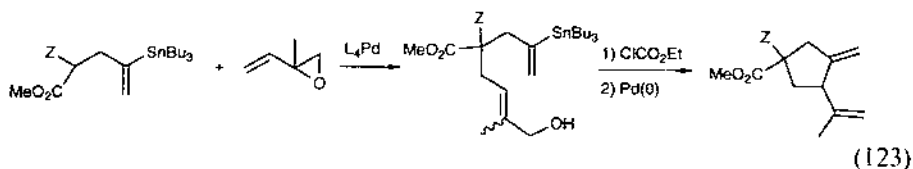
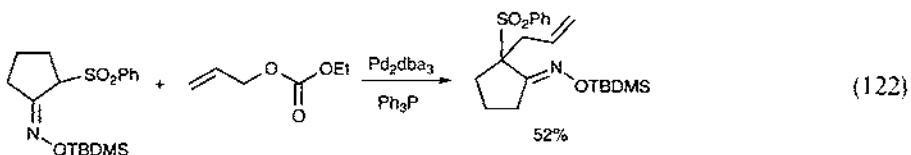
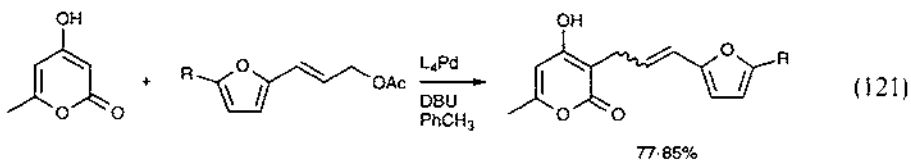
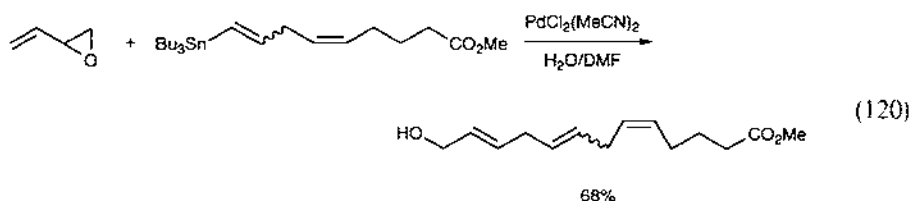
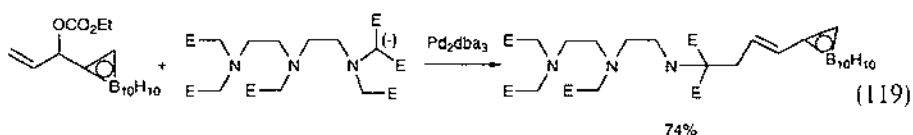
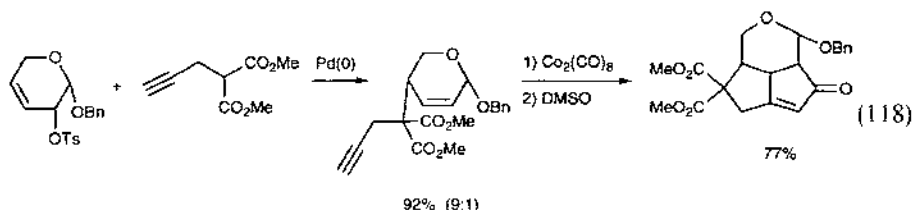


### 2.1.7. Alkylation of allyl, propargyl and allenyl systems

Palladium-catalyzed asymmetric allylic alkylation remains a growth industry with many new ligand systems for the asymmetric alkylation of racemic 3-acetoxy-1,3-diphenylpropene being reported [376–383]. Strangely, this system could already be alkylated with greater than 99% ee and would seem not to demand so much continuing attention. Palladium-catalyzed asymmetric allylic alkylation has been reviewed a number of times [384] (183 references), [385] (47 references), [386] (30 references), [387]. A careful study showed that the intermediate for this process need not be symmetrical [388]. Diphenyl imines of  $\alpha$ -acetoxy- $\alpha$ -amino acid esters were alkylated with low ee [389]. Using palladium catalyst, asymmetry could be induced at the  $\alpha$ -position of the stabilized carbanion used in alkylation of allyl acetates as long as a quaternary center was formed to preclude epimerization of the product [390, 391].

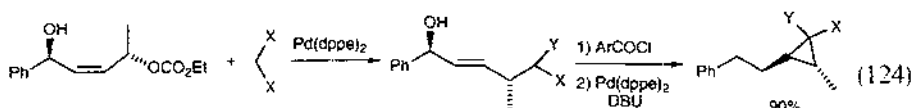
A supported aqueous-phase catalyst consisting of palladium acetate and water-soluble phosphine ligands on porous silica catalyzed the alkylation of cinnamyl carbonates by acetoacetates [392]. Allyl alcohols were treated with ethyl chloroformate then nitromethane and a palladium catalyst to alkylate the thus formed allyl carbonate [393]. The palladium-catalyzed alkylation of allyl acetates by the anion of 2-methyl-1,3-cyclopentanedione was reversible [394]. Intermediates for carbocyclic nucleoside analogs were made by palladium-catalyzed alkylation of 3-acetoxycyclopentenones [395, 396]. Carbohydrate-related systems also underwent efficient palladium-catalyzed allylic alkylation (Eq. (118)) [397, 398]. 1,4-Diacetoxy-2-triethylsilyl-2-butene underwent palladium-catalyzed allylic alkylation  $\alpha$  to the silyl group [399]. Quite complex systems were allylically alkylated, see Eq. (119) [400], Eq. (120) [401], Eq. (121) [402], Eq. (122) [403] and Eq. (123) [404]. Palladium(0) catalyzed the alkylation of allyl carbonates by allyl tins with loss of allyl

stereochemistry [405].

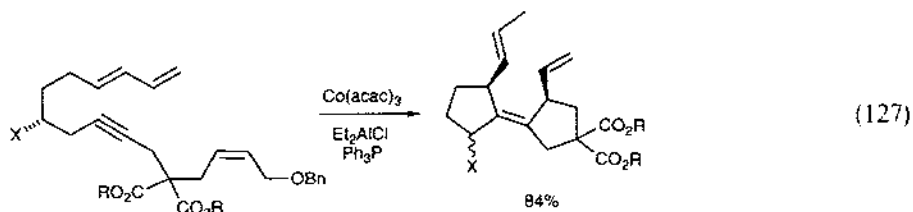
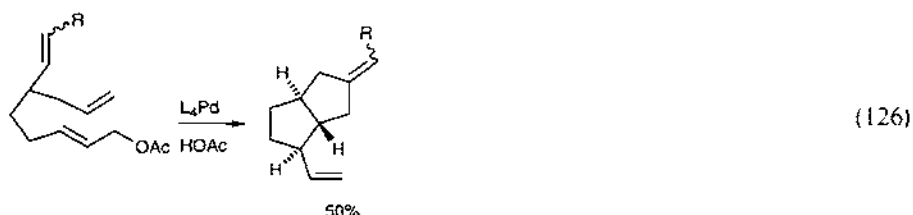
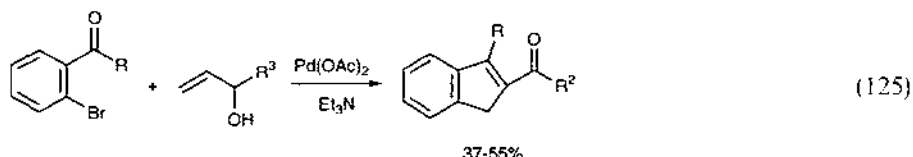


Cyclopropanes were synthesized by the palladium-catalyzed alkylation of 1,4-dichloro-2-butene with the anion of the diphenyl imine of  $\alpha$ -amino acetonitrile.

in a two-step allylic alkylation [406,407]. An example is shown in Eq. (124) [408,409].



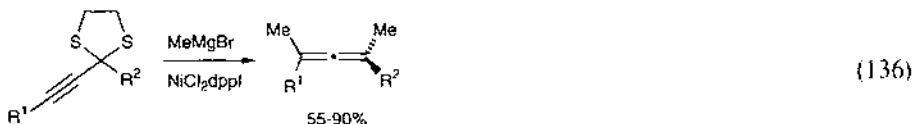
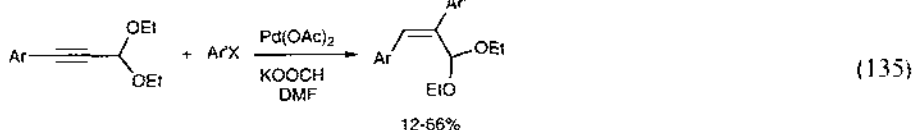
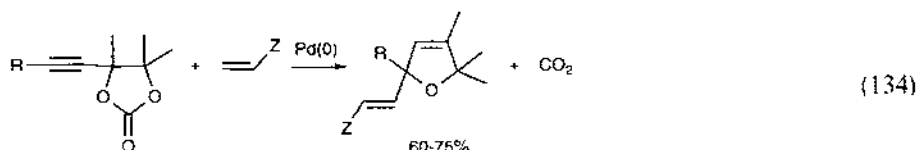
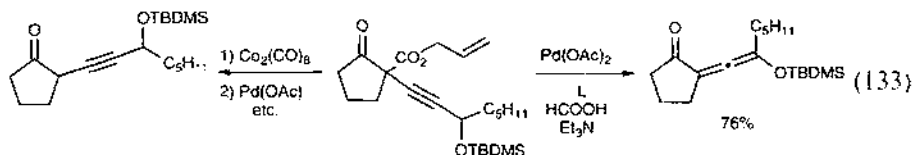
Enol ethers directly alkylated allyl alcohols in the presence of palladium catalyst and trifluoroacetic acid [410].  $\alpha$ -Naphthol underwent polyallylation by allyl alcohols in the presence of palladium(0) catalysts [411]. The alkene portion of allyl alcohols was arylated by arylodonium salts in the presence of palladium(0) catalysts, giving the arylated allylic alcohol, rather than the ketone from  $\beta$ -elimination into the alcohol group [412]. Palladium also catalyzed cascade polycyclization sequences of allyl systems (Eq. (125)) [413] and Eq. (126)) [414], as did reduced cobalt species (Eq. (127)) [415].



Ruthenium complexes catalyzed the alkylation of allyl alcohol by terminal alkynes (Eq. (128)) [416], while nickel phosphine complexes catalyzed the alkylation of allyl carbonates by aryl and alkenyl boronates [417] and the ring-opening of cyclic allyl ethers (Eq. (129)) [418]. Phenyl copper in the presence of  $\text{BF}_3$  performed an  $\text{Sn}2'$  ( $\delta$ ) displacement of chiral ketals of diene aldehydes [419]. Butylcopper effected  $\text{Sn}2'$  displacement of chiral sulfonamides with good ee [420]. The chiral zirconium

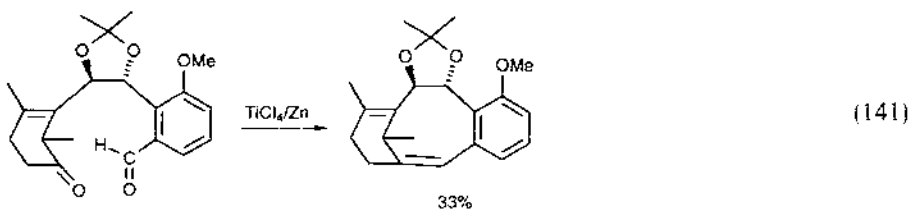
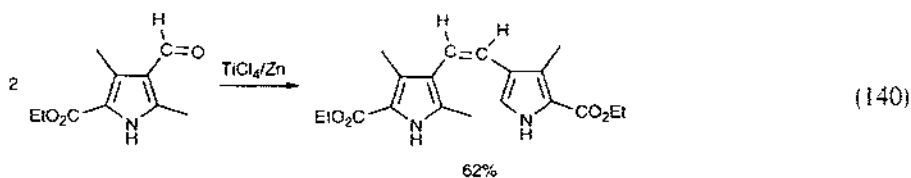
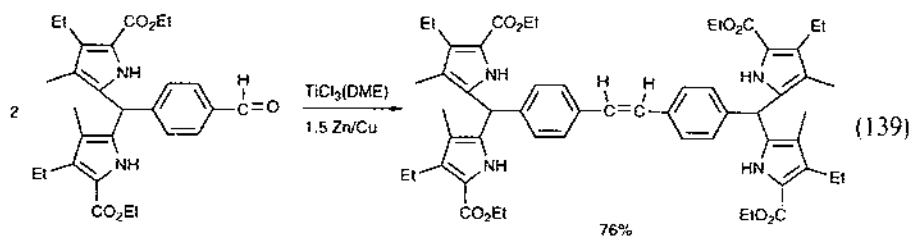
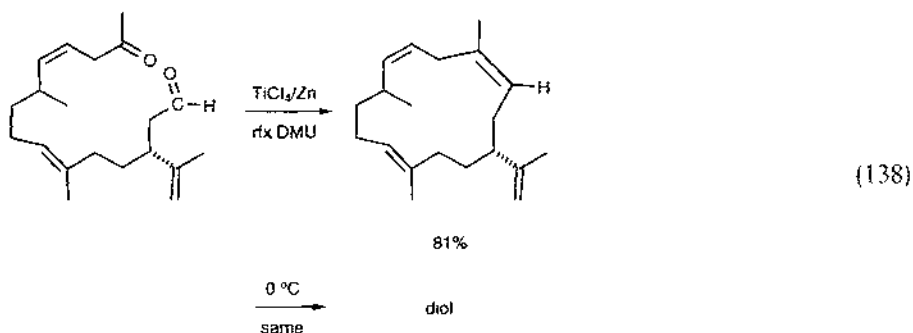
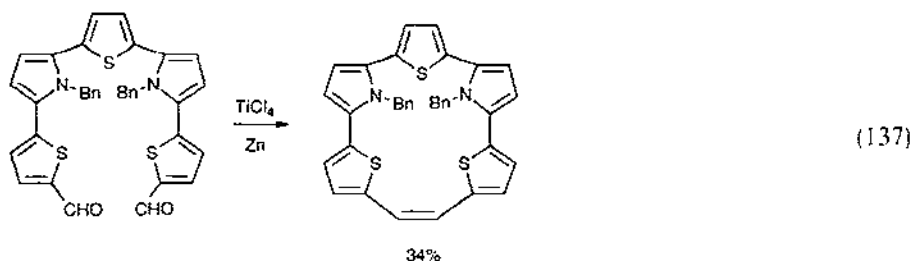


during palladium-catalyzed alloc deprotection was efficient (Eq. (133)) [427]. Palladium catalyzed the alkylation of propargyl carbonates by indole-2-borates [428]. The allene ( $\text{Sn}2'$ ) was the major product with  $\text{Pd}(\text{dba})_2$  catalysts while the alkyne resulted with  $\text{L}_4\text{Pd}$  catalysis. Palladium(0) complexes catalyzed the alkylation of 1,3-enynes by stabilized anions at the terminal olefin position to give allenes [429]. Propargyl carbonates were transformed into dihydrofurans (Eq. (134)) [430] and propargyl ketals were  $\alpha$  alkylated (Eq. (135)) [431] using palladium catalysts. Copper(I) cyanide catalyzed the alkylation of propargyl bromide by  $\alpha$ -( $\text{Cp}_2\text{ZrCl}$ ) alkoxyboranes to give allenyl(alkoxy)boranes [432]. Nickel(II) phosphine complexes catalyzed the conversion of propargyl dithianes to allenes by methyl magnesium bromide (Eq. (136)) [433].



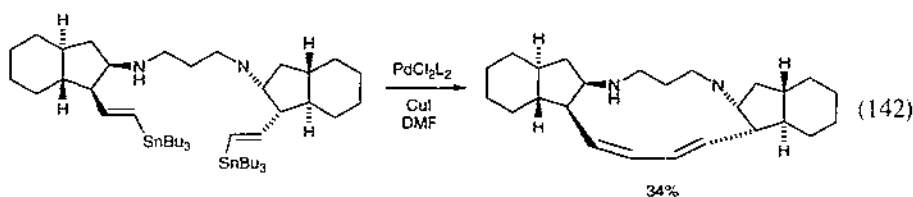
### 2.1.8. Coupling reactions

Papers dealing with the effect of external ligands on McMurry coupling [434], a review (142 references) on new developments in the chemistry of low-valent titanium [435], and a paper on the use of electrochemically generated titanium nanoclusters for McMurry coupling [436] have all appeared. *Cis*-stilbene-3,3'-dialdehyde was cyclocoupled at both aldehydes to give the cyclic tetraene by  $\text{TiCl}_4/\text{Zn}$  [437]. Nickel porphyrins with a peripheral aldehyde group were dimerized through this group by McMurry coupling [438]. McMurry cyclocoupling was also effective (Eq. (137)) [439] and Eq. (138)) [440]. McMurry coupling was used to make calix[4]arene derivatives by aldehyde coupling across the ring [441]. Other highly functionalized McMurry couplings are seen in Eq. (139) [442] and Eq. (140) [443].  $\text{Cp}_2\text{TiCl}(\text{THF})$  reductively coupled aldehydes to give diols [444] as did  $\text{TiCl}_4/\text{Zn}$  (Eq. (141)) [445] although samarium iodide was more efficient.

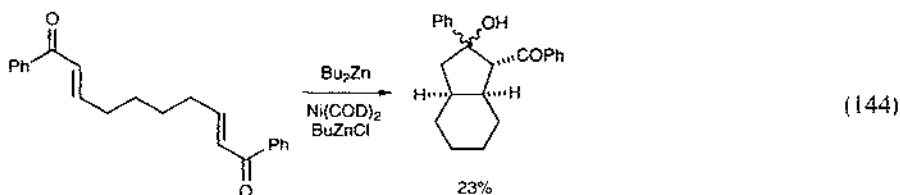
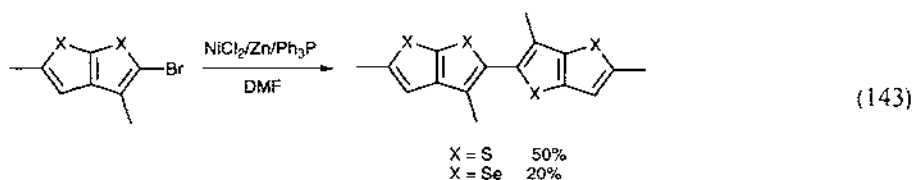


Excess copper(I) chloride in DMF coupled vinyl stannanes to give dienes both inter- [446] and intramolecularly [447] (Eq. (142)) [448]. Hydrozirconation of alky-

nyl boranes followed by copper(I) catalyzed coupling produced 2,3-diborane containing 1,3-dienes [449]

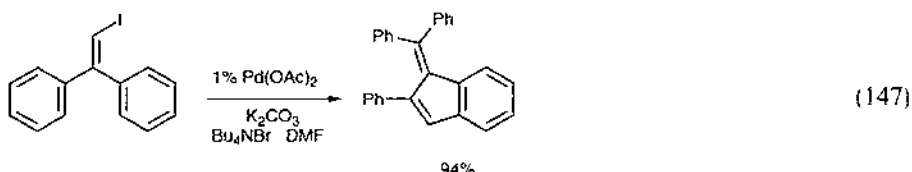
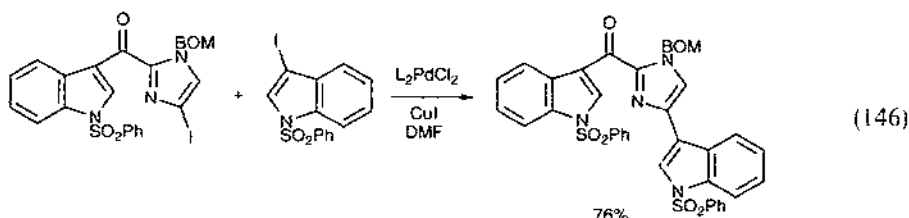
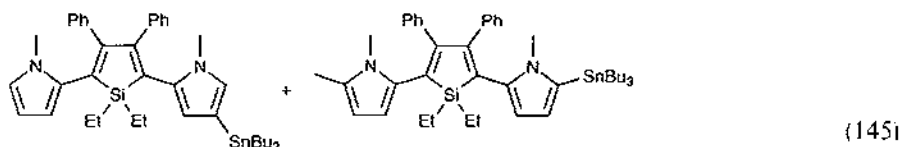


Nickel(0) catalysts coupled *o*-bis-(dibromomethyl)benzenes to produce dibromobenzocyclobutanes [450], 2-chloropyridines to give 2,2'-dipyridines [451] and 2-bromothio and selenophanes to give dimers (Eq. (143)) [452]. Nickel complexes also catalyzed the intramolecular coupling of *bis*-enones (Eq. (144)) [453] and the intermolecular coupling of  $\alpha$ -sulfonyl allyl Grignard reagents to give trienes [454].



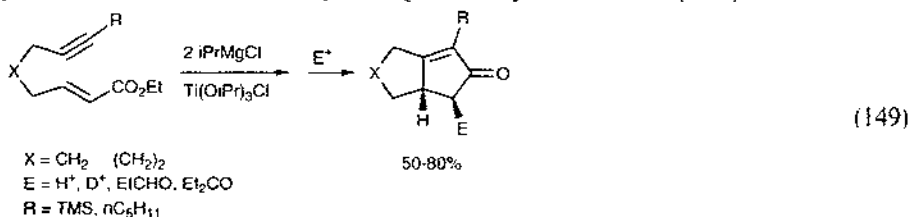
The mechanism of Suzuki self-coupling of arylboronic acids has been studied [455] and used to make biaryls and BINAPs [456]. Palladium chlorides coupled alkenylmercuric halides [457] and cross coupled heteroaryl stannanes (Eq. (145)) [458]. Palladium/copper systems coupled 2-lithiated dihydropyrans [459], heteroaryl-halides (Eq. (146)) [460] and alkenyl bromides [461] (Eq. (147)) [462]. Palladium acetate/phosphine systems coupled styrene oxides (Eq. (148)) [463] and head-to-tail dimerized terminal alkynes to give enynes [464].

Reduced titanocenes cyclocoupled dienes to give titanacyclopentanes [465] and enynes to give titanacyclopentenes [466] which could be further elaborated (Eq. (149)) [467]. Imines with remote alkenes could be similarly cyclized to give amines with a carbocyclic alkyl group [468]. 3,4-Dilithio-2,5-dimethylhexa-2,4-diene was converted to 2,5-dimethyl-1,3,4-hexatriene by treatment with  $\text{Cp}_2\text{TiCl}_2$  [469].



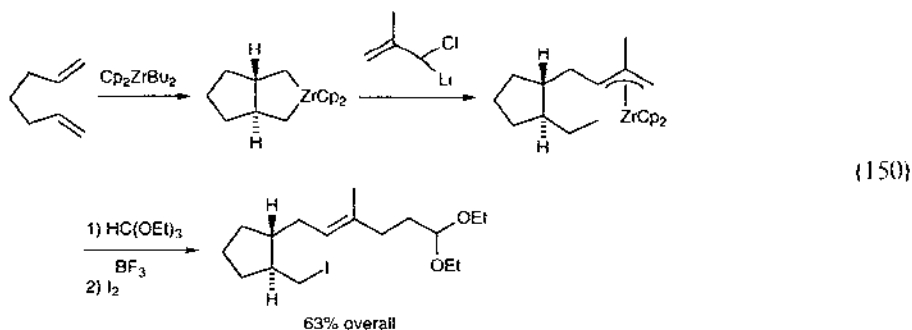
Ar = Ph, pMePh, pFPh, pMeOPh, oMeOPh

Reduced zirconocenes similarly coupled dienes (Eq. (150)) [470] and alkenes with alkynes [471]. Samarium iodide coupled the aldehydes of chromium tricarbonyl complexes of *o*-bromobenzaldehyde to give the symmetric diol [472].

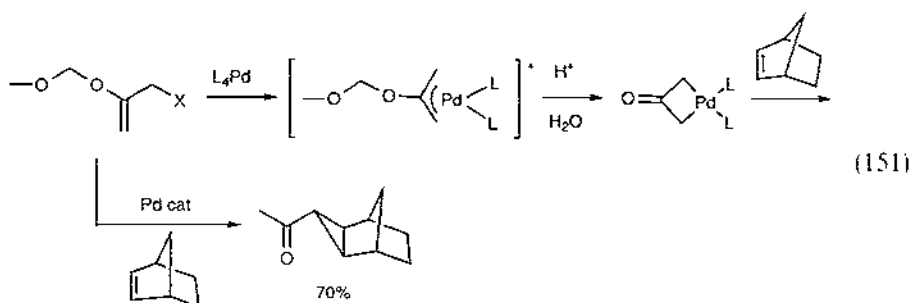


### 2.1.9. Alkylation of $\pi$ -allyl complexes

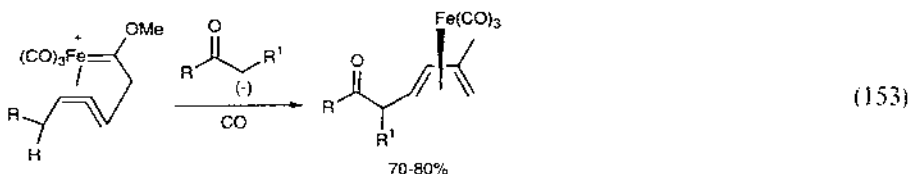
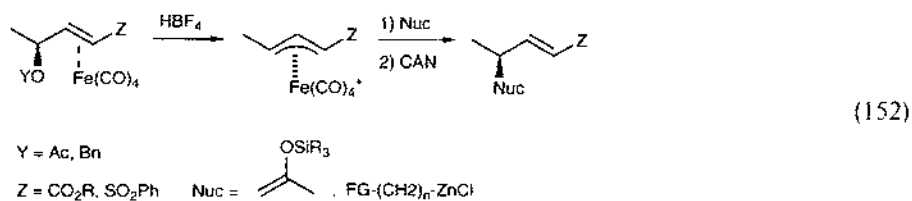
Theoretical papers dealing with the effects of auxiliary ligands on the regiochemistry of nucleophilic addition to  $\pi$ -allylpalladium complexes [473] and on palladium-carbon bonding in  $\pi$ -allyl-palladium complexes [474] have appeared. The origin



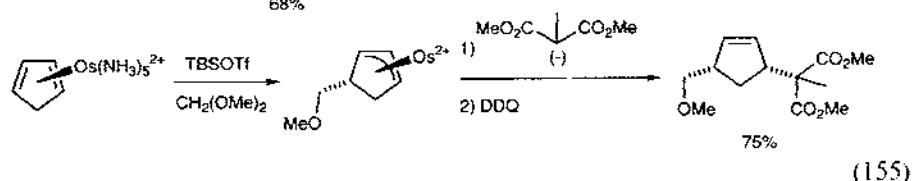
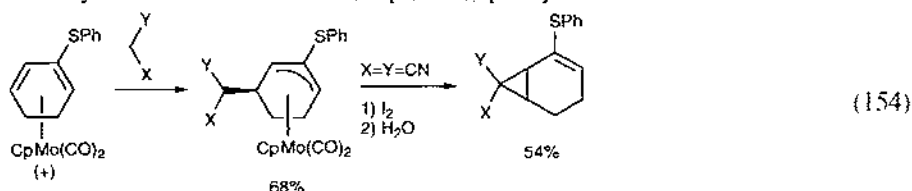
of enantioselectivity in the alkylation of  $\pi$ -allylpalladium (*S*)BINAP complexes was derived from an X-ray crystal structure of the complex [475]. 2-Oxoallyl palladium complexes reacted with norbornene to give cyclopropanes (Eq. (151)) [476].



A review entitled “New Applications of Chiral *N*-Acyliminium Precursors” (15 references), has appeared and deals with cationic iron  $\pi$ -allyl complexes [477]. These undergo alkylation with a high degree of regio- and stereocontrol (Eq. (152)) [478–480]. Enolates alkylated  $\pi$ -allyl iron carbene complexes (Eq. (153)) [481–483].

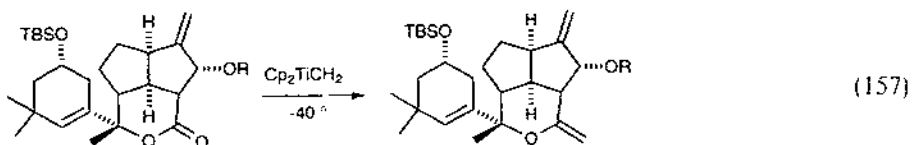
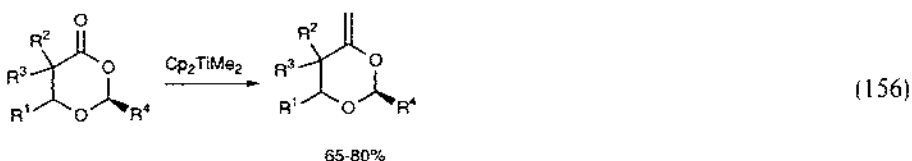


$\pi$ -Allylmolybdenum complexes, formed by nucleophilic addition to cationic 1,3-diene complexes, underwent nucleophilic attack under oxidizing conditions (Eq. (154)) [484,485]. Cationic osmium and ruthenium  $\pi$ -allyl complexes also were alkylated by stabilized carbanions (Eq. (155)) [486].

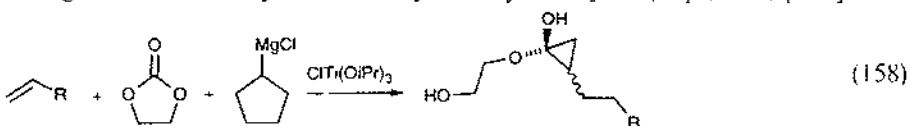


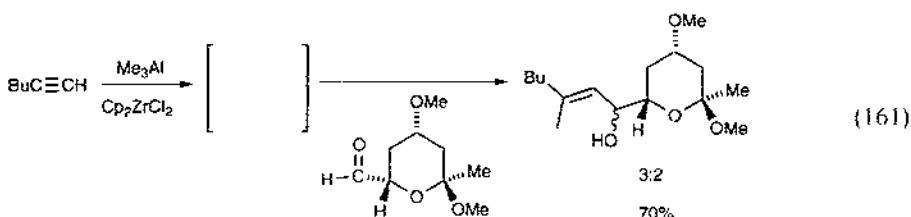
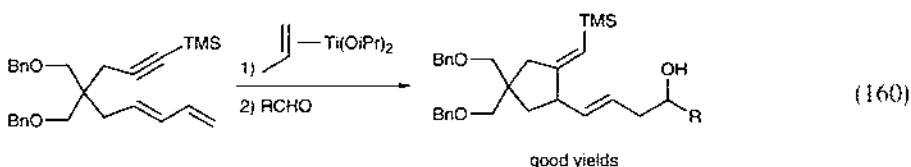
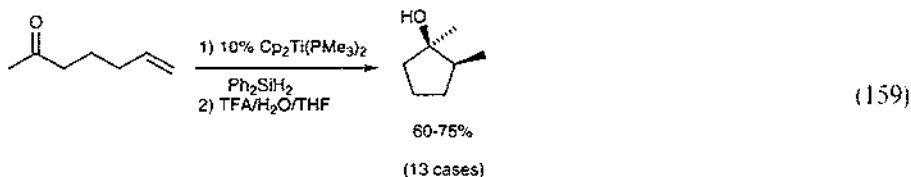
#### 2.1.10. Alkylation of carbonyl compounds

Petasis reagent ( $\text{Cp}_2\text{TiMe}_2$ ) methylenated  $\beta$ -lactones [487] and six-membered lactones (Eq. (156)) [488]. Tebbe's reagent was used to methylenate more complex systems (Eq. (157)) [489].

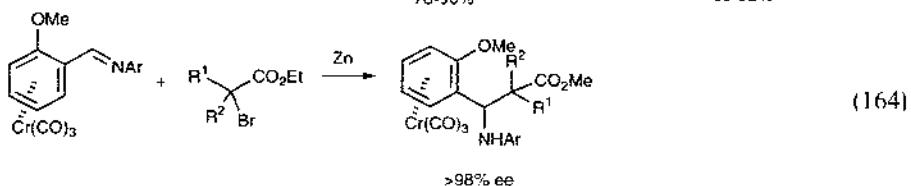
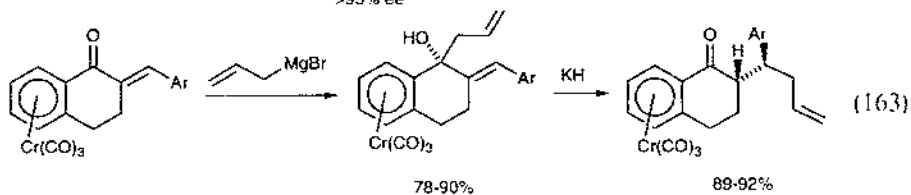
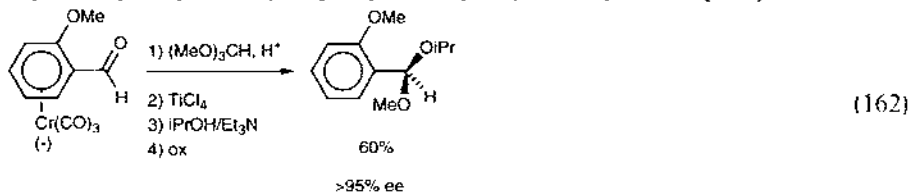


Titanium isopropoxide reacted with Grignard reagents followed by esters to give cyclopropanols (Eq. (158)) [490,491].  $\omega$ -Unsaturated ketones cyclized when treated with reduced titanium species (Eq. (159)) [492] [[493] for alkynes]. Dienes [494] and dienyynes [495] were cyclized then alkylated with aldehydes by reduced titanium species (Eq. (160)). Allenes [496] and alkynes were hydrozirconated or carbometalated using zirconium catalysts, then alkylated by aldehydes (Eq. (161)) [497].

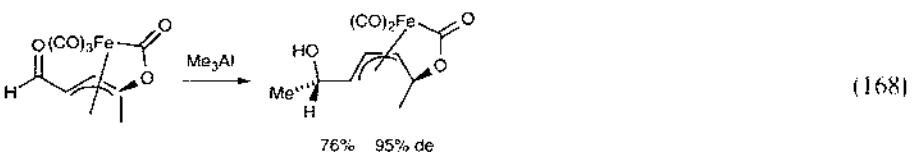
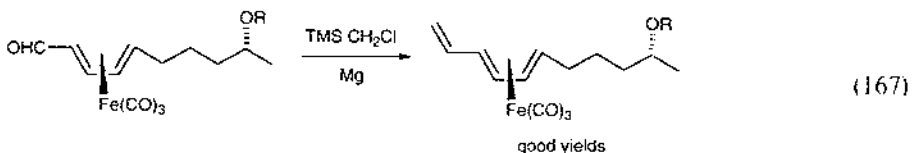
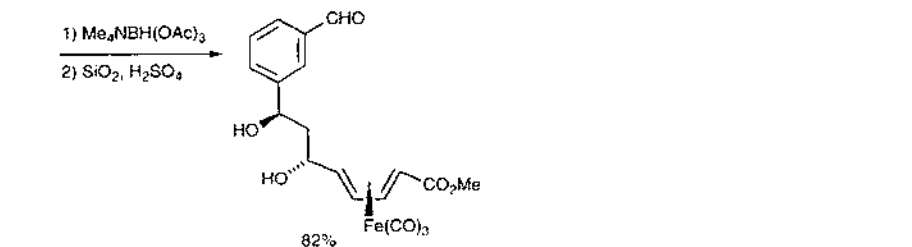
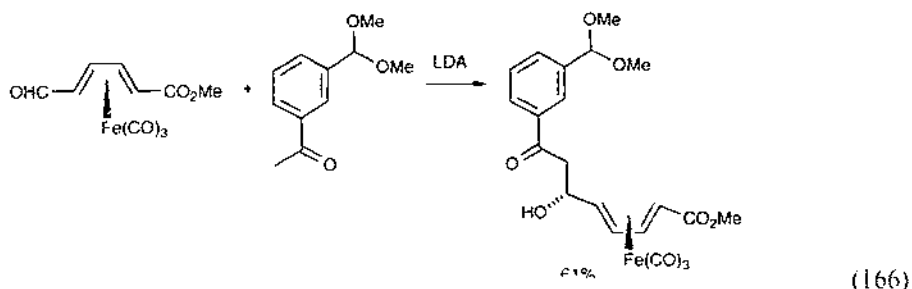
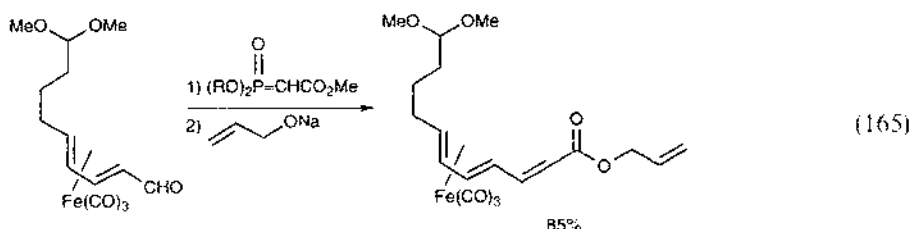




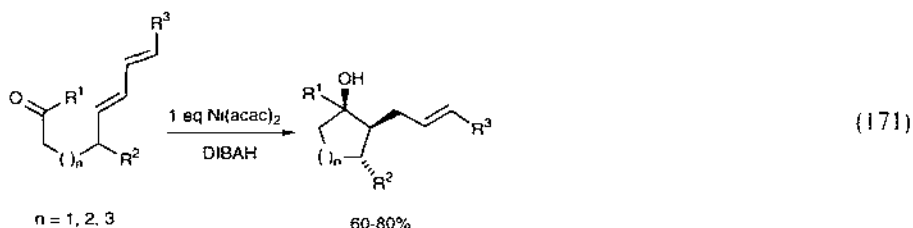
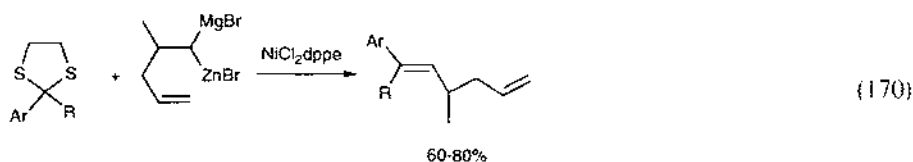
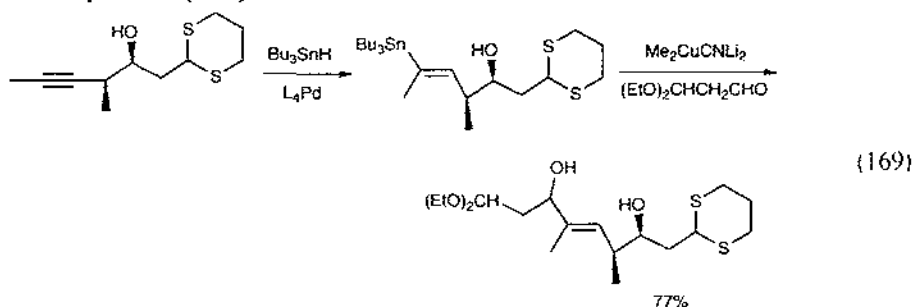
Chromium arene complexes having carbonyl groups were alkylated with high stereospecificity (Eq. (162) [498], Eq. (163) [499] and Eq. (164)) [500].



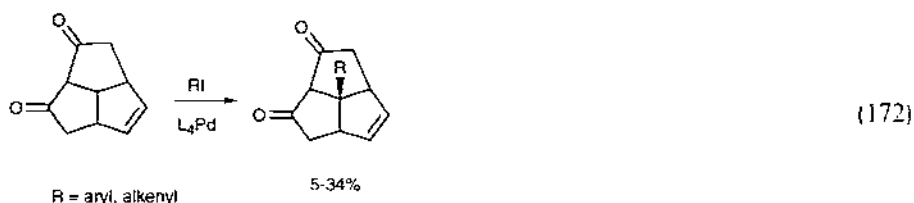
The complexation of dienals to iron tricarbonyl fragments allows normal aldehyde chemistry to be carried out on this usually over-reactive aldehyde. This includes Wittig reaction chemistry (Eq. (165)) [501,502], aldol chemistry (Eq. (166)) [503], alkylation by dialkyl zinc reagents in the presence of optically active aminoalcohols (98% ee) [504] and Peterson olefination (Eq. (167)) [505]. Ferrilactone complexes having carbonyl groups were easily alkylated (Eq. (168)) [506,507].



The copper complex  $\text{ArCu}(\text{Me})(\text{CN})\text{Li}_2$  arylated aldehydes as well as a wide range of other electrophiles [508].  $\alpha$ -Silyloxyaldimines were alkylated by cuprates in the presence of  $\text{BF}_3$  with high *anti* selectivity [509]. 1,2-Diols were prepared by the alkylation of aldehydes with the reagent  $\text{IZn}(\text{CN})\text{CuCH}_2\text{B}(\text{OR})_2$  followed by oxidation of the borane [510]. Vinyl cuprates, from transmetalation from tin, alkylated aldehydes (Eq. (169)) [511]. A review dealing with the alkylation of dithianes by Grignard reagents in the presence of nickel catalysts has appeared (27 references) [512]. An example is in Eq. (170) [513]. Reduced nickel species cyclized olefinic ketones (Eq. (171)) [514].

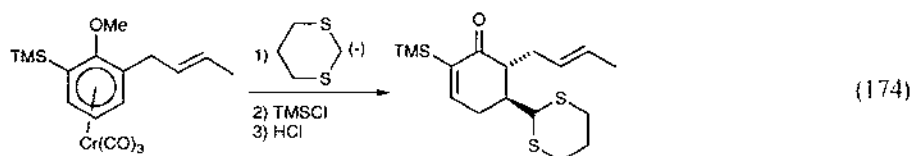
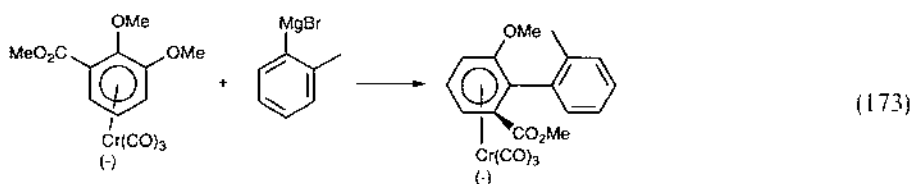


Palladium(0) complexes catalyzed the alkylation of aldehydes by allyl phosphonates [515], allyl alcohols [516] and propargyl benzoates [517] with only small amounts of allenes being formed. Aryl aldehydes were converted to diaryl ketones by reaction with aryl iodides in the presence of palladium(II) chloride [518]. Palladium complexes catalyzed the selective allylation of aldimines over aldehydes by allyl tin reagents [519,520]. Palladium(0) also promoted the strange alkylation shown in Eq. (172) [521]. Manganese-complexed conjugated enones could be specifically alkylated at the  $\alpha$ - $\text{sp}^3$  center by formation of the enolate followed by reaction with aldehydes [522]. Chromium(II) chloride catalyzed the alkylation of aldehydes by aryl, alkenyl and allyl bromides, as well as alkenyl triflates, in the presence of manganese metal [523].



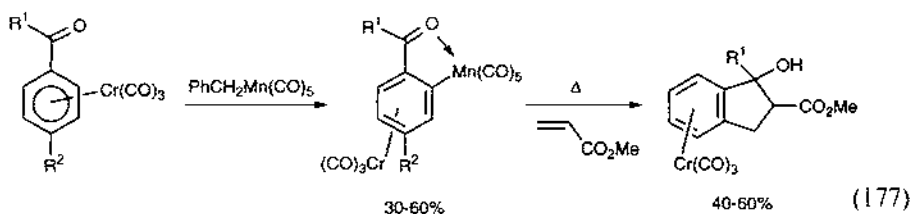
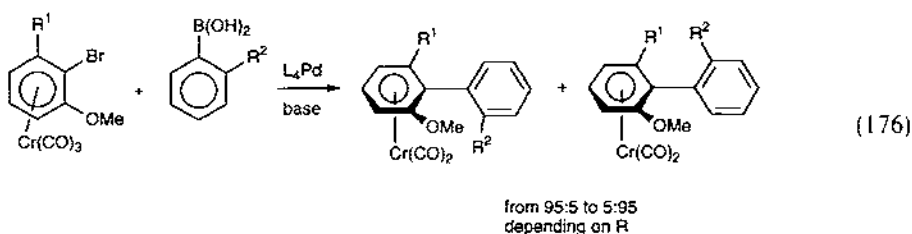
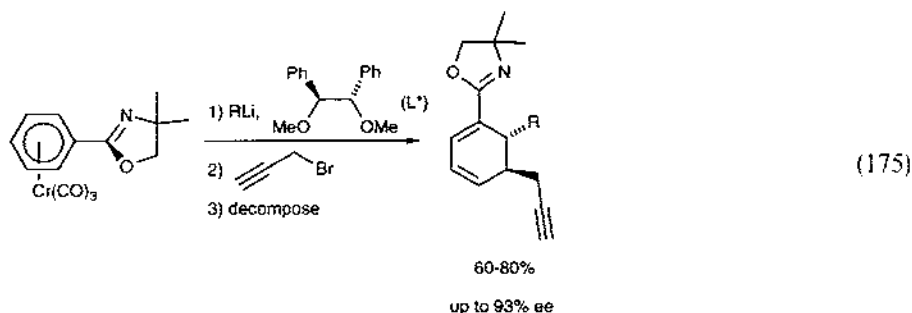
### 2.1.11. Alkylation of aromatic compounds

Chromium arene complexes continued to figure prominently in the alkylation of arenes. The use of chiral CO-emulating ligands in arene-chromium tricarbonyl chemistry has been reviewed (23 references) [524]. An optically active tetrahydroquinoline complexed stereoselectivity to chromium [525]. Optically active biphenyls were made by the alkylation of chromium-complexed anisoles by Grignard reagents (Eq. (173)) [526]. Alkylation of these complexes by other carbanions led to cyclohexenones [527] and Eq. (174) [528]. Intramolecular alkylations of arenechromium tricarbonyl complexes by pendant nitrile-stabilized carbanions went in low yield [529].

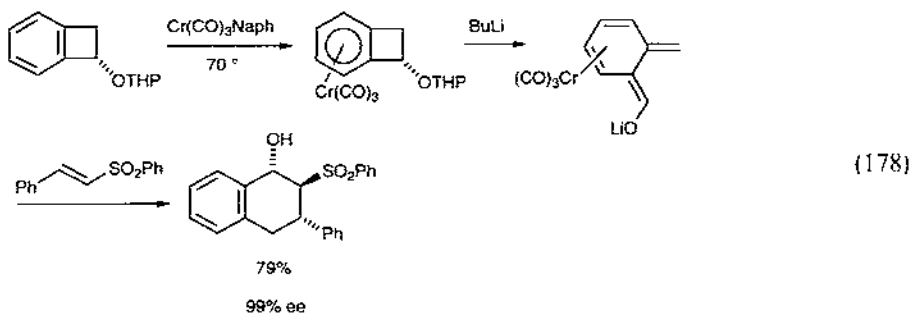


Benzenechromium tricarbonyl was ring-lithiated, alkylated with *p*-chlorobenzoyl chloride, and the resulting complexed benzophenone was reduced with high ee by chiral boranes [530]. Complexed aryl oxazolines were ring lithiated/alkylated with high ee (Eq. (175)) [531,532]. The chromium tricarbonyl complex of benzaldehyde dimethyl ketal was  $\alpha$ -lithiated/alkylated in up to 80% ee using a chiral base for the deprotonation [533].

Palladium(0) complexes catalyzed the alkylation of chromium-complexed chlorobenzene by a wide range of terminal arylacetylenes [534] by  $\beta$ -zincated alanine ester to produce aryl alanines [535] and by aryl boronates to produce axially disymmetric biphenyls (Eq. (176)) [536]. Chromium-complexed benzophenones were  $\alpha$ -manganated, then alkylated (Eq. (177)) [537].



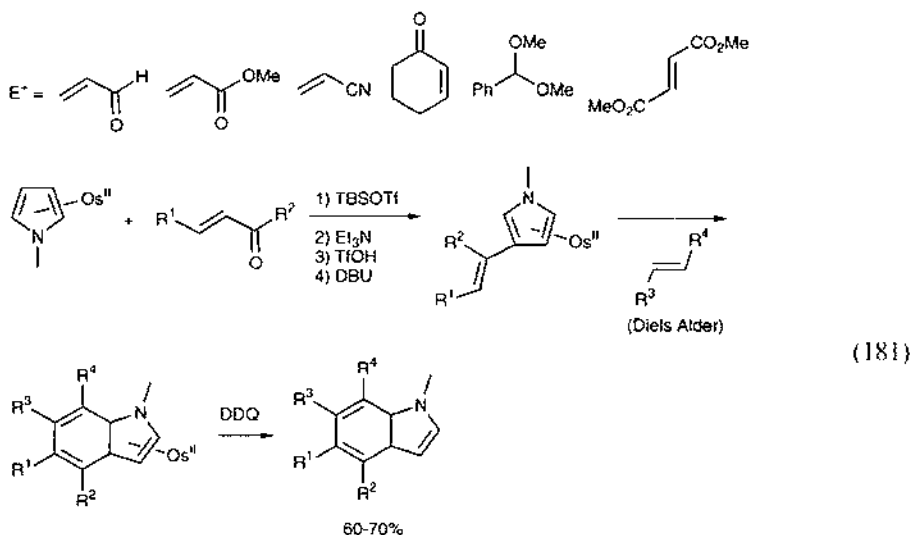
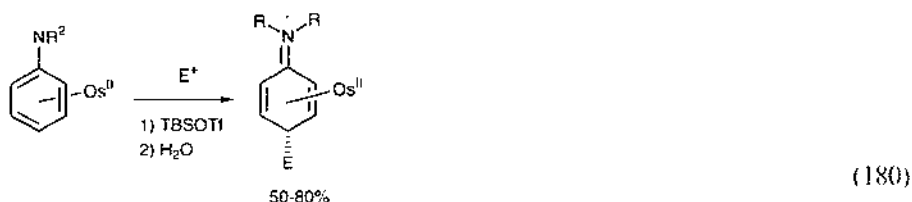
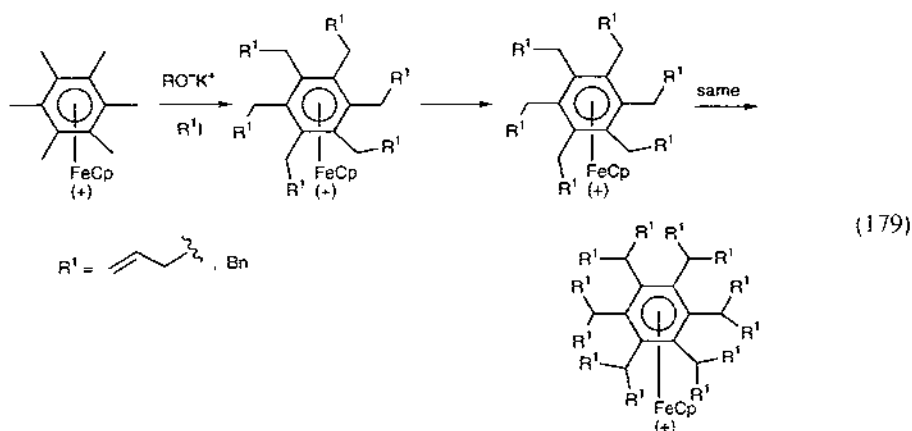
Chromium-complexed benzyl ethers were alkylated at the benzyl carbon with very high ee using a chiral base for the formation of the complexed benzyl anion [538–540]. Styrenechromium tricarbonyl was cyclopropanated by sulfur ylides with high de [541]. Benzaldehyde ketal chromium tricarbonyl was monoalkylated (one ketal OR replaced) by treatment with biphenyl radical anion followed by electrophiles



[542]. Unusual chromium benzocyclobutane chemistry is seen in Eq. (178) [543].

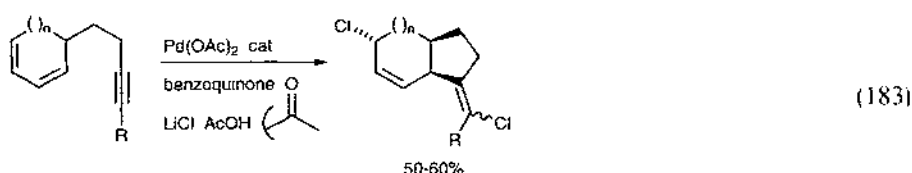
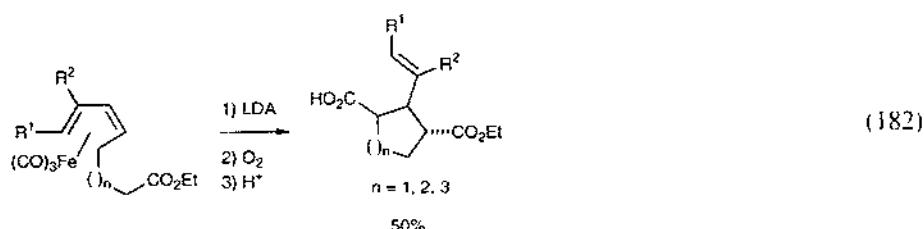
Iron-complexed hexamethyl benzene was polyalkylated by treatment with base

followed by an electrophile (Eq. (179)) [544,545]. Even triple branching could be achieved. Manganese tricarbonyl complexes of aryl silanes were  $\alpha$ -methylated by methylmagnesium bromide. Decomplexation gave 1-silyl-6-methylcyclohexa-1,3-diene [546]. Osmium-complexed anilines (Eq. (180)) [547] and pyrroles (Eq. (181)) [548] alkylated electrophiles.

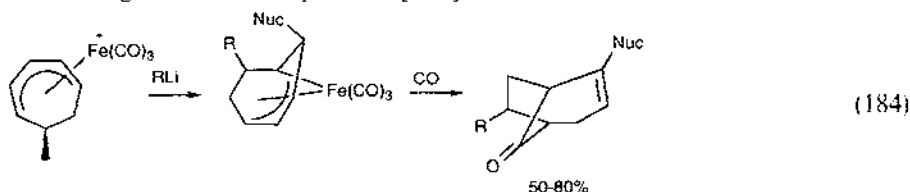


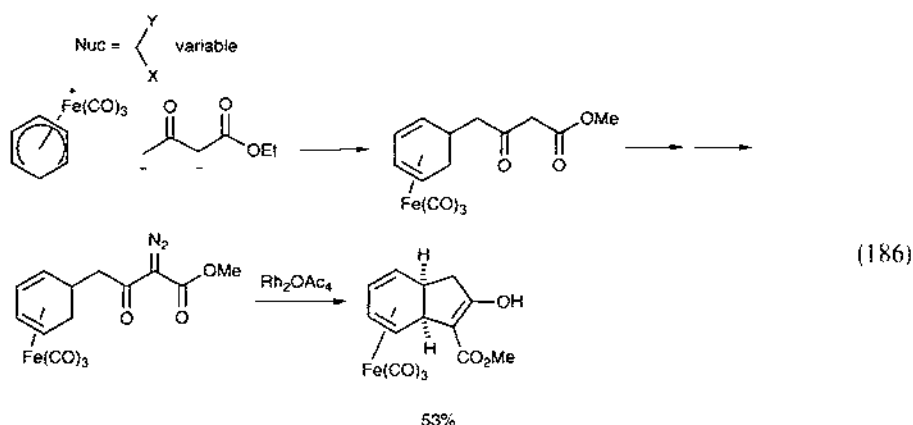
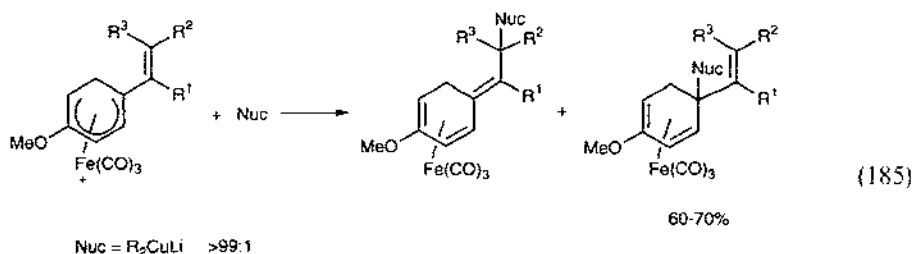
### 2.1.12. Alkylation of diene and dienyl complexes

Chiral HPLC was used to separate the enantiomers of planar chiral  $\text{Fe}(\text{CO})_3$  diene complexes [549]. The use of iron tricarbonyl diene complexes in asymmetric synthesis of natural products has been reviewed (41 references) [550]. An optically active sulfoxide on one terminus of an iron diene complex directed the stereochemistry of alkylation of an aldehyde on the other terminus [551]. Dieneiron tricarbonyl complexes underwent intramolecular alkylation (Eq. (182)) [552] and intramolecular Friedel-Crafts acylation [553]. Palladium(II) salts catalyzed the diene reaction in Eq. (183) [554].



Cationic cyclohexadienyliron tricarbonyl complexes were alkylated by  $\alpha$ -aminomalonate anions in low yield [555], and cyanoacetic esters in good yield [556]. They were alkylated by allyl and alkenecuprates, and the iron remained complexed to the resulting diene to protect it during side-chain oxidation [557]. Cationic iron tricarbonyl cycloheptadienyl complexes were alkylated by functionalized organocuprates, then remote  $\alpha$ -carbanions were generated and attacked the complexed diene [558]. The same types of complexes were alkylated and carbonylated (Eq. (184)) [559]. Nucleophiles attack cationic cyclohexadienyl complexes with conjugated exocyclic olefins both at the ring and the alkene (Eq. (185)) [560]. Iron dienyl complex chemistry was combined with rhodium(I) catalyzed diazodecomposition chemistry to result in ring formation (Eq. (186)) [561].

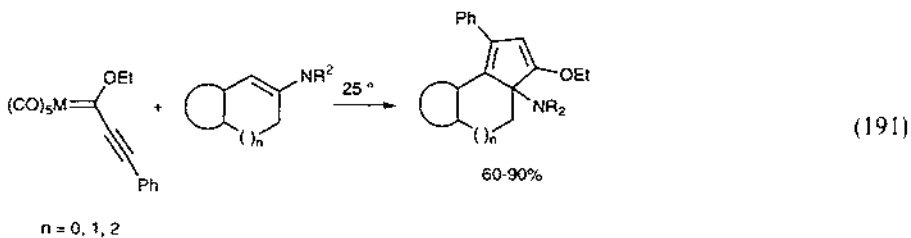
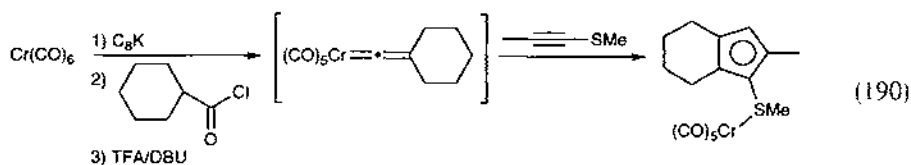
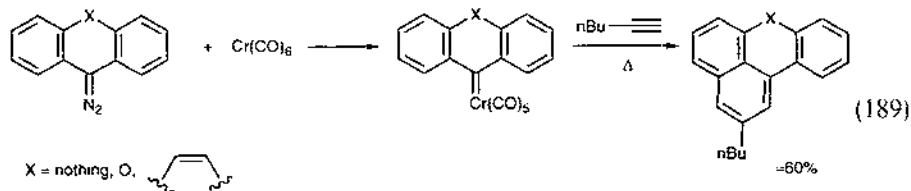
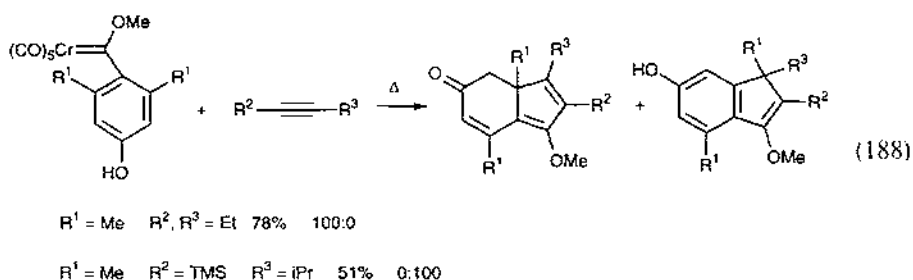
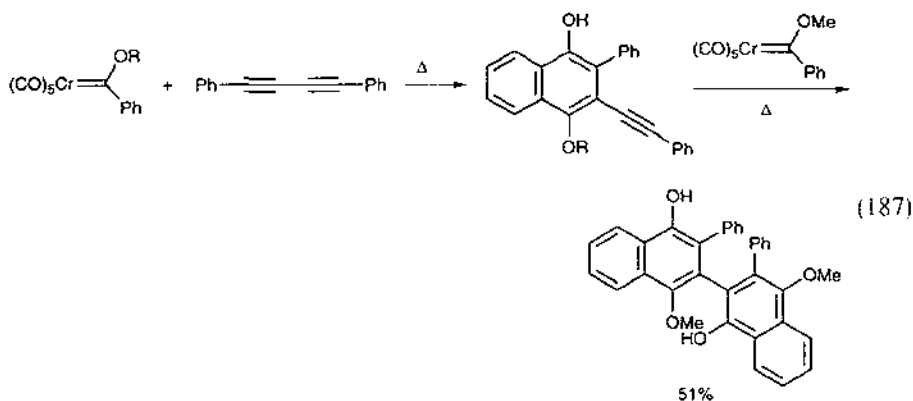


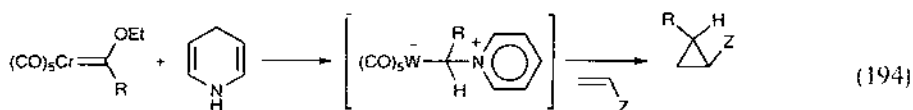
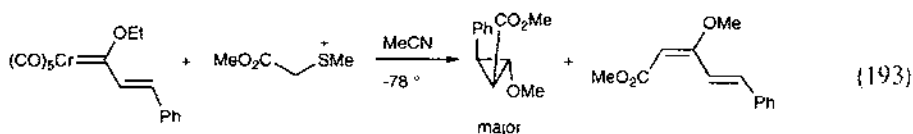
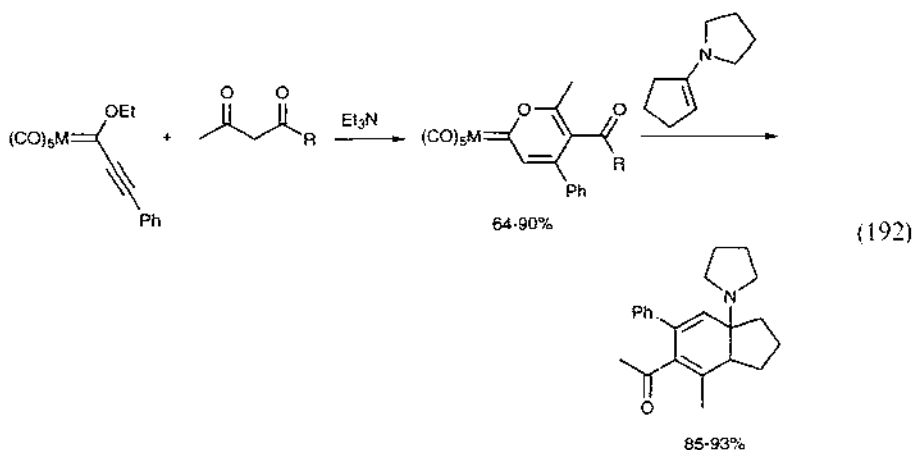


### 2.1.13. Metal carbene reactions

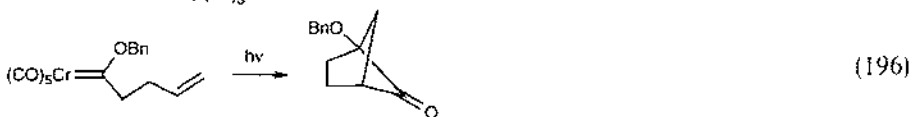
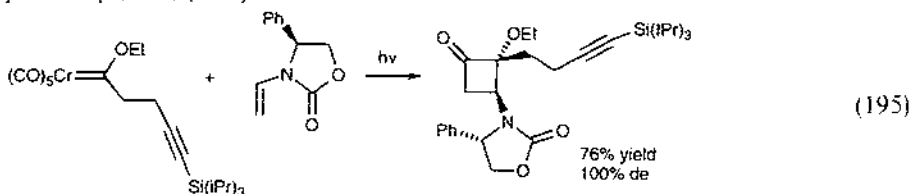
Chromium aminocarbene complexes were made by the alkylation of chromium (pentacarbonyl)(isocyanide) complexes followed by N-alkylation [562]. Density function calculations of the Dötz reaction showed the reaction to proceed by rate limiting loss of CO, and a highly energetic carbene/alkyne complex which directly inserts to give an  $\eta^3$ -allylidene intermediate [563]. Carbene alkyne alkene cyclization reactions have been reviewed (86 references) [564]. BINAPs were synthesized by the sequential reaction of 1,3-diynes with chromium alkoxy carbene complexes (Eq. (187)) [565]. Vaulted biaryls were synthesized starting with naphthylcarbene complexes [566]. An unusual methyl migration was observed in the Dötz reaction of hindered alkynes (Eq. (188)) [567]. Optically active cyclohexenylcarbene complexes underwent benzannulation with alkynes to give optically active arene complexes with high selectivity [568]. Carbene complexes made from diazo compounds also underwent the benzannulation reaction (Eq. (189)) [569]. Propargyl silanes (terminal alkyne) underwent thermal reactions with alkoxy carbene complexes to give 1-alkoxy-3-silyl-1,3-dienes by an insertion/double bond rearrangement [570].

Reviews entitled “ $\beta$ -Amino Substituted  $\alpha,\beta$ -Unsaturated Fischer Carbene Complexes as Chemical Multitalent” (35 references) [571] and “Selective Organic Synthesis via Group 6 Metal Carbene Complexes” (20 references) [572] have appeared. Other unusual reactions of group 6 carbene complexes are shown in Eq. (190) [573], Eq. (191) [574], Eq. (192) [575], Eq. (193) [576] and Eq. (194) [577]. The mechanism for the ring expansion of  $\alpha$ -vinyl cyclopropyl carbene complexes to give  $\alpha$ -vinyl cyclopentenones has been elucidated [578].





Photochemical reactions of chromium carbene complexes are shown in Eq. (195) [579] and Eq. (196) [580].



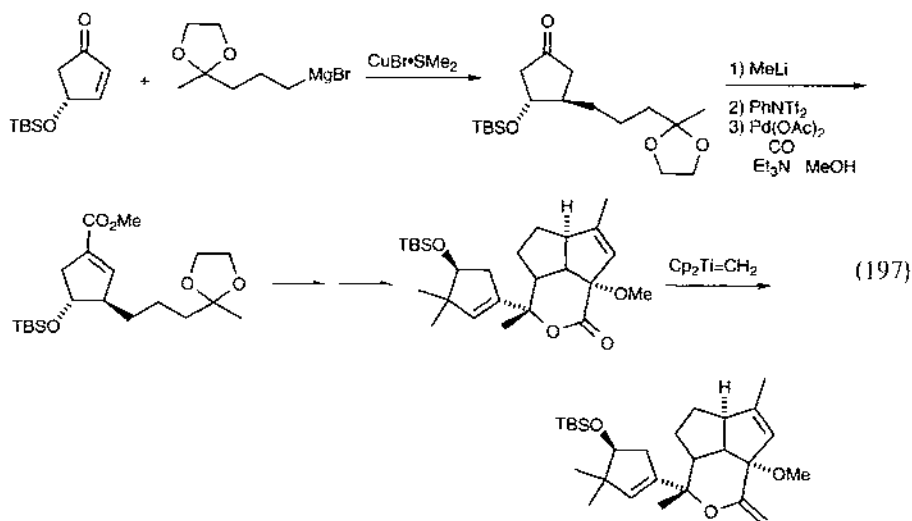
## 2.2. Conjugate addition

The cyanide in “higher order” cuprates  $R_2CuCNLi_2$  was claimed not to be directly bonded to copper (based on several spectroscopic techniques as well as calculations) but rather to reside between the two lithiums [581,582]. The silyl-containing mixed cuprates  $RCu(CH_2TMS)Li$ ,  $RCu(NTMS)_2Li$  and  $RCu(STMS)Li$  were thermally

stable, efficient in 1,4-addition reactions and only transferred the R group [583]. The reagent  $(\text{Me}_3\text{SnCH}=\text{CH})_2\text{CuCNLi}_2$  efficiently added 1,4 to enones [584]. Polyisoprenyl copper reagents added to camphor esters of acrylic acid with high ee [585].

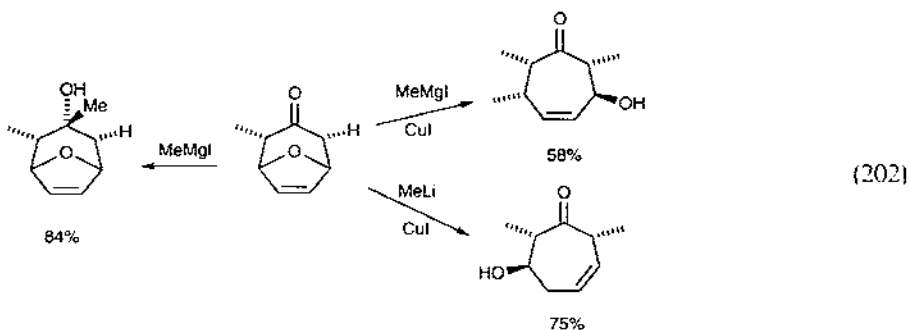
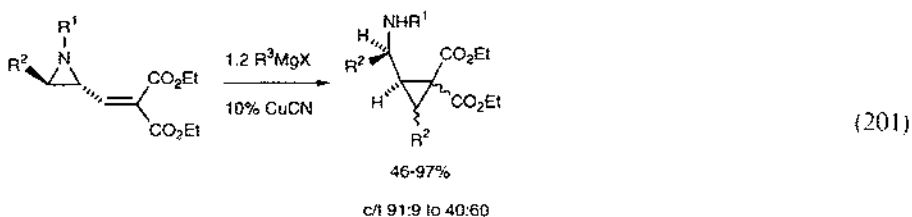
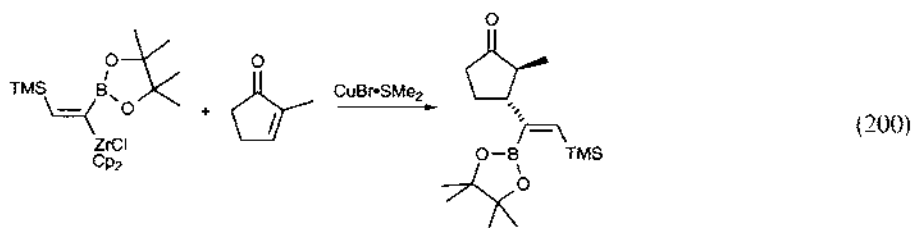
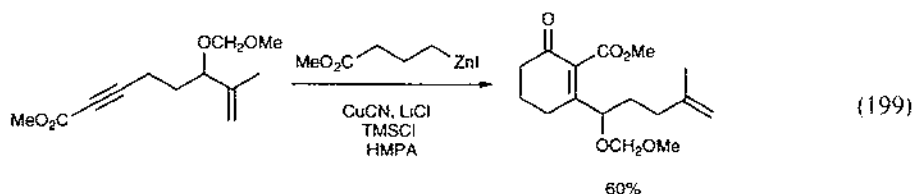
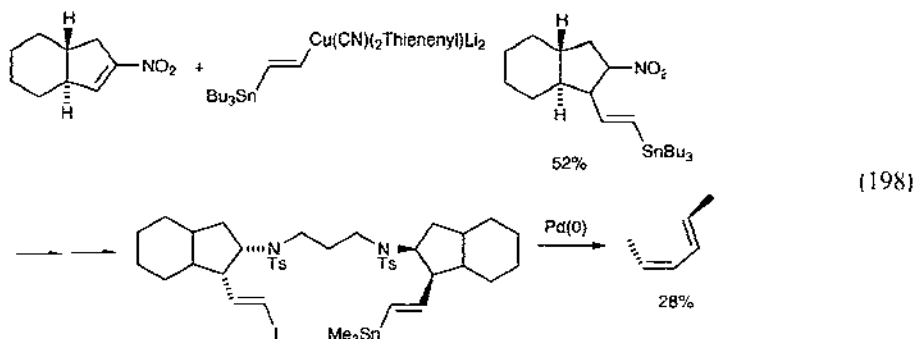
The role of trimethylsilyl iodide in the conjugate addition of cuprates to enones involved direct activation of the carbonyl group, and no enolate intermediate [586]. Conjugated esters were  $\beta$ -alkylated by  $\text{RCu} \cdot \text{TMEDA}$  in the presence of trimethylsilyl chloride [587], and by Grignard reagents in the presence of copper iodide and trimethylsilyl chloride [588].

Cuprates added with high stereoselectivity to camphor sultam esters of  $\alpha,\beta$ -unsaturated- $\beta$ -ketoesters [589], to  $\delta$ -amino- $\alpha,\beta$ -unsaturated- $\beta$ -ketoesters ( $\beta$  to keto group) [590] and to optically active cyclohexenones [591] and cyclopentenones (Eq. (197)) [592]. Cyclohexenones were  $\beta$ -alkylated with good ee by  $\text{Me}_3\text{Al}[\text{CuOTf}]/\text{TBDMSOTf}$  [593] and by *n*-butyl Grignard reagents in the presence of copper iodide [594] with chiral oxazolines as ligands.



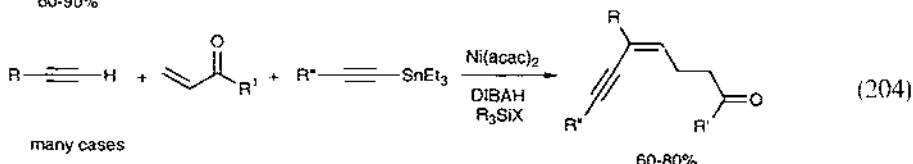
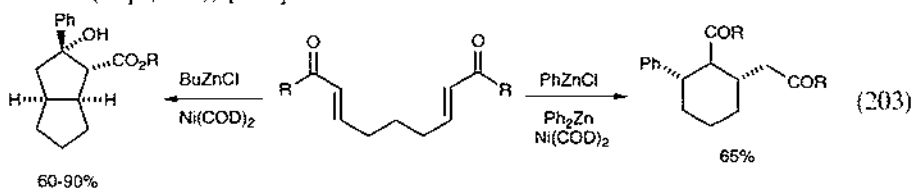
$\alpha$ -Alkoxy-stannanes [595] and alkenylstannanes [596] transmetallated to copper cyanide then 1,4 alkylated enones. Stannylcuprates Michael added to nitroolefins (Eq. (198)) [597]. Copper-catalyzed conjugate addition of functionalized organozinc reagents to alkynoic esters (Eq. (199)) [598] and to cyclic enones [599]. Alkenyl (Eq. (200)) [600] and alkylzirconium species [601] transmetallated to copper then Michael added to enones.

Alkynyl triflates alkylated  $\beta$  to the triflate with organocopper reagents [602]. Enol triflates of  $\beta$ -ketoesters underwent triflate replacement by *t*-butyl and cyclopropyl cuprates [603]. Allyl epoxides bearing chiral sulfoxides  $\alpha$  to the epoxide underwent  $\text{Sn}2'$  ring opening by organocuprates in an *anti* sense with high stereoselectivity [604]. Other stereoselective ring openings are shown in Eq. (201) [605] and Eq. (202)

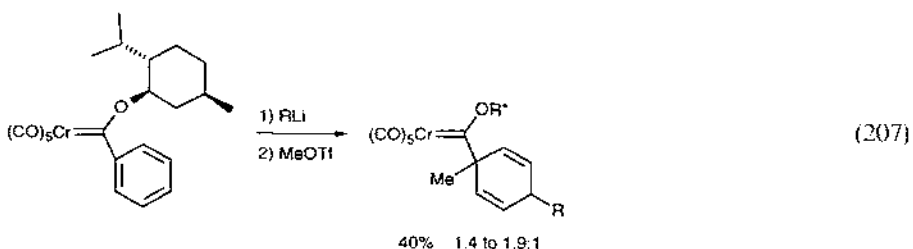
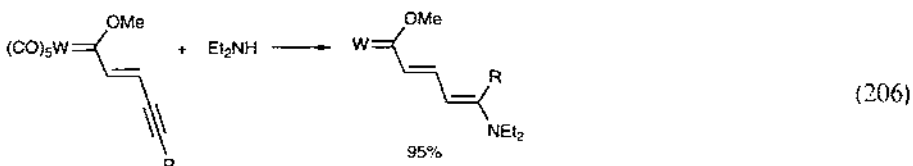
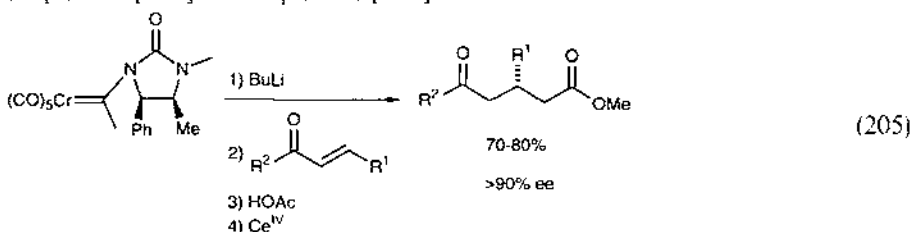


[606]. Allyl thioamidates were  $\text{Sn}2'$  alkylated by Grignard reagents in the presence of copper salts [607].

Nickel boride on a borohydride exchange resin catalyzed the 1,4 alkylation of conjugated esters by alkyl iodides [608]. Nickel complexes also catalyzed the cyclization of *bis*-enones (Eq. (203)) [609] and the coupling of alkynes, alkynylstannanes and enones (Eq. (204)) [610].



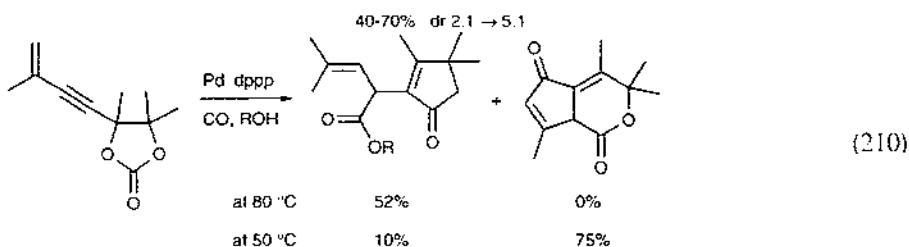
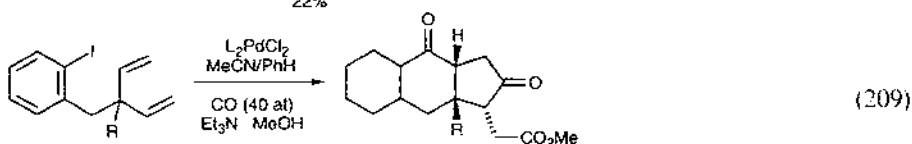
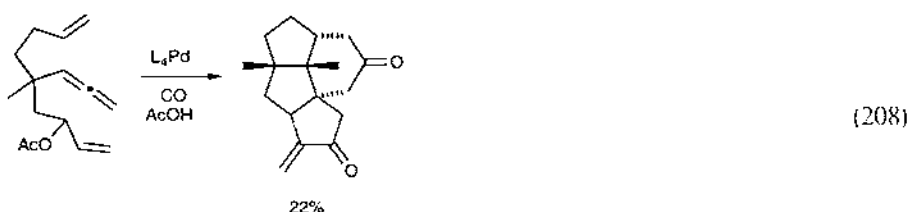
Palladium(0) complexes catalyzed the  $\beta$ -stannylation of ynones [611], the Michael arylation of enones by triarylstilbenes [612] and the Michael addition of terminal [613] and internal [614] alkynes to enones. The  $\alpha$ -anion of optically active amino-carbene chromium complexes added to enones with high stereoselectivity (Eq. (205)) [615]. Unsaturated carbene complexes underwent facile conjugate addition (Eq. (206) [616] and Eq. (207) [617]).



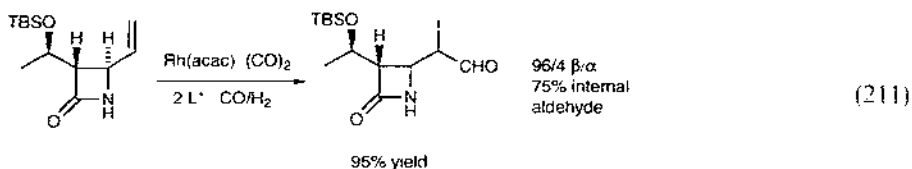
## 2.3. Acylation reactions excluding hydroformylation

### 2.3.1. Carbonylation of alkenes and arenes

Palladium-catalyzed ring forming reactions of organic halides with unsaturated substrates and CO has been reviewed (37 references) [618]. The cyclocarbonylation of allyl acetates with allenes was catalyzed by palladium(0) complexes (Eq. (208)) [619,620]. The full papers on palladium(0) cyclocarbonylation of alkenyl iodides [621] and aryl iodides [622] onto alkenes have appeared, as has a polycarbonylation (Eq. (209)) [623]. Palladium-catalyzed the triple carbonylation of terminal alkenes to give 2-keto-1,4-diester [624]. In the presence of chiral ligands, high ee was obtained. Palladium(0) complexes catalyzed the carbonylative rearrangement shown in Eq. (210) [625].

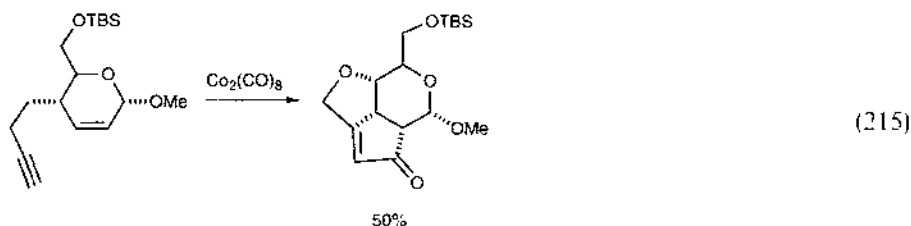
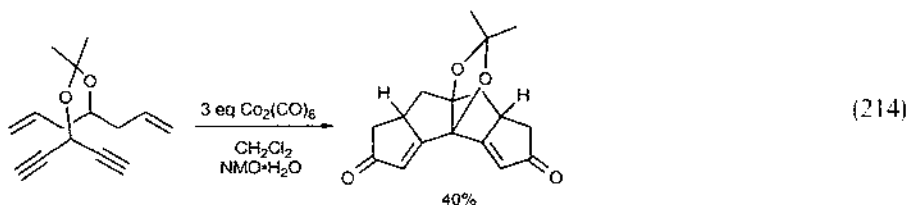
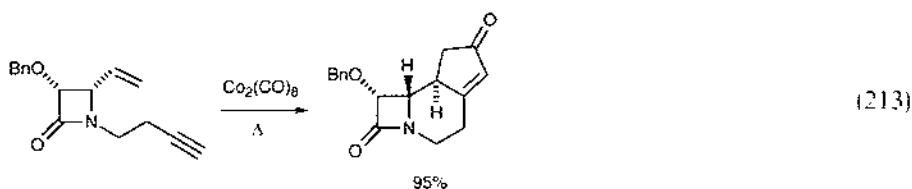


The mechanism of the conversion of vinylcyclopropanes to cyclohexenones by iron pentacarbonyl has been described [626], as has the cyclocarbonylation of 1,2,3,4-tetraenes (*bis* allenes) to 2,4-bismethylenecyclopent-3-enone [627]. The asymmetric hydroformylation of a vinyl  $\beta$ -lactam by rhodium was highly stereoselective (Eq. (211)) [628]. Ruthenium carbonyl catalyzed the acylation of *N*-methylimidazole by terminal alkenes and CO [629].

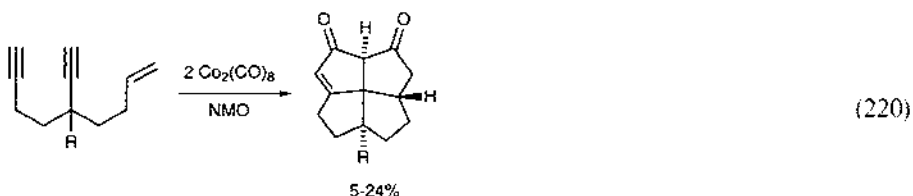
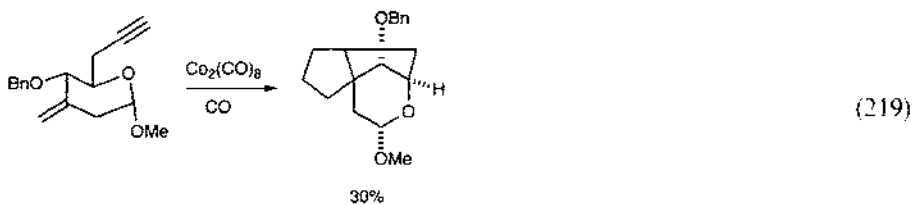
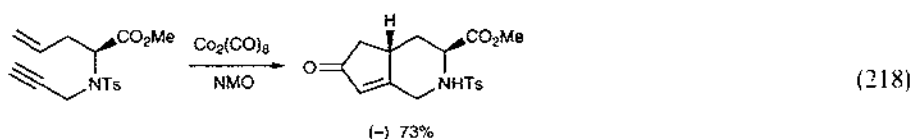
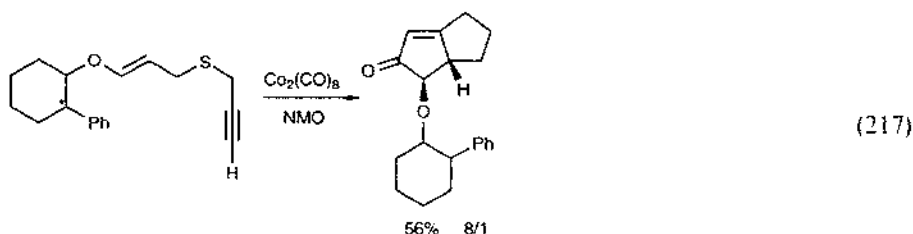
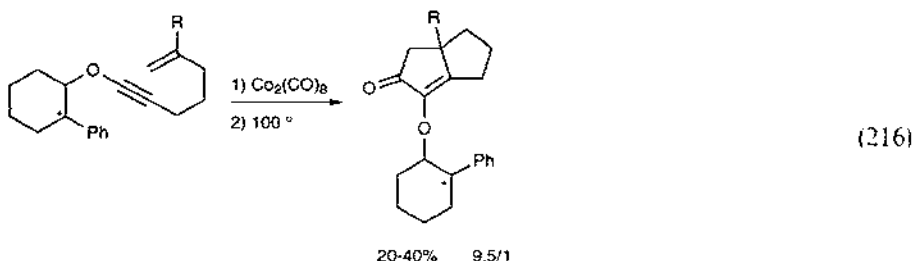


### 2.3.2. Carbonylation of alkynes (including the Pauson–Khand reaction)

The Pauson–Khand reaction between norbornadiene and terminal alkynes was catalyzed by  $\text{Co}(\text{acac})_2 \cdot \text{NaBH}_4$  [630]. Intramolecular Pauson–Khand reactions of enynes were catalyzed by  $\text{Co}_2(\text{CO})_8$  in the presence of high intensity visible light [631]. Treatment of the dicobalt complex of alkynylcyclopropanes with ethene, carbon monoxide and an amine oxide led to the formation of 2-cyclopropyl-cyclopentenones by Pauson–Khand processes [632]. Treatment of dicobalt complexes of internal alkynes with trifluoroacetic acid produced cyclopentenones containing two equivalents of the alkyne [633]. Oxidation of cobalt enyne complexes led to truncated Pauson–Khand reactions (Eq. (212)) [634]. Other synthetically useful Pauson–Khand reactions are seen in Eq. (213) [635], Eq. (214) [636], Eq. (215) [637], Eq. (216) [638], Eq. (217) [639], Eq. (218) [640], Eq. (219) [641] and Eq. (220) [642].

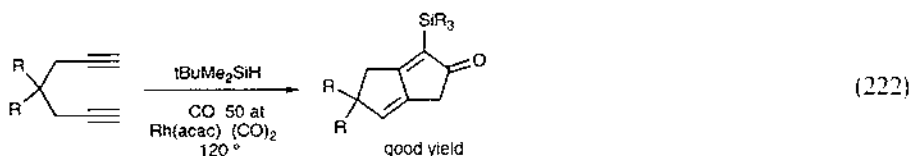
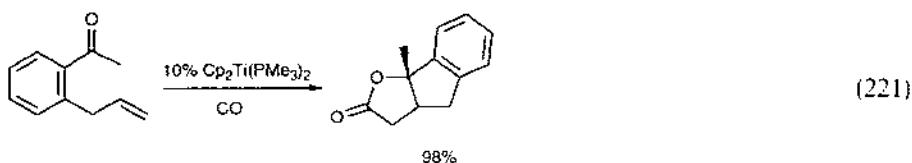


Enynes were converted to Pauson–Khand cyclopentenone products by a  $\text{Cp}_2\text{Ti}(\text{CO})_2$  catalyst [643]. With a chiral Cp system high ee was obtained [644]. Titanium complexes similarly cyclized alkenes onto ketones to give lactones (Eq. (221)) [645,646]. Cyclopentenones were also produced by the stepwise cycliza-

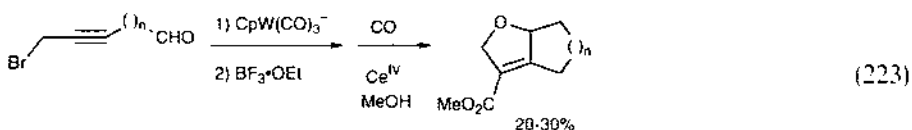


tion of enynes to zirconacyclopentenes followed by carbonylation [647]. Diynes were silylcarbonylated using rhodium catalysts, again giving cyclopentenones (Eq. (222)) [648].

Nickel cyclooctadiene complexes cocyclized enynes with isonitriles to give Pauson–Khand-like cyclopentenone imines [649]. The full paper on palladium(0) catalyzed cyclocarbonylation of alkenyl iodides onto alkynes with trapping by a pendant amine to give lactams has appeared [650]. Internal alkynes were converted into cyclopentenc-1,2-diones by reaction with  $\text{NaHFe}(\text{CO})_4$  and methyl iodide [651].

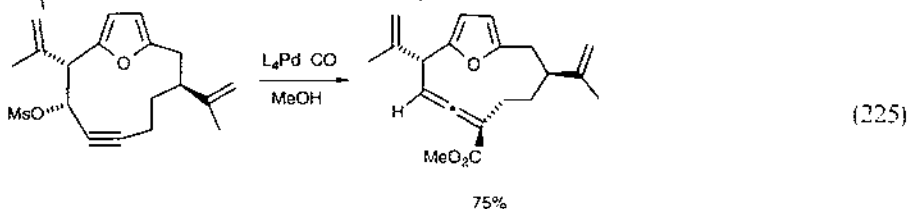
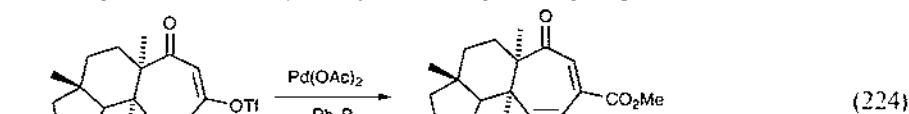


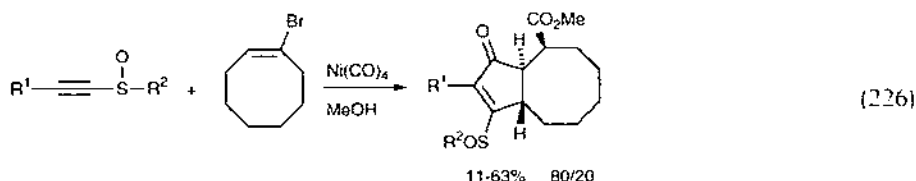
An unusual alkyne carbonylation is seen in Eq. (223) [652].



### 2.3.3. Carbonylation of halides and triflates

Palladium acetate catalyzed the conversion of aryl and vinyl triflates to aldehydes by silanes in the presence of CO [653]. Anodic reduction of alkyl halides in the presence of  $\text{Fe}(\text{CO})_5$  followed by an acidic workup produced aldehydes [654]. Cinnamyl carbonates were converted to the homocinnamyl ester by palladium catalyst in the presence of carbon monoxide and methanol [655]. Carbonylation of the racemic chromium tricarbonyl complex of *o*-chloroanisole produced complexed *o*-methoxy benzoates in low yield and low ee in the presence of BINAP [656]. Vinyl triflates were converted to esters using palladium catalyst [657] (Eq. (224)) [658]. Heteroaromatic triflates [659] and propargyl mesylates (Eq. (225)) [660] were carbonylated under similar conditions. Benzyl chloride was converted to phenyl glycine acetamide by reaction with acetamide,  $\text{Co}_2(\text{CO})_8$  and  $\text{H}_2$  at 100 °C [661]. Eq. (226) shows a complex nickel-catalyzed cyclocarbonylation [662].





### 2.3.4. Carbonylation of nitrogen compounds

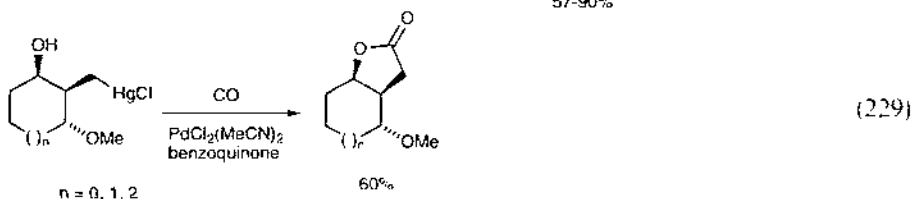
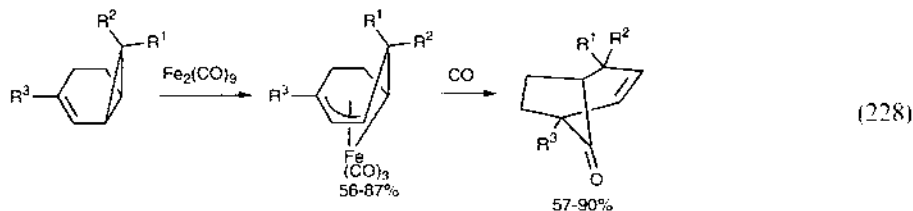
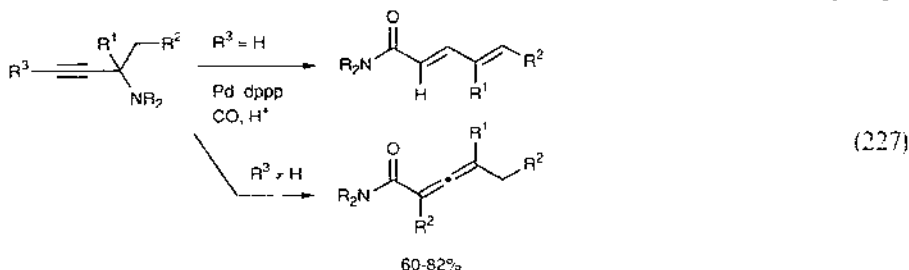
Sodium iodide accelerated the palladium-catalyzed aminocarbonylation of electron poor, *p*-substituted chlorobenzenes by aniline [663]. Palladium complexes catalyzed the conversion of propargyl [664] and allenyl [665] amines to unsaturated amides (Eq. (227)). 1,2-Amino alcohols were cyclocarbonylated to carbamates by palladium copper catalysts in the presence of carbon monoxide and air [666].

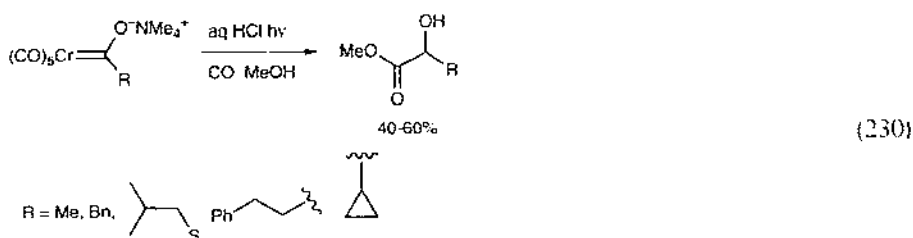
### 2.3.5. Carbonylation of oxygen compounds

There were no synthetically significant examples this year.

### 2.3.6. Miscellaneous carbonylations

Treatment of  $\text{Mn}_2(\text{CO})_{10}$  sequentially with two different organolithium reagents followed by addition of HMPA gave unsymmetrical ketones [667]. Other unusual carbonylations are shown in Eq. (228) [668], Eq. (229) [669] and Eq. (230) [670].

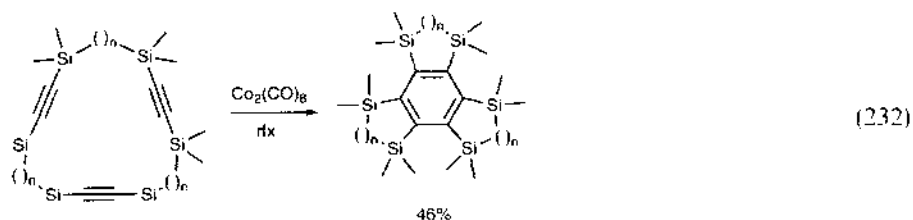
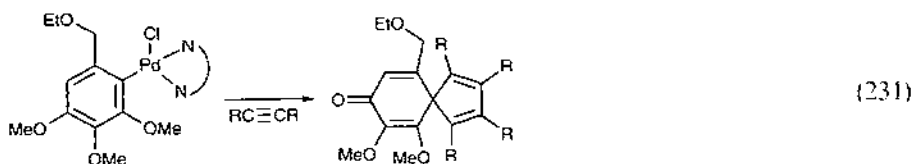


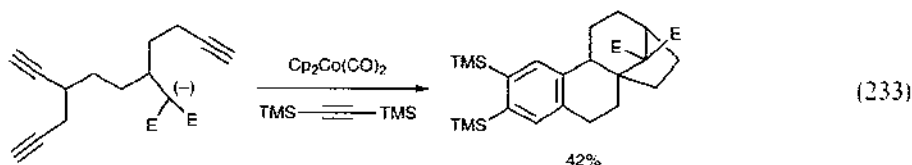


#### 2.4. Oligomerization (including cyclotrimerization and metathesis polymerization)

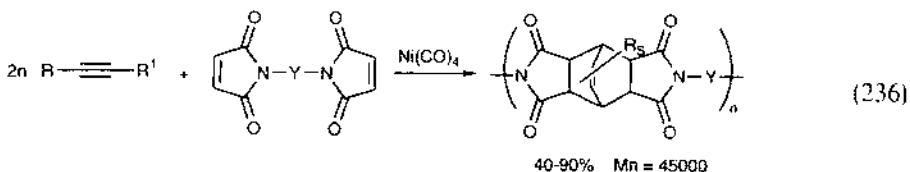
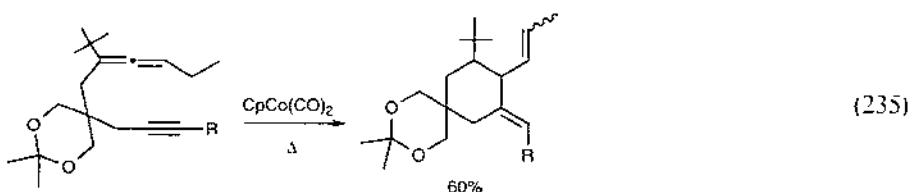
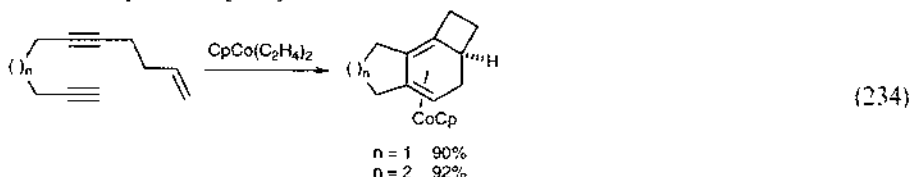
Ruthenium complexes catalyzed the head-to-head and head-to-tail dimerization of terminal alkynes [671,672]. Wilkinson's complex catalyzed the codimerization of vinylpyridines with terminal alkenes [673]. The mechanism for the codimerization of ethylene with methyl acrylate by cationic  $\pi$ -allylpalladium complexes has been published [674]. Copolymers of these two monomers were produced using palladium(II) *bis* amine complex catalysis [675]. The mechanism for the copolymerization of ethylene with carbon monoxide by palladium catalysts has been elucidated [676]. Stereoblock polyketones were synthesized using this knowledge [677]. Depending on the symmetry of the ligand, alternating copolymers of styrene and carbon monoxide were either highly isotactic or completely atactic, using palladium(II) catalysis [678]. The complex  $\text{Cp}_2^*\text{Ti}[\eta^2\text{C}_2\text{TMS}_2]$  catalyzed the head-to-tail dimerization of alkynes [679].

Arylpalladium(II) complexes cyclodimerized with alkynes (Eq. (231)) [680]. Palladium(0) complexes dimerized 1,3-enynes to give styrenes [681]. A review dealing primarily with cobalt-catalyzed cyclotrimerization of alkynes (109 references) has appeared [682]. A stable 19 e<sup>-</sup> complex ("18e<sup>-</sup> + d<sup>1</sup>") of cobalt-catalyzed cyclotrimerization of alkynes [683]. Other cyclotrimerizations are shown in Eq. (232) [684] and Eq. (233) [685].

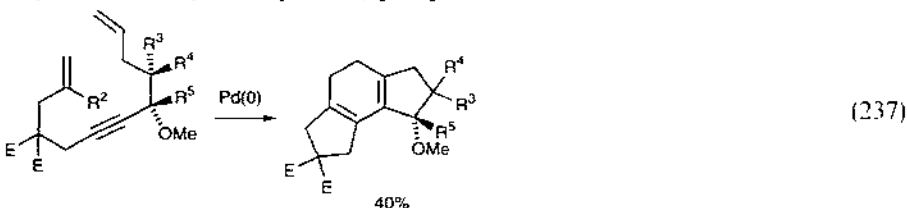


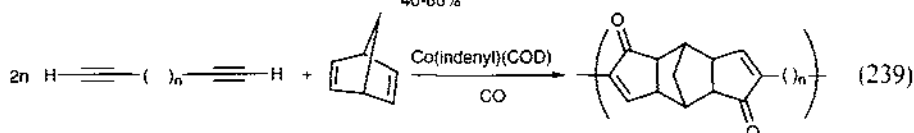
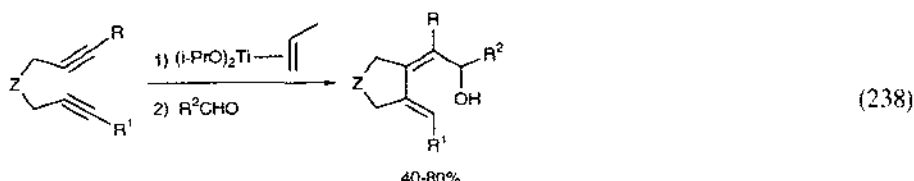


Nickel(0) catalyzed the cocyclotrimerization of diynes with propargyl amines to give benzylamines [686]. Nickel benzyne complexes reacted with alkynes to give naphthalene [687]. Cobalt complexes catalyzed the cocyclotrimerization of enediynes to give cyclohexadienes (Eq. (234)) [688] while ruthenium complexes cyclized 1,3,5-dienynes to benzenes [689] and rhodium complexes cyclized 1,3-diyne-2-enes to benzenes [690]. Cobalt complexes codimerized alkynes with allenes (Eq. (235)) [691] and nickel complexes catalyzed the cooligomerization of alkynes with *bis* maleimides (Eq. (236)) [692].



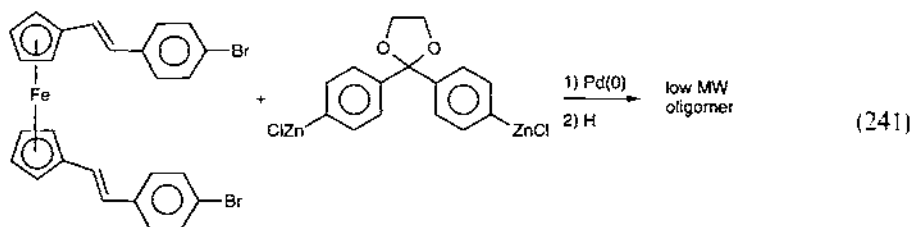
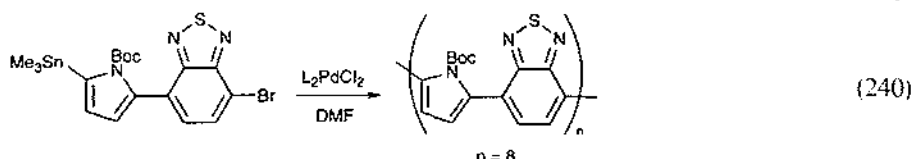
Palladium(0) complexes catalyzed the cocyclotrimerization of bromoenynes with alkenes (Eq. (237)) [693,694], while low-valent titanium cyclodimerized diynes (Eq. (238)) [695]. Oligo ladderanes were made by oxidation of iron-cyclobutadiene complexes [696]. Diynes were cooligomerized with norbornadiene and carbon monoxide by cobalt catalysts (Eq. (239)) [697].



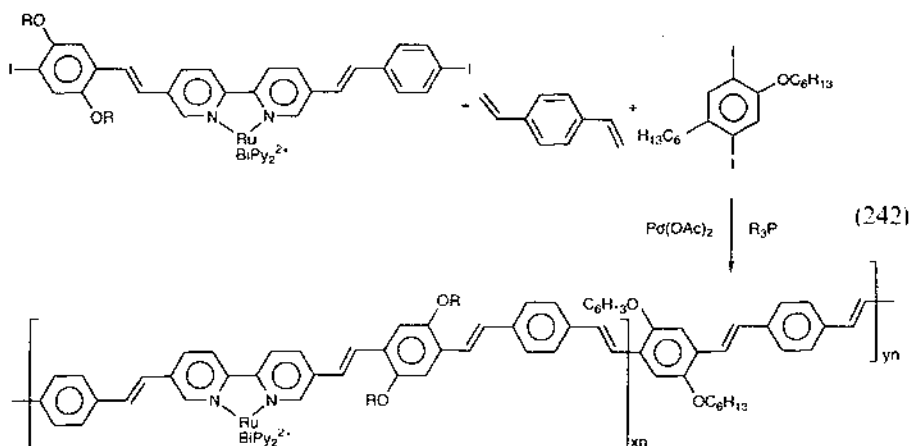


A review dealing with palladium-catalyzed coupling of thiophenes and alkynes to give macromolecules of precise length and constitution (60 references) has appeared [698]. Manganese tricarbonyl complexes of *m*-diiodobenzene were coupled to *para*-substituted aryl (*bis*)-boronic acids to give alternating oligomers of about 30 units [699]. 2,5-Dibromo-1,4-pyridazines coupled to 1,4-*bis* trimethylstannylbenzenes under palladium catalysis to give an oligomer of 9–18 units [700].

$\pi$ -Conjugated donor-acceptor molecules were made by the nickel(0) catalyzed oligomerization of 2,2'-dibromo(*bis*)-thiophenes, -selenophenes, 2,5-dibromopyridines, and the cross coupling of 1,4 dihaloarenes with 1,4-*bis* (tributylstannyl) arenes [701]. 3,3'-Dibromo-6,6'-dialkoxy biphenyls [702] and 2,2'-*bis* acetoxy-6,6'-dibromoBINAPs [703] were oligomerized (MW  $\approx$  5000) by nickel(0) cyclooctadiene complexes. Palladium(0) catalyzed oligomerization of heteroaromatic halostannanes (Eq. (240)) [704], the co-oligomerization of aryl halides and aryl zinc reagent (Eq. (241)) [705], and the production of photorefractive polymers (Eq. (242)) [706].



Ferroelectric liquid crystal oligomers were made by metathesis polymerization of long chain  $\alpha,\omega$ -dienes having aryl ether and ester linkers [707]. The Diels–Alder adduct between cyclopentadiene and *N*-methylmaleimide underwent living ROMP polymerization in aqueous solution with Grubbs ruthenium catalyst [708]. 1,2-Polybutadiene underwent quantitative ring-closing metathesis polymerization with the same catalyst to give polymethylcyclopentene [709]. The complex



Mo(*N*-2-*i*BuPh)(CH*t*Bu)/O<sub>2</sub>C(Ph)<sub>3</sub> polymerized 1,6-heptadiynes to give exclusively six-membered ring containing polymers [710].

Dicobalt octacarbonyl catalyzed the living polymerization of 1,1-dimethylallene via  $\eta^3$ -allyl complexes [711].  $\alpha$ -Olefins underwent living polymerization to give nearly monodispersed elastomeric  $\alpha$ -olefin base block copolymers with a nickel(II) diazine catalyst [712]. Cyclopropenes were linearly polymerized to polycyclopropanes (MW 99 000) by palladium(0)/spartein catalysts [713]. 1,2-Bisisocyanarenes were polymerized with high screw-sense selectivity by optically active binaphthylpalladium(II) complexes [714]. Achiral catalysts effected the same type of polymerization [715]. 1,2-Disilylcyclopentane was oligomerized to larger ring polysilanes by palladium isonitrile catalyst [716]. Reviews dealing with the synthesis of well-defined conjugated oligomers for molecular electronics (9 references) [717] and recent developments in inorganic polymer science (224 references) [718] have appeared.

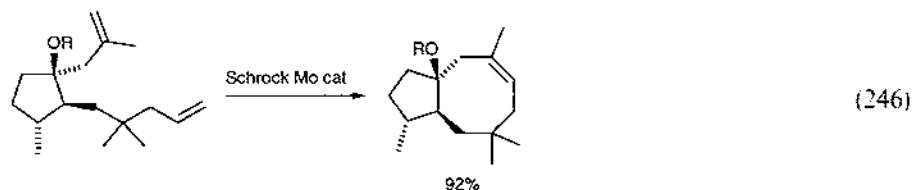
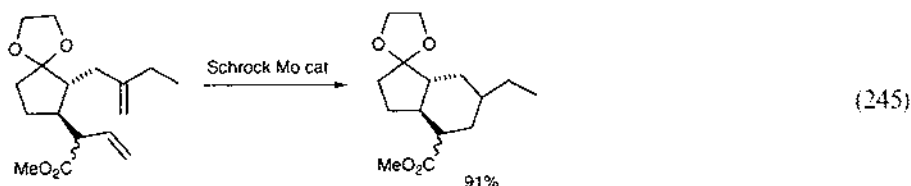
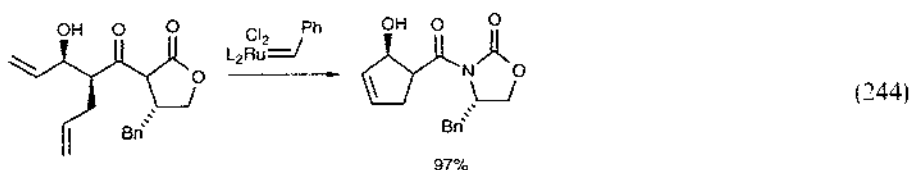
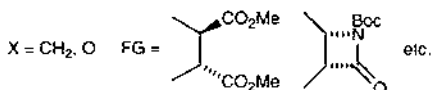
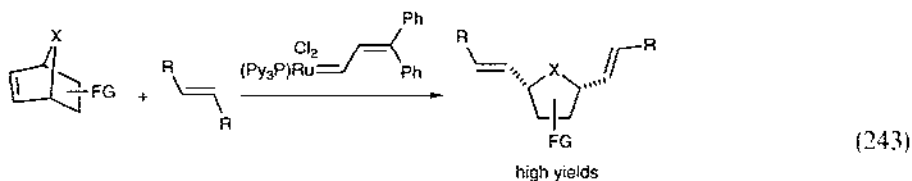
## 2.5. Rearrangements

### 2.5.1. Metathesis

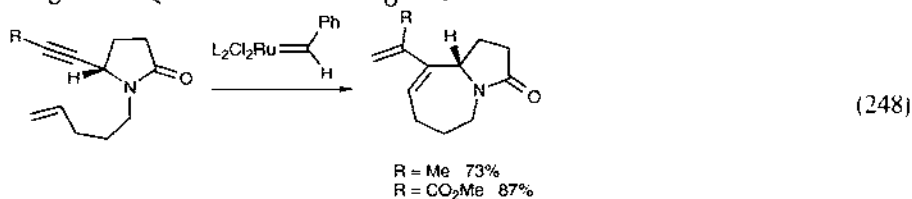
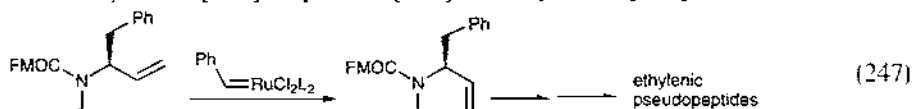
A water-soluble variant of the Grubbs metathesis catalyst has been reported [719]. Treatment of *bis*- $\pi$ -allylruthenium(*tris*)isonitrile complexes with ethyl diazoacetate produced a good ROMP catalyst [720]. The Schrock metathesis catalysis was used to metathesize substituted styrenes with allyl silane [721] and substituted silyl ethers of allyl alcohols [722] to give new allyl silanes or protected allyl alcohols. Another useful metathesis is seen in Eq. (243) [723].

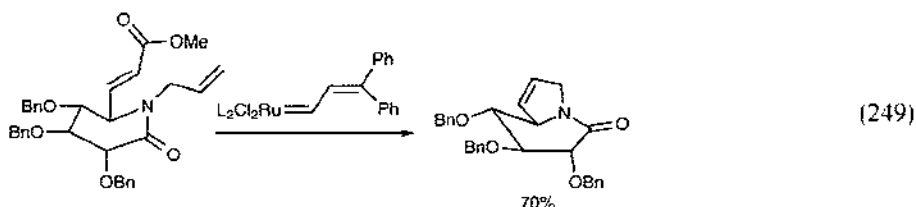
The complex W(O)(OAr)<sub>2</sub>Cl<sub>2</sub> metathesized 1,6-heptadienes to cyclopentenes [724]. The Schrock catalyst (but not the Grubbs catalyst) metathesized diallylsulfide to 2,5-dihydrothiophene [725]. Useful carbocycle-forming metatheses are shown in Eq. (244) [726], Eq. (245) [727] and Eq. (246) [728].

Ruthenium complexes catalyzed the metathesis of diallyl and longer chain unsaturated amines to dihydropyrroles and larger heterocycles attached to polymers [729].

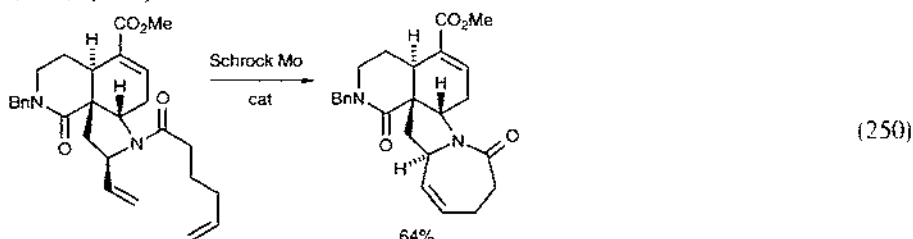


Cyclic amides were made by metathesizing *N*-allyl amides of unsaturated acids [730]. The process also is efficient in solution [731] and has been used to make useful molecules (Eq. (247) [732], Eq. (248) [733] and Eq. (249) [734]).

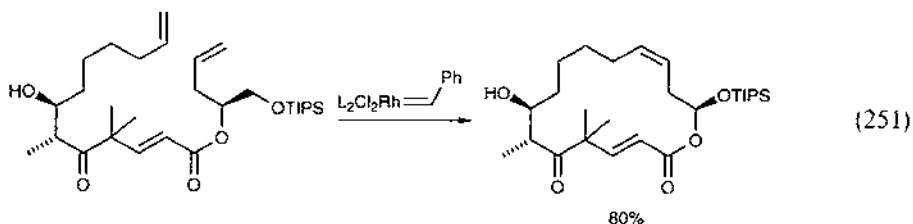




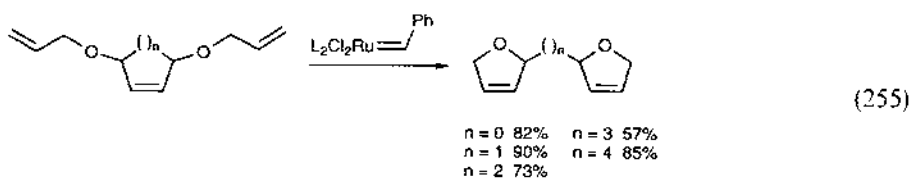
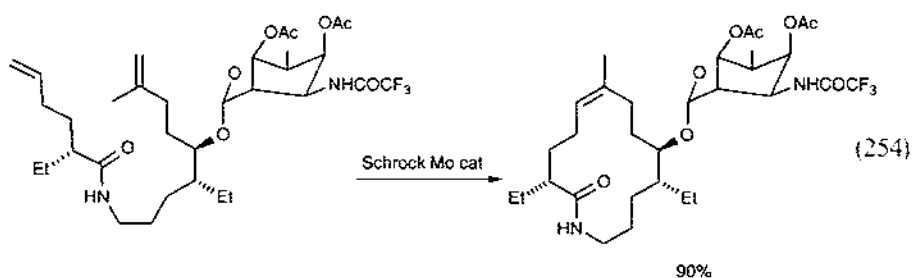
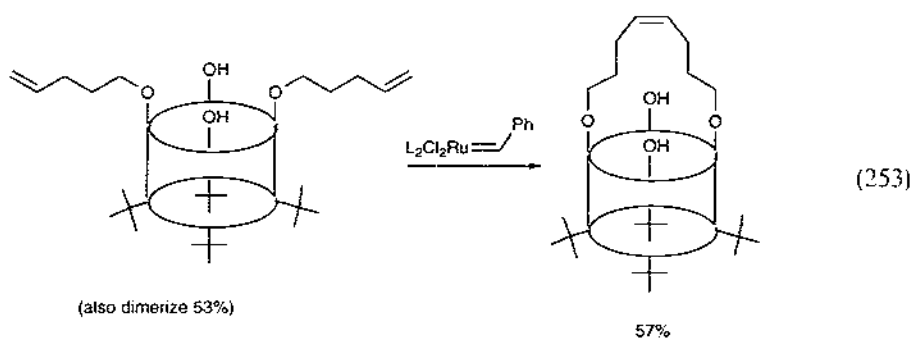
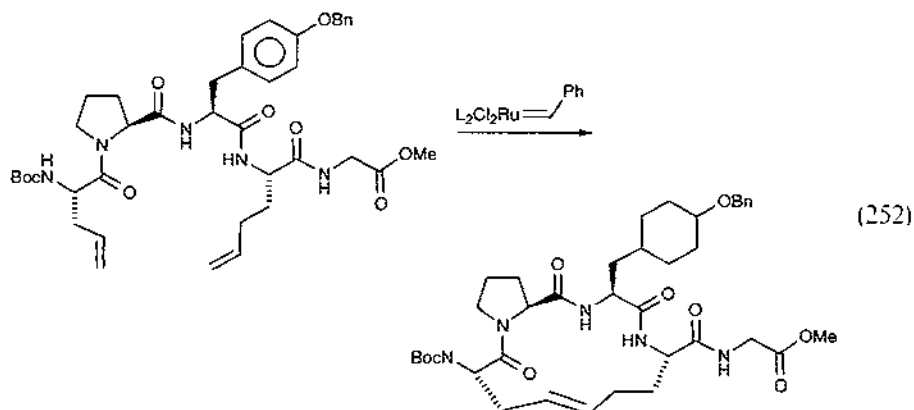
Dialkyl amines with two unsaturated alkyl groups metathesized to give unsaturated five-, six- [735]a, seven- [735]b and eight-membered nitrogen heterocycles (Eq. (250)) [736].

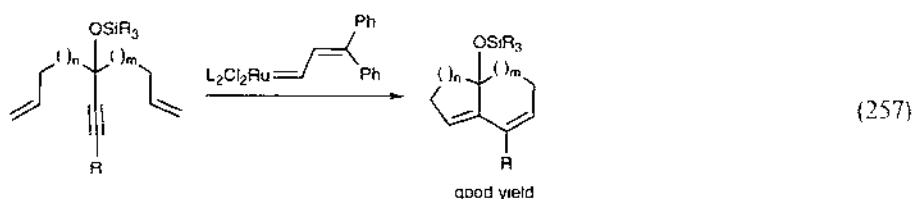
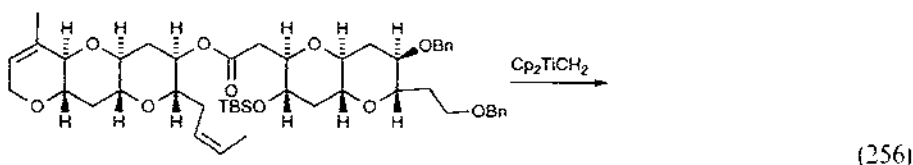


Ring-closing metathesis was used to synthesize macrocyclic lactones (Eq. (251)) [737] including unfunctionalized ones with up to 21 members in the ring [738], 14-membered lactones spanning the 1 and 6 positions of a monosaccharide [739] and ten-membered lactones bridging the *ortho* position of methoxy arenes [740]. Cyclic peptides were formed via this process (Eq. (252)) [741]. *N*-Allyl  $\beta$ -lactams having unsaturated ethers  $\alpha$ - to the nitrogen metathesis ring closed to give bicyclic  $\beta$ -lactams having 7- (84%), 8- (53%) and 9- (12%) membered fused cyclic ethers [742]. Ring-closing metathesis was used to cap or dimerize calixarenes (Eq. (253)) [743] and to synthesize antifungal agents (Eq. (254)) [744].



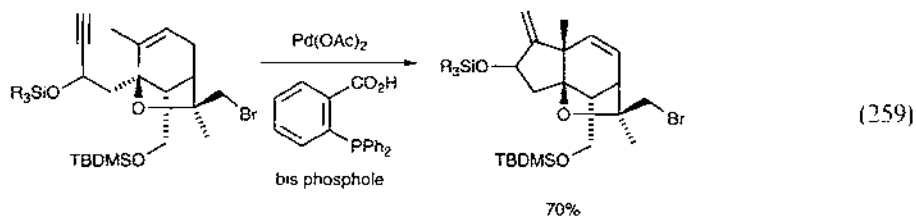
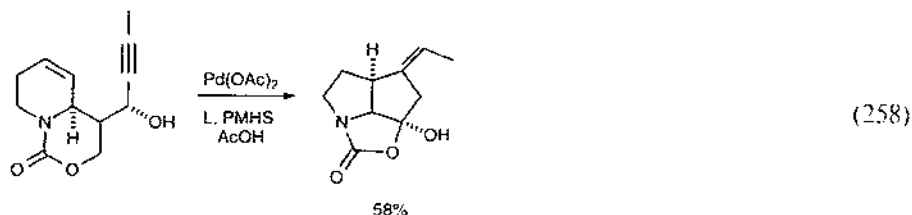
Optically active  $\alpha,\omega$ -dienes were kinetically resolved (up to 84% ee and 90% conversion) by ring-closing metathesis with a chiral carbene complex catalyst [745]. Combined ring opening/ring closing metathesis was used to make *bis* dihydrofurans (Eq. (255)) [746]. Other interesting metatheses are shown in Eq. (256) [747] and Eq. (257) [748].



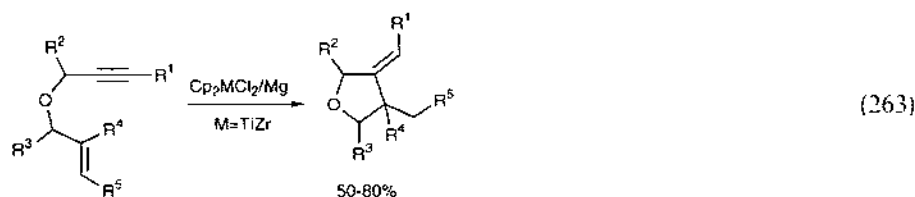
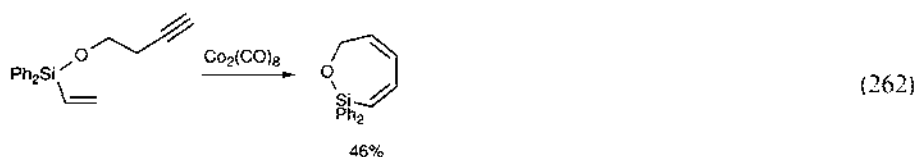
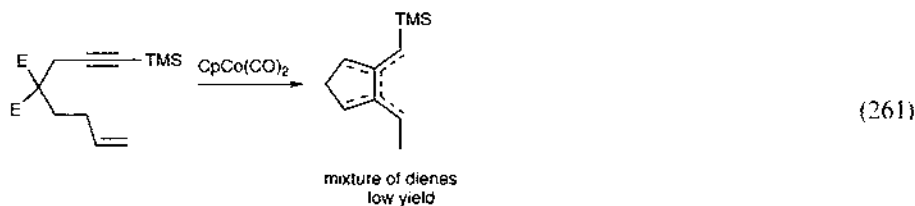
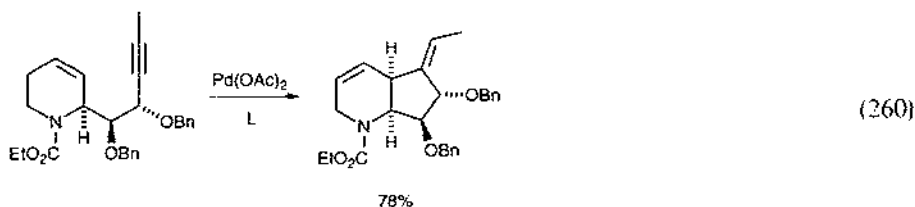


### 2.5.2. Olefin isomerization including cycloisomerization

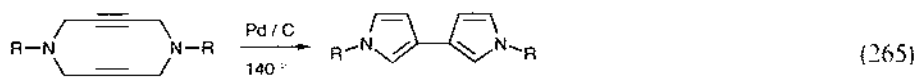
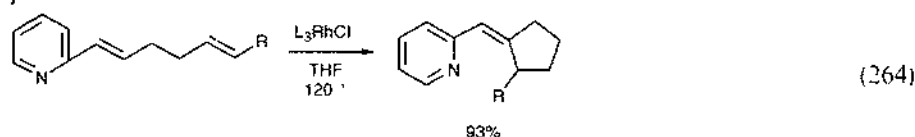
Palladium-catalyzed enyne cycloisomerizations with reduction formed cyclic alkenes [749,750], (Eq. (258)) [751]. Without reduction, dienes were formed [752], (Eq. (259)) [753]. By this method,  $\alpha,\beta$ -bis methylene lactones [754], bridged bicyclic dienes [755], alkaloids (Eq. (260)) [756] and 3,4-bis-methylenepyrroles [757] were made.

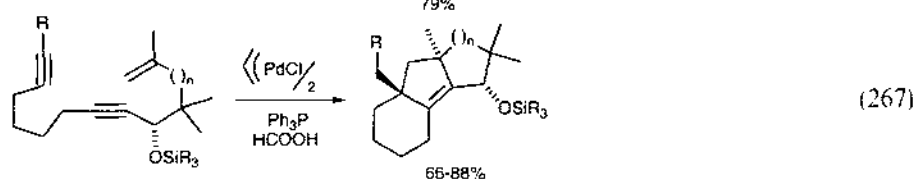
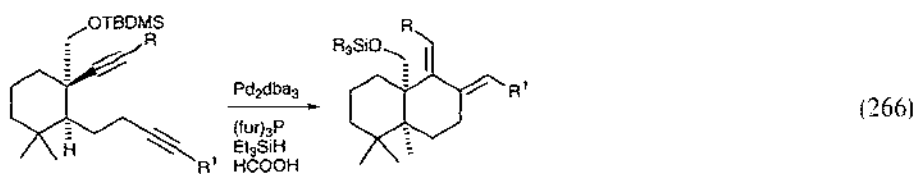


Hepta-1-ene-6-yne cyclized to 1-vinylcyclopentenes with platinum(II) chloride as a catalyst [758]. Other enyne cyclizations are shown in Eq. (261) [759], Eq. (262) [760] and Eq. (263) [761].



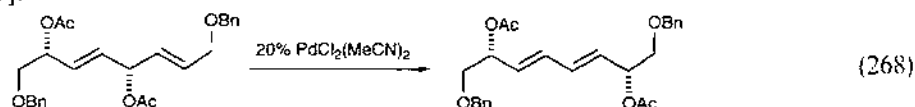
$\alpha$ -Diallylbenzene cyclized to the seven-membered ring with an exocyclic double bond under  $\text{Cp}_2\text{ZrCl}_2/\text{MAO}$  catalysis [762]. Other miscellaneous cycloisomerizations are shown in Eq. (264) [763], Eq. (265) [764], Eq. (266) [765] and Eq. (267) [766].





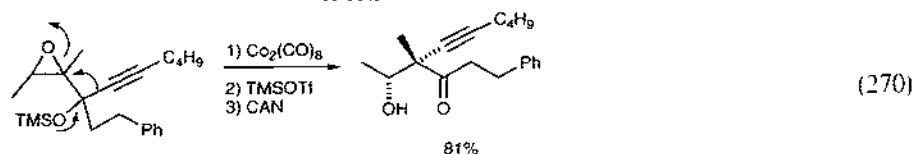
### 2.5.3. Rearrangements of allylic and propargylic compounds

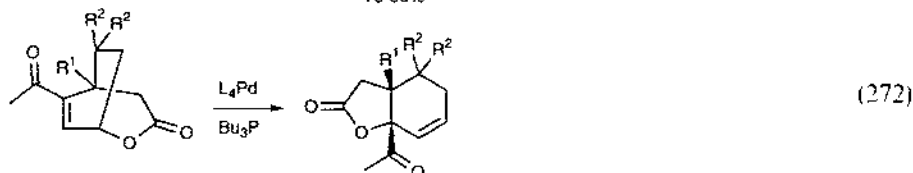
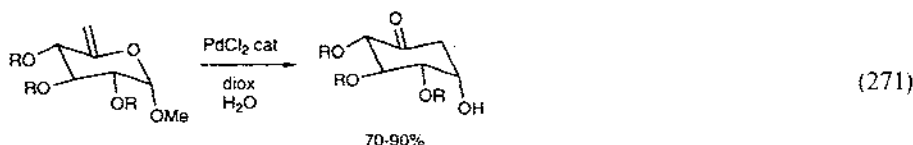
Propargyl alcohols were isomerized to  $\alpha,\beta$ -unsaturated aldehydes by  $\text{MoO}_2(\text{acac})/\text{Bu}_2\text{SnO}$  [767]. Allyl ethers were isomerized to enol ethers by  $\text{L}_3\text{RhCl}/\text{BuLi}$  [768,769]. Palladium(II) salts catalyzed the isomerization of allyl ethers of 2-hydroxypyridine to *N*-allylpyridones [770], and the isomerization of non conjugated dienyl acetates to conjugated ones (Eq. (268)) [771]. Palladium(0) isomerized allyl sulfonates to *N*-sulfonyl allyl amines [772]. Rhodium(I) complex catalyzed the isomerization of allyl vinyl ethers to give 4-alkenyl aldehydes (oxy Cope) followed by addition of the aldehyde to the alkene to give cyclopentanones [773].



### 2.5.4. Skeletal rearrangements

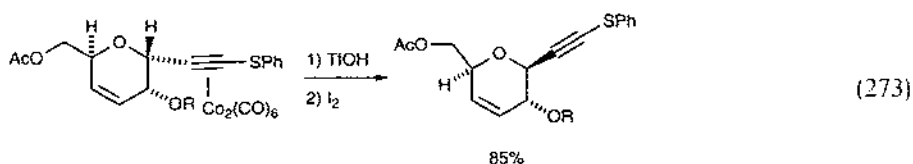
A paper dealing with skeletal rearrangements from the products of addition of perfluorophenyl palladium(II) to 1,4-pentadiene has appeared [774]. Optically active *trans*-2-silyl-3-vinyl epoxides rearranged to *anti*-3-silyl-4-hydroxy-1-alkenes in good yield and high ee [775]. Other skeletal rearrangements are shown in Eq. (269) [776], Eq. (270) [777], Eq. (271) [778] and Eq. (272) [779].





### 2.5.5. Miscellaneous rearrangements

“Molecular Gymnastics of Alkynes Orchestrated by Ruthenium Complexes” (37 references) was the title of a review [780]. Cobalt alkyne complexes of propargyl ethers underwent epimerization in the presence of triflic acid (Eq. (273)) [781]. 2-Vinyl aziridines were epimerized by palladium(0) complexes, with the *cis* compound being favored [782,783]. Complexation of 1,4-disubstituted cyclohexadienes to iron carbonyl in the presence of chiral binap diene ligands led to up to 66% ee (but 9% yield) of the optically active iron cyclohexadiene complex [784]. Iron pentacarbonyl rearranged methyl oleate to 4-ethyl-4-pentenoate [785].



## 3. Functional group preparation

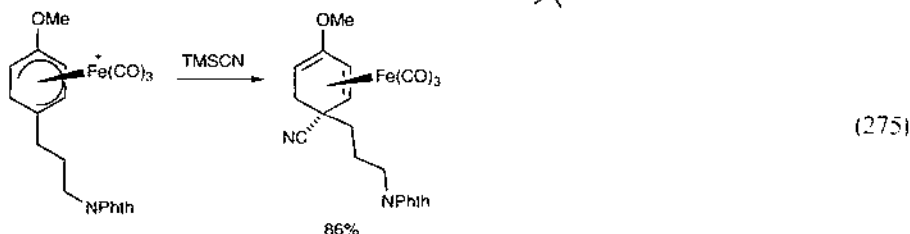
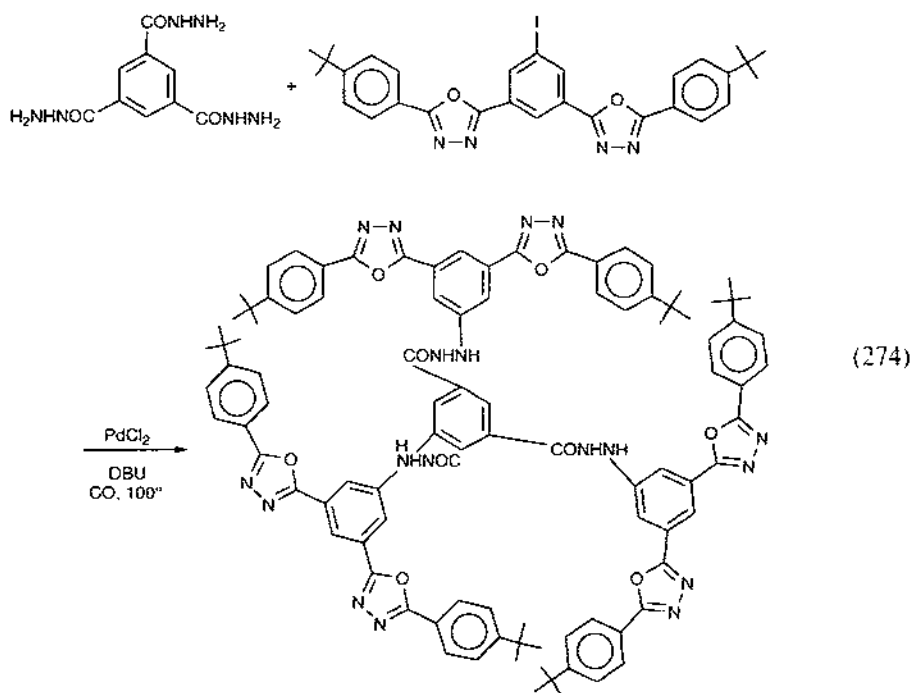
### 3.1. Halides

Tungsten hexachloride converted benzyl alcohols and benzaldehydes to benzyl chlorides [786]. It converted epoxides to 1,2-dichlorides. DAST replaced allyl OH groups on iron tricarbonyl diene complexes [787] and benzyl alcohols on chromium carene complexes of 1-hydroxy tetralin [788] with fluoride stereospecificity. Alkenyl iodides were made by hydrozirconation of alkynes (*syn* 4-methyl-5-methoxy-6-phenylhex-2-yne) followed by cleavage with iodine [789]. Alkynyl borates were hydrozirconated, alkylated at the Zr position, then the borate cleaved by NBS or NIS to give the *trans* alkenyl halide [790].

### 3.2. Amides, nitriles, azides

Nickel(0) complexes stoichiometrically converted cyclopentene to cyclopentene-3-carboxamide by treatment with phenyl isocyanate [791]. Palladium complexes

catalyzed the carbonylative coupling of aryl iodides with acid hydrazides (Eq. (274)) [792].  $\text{Cp}_2\text{ZrHCl}$  deoxygenated amides converting them into imines by elimination of the carbonyl oxygen and an N–H [793]. Iron tricarbonyl complexes of dialdehydes were cleanly converted to imines by treatment with primary amines [794]. Iron vinylketene complexes were converted to iron vinyl keteneimine complexes by reaction with isonitriles [795]. In the nickel(0) catalyzed addition of HCN to styrene to give the benzyl nitrile, the ee of the process was dramatically altered by changing the electronics of the aryl groups on the diphosphine ligand [796]. Trimethylsilyl cyanide cleanly alkylated iron diene complexes (Eq. (275)) [797]. Optically active chromium salen complexes catalyzed the asymmetric ring-opening of epoxides [798] including epichlorohydrin [799] by trimethylsilylcyanide, with very high ee (kinetic resolution). A review dealing with palladium-catalyzed addition of azide to allylacetates has appeared (10 references) [800]. An example is seen in Eq. (276) [801].

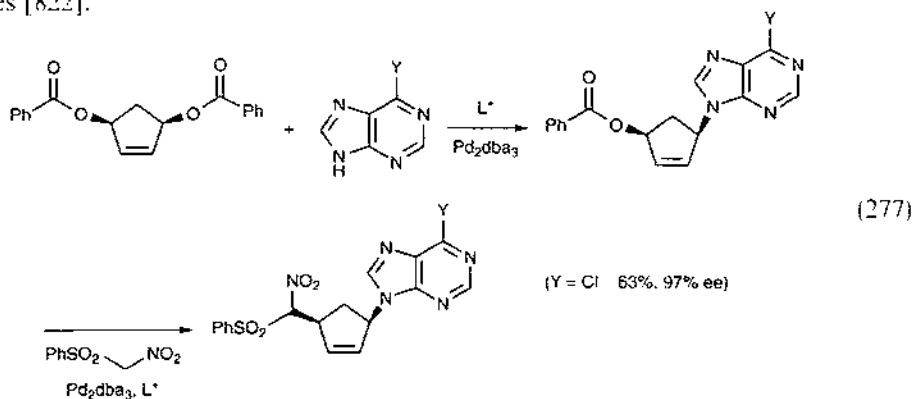




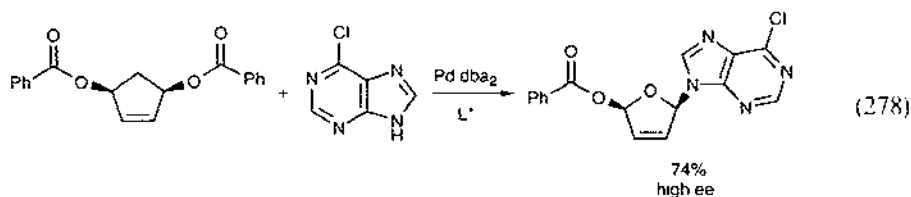
### 3.3. Amines, alcohols

Palladium(0) catalyzed amination of aryl bromides and iodides has rapidly developed into an efficient process and was the subject of numerous papers [802–805]. Heteroaromatic halides such as bromopyridines and bromoquinolines were aminated [806], and aryl halides were monoaminated by 1,4-diazacyclohexane [807]. Aryl dihalides and diamines oligomerized under these conditions [808]. Polymer-bound *p*-bromobenzamides were cleanly aminated and cleaved from the resins [809,810]. Aryl halides were aminated by optically active  $\alpha$ -amino acids without racemization utilizing  $\text{PdCl}_2(\text{otol}_3\text{P})/\text{K}_2\text{CO}_3/\text{DMF}/\text{H}_2\text{O}/\text{Et}_3\text{N}/\text{CuI}$ , TEBA catalysts [811].  $\text{Pd}_2(\text{dba})_3$  catalyzed the amination of aryl iodonium salts [812].

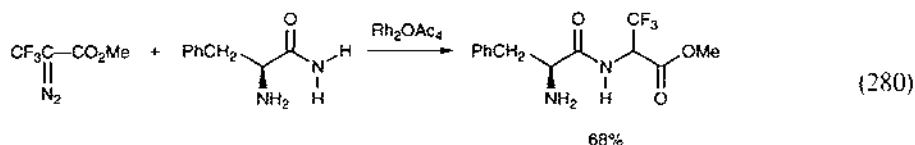
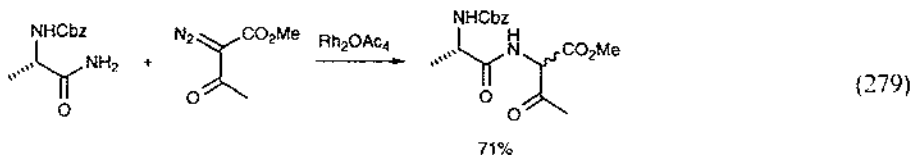
Palladium-catalyzed asymmetric allylic amination was the subject of a theoretical treatment [813]. Very high ee was obtained in the allylic amination of the all-time favorite substrate for asymmetric allylic reactions, 1,3-diphenyl-3-acetoxyprop-1-ene [814]. Allyl epoxides were asymmetrically aminated by phthalimide in the presence of palladium catalyst and optically active *bis*-phosphines [815,816]. This was extensively used in the synthesis of carbocyclic nucleoside analogs (Eq. (277)) [817–820], nucleoside analogs (Eq. (278)) [821] as well as simple *N*-allyl purine and pyrimidine bases [822].



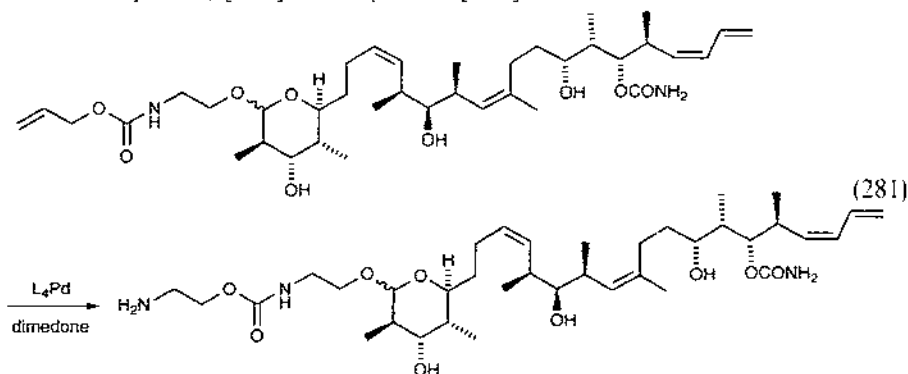
Palladium(0) catalyzed allylic amination of polymer-bound allyl acetates was also efficient [823]. Allylic amination of uracils occurred at  $\text{N}_1$ , but thiouracils underwent reaction both at nitrogen and sulfur, under palladium catalysis [824]. Allyl alcohols were converted to allyl amines by treatment with secondary amines and a palladium(0) catalyst in the presence of carbon dioxide [825]. Palladium(0) catalyzed the amination of optically active propargyl mesylates with retention [826].

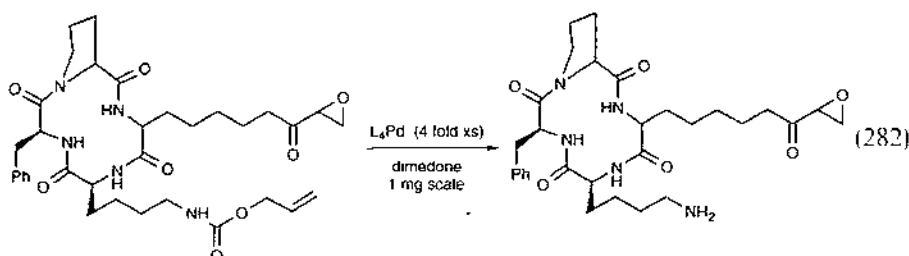


Rhodium(II) acetate catalyzed the insertion of  $\alpha$ -diazocarbonyl compounds into N–H bonds to make amines [827], (Eq. (279)) [828]. Trifluoroalanines were made this way (Eq. (280)) [829] as were  $\alpha$ -amino phosphoric acids [830]. Ruthenium(II) complexes catalyzed similar reaction of  $\alpha$ -diazocarbonyl compounds [831].

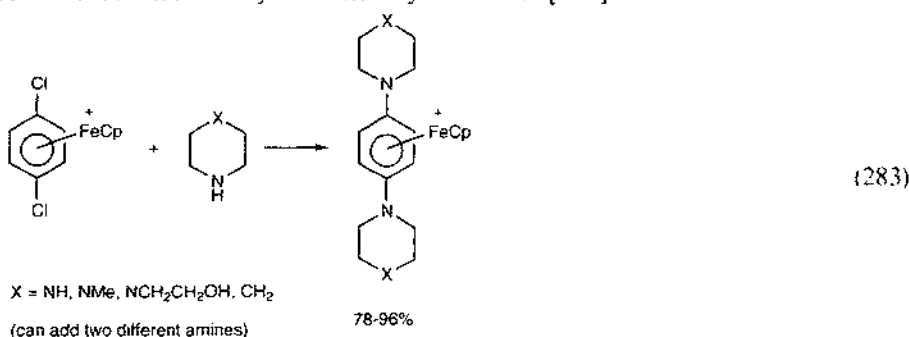


Palladium-catalyzed hydrogenolysis of allylic and propargylic compounds with various hydrides has been reviewed (134 references) [832]. Many systems for removing alloc protecting groups involving palladium catalysis have been developed, including  $L_2PdCl_2/Bu_3SnH$  to remove NH alloc groups from tricyclic homoleptic peptides [833],  $Pd(OAc)_2/tppts/H_2O/NaN_3/CH_3CN$  to remove *o*-alloc groups [834], and  $Pd_2dba_3/dppb/o$ -thiobenzoic acid for the selective monodeallylation of diallyl amines in the presence of Boc- and Troc groups [835]. Examples of alloc deprotection are shown in Eq. (281) [836] and Eq. (282) [837].

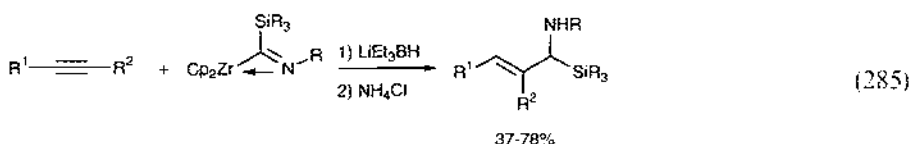
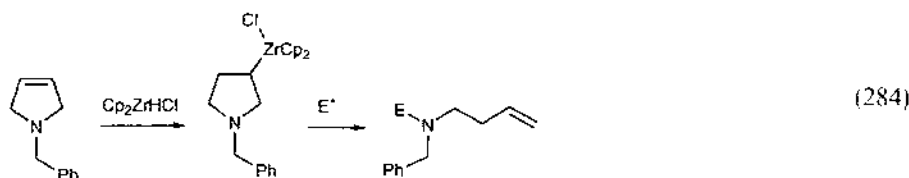




Racemic phenethylamine was partially resolved (38% ee) using optically active dienyiron tricarbonyl complexes [838]. Iron-complexed 1,4-dichlorobenzene could be differentially diaminated in good yield (Eq. (283)) [839]. Ammonia aminated cyclohexadienyl iron tricarbonyl complexes twice, to give the symmetrical 2 amine having two cyclohexadiene iron groups [840]. Chromium tricarbonyl complexed fluorobenzenes were readily aminated by 2 amines [841].



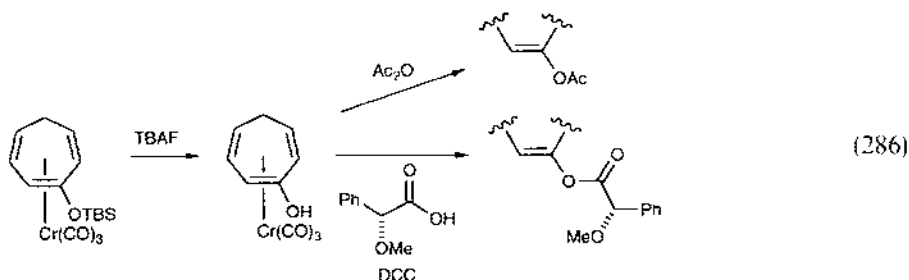
Imines were reduced to optically active amines by phenylsilane in the presence of an optically active titanocene difluoride [842]. 2,4-Dihydropyrroles were ring-opened by hydrozirconation (Eq. (284)) [843]. Dihydrofurans underwent a similar process. Homoallyl amines were made as in Eq. (285) [844].



Alkyl(cyano)cuprates converted lithiated secondary amines to tertiary amines under oxidizing conditions [845]. Cyclohexene was allylically aminated by reaction

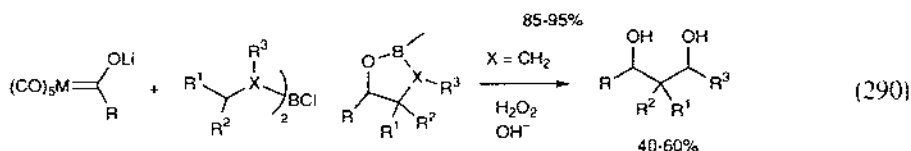
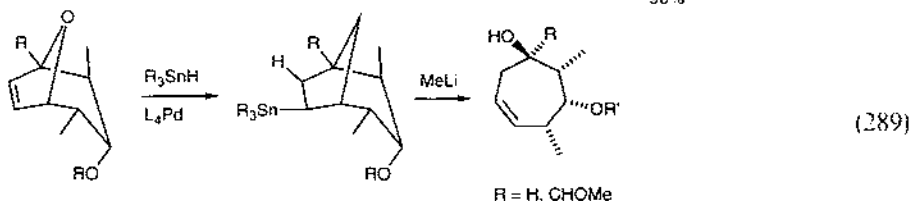
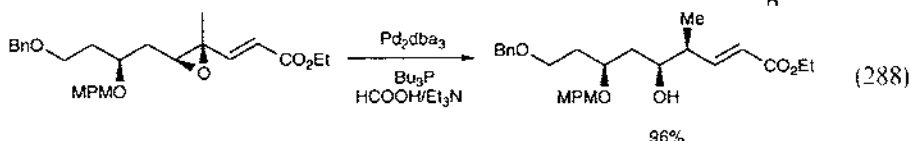
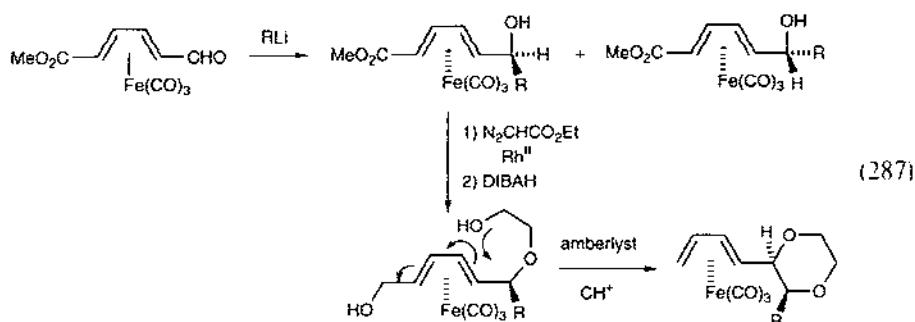
with nitrobenzene and carbon monoxide in the presence of  $\text{Ru}_3(\text{CO})_{12}$  [846]. Other systems which allylically aminated olefins were  $\text{LMoO}(\text{O}_2)_2/\text{R}'\text{OOH}/\text{ArNH}_2$  [847] and  $\text{ArNHOH}/\text{FeCl}_3$  [848]. Chromium complexes catalyzed the ring-opening of *N*-tosyl aziridines by trimethylsilylazide [849]. Iron tricarbonyl complexes of benzalacetone imines were converted to iron tricarbonyl complexes of 2-amino-1,3-dienes by reaction with  $\text{PhCH}_2\text{NHLi}$  [850].

Aryl ketones were asymmetrically reduced to alcohols with high enantioselectivity by rhodium(I) [851,852] and iridium(I) [853] catalyzed hydrosilylation-hydrolysis. Ruthenium(II)-catalyzed transfer hydrogenation of aryl ketones to alcohols also proceeded with high enantioselectivity [854]. Optically active allyl alcohols were prepared by the ruthenium(II) catalyzed cleavage of the corresponding allyl carbonate [855]. TBDMS ethers of phenols were deprotected by treatment with acetone, water and palladium(II) chloride catalysts [856]. Aliphatic ethers of phenol were readily cleaved to the phenoxide by forming the cationic  $\text{CpFe}$  arene complex followed by treatment with potassium *t*-butoxide [857]. Deprotection of the chromium tricarbonyl complex of 3-silyloxy-1,3,5-cycloheptadiene gave the complexed enol, which could be utilized synthetically (Eq. (286)) [858].



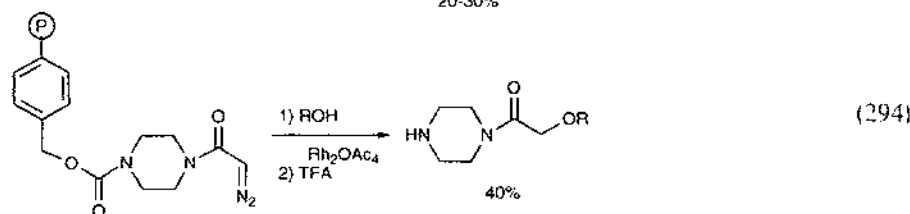
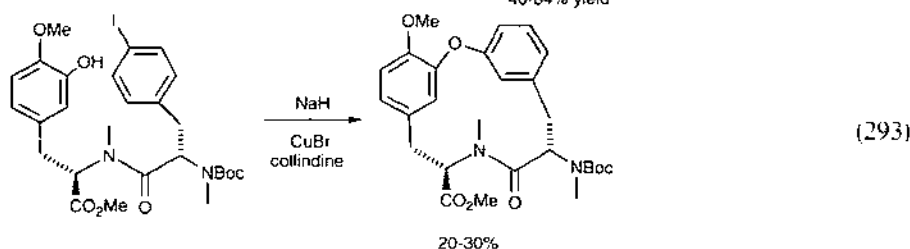
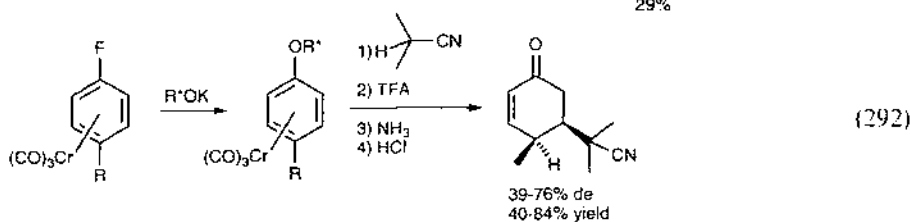
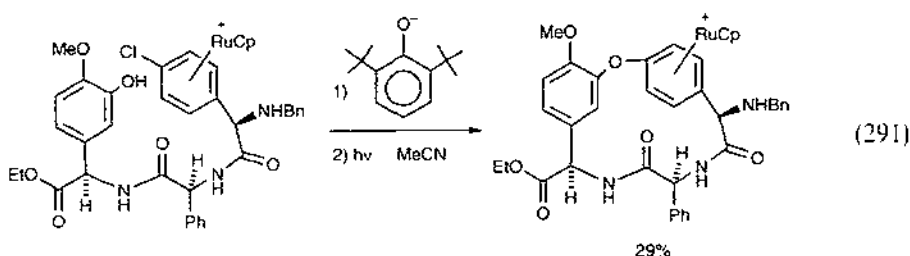
Bakers yeast reduced iron-complexed deuterodienals to optically dienol complexes with 100% ee [859]. Racemic allyl acetates were dynamically resolved into a single optically active allyl alcohol by an enzyme, while palladium catalysts were used to equilibrate the racemic acetate to supply the correct enantiomer to the enzyme [860].

Alcohols were made by alkylation of iron dienal complexes, and underwent further useful reaction chemistry (Eq. (287)) [861]. Iron tricarbonyl complexes of dienes with adjacent alkenes underwent moderate yield *cis* dihydroxylation of the alkene with  $\text{OsO}_4$  [862].  $\text{Pd}_2\text{dba}_3/\text{HCOOH}$  regio and stereospecifically reduced allyl epoxides (Eq. (288)) [863]. Cyclohexenals were made by ring-opening of furan Diels Alder adducts (Eq. (289)) [864]. Osmium (2+) complexed enol ethers were subject to nucleophilic substitution and were converted to complexed enols ( $\text{H}_2\text{O}$ ), enol ethers ( $\text{ROH}$ ), enamines ( $\text{PhNH}_2$ ) and acetates ( $\text{AcO}$ ) [865].  $\text{Rh}_2\text{Cl}_2(\text{COD})_2$  catalyzed the hydrogenolytic ring-opening of cyclobutanones to butanols [866].  $\alpha$ -Keto phosphonates were reduced to  $\alpha$ -hydroxyphosphonates with high ee using ruthenium BINAP catalysts [867]. Rhodium(I) complexes catalyzed the hydroboration of alkenyl boranes to give 1,2-*bis* boranes, oxidation of which gave diols [868]. 1,3-Diols were prepared by the unusual chemistry in Eq. (290) [869].

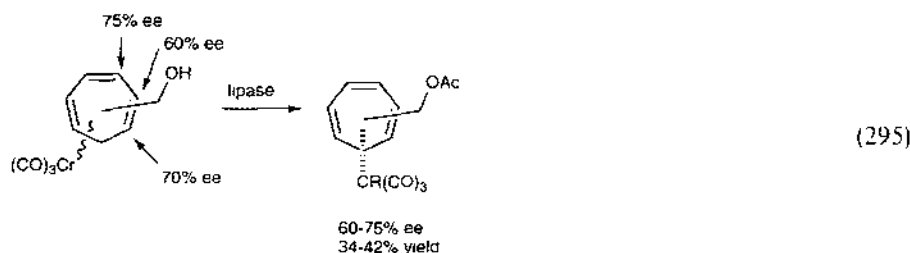


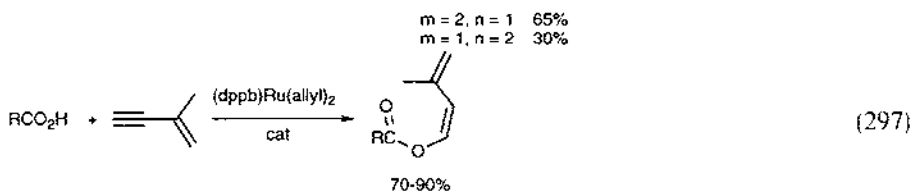
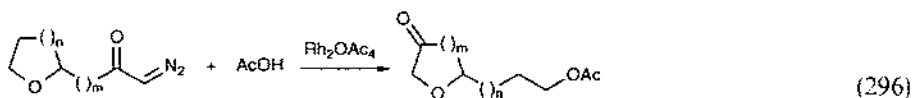
### 3.4. Ethers, esters, acids

The cationic  $\text{CpRu}$  complexes of haloarenes underwent reaction with phenoxides to give diaryl ethers [870]. This was used intramolecularly (Eq. (291)) [871] and intermolecularly [872] in the context of vancomycin synthesis. Fluorobenzenechromium tricarbonyl complexes underwent reaction with alkoxides of optically active alcohols to give ethers which were further functionalized (Eq. (292)) [873]. The cationic iron dienyl complex of cyclohexadiene phenyl sulfone underwent nucleophilic attack by alkoxide to form aryl ethers after oxidation [874]. Aryl ethers were formed by the arylation of phenols by aryl iodides using copper(I) bromide in collidine (Eq. (293)) [875]. Palladium(0) catalyzed the coupling of aryl bromides with sodium *t*-butoxide to make aryl *t*-butyl ethers [876]. Cyclopentadiene monoxide underwent palladium(0) catalyzed ring-opening with phenol to give 1-phenoxy-4-hydroxycyclopent-2-ene [877]. Rhodium(II) catalyzed the insertion of  $\alpha$ -diazoketones into alcohol O–H bonds (Eq. (294)) [878].



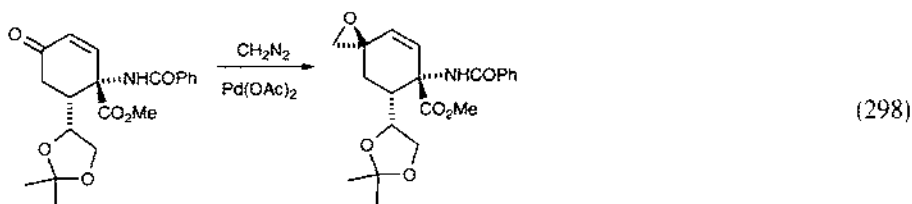
The racemic chromium-complexed “benzyl” alcohol of cycloheptatriene was acetylated by lipase, and resolved in this way (Eq. (295)) [879]. Rhodium(II) acetate catalyzed insertion of diazo ketones into the OH bond of carboxylic acids (Eq. (296)) [880]. Ruthenium(II) complexes catalyzed the addition of carboxylic acids to enynes to give dienol esters (Eq. (297)) [881].



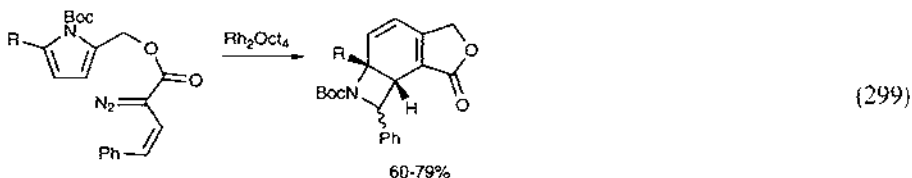


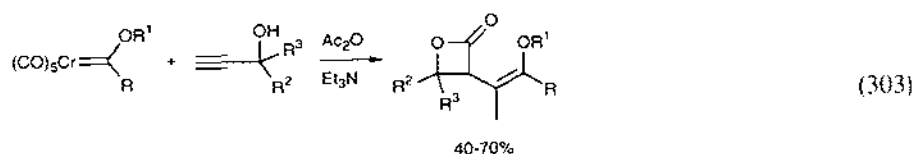
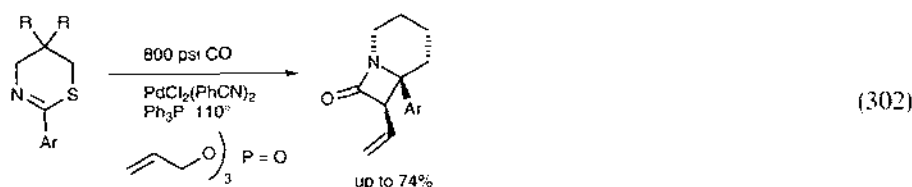
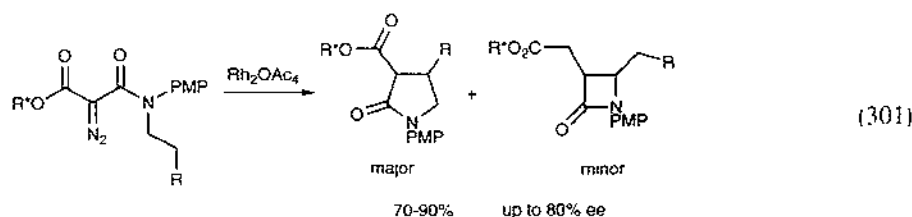
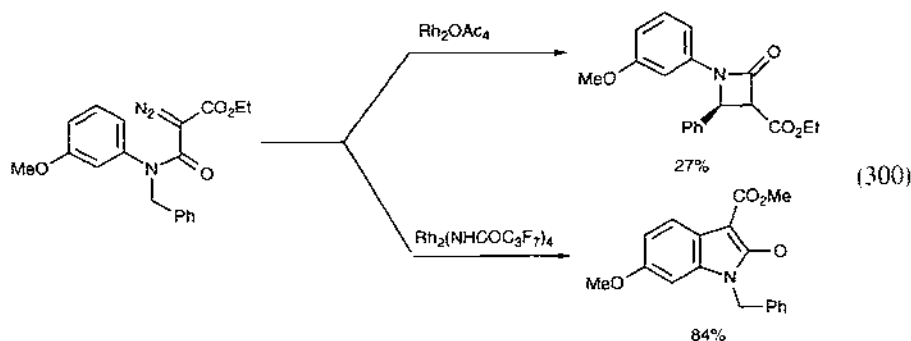
### 3.5. Heterocycles

Azirenes were made by the rhodium(II) acetate catalyzed reaction of alkenes with  $\text{NsN} - \text{I}^{\text{Ph}}$  in 40–80% yield [882], and by the rhodium(II) acetate catalyzed reaction between imines and phenyl diazomethane [883]. A wide range of olefins were converted to epoxides by hydrogen peroxide/1%  $\text{MeReO}_3$  catalyst [884]. Palladium acetate catalyzed the conversion of a functionalized cyclohexenone to its epoxides (Eq. (298)) [885]. Thiiranes were made by the rhodium(II) catalyzed decomposition of stable diazo compounds with thioketones [886] and thioketenes [887] to give methylenethiiranes.



Azetidines were prepared by the rhodium(II) catalyzed decomposition of diazoketones adjacent to pyrroles (Eq. (299)) [888].  $\beta$ -Lactams were made by rhodium(II) catalyzed insertions of diazoamides (Eq. (300) [889] and Eq. (301) [890]), by dicobalt octacarbonyl carbonylation of aziridines [891] and by the palladium-catalyzed allyl carbonylation of thiazines (Eq. (302)) [892]. The use of  $\pi$ -allyl iron tricarbonyl complexes in the synthesis of lactams and lactones has been reviewed (64 references) [893]. Propargyl alcohols reacted with chromium carbene complexes to give  $\beta$ -lactones (Eq. (303)) [894].

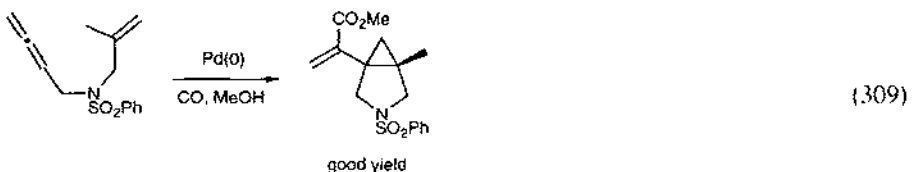
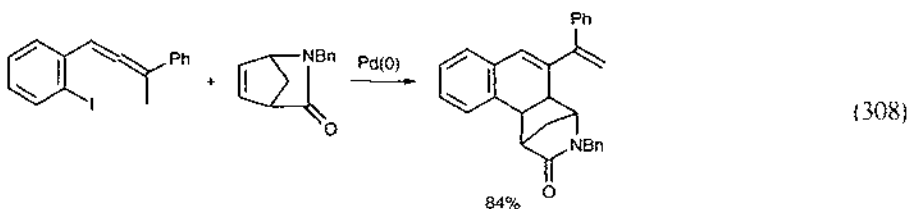
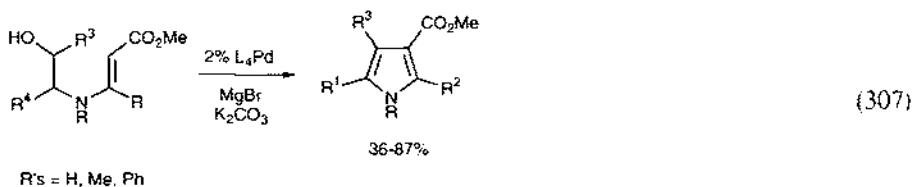
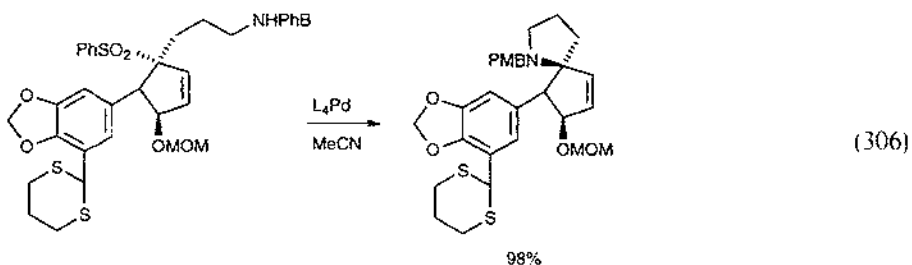
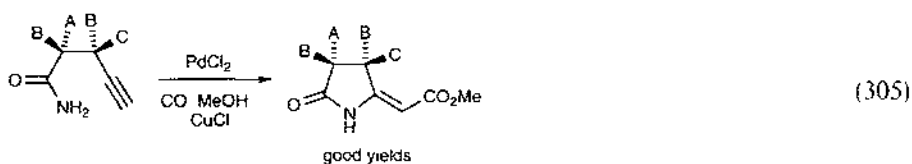
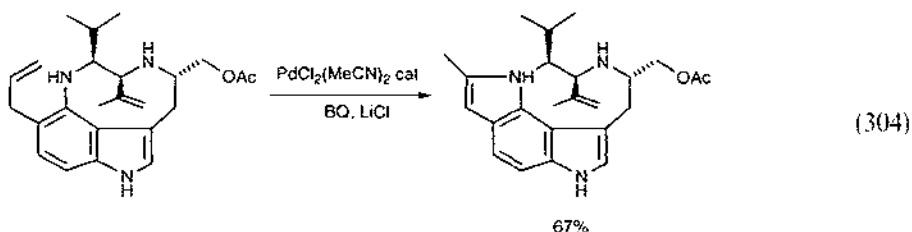




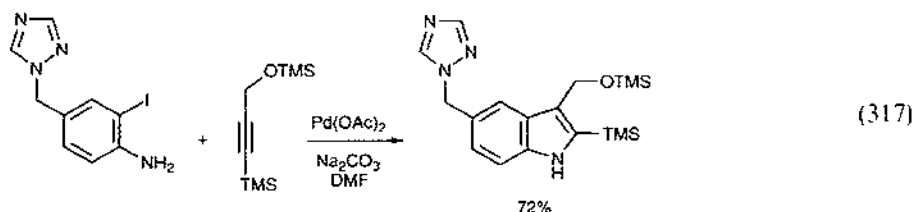
Five-membered nitrogen heterocycles were synthesized by palladium(II) catalyzed amination of olefins (Eq. (304)) [895], aminocarbonylation of alkynes (Eq. (305)) [896], the palladium(0) catalyzed amination of allylic sulfones (Eq. (306)) [897] and the palladium(0) catalyzed cyclization of amino alcohols (Eq. (307)) [898]. They were also prepared by palladium(0) catalyzed cascade reactions (Eq. (308) [899], Eq. (309) [900] and Eq. (310) [901]).

Other syntheses of five-membered nitrogen heterocycles are shown in Eq. (311) [902], Eq. (312) [903], Eq. (313) [904], Eq. (314) [905], Eq. (315) [906] and Eq. (316) [907].

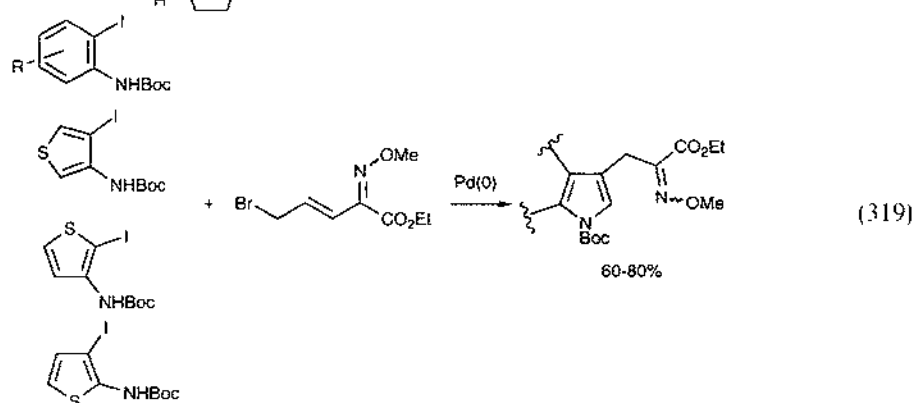
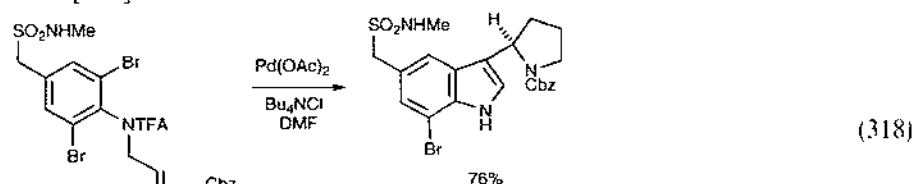
Indoles were synthesized by the palladium-copper catalyzed reaction of *o*-iodoanilines with alkynes [908] (Eq. (317)) [909,910], while allenes produced 3-methylene indolines [911,912] and vinyl cyclopropanes produced 2-ethenyl indolines [913].





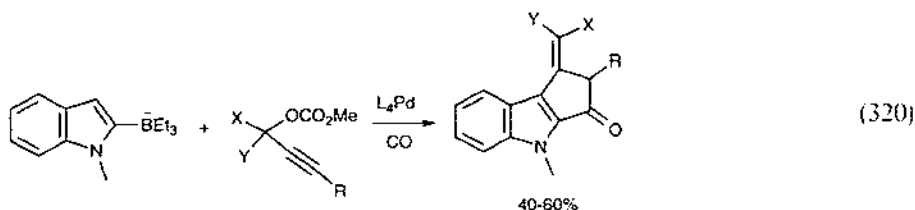


2-Bromo-*N*-allylanilines underwent palladium(0) catalyzed cyclization to indoles [914,915], (Eq. (318)) [916], and 2-iodoanilides reacted with allyl bromides to give indoles (Eq. (319)) [917] and with enol triflates and carbon monoxide to give oxindoles [918].

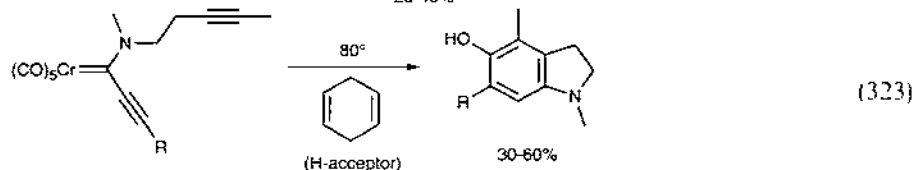
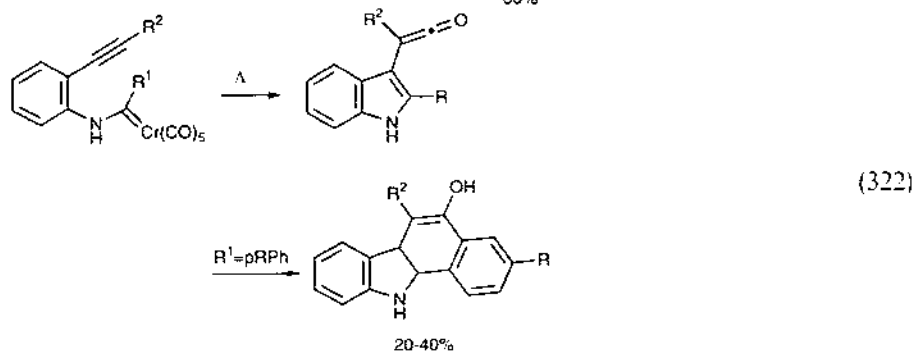
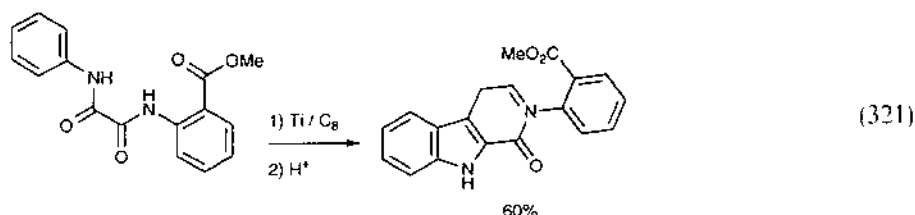


A range of nitrogen heterocycles were prepared by the  $\text{Pd}(\text{OAc})_2 \cdot \text{O}_2 \cdot \text{DMSO}$  catalyzed cyclization of olefinic tosamides ( $\text{Pd}^{\text{II}}$  catalyzed amination of olefins) [919]. These included indoles and dihydroquinolines. Indolines and tetrahydroquinolines were prepared by the intramolecular amination of aryl halides using palladium catalysts [920]. Indolines were also prepared by the stoichiometric reaction between 2-phenyl-2-methylpropylmagnesium chloride, nickel(II) chloride *bis* phosphines and aryl or tosyl azide [921]. A synthesis of elaborated indoles is shown in Eq. (320) [922].

Another spate of papers deal with the synthesis of carbazoles by the alkylation of cationic iron cyclohexadiene complexes with anilines, followed by oxidative cyclization to form the indole ring [923–926]. *Ortho* acyl acetanilides were reductively cyclized to indoles using McMurry coupling (Eq. (321)) [927]. Excess  $\text{Ni}(\text{COD})_2$

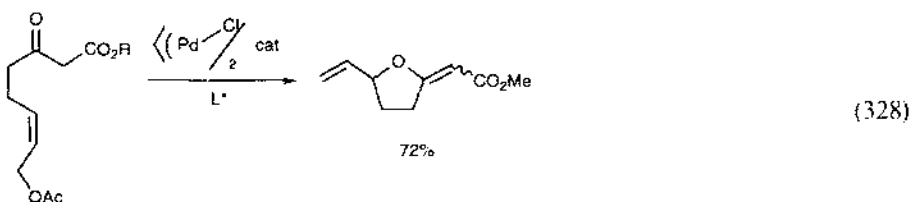
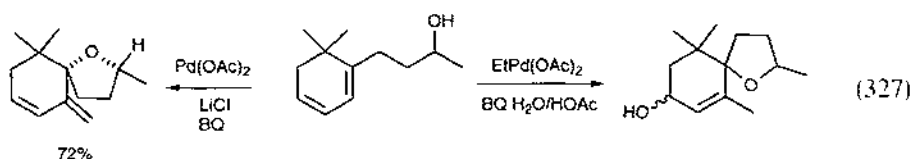
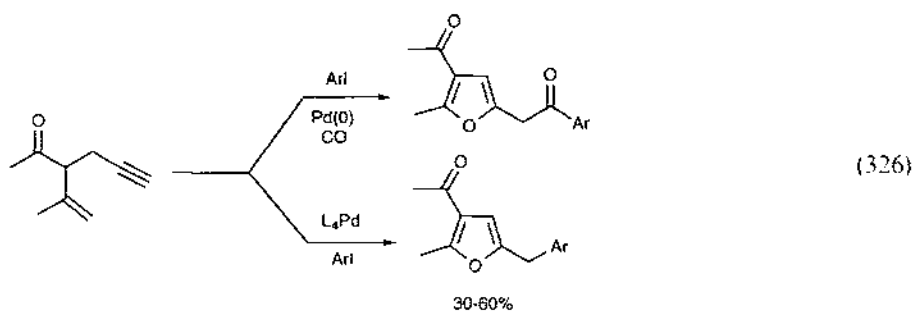
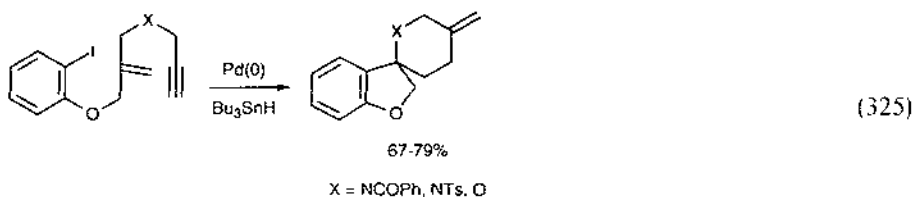
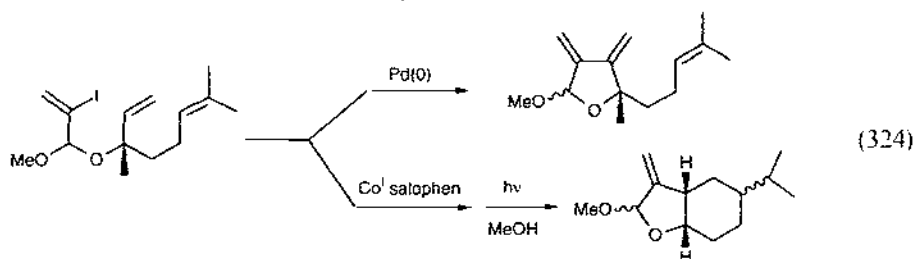


promoted the formation of indolines by the reductive coupling of nitroarenes with *o*-attached cyclohexenones [928]. Indoles (Eq. (322)) [929] and indolines (Eq. (323)) [930] were available from chromium carbene chemistry.

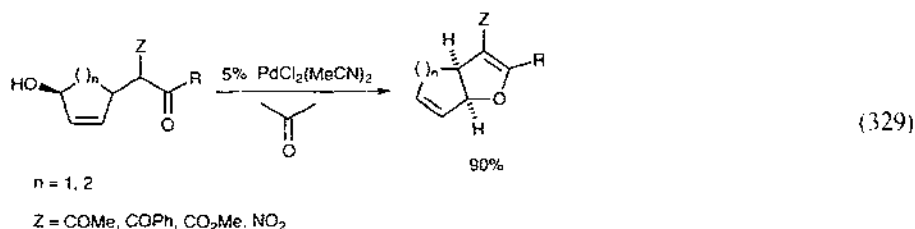


Saturated, benz-fused oxygen heterocycles were synthesized by the palladium(0) catalyzed alkoxylation of aryl halides by *ortho*-aliphatic alcohol groups [931]. Palladium/copper systems catalyzed the reaction of *o*-halophenols with alkynes to give benzofurans [932]. Ruthenium carbene complexes catalyzed the conversion of pent-1-yne-2-ene-5-ol to 2,3-dimethylfuran [933]. Oxygen heterocycles were made by palladium(0) catalyzed cascade reactions (Eq. (324) [934], Eq. (325) [935] and Eq. (326) [936]), by palladium(II) catalyzed alkoxylation of alkenes (Eq. (327)) [937], and by palladium(II) catalyzed  $\pi$ -allyl chemistry (Eq. (328) [938] and

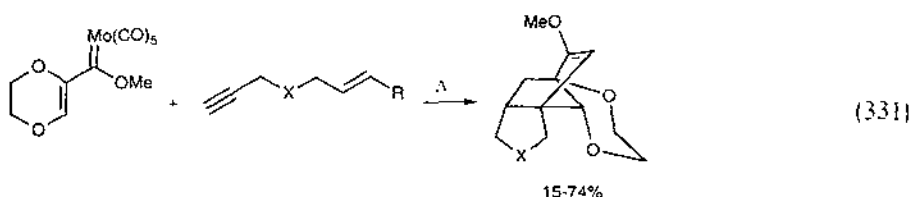
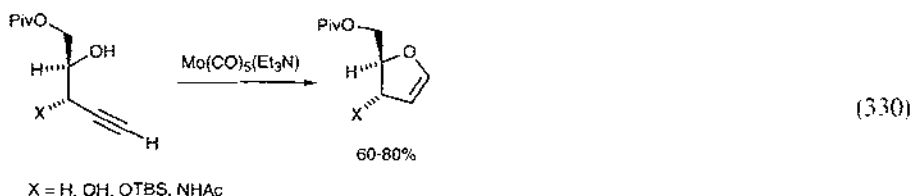
Eq. (329) [939]). The regiospecific synthesis of 3,4-disubstituted furans via Stille coupling was reviewed (24 references) [940].



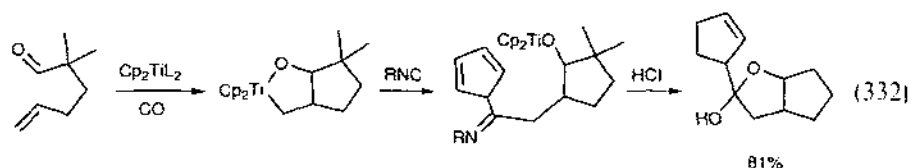
Tetrahydrofurans [941] and dihydrofurans [942] were synthesized by the rhodium(II) catalyzed CH insertion of diazoalkanes in ethers. Molybdenum car-



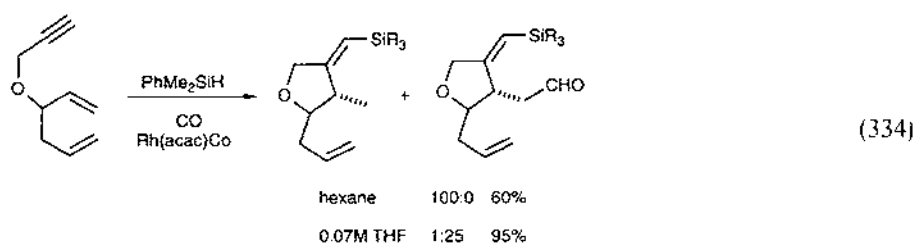
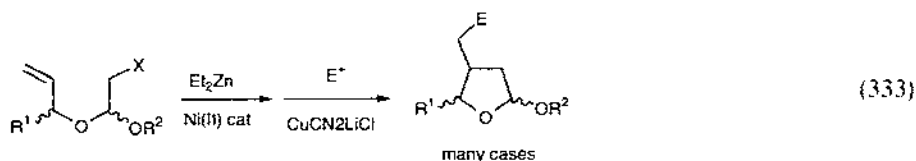
bonyl (Eq. (330)) [943] and chromium carbene complexes [944] converted homopropargyl alcohols to dihydrofurans. Molybdenum carbene complexes converted allyl propargyl ethers to tetrahydrofurans (Eq. (331)) [945]. The full paper on the reaction of propargyl tungsten complexes with aldehydes to give furan derivatives has appeared [946].



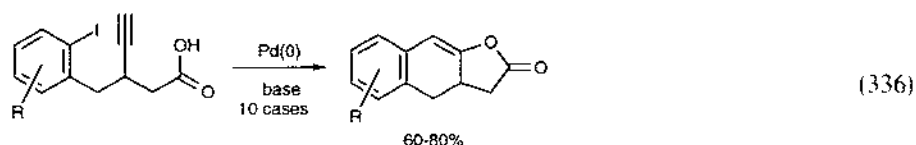
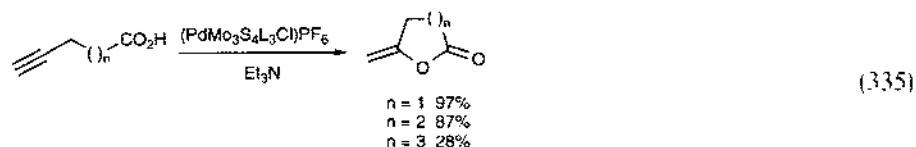
Cyclohexadieneiron complexes having an exocyclic double bond underwent 1,3-dipolar cycloaddition reactions to give heterocycles [947]. Oxidation of cyclobutadieneiron complexes having pendent ether groups produced tetrahydrofurans by 2+2 cycloaddition of the alkene to cyclobutadiene [948]. Reduced titanocenes cyclized  $\delta$ -olefinic aldehydes with isonitriles and CO to give tetrahydrofurans [949] (Eq. (332)), and epoxy propargyl ethers to give methylene tetrahydrofurans [950]. Other syntheses of tetrahydrofurans are seen in Eq. (333) [951] and Eq. (334) [952].



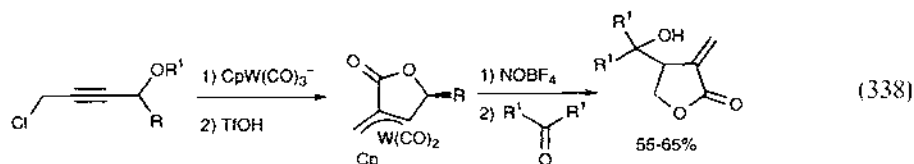
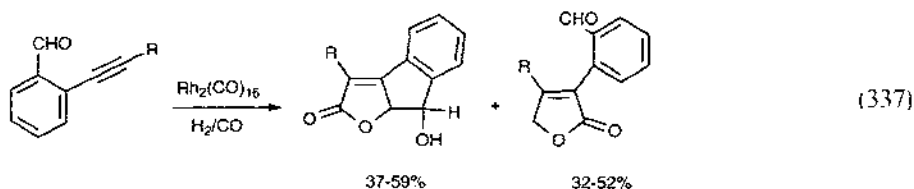
Palladium/copper systems catalyzed the cyclocarbonylation of homoallyl alcohols to give butyrolactones [953] and the cyclization of homopropargyl alcohols to



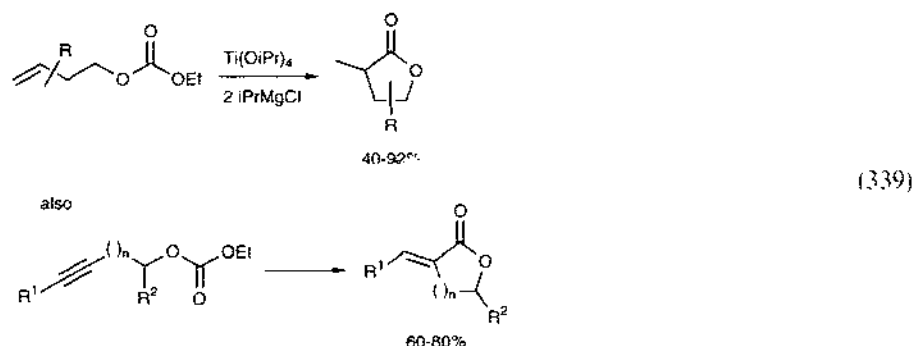
butyrolactones [954], while palladium(0) complexes catalyzed the reaction of aryl halides with  $\gamma$ -hydroxy acrylates to give 3-aryl butenolides [955].  $\gamma$ -Methylene lactones were available by the palladium(II) catalyzed oxidative cyclization of terminal olefins with acrylic acid [956] and the palladium(II) catalyzed halocyclization of chloroallyl esters of propionic acids [957]. Alkynoic acids were cyclized to methylene lactones by palladium/molybdenum systems (Eq. (335)) [958] and by the palladium(0) cascade reaction (Eq. (336)) [959].



Palladium(II) complexes catalyzed the cyclocarbonylation of *o*-allylphenols to benz-fused five- (up to 77%), six- (up to 70%) and seven- (up to 95%) membered lactones, depending on conditions [960].  $\alpha$ - and  $\beta$ -naphthols underwent reaction with aldehydes in the presence of palladium acetate catalysts to give butyrolactones benz-fused at the  $\alpha$ - and  $\beta$ -position [961]. 1-Iodo-2,3-dimethoxybenzene was oxidatively dimerized (Pd(OAc)<sub>2</sub>/PCC) to tetramethoxydibenzo six-membered lactones [962]. Rhodium carbonyls catalyzed the production of butenolides from *o*-ethynylbenzaldehydes via the water gas shift reaction (Eq. (337)) [963]. (2-Cyclohexadienecirontricarbonyl)furan was oxidized by singlet oxygen to the 4-methoxy-4-cyclohexadienyl butenolide in low yield [964].  $\alpha$ -Methylene lactones were prepared via  $\pi$ -allyltungsten chemistry (Eq. (338)) [965].



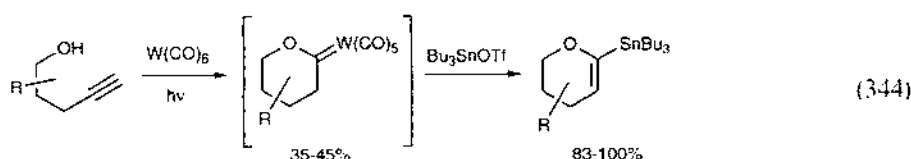
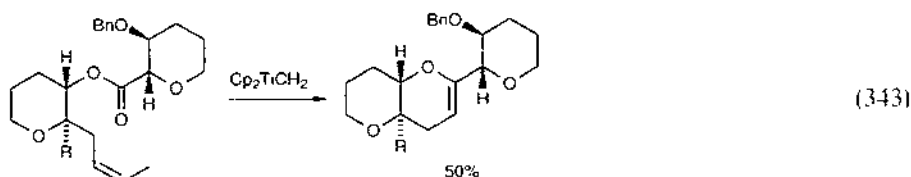
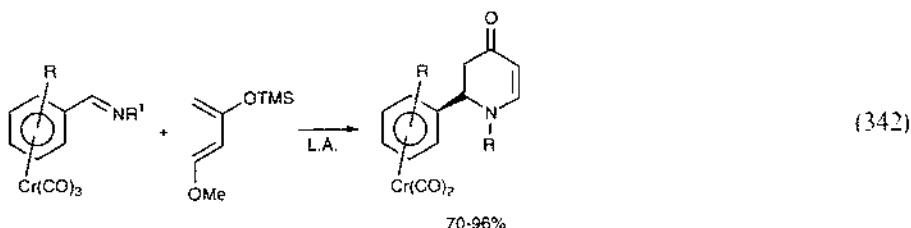
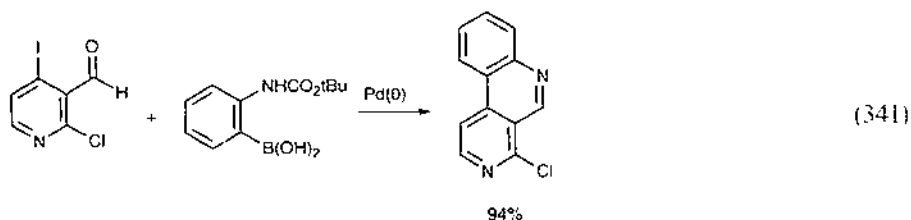
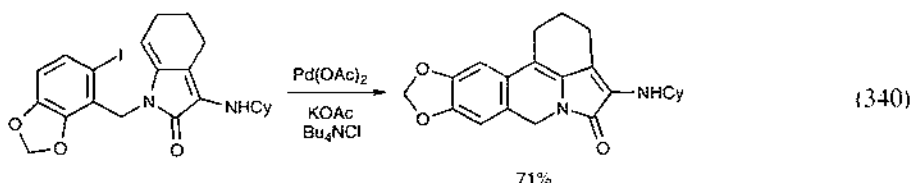
A full paper on the synthesis of butyrolactones by rhodium(II) carboxylate catalyzed C–H insertion reactions of diazo esters [966], including diastereoselection with chiral complexes, has appeared [967]. Lactones were prepared by low-valent titanium cyclization of unsaturated esters (Eq. (339)) [968].



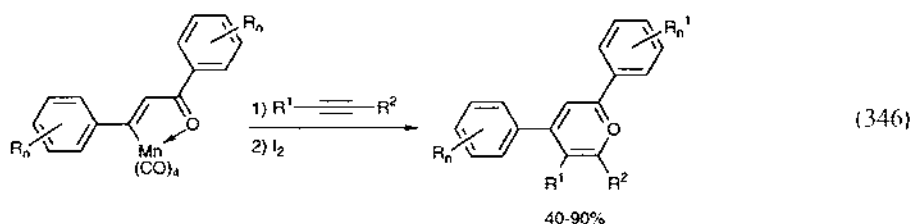
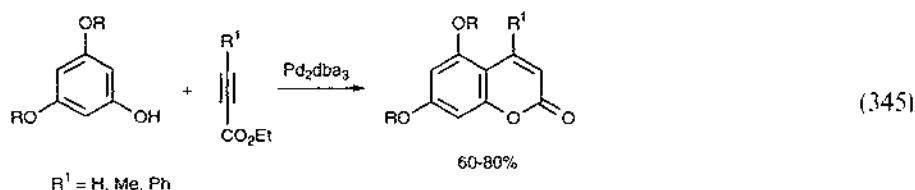
Six-membered nitrogen heterocycles were prepared by palladium(0) catalyzed oxidative addition/insertion chemistry (Eq. (340)) [969–972], oxidative addition/transmetalation chemistry (Eq. (341)) [973] and orthopalladation/insertion chemistry [974]. Pyridones were made by hetero Diels–Alder reaction of chromium tricarbonyl complexed benzaldimines (Eq. (342)) [975] and rhodium(II) carboxylate catalyzed diazoester N–H insertion chemistry [976]. Iron  $\eta^6$ -phosphimine complexes catalyzed the co-cyclotrimerization of alkynes with nitriles to give pyridines [977].

Palladium(II) complexes catalyzed the hetero Diels–Alder reaction between dienes and aldehydes to form dihydropyranes [978]. Dihydropyrans were made by metathesis chemistry after Tebbe olefination (Eq. (343)) [979], palladium(0) catalyzed intramolecular hydroxylation of propargyl acetates [980] and tungsten hexacarbonyl cyclization of acetylenic alcohols (Eq. (344)) [981].

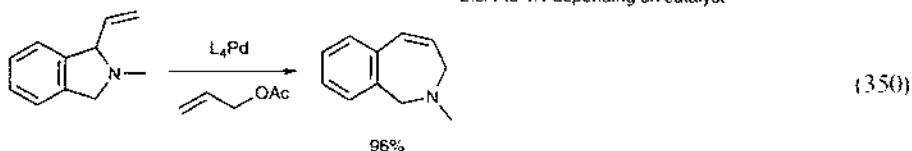
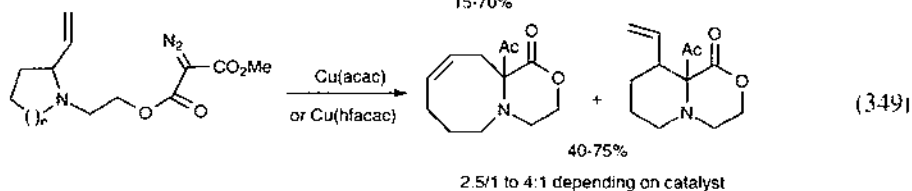
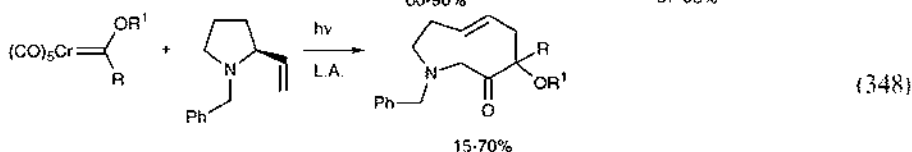
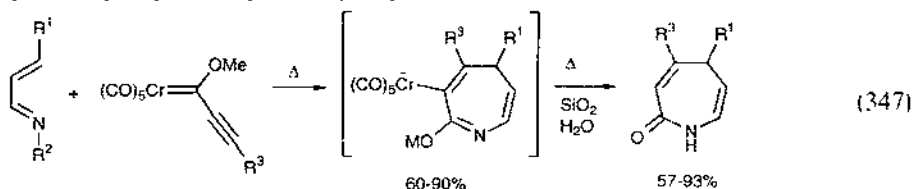
Coumarins were made by palladium(0) oxidative addition/insertion processes with



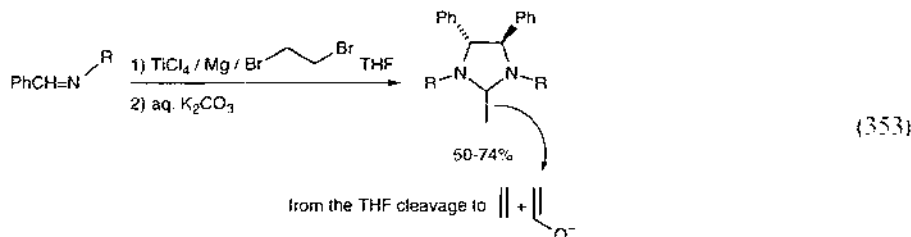
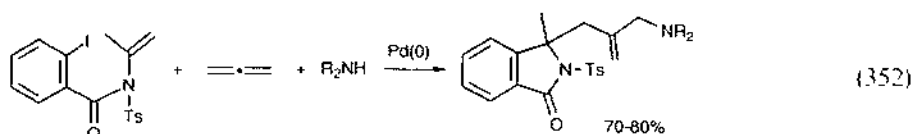
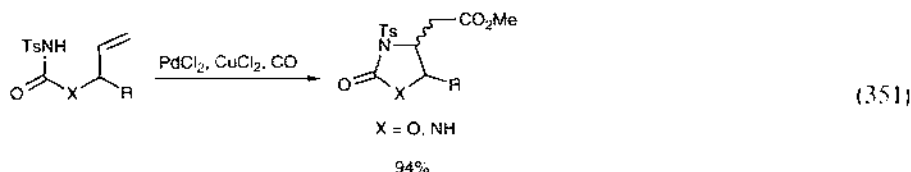
$\beta,\gamma$ -unsaturated esters of *o*-iodophenol [982] and by the palladium(0) catalyzed reaction of 1,3,5-trihydroxybenzene with alkynoic esters (Eq. (345)) [983]. Oxidative addition of  $\gamma$ -acetoxy- $\alpha,\beta$ -unsaturated esters could go with either retention or inversion, depending on conditions [984]. Dihydro- $\gamma$ -pyrones were made by Diels-Alder reactions of Danishefsky's diene with chromium tricarbonyl complexed benzaldehydes (see Eq. (342)) [985]. Pyrilium salts were made by the reaction of alkynes with manganese enone complexes (Eq. (346)) [986]. Acetylacetone reacted with tungsten alkyne carbene complexes to give  $\alpha$ -pyrone carbene complexes [987]. Macrocyclic diethers were made by rhodium(II) catalyzed aryl CH insertion of diazoketones [988].



Cinnamaldehyde imines cycloadded to alkynylcarbene chromium complexes to give azepinones (Eq. (347)) [989,990]. Photolysis of chromium carbene complexes with 2-vinylpyrrolidines gave nine-membered rings via zwitterionic aza Cope chemistry (Eq. (348)) [991]. Other approaches to larger nitrogen heterocycles are shown in Eq. (349) [992] and Eq. (350) [993].

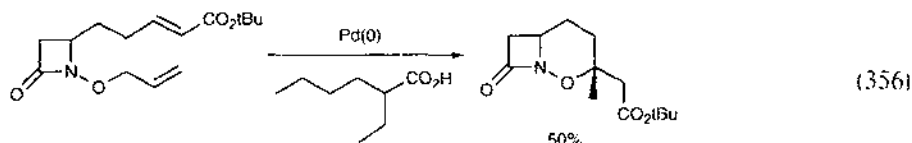
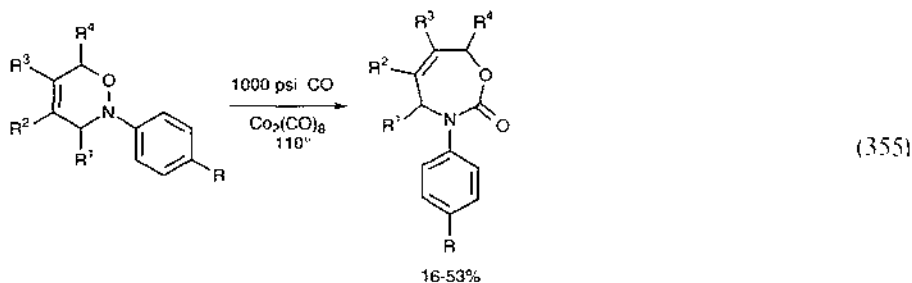
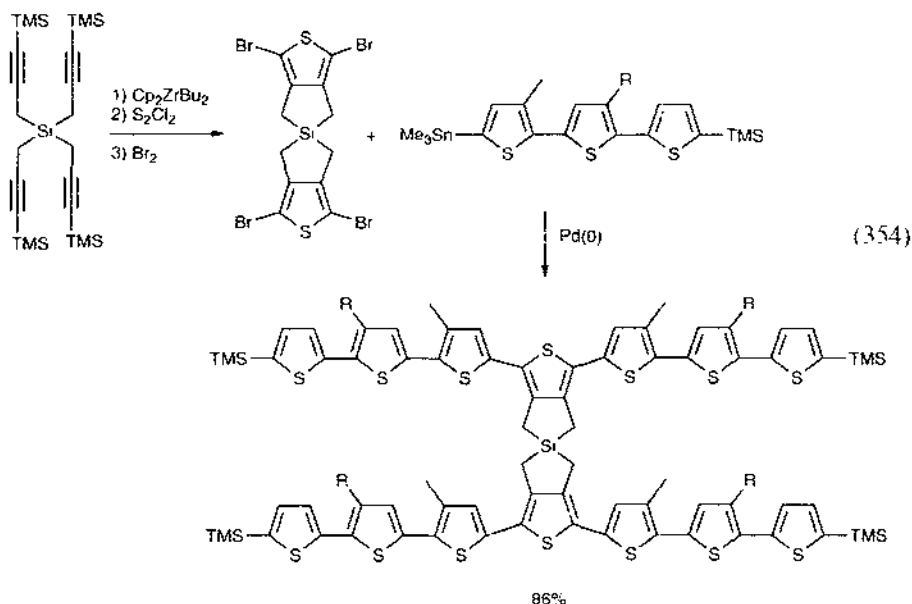


Palladium(II) catalyzed intramolecular olefin amination to make cyclic carbonates or carbamates (Eq. (351)) [994]. Oxazoles were prepared by rhodium(II) catalyzed NH insertion of  $\alpha$ -diazoketones into amides followed by cyclizations [995]. Oxazolines of *o*-bromobenzoic acids cyclized to the five-membered lactam when treated with combined palladium/nickel catalysts and CO [996]. Cobalt complexation of the alkyne position of enynes protected the alkyne while the ene portion underwent 1,3-dipolar cycloaddition with nitrile oxides to form the heterocycle [997]. Palladium(0) catalyzed the asymmetric cycloaddition of nitrones to methacrylamides with good ee, but poor *endo/exo* ratios [998]. Other reactions forming nitrogen heterocycles are seen in Eq. (352) [999] and Eq. (353) [1000].



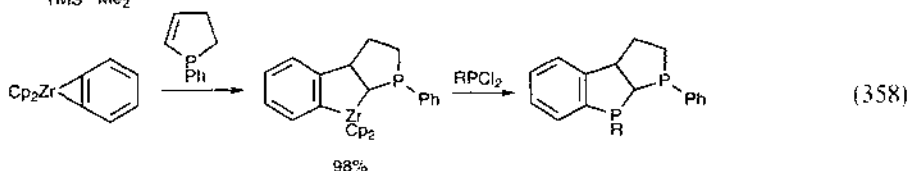
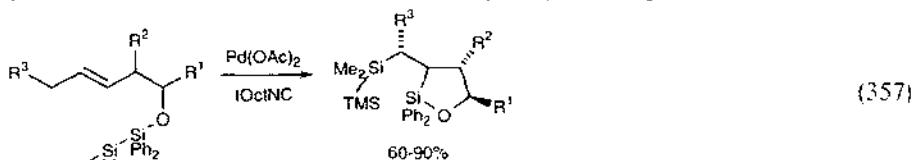
Polythiophenes were made by zirconium promoted alkyne cyclization followed by palladium-catalyzed coupling (Eq. (354)) [1001]. Tetraryl thiophenes were produced by palladium acetate catalyzed dimerization of diaryl alkynes with  $\text{S}_8$  [1002]. Diynes gave polythiophenes. Cyclic hydroxylamine ethers were expanded to carbamates by dicobalt octacarbonyl catalyzed high pressure carbonylation (Eq. (355)) [1003]. *Ortho* aminophenols reacted with 1,4-dihalo-2-butenes in the presence of palladium(0) and chiral ligands to give 2-vinylbenzomorpholines in good yield and modest ee's [1004]. Bicyclic  $\beta$ -lactams were made via palladium(0) chemistry (Eq. (356)) [1005]. Benzo-1,4-dioxenes were made by the palladium(0)/copper(I) catalyzed cyclization of monopropargyl ethers of *o*-hydroquinone [1006]. Rhodium(I) complexes catalyzed the cyclization of allyl ethers of *o*-hydroxybenzylamines to the saturated N,O heterocycle [1007]. Cationic ruthenium complexes of *o*-difluoro- [1008] and *o*-dichlorobenzene [1009] coupled efficiently to *o*-hydroquinone or *o*-phenylenediamine to give dibenzodioxenes or dibenzodiazines in good

yield. 1,2,4,5-Tetrahaloarene complexes readily coupled on both sides to give the pentacyclic system.



Rhodium(I) complexes catalyzed the intramolecular hydrosilyloxylation of allylic alcohols to make five-membered Si-O heterocycles [1010]. Palladium acetate isonitrile complexes catalyzed the intramolecular addition of disiloxanes to the olefin of homoallylic alcohols to give Si-O heterocycles (Eq. (357)) [1011]. Silacyclobutanes underwent reaction with organic halides and carbon monoxide in the presence of palladium(II) catalysts to give 2-silahydropyrans [1012]. *Bis*-propar-

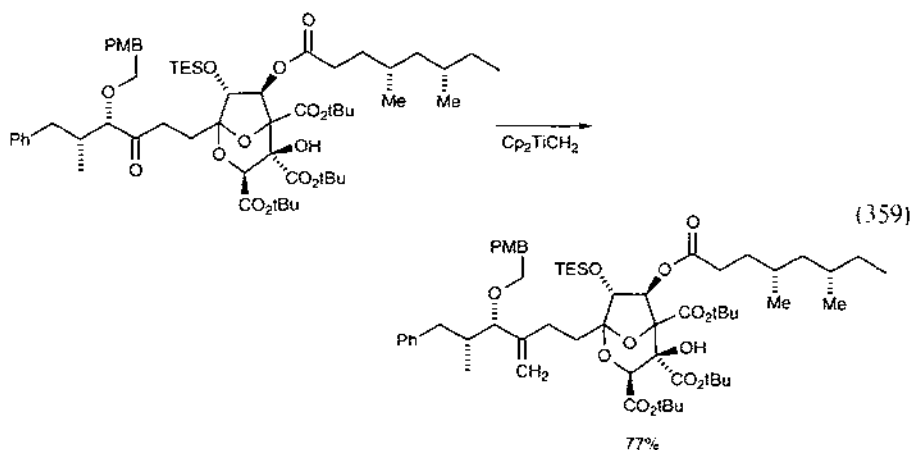
gyl silanes were cyclized to 3,4-bismethylene silacyclopentanes by treatment with “Cp<sub>2</sub>Zr” followed by hydrolysis [1013]. Palladium(0) alkene complexes catalyzed the co-cyclization of acetylene with stannenes (R<sub>2</sub>Sn:) to give stannols [1014]. Phospholes were made via zirconium chemistry (Eq. (358)) [1015].

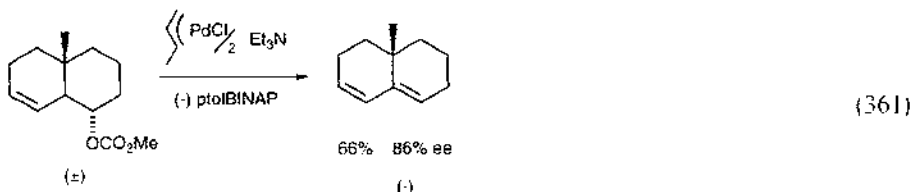
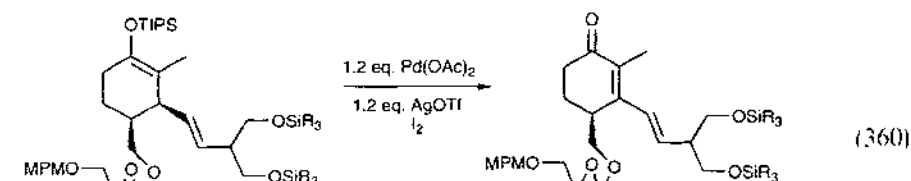


### 3.6. Alkenes, alkanes

Alkenes were made by Tebbe olefination of complex ketones (Eq. (359)) [1016], and stoichiometric palladium assisted oxidation of enol ethers (Eq. (360)) [1017] or enol triflates to convert indanones to indenones [1018]. Optically active dienes were prepared by palladium(0) catalyzed elimination of allyl acetates [1019], (Eq. (361)) [1020]. Samarium iodide in the presence of palladium catalysts eliminated propargyl acetates to give enynes [1021].  $\alpha$ -Diazoketones were converted to  $\alpha,\beta$ -unsaturated enones by treatment with rhodium(II) trifluoroacetate [1022]. Reduced titanocene species eliminated  $\alpha$ -bromohexose acetates to give corresponding dihydropyran [1023]. Cp\*ReO<sub>3</sub> catalyzed the deoxygenation of 1,2 diols to give alkenes [1024].

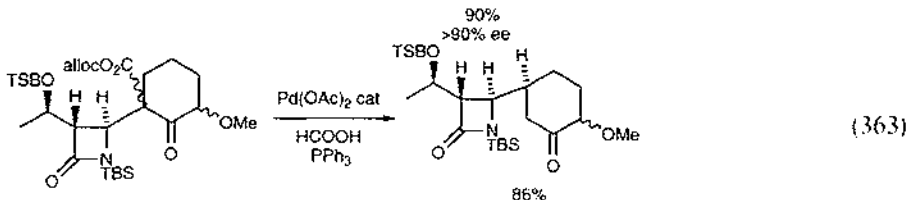
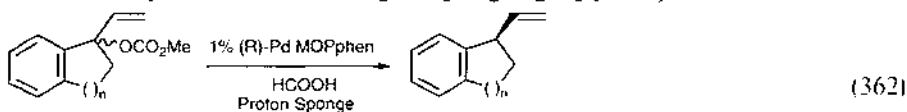
Chiral ruthenium complexes catalyzed the asymmetric reduction of 3,4-dihydroisoquinolines to the tetrahydroisoquinoline with high ee [1025].





Rhodium(I) DUPHOS complexes catalyzed the hydrogenation of either *E* or *Z* dehydroamino acids with very high ee [1026].  $\alpha$ -Methyl methacrylic acid was reduced with high ee in supercritical  $\text{CO}_2$  using chiral ruthenium catalyst [1027]. *Bis* chromium fulvalene complexes catalyzed the mono 1,2-reduction of 1,3-dienes [1028].

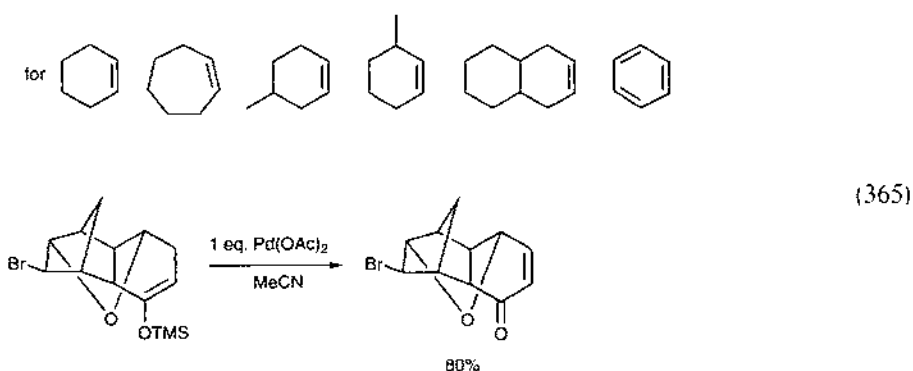
A very wide range of aryl and alkyl halides were reduced to alkanes by trialkylsilanes in the presence of palladium(II) chloride catalysts [1029]. 1,1-Dibromoalkenes were reduced to *Z*-1-bromoalkenes by tributyl stannane in the presence of palladium(0) catalysts [1030,1031]. Racemic allylcarbonates were reduced to optically active alkenes by chiral palladium(0) catalyst (Eq. (362)) [1032]. Similar conditions led to the reductive dicarboxylation of allox-protected  $\beta$  lactams (Eq. (363)) [1033]. Excess ethyl Grignard reduced allyl ethers to alkenes in the presence of nickel(II) chloride [1034]. Perfluorinated arenes (benzene, naphthalene) were mono reduced (loss of one F) by treatment with  $\text{Ph}_2\text{ZrCl}_2 \cdot \text{Mg} \cdot \text{HgCl}_2$  [1035].



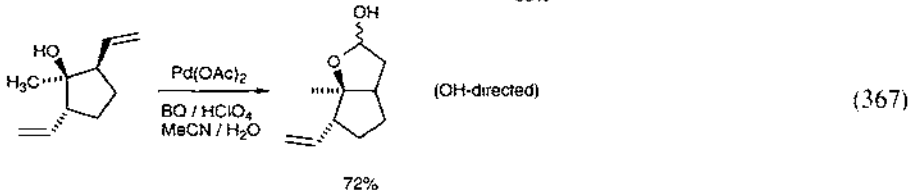
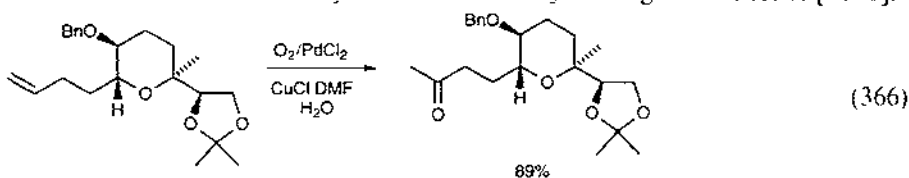
### 3.7. Ketones, aldehydes

The system  $\text{Pd}_4\text{Phen}_2(\text{OAc})_4/\text{O}_2$  was a very selective catalyst for the oxidation of allylic alcohols to aldehydes [1036]. The system 5%  $\text{PdCl}_2(\text{MeCN})_2/$

H<sub>2</sub>O/DMF/bromomesitylene deprotects a range of silyl-protected alcohols then oxidizes them to aldehydes [1037]. Arene chromium tricarbonyl complexes of alcohols could be oxidized to aldehydes without oxidation of the metal [1038].  $\beta$ -Bromoallyl alcohols were rearranged to unsaturated aldehydes by palladium(I) complexes (Eq. (364)) [1039]. *Para*-Alkylphenols were oxidatively rearranged to 2-alkyl-1,4-benzoquinones by RuCl<sub>2</sub>L<sub>3</sub> and *t*-butylhydroperoxide [1040]. Palladium acetate oxidized silylenol ether to enones (Eq. (365)) [1041].



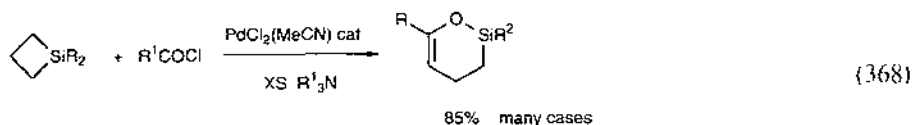
Terminal alkenes were oxidized to methyl ketones using palladium(II) catalyzed oxidation chemistry [1042]. (Eq. (366) [1043–1045] and Eq. (367) [1046]). Ruthenium chloride/*t*-butyl hydroperoxide oxidized  $\Delta$ -5-steroids at the allylic (7) position to give the conjugated cyclohexenone [1047]. Rhodium(I) complexes catalyzed the reaction between benzoyl chloride and alkynes to give indenones [1048].



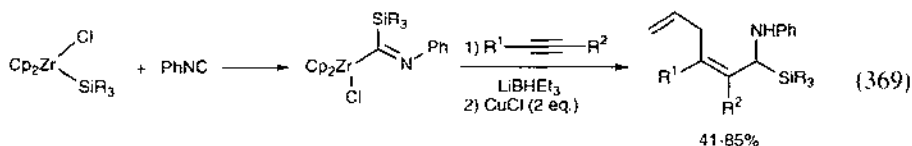
### 3.8. Organoboranes, silanes, germanes and stannanes

Palladium(0) complexes catalyzed the addition of  $(\text{RO})_2\text{BB}(\text{OR})_2$  species to allyl carboxylates to give allylboronates [1049], to alkynes to give *cis*-(*bis*)(boryl)alkenes [1050] and to 1,3-dienes to give 1,4-(*bis*)(boryl)-2-butenes [1051]. Palladium(II) complexes catalyzed the addition of  $\text{R}_3\text{SiB}(\text{OR})_2$  to alkynes to give (silyl)(boryl)alkenes [1052]. Rhodium(I) or  $\text{Cp}_2\text{Zr}(\text{H})\text{Cl}$  catalyzed the hydroboration of alkenes [1053] and  $\text{Cp}_2\text{Ti}(\text{CO})_2$  catalyzed the hydroboration of both alkynes and alkenes [1054].

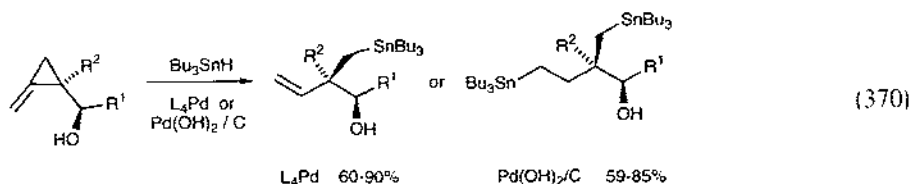
Asymmetric hydrosilylation of olefins catalyzed by MOP palladium catalysts has been reviewed (36 references) [1055] and was modestly (51–81% ee) stereoselective in the hydrosilylation of cyclized dienes to give allyl silanes [1056,1057]. Palladium(0) complexes catalyzed the addition of  $\text{R}_3\text{Si-SiR}_3$  to alkynes to give *cis*-1,2-(*bis*)silyl alkenes [1058] and to allyl triflates and acetate to give allyl silanes [1059]. The intramolecular *bis*-silylation of alkenes catalyzed by palladium(0), tertiary alkylisocyanide complexes has been reviewed (4 references) [1060]. This catalyst system converted the  $\text{O-SiR}_2\text{-SiR}_3$  protected propargyl alcohols to silylallenes ( $\text{Sn}^{2+}$ ) [1061] and similarly protected allyl alcohols to allylsilanes ( $\text{Sn}^{2+}$ ) [1062]. The same system catalyzed the *cis-bis* silylation of alkynes [1063]. Palladium(II) complexes catalyzed the addition of acid chlorides to silacyclobutanes (Eq. (368)) [1064] while palladium(0) complexes catalyzed the addition of  $\text{R}_3\text{SnSiR}_3$  to allenes to give 2-silyl allylstannanes [1065].



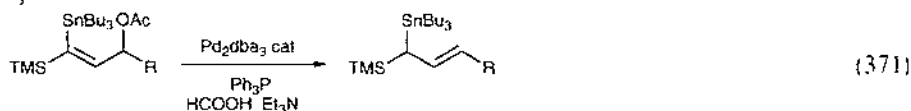
$\text{Rh}_3(5\text{-MEPY})_4$  catalyzed the asymmetric insertion of  $\alpha$ -diazoesters into Si-H bonds to give  $\alpha$ -silyl esters in good yield and 47% ee [1066]. The chromium tricarbonyl complex of 1,4-*bis*-(chlorodimethylsilyl)benzene was dimerized by sodium to give the cyclic tetrasilane having the  $\text{Cr}(\text{CO})_3$  groups on the outside [1067]. Chromium hexacarbonyl catalyzed the 1,4-hydrosilylation of 1,3-dienes under photolytic conditions [1068].  $\alpha$ -Silylamines were made via zirconium chemistry (Eq. (369)) [1069]. A full paper on the rhodium(I) catalyzed silylformylation of aldehydes has appeared [1070]. Palladium(0) catalyzed the conversion of allyl chlorides to allylgermanes by reaction with  $\text{R}_3\text{GeSnR}_3$  [1071].



Pearlman's catalyst ( $\text{Pd}(\text{OH})_2/\text{C}$ ) catalyzed the hydrostannylation of allyl acids, amines, alcohols and ketals to give terminal stannanes, and of *N*-vinylphthalimide and acrylonitrile to give secondary stannanes [1072]. Palladium(0) complexes catalyzed the hydrostannylation of bromo [1073] and boryl alkynes [1074] to put tin  $\alpha$  and  $\beta$  respectively to the heteroatom, the hydrostannylation of ene diynes to stannylate the less hindered alkyne [1075], the hydrostannylation of 1-chloroenynes to give 1-chloro-3-stannyl-1,3-dienes [1076], and methylene cyclopropanes (Eq. (370)) [1077].

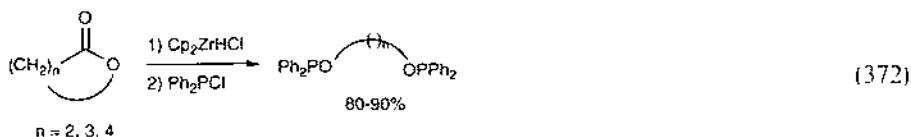


Palladium(0) complexes catalyzed the 1,2-*bis*-stannylation of alkynes [1078] and cyclic allenes [1079] by  $\text{R}_3\text{SnSnR}_3$ , the conversion of 3-iodo-4-methoxyphenylalanine to the 3-tributylstannylarene by  $\text{R}_3\text{SnSnR}_3$  [1080], the stannylborylation of alkynes by  $\text{R}_3\text{SnBR}_2$  [1081] and tin  $\text{Sn}2'$  reduction of stannyl silyl allyl acetates (Eq. (371)) [1082].



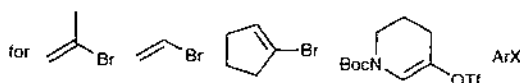
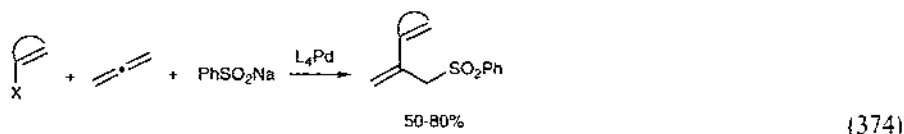
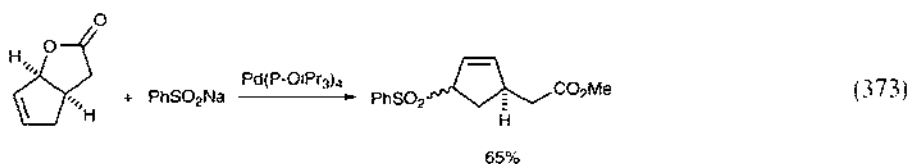
### 3.9. Organophosphorous and sulfur compounds

Palladium(0) complexes catalyzed the hydrophosphinylation of terminal alkynes by  $\text{Ph}_2\text{P}(\text{O})\text{H}$  to give terminal phosphination [1083], by  $(\text{RO})_2\text{P}(\text{O})\text{H}$  to give internal phosphination [1084] and by  $\text{PhSeP}(\text{O})(\text{OPh})_2$  to give 1-P-2-Se alkenes [1085]. Palladium(0) complexes also catalyzed the conversion of aryl iodides to aryl phosphines by reaction with  $\text{Ph}_2\text{PH}$  [1086,1087] and by  $\text{H}_2\text{P}(\text{O})(\text{OR})$  [1088]. Lactones were converted to  $\alpha,\omega$ -*bis* phosphorous compounds utilizing zirconium chemistry (Eq. (372)) [1089].

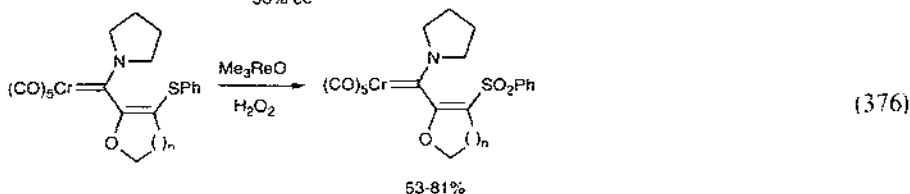
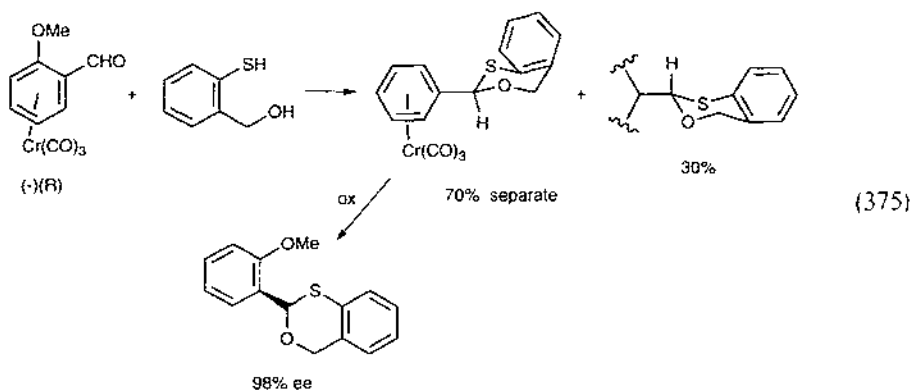


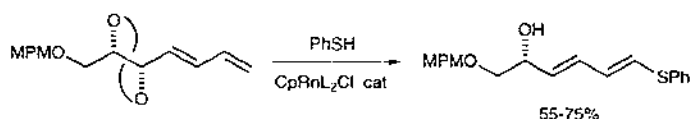
Allyl esters were converted to allyl sulfones by reaction with  $\text{PhSO}_2\text{Na}$  in the presence of palladium(0) catalysts (Eq. (373)) [1090] (asymmetric induction) [1091]. Allyl sulfones were also produced by palladium(0) cascade processes (Eq. (374))

[1092]. Palladium(0) complexes catalyzed the addition of thiophenol to allenes to give vinylsulfides [1093], the reaction of aryl and alkenyl halides with  $\text{RSBR}_2$  to give aryl and alkenylsulfides (thioethers) [1094], the reaction of allyl thiocarbamates with aryl iodides to give (aryl)allyl thioethers [1095], the coupling  $\text{RSCl}$  to  $\text{RSSS}$  by tetramethylstannane [1096], the reaction of 4-iodophenylalanine with *t*-butylthiol to give the 4-*t*-butylthiol species [1097] and the cross coupling of  $\text{ArSCN}$  with aryl halides to give  $\text{ArSAr'}$ , in the presence of samarium iodide [1098].

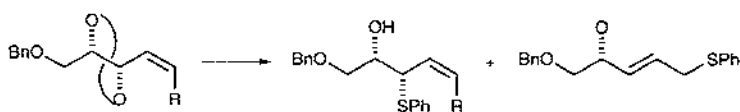


Alkynes were converted to alkenyl selenanes by hydrozirconation followed by treatment with  $\text{ArSeSeAr}$  [1099] or  $\text{ArSeBr}$  [1100]. Reactions producing sulfur compounds are shown in Eq. (375) [1101], Eq. (376) [1102] and Eq. (377) [1103].





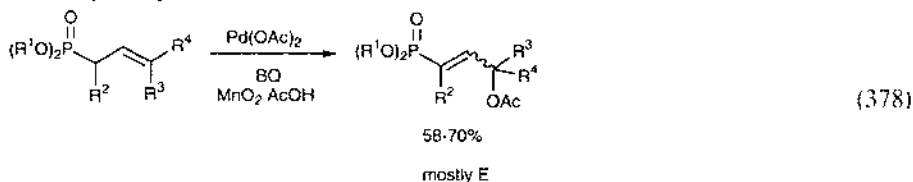
and



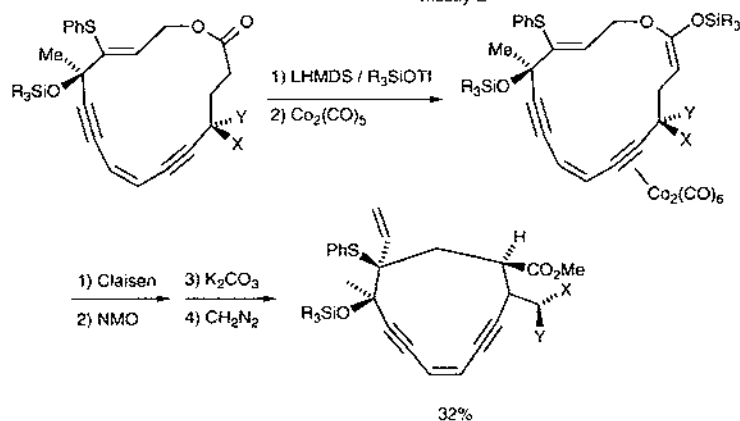
(377)

### 3.10. Miscellaneous

An improved procedure for the resolution of racemic ethylene *bis*-(tetrahydroindenyl)titanium derivatives has been devised [1104]. Secondary amines were oxidized to nitrones by hydrogen peroxide and  $\text{MeReO}_3$  catalysts [1105]. Palladium(II) catalyzed the allylic acetoxylation of allylphosphonate (Eq. (378)) [1106]. Complexation to cobalt was used to protect an alkyne in a complex synthesis (Eq. (379)) [1107].



(378)



(379)

## 4. Reviews

The following reviews have appeared:

- (1) Highly selective addition of organic dichalconides to carbon carbon unsaturated bonds (19 references) [1108].

- (2) Use of  $\text{Rh}(\text{CO})\text{I}_2\text{Cl}$  as catalyst for pinacol borane hydroboration of alkenes and alkynes [1109].
- (3) Toward direct synthesis of organonitrogen compounds from dinitrogen: the chemistry of diazoalkane complexes derived from dinitrogen complexes (49 references) [1110].
- (4) Catalysts for the reactions of epoxides and carbon dioxide (50 references) [1111].
- (5) Heterogeneous catalysis of C–C bond formation: black art or organometallic science? (45 references) [1112].
- (6) Enantioselective C–C and C–H bond formation mediated or catalyzed by chiral ebThi complexes of Ti and Zr (86 references) [1113].
- (7) Les polyenones à conjugaison croisés en chimie moléculaire (82 references) [1114].
- (8) The versatile chemistry of arenemanganese carbonyl complexes (77 references) [1115].
- (9) Design of chiral ligands for asymmetric catalysis: from  $\text{C}_2$ -symmetric semicorrins and bisoxazolines to nonsymmetric phosphinooxazolines (53 references) [1116].
- (10) Catalytic conversions in water: environmentally attractive processes employing water soluble transition metal complexes (65 references) [1117].
- (11) Annual survey 1994. Transition metals in organic synthesis (866 references) [1118].
- (12) Annual survey 1994. Transition metals in hydroformylation (102 references) [1119].
- (13) Transformations of  $\beta$ -dicarbonyl compounds by reactions of their transition metal complexes with carbon and oxygen electrophiles (66 references) [1120].
- (14) Nonstabilized alkyl complexes and alkyl cyano ate complexes of iron(II) and cobalt(II) as new reagents in organic synthesis (80 references) [1121].
- (15) Asymmetric catalytic routes to chiral building blocks of medicinal interest (20 references) [1122].
- (16) Regiocontrolled intramolecular reductive cyclization of diynes (12 references) [1123].
- (17) A new way for efficient catalysis by using low valent ruthenium complexes as redox Lewis acid and base catalysts (79 references) [1124].
- (18) Exploration of synthetic reactions via C–H bond activation by transition metal catalysts. Carboxylation and aminomethylations of alkanes or arenes (41 references) [1125].
- (19)  $\text{Sn}2'$  and  $\text{Sn}2'$  like ring openings of oxa-*n*-cyclosystems (65 references) [1126].
- (20) Application of  $\pi$ -arene ruthenium complexes in peptide labeling and peptide synthesis (25 references) [1127].
- (21) Recent developments in Pd-catalyzed oxidations (7 references) [1128].
- (22) Organotitanium compounds in organic synthesis (30 references) [1129].
- (23) Regio and stereoselective metal-mediated syntheses of polyfunctionalized alkenes (19 references) [1130].

- (24)  $\text{HSiR}_3/\text{CO}$  as the potent reactant combination in developing new transition metal catalyzed reactions (68 references) [1131].
- (25) Catalytic applications of rhodium complexes containing trialkylphosphines (200 references) [1132].
- (26) From Hein to hexene: recent advances in the chemistry of organochromium  $\pi$ -complexes (49 references) [1133].
- (27) Synthetic applications of organochlorozirconocene complexes (235 references) [1134].
- (28) Organic products from oxidation of metal carbynes (32 references) [1135].

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