

Transition metals in organic synthesis. Highlights for the year 1995

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Keywords: Transition metals; Organic synthesis

1. General comments

This review offers a broad survey of the literature dealing with the use of transition metals in organic synthesis for the year 1995. Although the coverage is relatively all-inclusive, only those reactions of unusual novelty, complexity or significance will be presented in equation form. The remainder will be referenced but not explicitly discussed. In this way this review can serve as a rather complete reference source for the field without being an inordinate burden to write, type, print and read.

2. Carbon–carbon bond forming reactions

2.1. Alkylations

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

New chiral ferrocenylphosphine-amine ligands for use in nickel and palladium catalyzed Grignard reactions have been synthesized [1]. Nickel–phosphine complexes were used to catalyze the alkylations of vinyl carbamates by acetylenic Grignard reagents [2], while palladium complexes catalyzed the coupling of gem-vinyl dihalides with alkynyl Grignard reagents [3]. Bridgehead [1.1.0.3] tricycloalkyl Grignard reagents coupled to alkynyl chlorides under nickel catalysis [4]. Nickel–phosphine complexes catalyzed the coupling of 1-naphthyl Grignard reagents with 1,2-dichloro benzene [5]. Nickel–phosphine complexes catalyzed aryl–aryl coupling (Eq. (1)) [6], while optically active palladium–phosphine complexes catalyzed the alkylation of aryl triflates (Eq. (2)) [7]. Copper(II) salts catalyzed the *mono* alkylation of chlorobromo alkanes and aryl systems by allyl Grignard reagents [8].

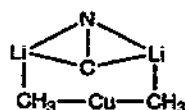
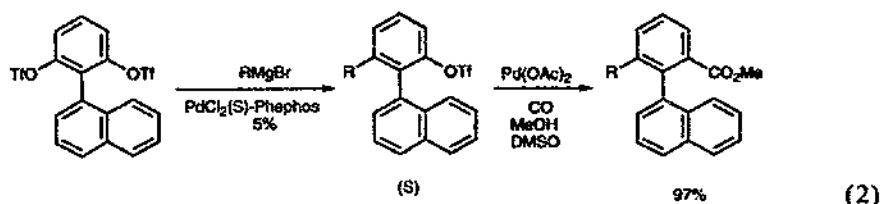
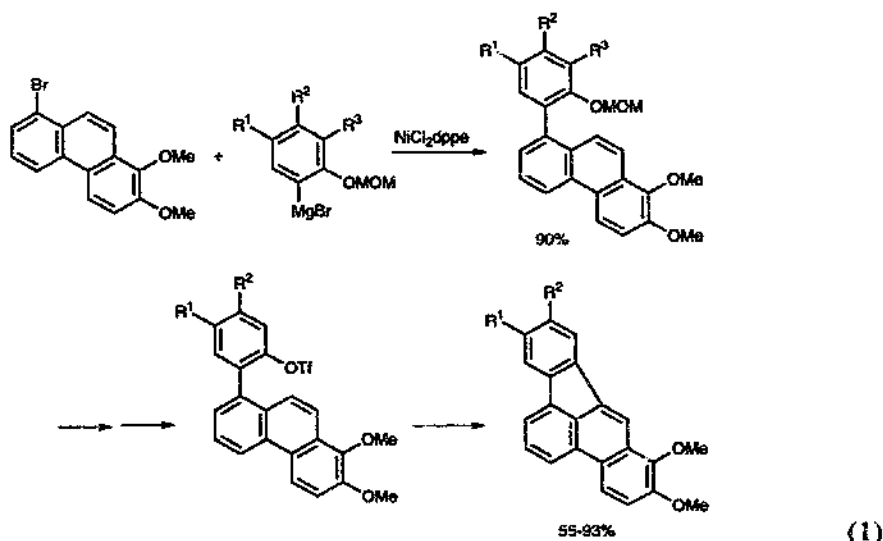


Fig. 1.

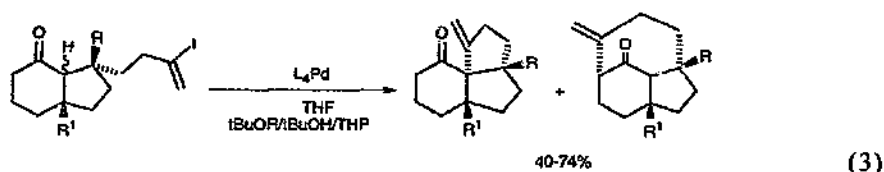
The controversy over the structure of higher order cyanocuprates continued, with EXAFS, XANES [9] and ab initio calculations [10,11], all indicating that the cyanide is *not* coordinated to copper, but rather to two lithiums (Fig. 1). The regiochemistry of the reaction of aryl cuprates with β,γ -epoxy- β -unsaturated esters depended on the nature of the cuprate [12].

Nickel(II) acetylacetonate catalyzed the alkylations of alkyl iodides by functionalized organozinc compounds [13]. Copper(I) chloride catalyzed the alkylation of α -haloethers by alkyl and vinyl zirconium species, generated by hydrozirconation of alkenes and alkynes, respectively [14]. Vinyl halides reacted with $\text{Cp}_2\text{Zr}(\text{Bn})_2$ to produce vinyl zirconium species, which underwent an array of copper and palladium catalyzed coupling processes [15]. Palladium(0) complexes catalyzed the cyanation

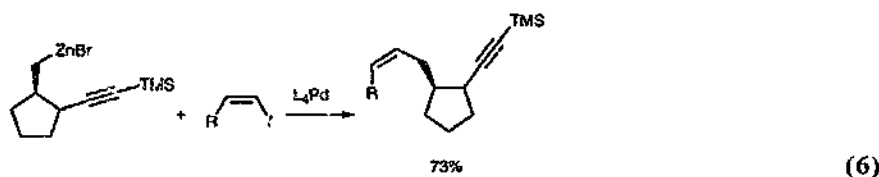
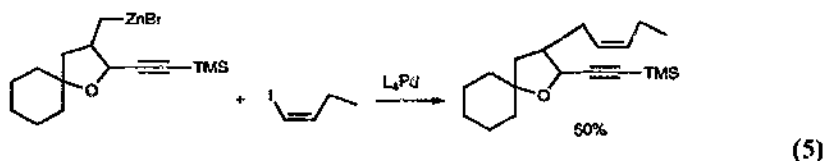
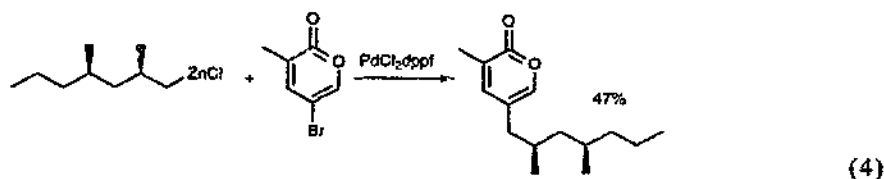
of aryl triflates by zinc cyanide [16] while nickel(0) complexes catalyzed the same process for vinyl chlorides and bromides [17].

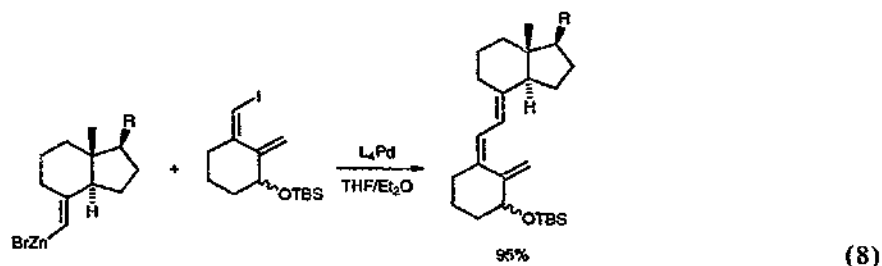
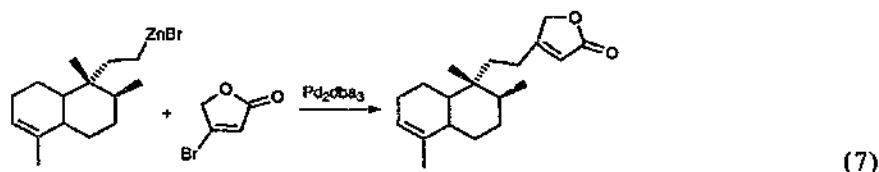
The reduction of palladium acetate to palladium(0) by phosphines has been studied in detail [18], as have the rates and mechanism of oxidative addition of aryl triflates to palladium(0) [19].

Oxidative addition/transmetallation processes involving palladium continues to be extensively used to alkylate organic halides. Palladium(0)/copper(II) catalyzed the arylations of α -lithiated cyclic carbamates by aryl halides [20]. Palladium(0) complexes catalyzed the intramolecular alkylation of a vinyl halide by a potassium enolate (Eq. (3)) [21].

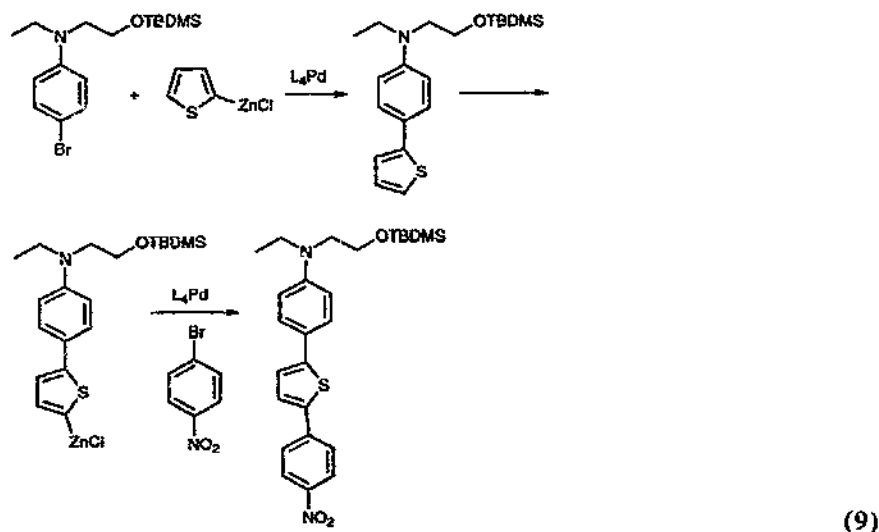


Palladium catalyzed the coupling of a functionalized aryl zinc reagent with vinyl halides [22], chloropurines with vinyl and aryl main group organometallics [23], β -iodo- α,β -unsaturated acids with alkyl, allyl and alkynyl zinc and tin reagents [24], *cis*-dideutero vinyl iodides with alkynyl zinc reagents [25], and benzyl bromide with 2-zincated indoles [26]. *o*-iodostilbene was lithiated, transmetalated to zinc chloride, and coupled to *o*-iodo-1,4-diphenyl butadiene under palladium catalysis [27]. Other synthetically useful transformations of this sort are shown in Eq. (4) [28], Eq. (5) [29], Eq. (6) [30], Eq. (7) [31] and Eq. (8) [32].

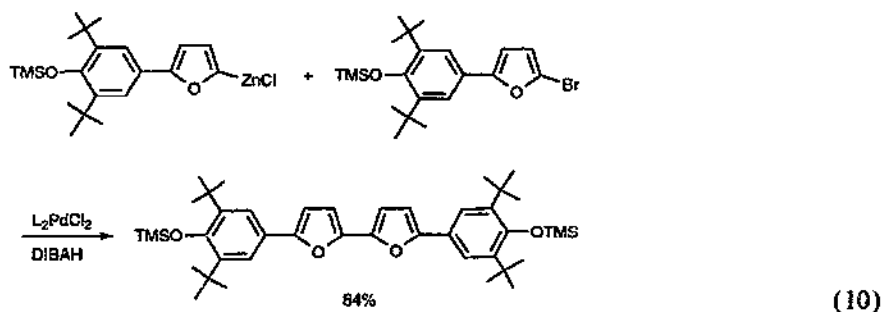




Donor-acceptor oligothiophenes were prepared by palladium catalyzed coupling of 2-iodothiophenes with 2-zincated thiophenes [33]. Terminally zincated oligothiophenes were capped by aryl containing iodothiophenes using palladium catalysts [34]. 3-Aryl thiophenes were prepared from aryl iodides and 3-zincated thiophenes [35]. Compounds for nonlinear optical materials were prepared by palladium catalyzed coupling (Eq. (9)) [36] and (Eq. (10)) [37].

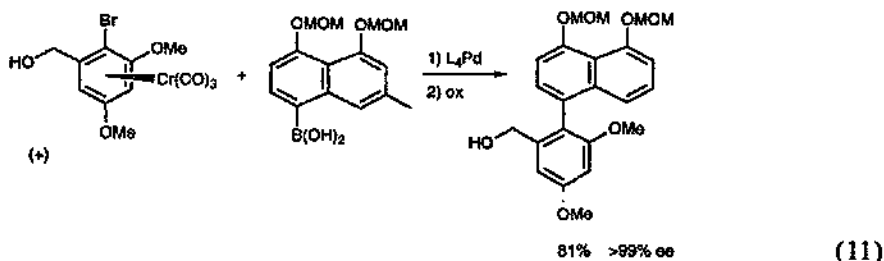


Palladium catalyzed oxidative addition/transmetalation from boron continues to grow in popularity. Palladium catalyzed cross-coupling reactions of organoboron compounds has been reviewed (246 references) [38]. Treatment of mono- or bis-



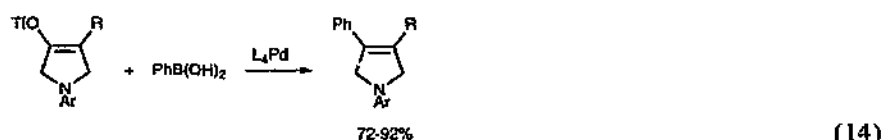
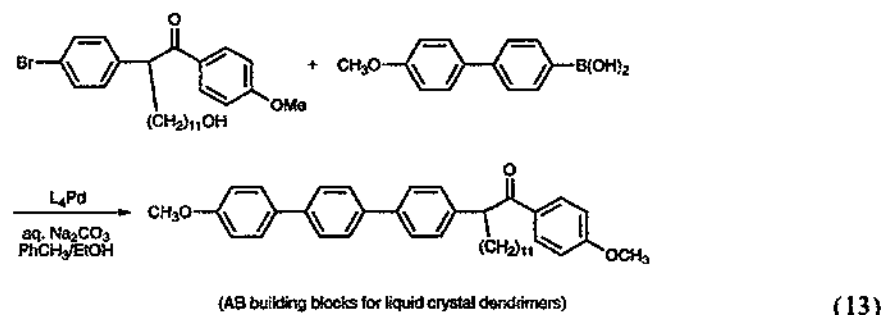
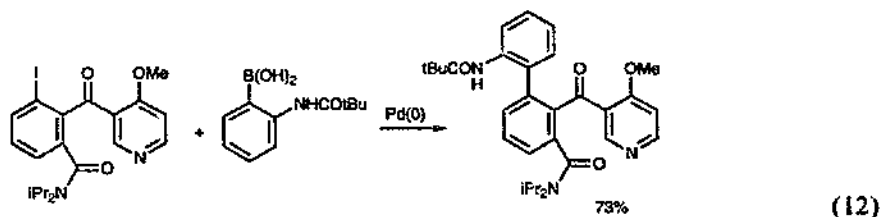
alkoxyboranes with lithium acetylides followed by organic halides and a palladium(0) catalyst resulted in alkylation of the acetylide [39,40]. Vinyl boranes coupled with vinyl and aryl halides ([41], used for retinoid synthesis, [42,43]), while alkyl and aryl boronic acids coupled to vinyl triflates [44], and vinyl halides ([45], and α,ω -diboranes coupled with gem dihalides to give cyclic compounds, [46]) under palladium catalysis.

Treatment of 9BBN(OMe) with acetylides followed by functionalized aryl halides and PdCl_2dppf resulted in efficient coupling [47]. Aryl boronic acids coupled efficiently to aryl halides with palladium catalysis [48–50]. Aryl fluorosulfonates also coupled with nickel catalysis while aryl triflates required palladium catalysts [51]. Optically active biaryls were prepared by palladium catalyzed coupling of naphthyl boronic acids with bromoarenechromium tricarbonyl complexes (Eq. (11)) [52]. The palladium catalyzed coupling of *p*-tolyl boronic acid to *o*-bromobenzene sulfonamides was carried out in 87% yield on a 7.3-kg scale [53].

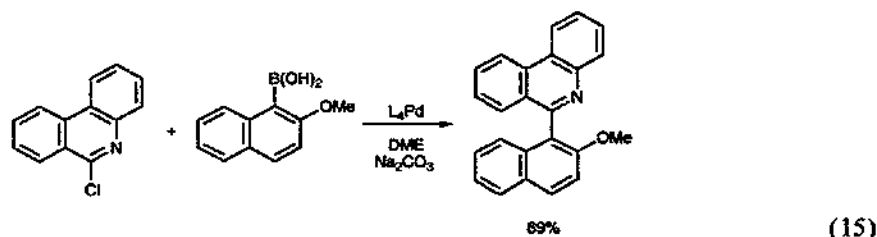


Polyhaloanilines were arylated by phenylboronic acid using palladium catalysis [54]. Other interesting couplings are shown in Eq. (12) [55] and Eq. (13) [56] (see Ref. [57] for another application to liquid crystals).

Palladium complexes catalyzed the coupling of quaternized chloropyridines to pyridinyl boranes [58–60], aryl boranes to bromotetrazoles [61] and bromopyrroles [62], and aryl halides to pyrrolaboranes [63]. Tetraphenylporphyrins with up to eight aryl halide groups were polyarylated by arylboronic acids in the presence of palladium catalysts [64]. Heterocyclic vinyl triflates arylated under similar conditions (Eq. (14)) [65].

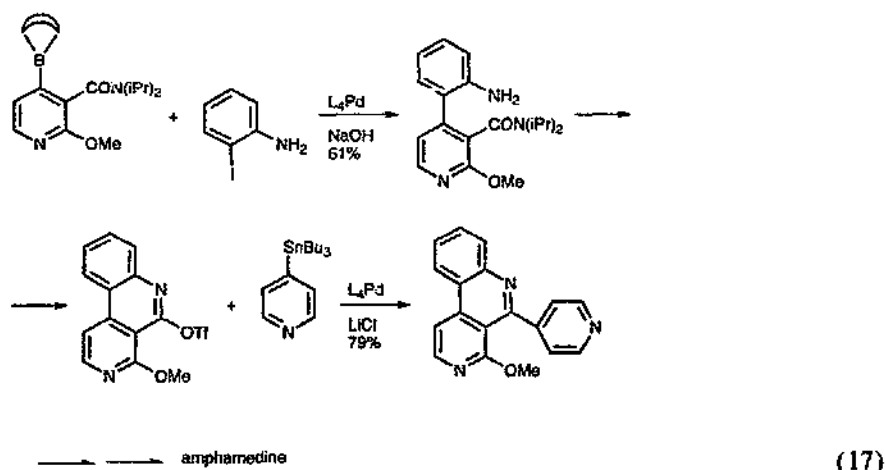
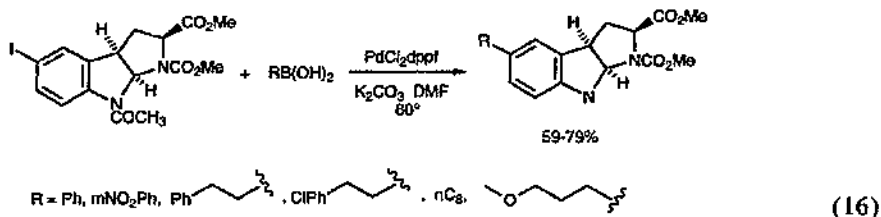


Ortho acetamidoaryl boronic acids were coupled to 3- [66] and 6-bromoquinolines using palladium catalysts [67]. Other useful couplings of this type are shown in Eq. (15) [68], Eq. (16) [69], and Eq. (17) [70].

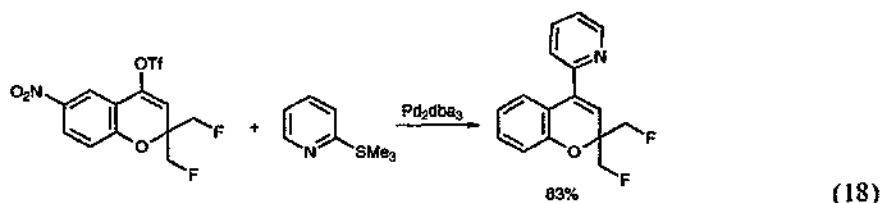


Oxidative addition/transmetalation from tin remains the most popular form of alkylating halides and triflates. Trichloroorganostannanes successfully coupled with aryl and vinyl halides with palladium catalysis if the reaction was run in the presence of aqueous base [71, 72]. Aryl mesylates coupled to stannanes but in low yield [73]. Compounds containing ^{11}C (half-life = 20 min) were synthesized by the palladium catalyzed coupling of $^{11}\text{CH}_3\text{I}$ with stannanes [74].

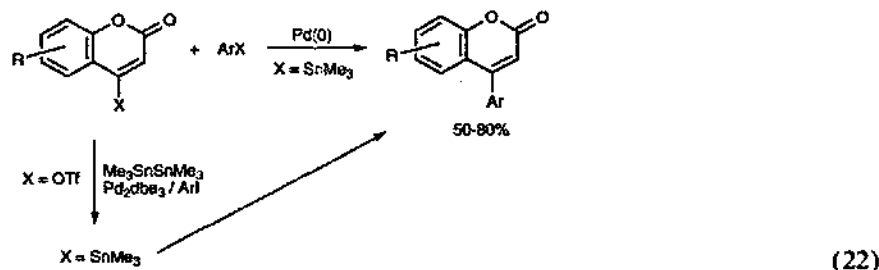
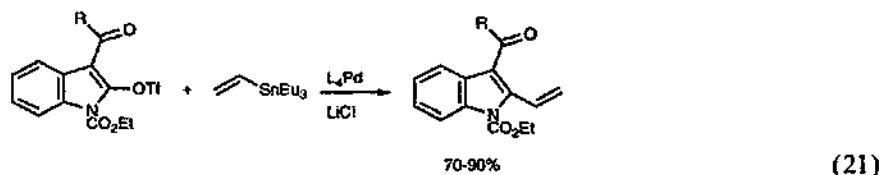
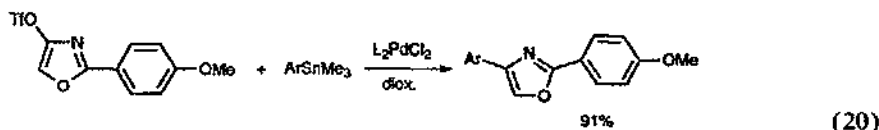
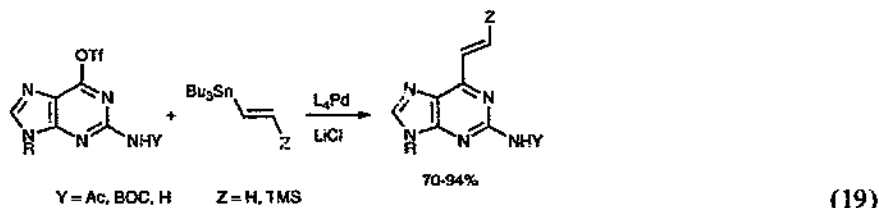
Vinyl triflates of six-membered cyclic ketones [75, 76], of cyclic β -ketoesters



[77,78], and of acyclic ketoesters [79] all coupled to vinyl, aryl or alkynyl stannanes in the presence of palladium catalysts. Palladium(0) complexes catalyzed the alkylations of 1-naphthol triflates by vinyl stannanes [80], and aryl triflates by Bu₃SnCFC(OMe)(CO₂Me) [81]. In an attempt to vinylate the 1,3-triflate of a tetraphenol cryptand, it was discovered that palladium catalyzed the redistribution of the triflate groups to give a 1:1 mixture of the 1,2,3-OTf-4-OH and 1,2,3-OH-4-OTf compounds [82]. Palladium complexes also catalyzed the coupling of hetero-aromatic triflates with stannanes (Eq. (18)) [83], (Eq. (19)) [84], (Eq. (20)) [85], (Eq. (21)) [86], and (Eq. (22)) [87].

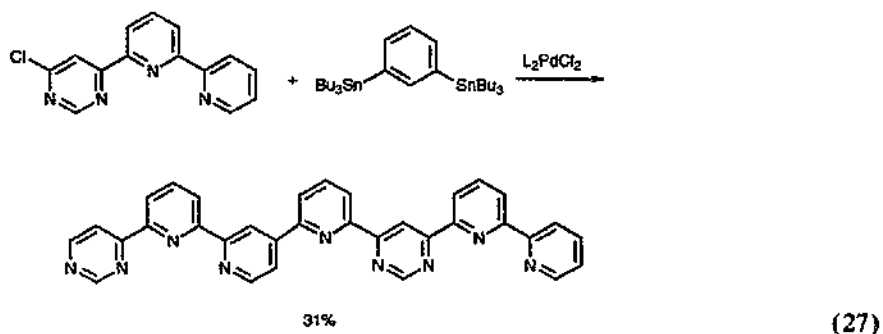
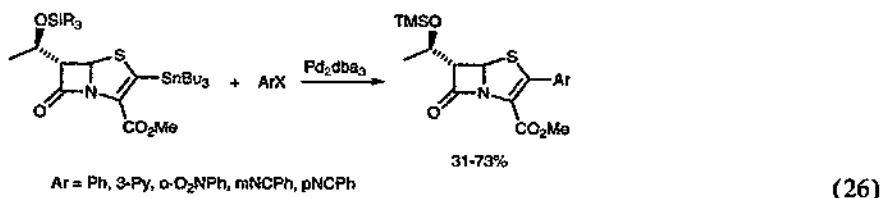
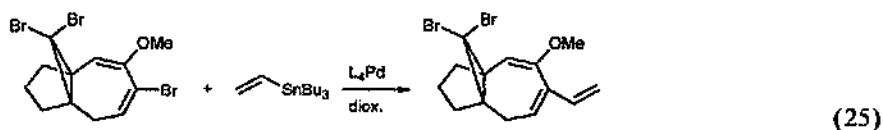
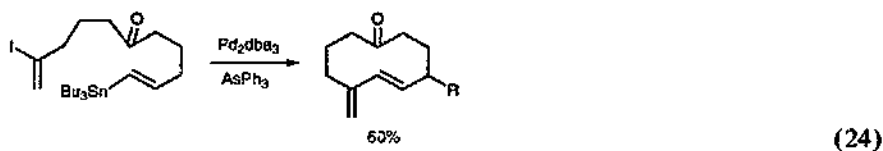
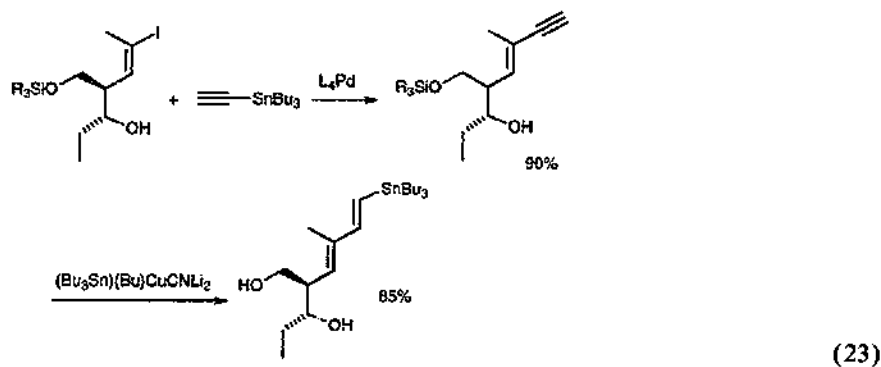


Polyenes were prepared by the palladium catalyzed coupling of 1-stannyl dienes [88] or 1,4-bis-stannyl dienes [89] with vinyl halides. Palladium catalyzed the coupling of α -bromocinnamates to α -stannylcinnamates to give diene diesters [90].

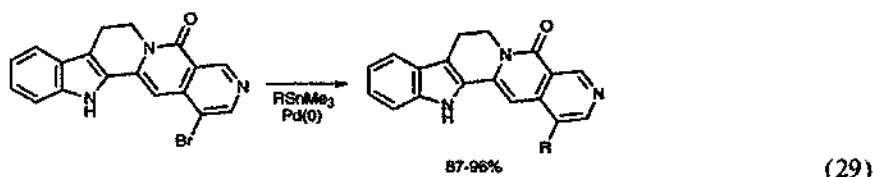


β -Stannyl- α,β -unsaturated ketones were β -alkylated by aryl or benzyl halides in the presence of palladium(0)/copper(I) catalysts [91]. Imines of α -amino acid esters bearing a β - or γ -vinylstannane were alkylated by vinyl halides in the presence of palladium catalysts [92]. α -Halo-optically active vinyl sulfoxides were alkylated by vinylstannanes to give the 2-sulfoxy butadiene using palladium catalyst [93]. Other useful vinyl-vinyl couplings are shown in Eq. (23) [94], Eq. (24) [95], and Eq. (25) [96].

The Stille coupling of aryl and vinyl halides and triflates to aryl and vinyl stannanes was optimized utilizing 0.5% palladium from palladium on carbon, 10% copper(I) iodide and 20% triphenylarsine [97]. A solid-phase synthesis of biaryls utilizing Stille coupling has been developed [98]. Palladium also catalyzed the coupling of β -methoxy- α -stannyl acrylates with aryl halides [99], stannyl stilbenes with aryl halides [100] and 1-trimethylsilyl-4-tributylstannyl-1,3-butadiene with *o*-trimethylsilyl bromobenzene [101].

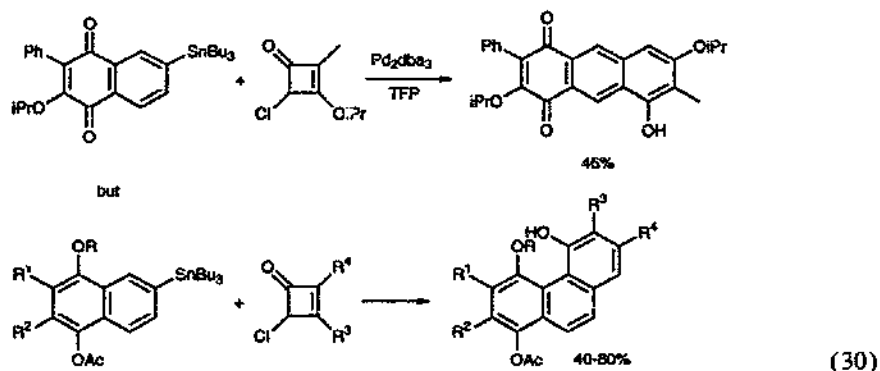


Palladium catalyzed the coupling of a wide range of heteroaromatic halides to stannanes including iodoimidazoles to vinylstannanes [102,103], iodopyrroles [104] and 3-bromoquinolines [105] to vinylstannanes, and 4-stannylpyridines to polybromoarenes [106]. 2,2-Linked bis-thiophenes [107], methyl-capped α -oligothiophenes [108] and pyridine-capped α -oligothiophenes [109] were all synthesized by palladium catalyzed coupling processes. Other useful coupling of heterocyclic systems are shown in Eq. (26) [110], Eq. (27) [111], Eq. (28) [112], and Eq. (29) [113].



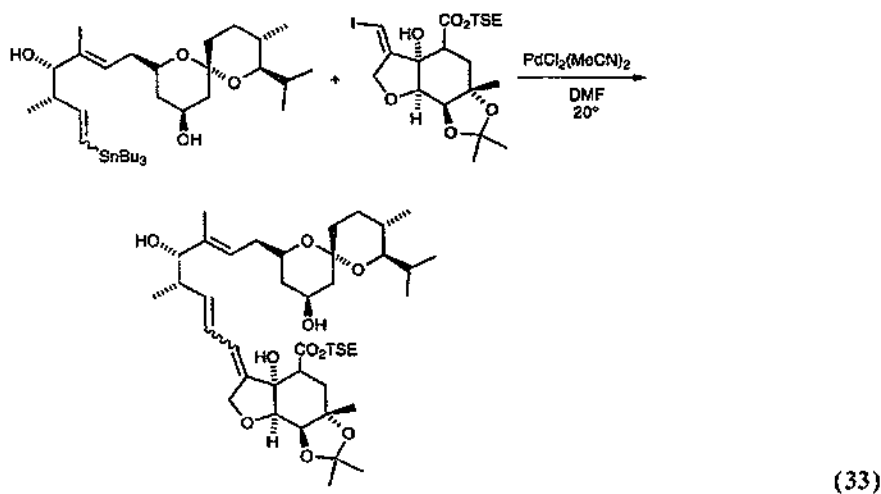
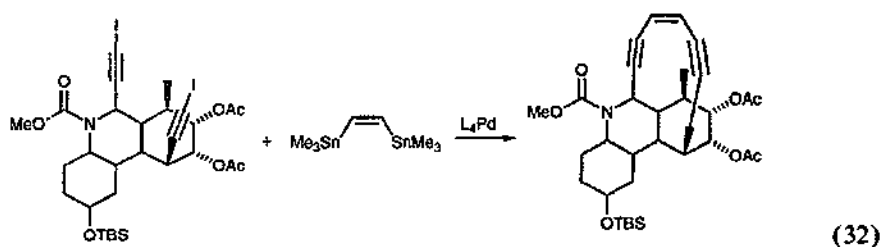
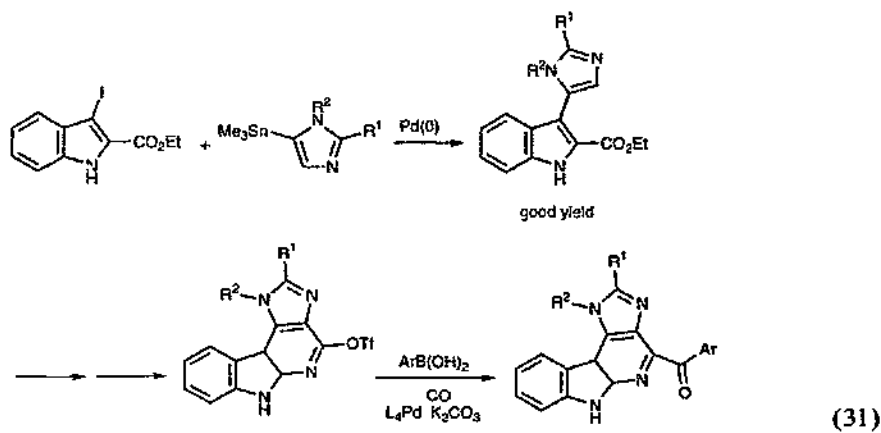
Palladium catalyzed the coupling of 2-stannyl indoles with a wide range of aryl and vinyl halides [114]. Other applications to the synthesis of more complex molecules are shown in Eq. (30) [115], Eq. (31) [116,117], Eq. (32) [118], Eq. (33) [119], Eq. (34) [120] and Eq. (35) [121].

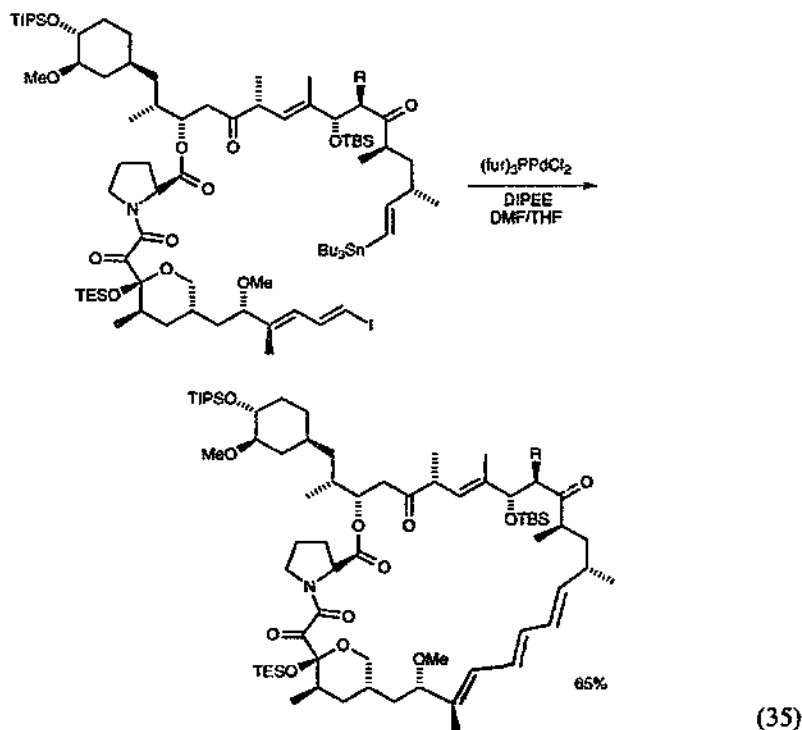
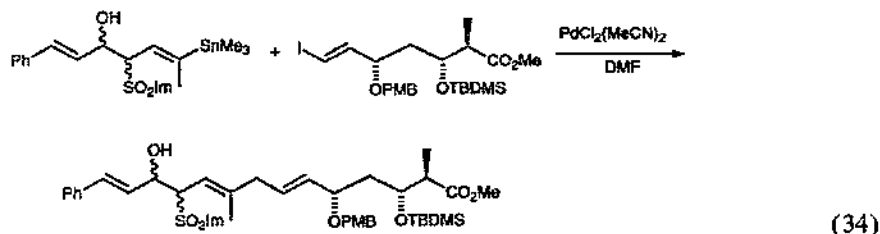
Palladium catalyzed the coupling of vinyl silanes with aryl halides [122].



2.1.2. Alkylation of acid derivatives

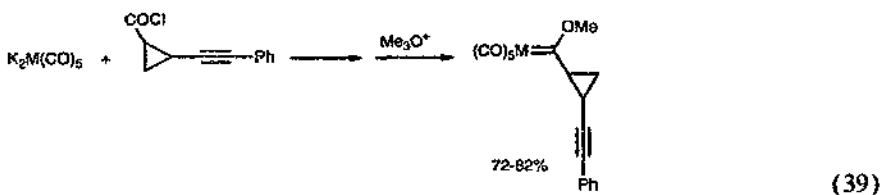
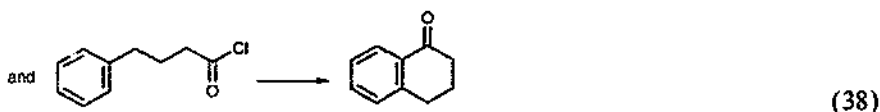
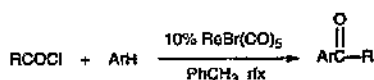
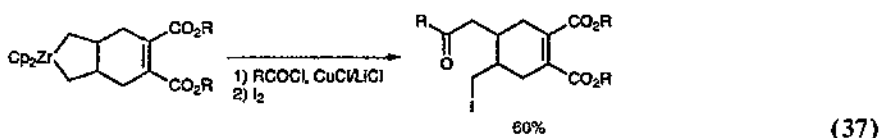
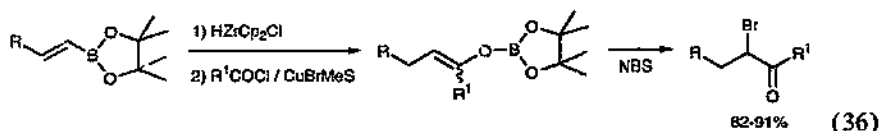
Palladium(0) catalyzed the alkylation of *m*-methoxyphenylacetyl chloride by the zinc reagent from γ -iodobutyric acid ester [123]. Arenes containing *ortho* directing groups were *ortho* lithiated, transmetalated to zinc chloride, then acylated by acid chlorides using palladium catalysts [124]. Trifluoroacetyl chloride was arylated by





arylstannanes using palladium catalysis [125]. Polymer-bound *ortho* stannylated aniline was arylated in a similar fashion [126]. Copper(I) iodide promoted the acylation of α -alkoxystannanes by acid chlorides only if the protecting group or the acylating agent (e.g., $\text{Cl}(\text{CO})\text{SEt}$) contained sulfur [127]. Acid halides were incorporated into vinyl boranes in the strange reaction shown in Eq. (36) [128]. Nickel(0) complexes catalyzed the reaction of Grignard reagents with acid halides to give ketones [129]. Optically active α -acyloxyacid chlorides were converted to ketones without racemization by treatment organomanganese(II) halides [130]. Other more peculiar reactions of acid halides are shown in Eq. (37) [131], Eq. (38) [132], and Eq. (39) [133]. Acid chlorides were alkylated by aryl cuprates made from activated

copper (NaNaph reduction of CuI). Even *ortho* haloaromatics did not form benzyne [134].



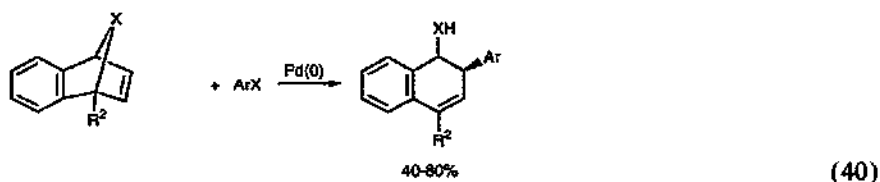
2.1.3. Alkylation of olefins

The Heck reaction (palladium catalyzed oxidative addition/olefin insertion) continued to be the method of choice for the alkylation of olefins. High pressure (10 000 atm) was found to accelerate the Heck reaction [135]. Water-soluble ligands, phosphines with pendant guanidinium groups, have been developed for use in the Heck reaction, and work in 1:1 water-acetonitrile [136]. The phenyl groups of triphenyl phosphine get involved in both the Heck reaction and Stille coupling [137] and tetraphenylphosphonium salts will arylate olefins under Heck reaction conditions [138]. Preformed palladium catalysts $[\text{Ph}_2\text{PCH}_2\text{PdOAc}]_2$ [139] and $[\text{L}_2\text{PdPh}]^+$ [140] are more active and more stable in the Heck reaction. The Heck reaction in super heated (260°C) and super critical (400°C) water has been studied in detail [141,142].

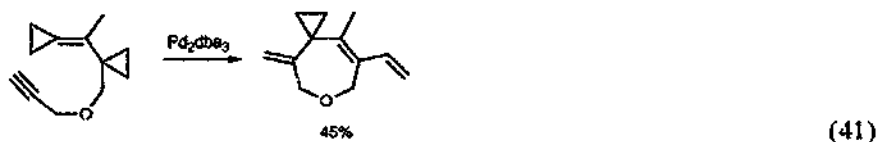
Aryl halides were replaced by aryl diazonium salts for use in the Heck reaction [143,144]. Azoarenes [145] and quaternary benzyammonium bromides [146] also alkylated olefins. Palladium(0) catalyzed the alkylation of N-Boc allyl amines by β -bromomethacrylates [147] and *para*-methoxyiodobenzene [148] and the arylation

of acrylates by *o*-bromobenzyl amines [149]. *Para*-iodobenzamide coupled to a resin through a lysine linker arylated acrylamides under palladium catalysis [150]. Dihydrofuran [151] and glucals [152] were arylated and olefinated under Heck conditions.

β -Iodo- α,β -unsaturated phosphonates were olefinated under Heck conditions [153]. Cyclic polyethers of *m*-iodo-*o*-hydroquinone were vinylated under Heck conditions [154]. Vinyl boronates were coupled to vinyl halides to give dienyl boronates [155] (silver iodide was required to suppress Suzuki coupling) while vinyl siloxanates ($\text{CH}_2\text{CHSi(OR)}_4$) arylated and olefinated with elimination of the silicon [156]. Vinyl triflates coupled to vinyl phosphonates [157], and enol ethers [158]. The triflate of 3-hydroxypyridine arylated acrylates under Heck conditions [159]. Heteroatom bridged bicyclic olefins were arylated by aryl halides with ring opening (Eq. (40)) [160,161].

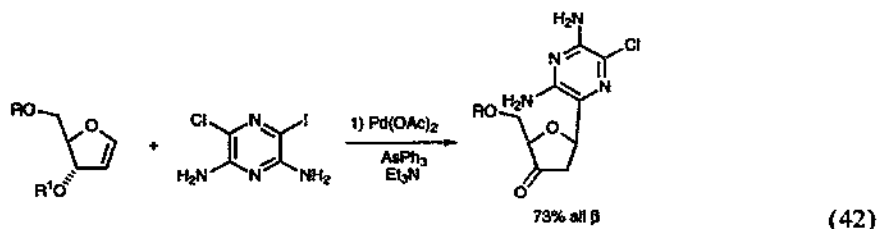


Clavicipitic acid was synthesized utilizing Heck type chemistry extensively [162] in a synthesis patterned after a previous synthesis of Harrington and Hegedus. A methylene cyclopropane ring opened under Heck conditions (Eq. (41)) [163].

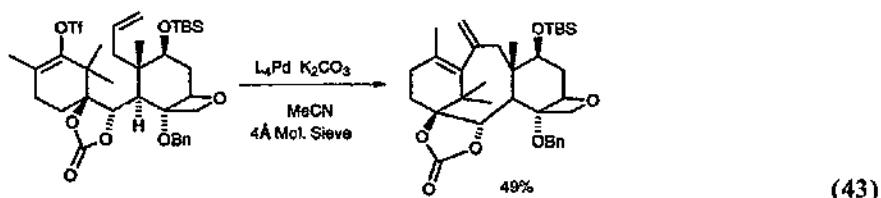


Palladium catalyzed arylation of allylic alcohols gave saturated (phenethyl) aldehydes [164], by β -elimination to give first the enol. β -Elimination away from the OH group, to give back an arylated allylic alcohol, could be promoted by a change in conditions [165]. This chemistry was used to make nucleoside analogs (Eq. (42)) [166]. Palladium catalyzed the reaction of 3-mercuriindole with allyl bromide to give 3-allylindoles [167].

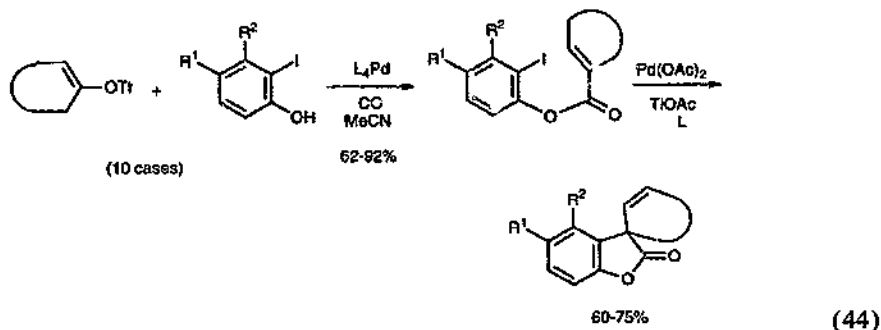
The mechanism of the intramolecular Heck reaction was studied in detail [168].



The α,β -unsaturated amide of 7-bromodihydroindole cyclized exclusively 6-endo-trig in 88–95% yield under Heck conditions [169]. Macrocyclization was effected under high dilution conditions [170] or on a solid support [171]. Intramolecular Heck chemistry was used in the context of taxol synthesis (Eq. (43)) [172–175] and other related systems.



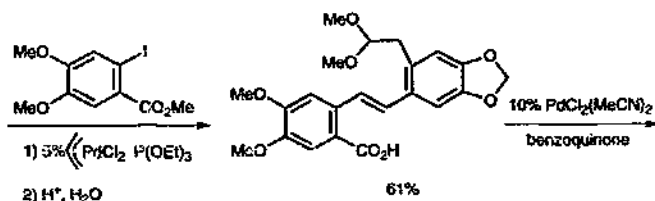
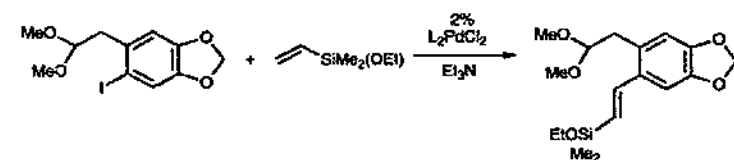
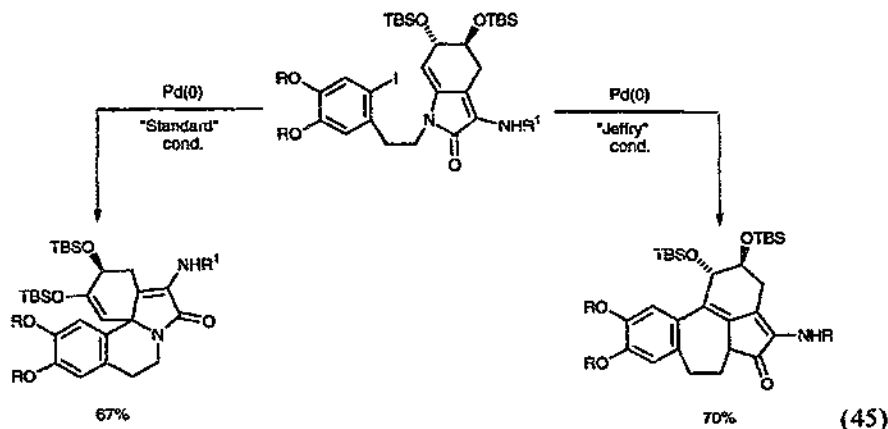
Intramolecular Heck reactions have been used to make six-membered fused carbocyclic rings [176], five-membered fused carbocyclic rings [177], seven-membered fused carbocyclic rings [178] and spirofused five-membered furanones (Eq. (44)) [179]. Depending on reaction conditions, spiro or fused ring systems could be prepared from the same starting material (Eq. (45)) [180]. In the presence of sodium formate reduced cyclization products were formed [181]. The Heck reaction, along with Pd(II) catalyzed carboxylation of alkenes was used in the synthesis of complex polycyclic systems (Eq. (46)) [182].



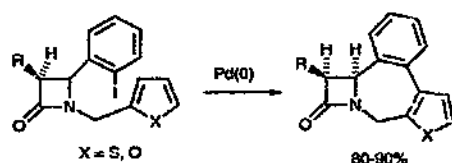
Heck insertion can even take place into aromatic [183,184] and heteroaromatic (Eq. (47)) [185] bonds ([186], for insertion into the indole double bond). By using chiral ligands, asymmetric induction in the Heck reaction was achieved (Eq. (48)) [187–189].

The Heck reaction need not be terminated by β -elimination. Rather the resulting σ -alkylpalladium complex can be trapped by stabilized carbanions [190], or by transmetalation from tin [191], (Eq. (49)) [192], or by another olefin insertion (Eq. (50)) [193].

Palladium(II) complexes promoted the stoichiometric alkylations of chiral ene carbamates with high diastereoselection [194]. Nickel acetylacetonate catalyzed the hydrozincation of the double bond of homoallylic alcohols to give terminal alkylzinc reagents, which coupled to a variety of carbon electrophiles [195]. The same reagent

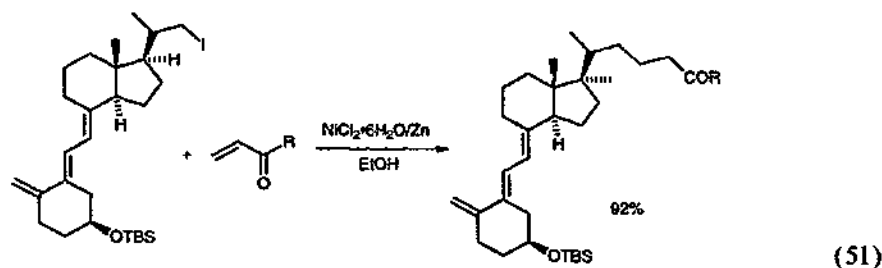
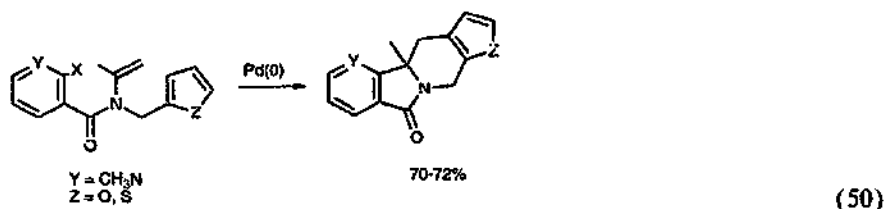
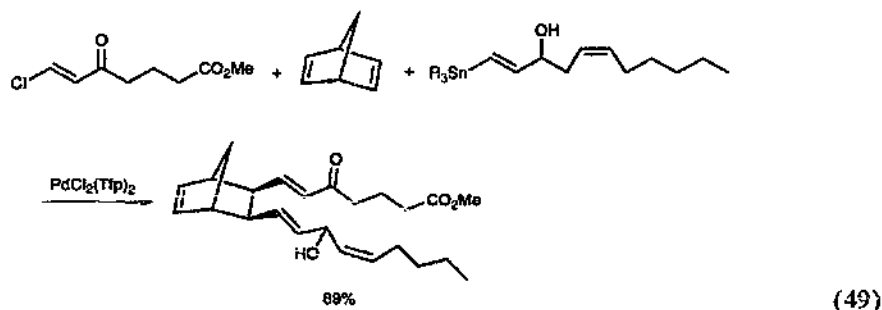
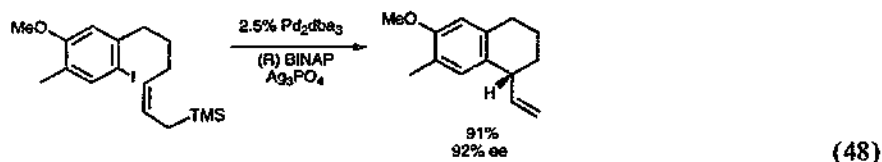


(46)

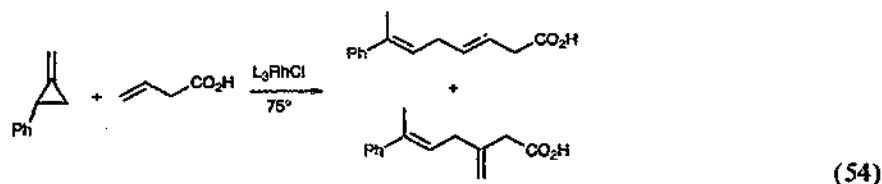
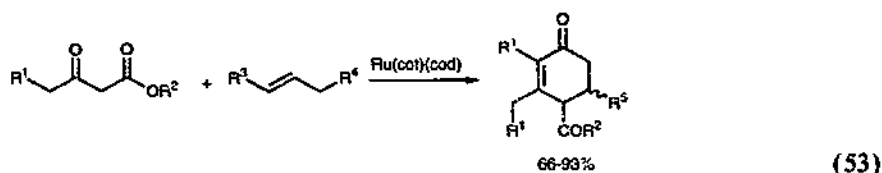
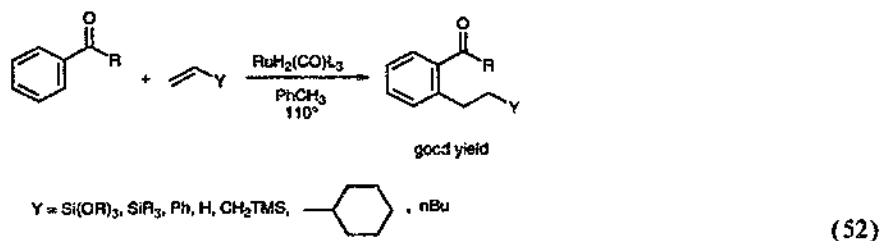


(47)

system catalyzed the intramolecular carbometallation of alkenes by a remote alkyl bromide [196] and the alkylation of conjugated enones by alkyl iodides (Eq. (51)) [197]. Aryl halides added to activated alkenes under electrochemical reducing conditions in the presence of nickel bromide [198].



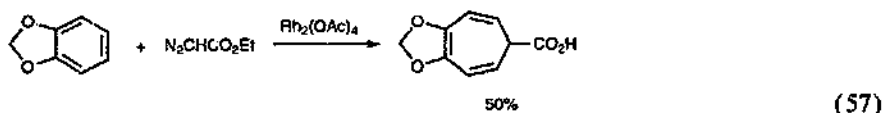
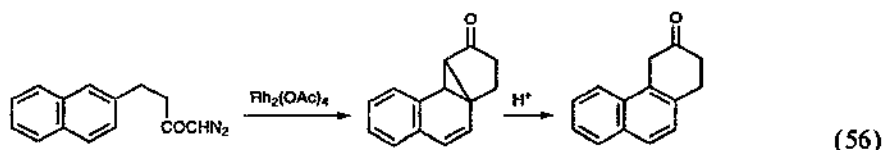
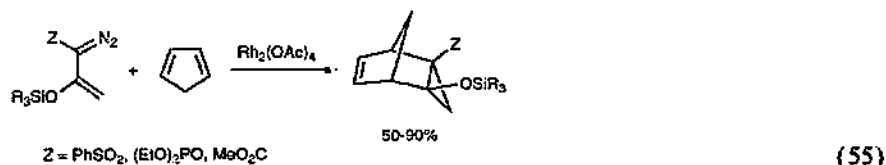
Ruthenium hydride complexes catalyzed the alkylation of arenes (C–H insertion) *ortho* to carbonyl groups by alkenes (Eq. (52)) [199,200]. Conjugated enones were similarly alkylated in the β -position by the same catalyst system [201,202]. Ruthenium(0) complexes catalyzed the cyclization of acetoacetates with alkenes (Eq. (53)) [203], while rhodium(I) complexes catalyzed the addition of methylene-cyclopropanes to allyl carboxylic acids (Eq. (54)) [204].



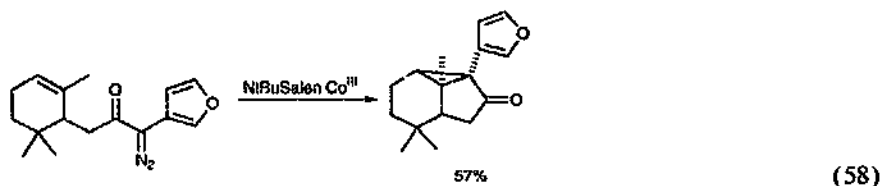
2.1.4. Decomposition of diazoalkanes, and other cyclopropanations (see heterocycles (Section 3.5) for additional examples of diazo decomposition)

Rhodium(II) catalyzed decomposition of diazo compounds continued to be extensively studied for making cyclopropanes, and studies examining the effect of the carboxylate ligand on the reaction path were many. *Trans* stereochemistry in the cyclopropanation reaction was observed with $\text{Rh}_2(\text{OAc})_4$ and $\text{Rh}_2[2\text{S-MEPY}]_4$, while $\text{Rh}_2[(4\text{S})\text{Phox}]_4$ led to *cis* cyclopropanes [205]. The system $\text{Rh}_2[(4\text{S-MPPIM})]_4$ (read the paper to figure out the acronyms) enhanced enantiocontrol in intramolecular cyclopropanations of allylic diazoacetates and provided optimal enantio and diastereocontrol for intramolecular CH insertion reactions of these substrates [206]. By varying the rhodium ligand from pfb, which gave 100% CH insertion, to cap, which gave 100% intramolecular cyclopropanation with 1-diazo-2-phenylhex-5-ene-2-one, product distribution could be effectively controlled [207]. Iodonium ylides ($\text{X}_2\text{C}=\text{IPh}$) gave the same products with the same distribution as did diazo compounds when treated with rhodium(II) carboxylates, indicating the intermediacy of metallocarbenes [208]. Decent asymmetric induction (71% ee) in the intramolecular cyclopropanation of isoprene esters of diazopropionic acid was observed using rhodium(II) 4S-MEOX catalyst [209]. Styrene was cyclopropanated by ethyl diazoacetate in the presence of rhodium-chiral 2,6-bis-substituted pyridine ligands to give 1:1 *cis* to *trans*, 16–80% ee with (S,R) stereochemistry, while the corresponding copper complex gave 2:1 *trans* to *cis*, with (R,S) stereochemistry. The yields were 40–70% [210]. Some less obvious reactions of diazo compounds in

the presence of rhodium(II) catalyst are shown in Eq. (55) [211], Eq. (56) [212], and Eq. (57) [213]. Alkynes were converted to cyclopropenes by rhodium(II) catalyzed diazodecomposition. Their rearrangements in the presence of other rhodium complexes were studied [214].

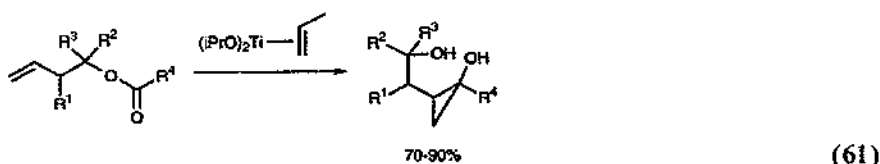
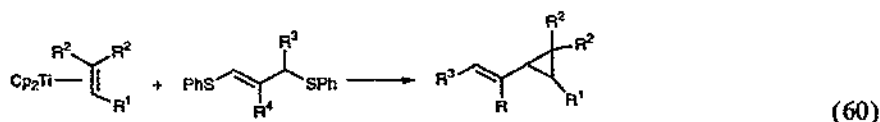


Copper triflate in the presence of a chiral oxazoline cyclopropanated styrene with ethyl diazoacetate in low yield with low ee [215]. However chiral bis-imines [216] and binap-bis-oxazolines [217] and copper triflate were very efficient catalysts for intramolecular cyclopropanations. In contrast 2,6-bis-chiral oxazoline pyridine-ruthenium complexes were highly efficient asymmetric catalysts for both inter- and intramolecular cyclopropanations of alkenes by diazo esters [218,219]. The achiral complex RuH₃[Si(OEt)₃]₂L₂ catalyzed the cyclopropanation of substituted styrenes by ethyl diazoacetate [220]. Copper(II) salen complexes catalyzed the intramolecular cyclopropanation shown in Eq. (58) [221], while chiral cobalt(III) salen complexes catalyzed the cyclopropanation of alkenes by *t*-butyldiazoacetate with 95:5 *trans/cis* selectivity and 70% ee [222]. Iron tetraphenylporphyrin catalyzed the cyclopropanation of alkenes by ethyl diazoacetate with a selectivity for terminal olefins, and electron deficient olefins, giving predominantly the *trans* cyclopropane [223]. Palladium catalyzed cyclopropanation by diazomethane (Eq. (59)) [224].

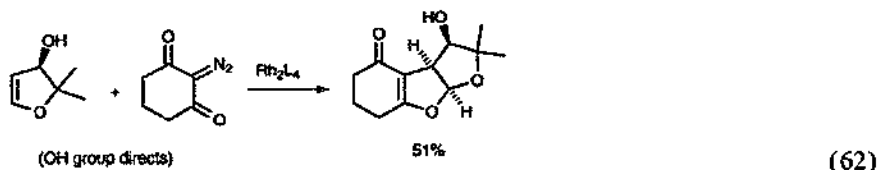




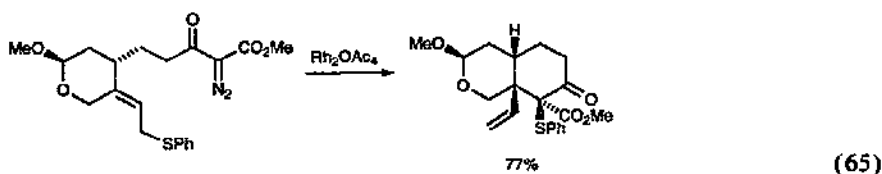
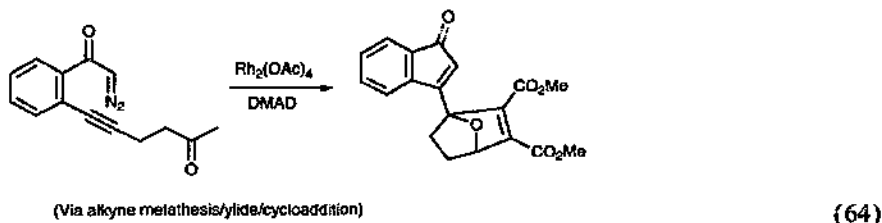
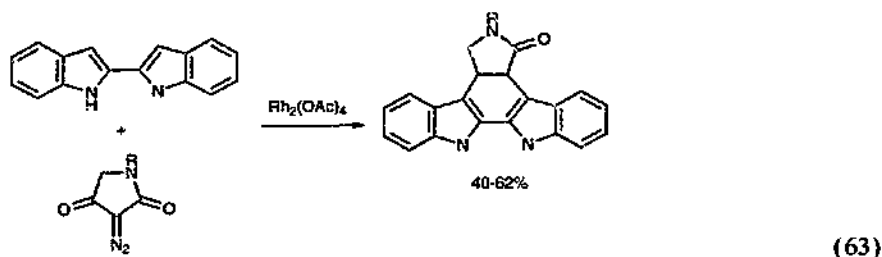
An EHMO study of the cyclopropanation of alkenes by $(\text{CO})_5\text{Cr}=\text{CPh}(\text{H})$ and $\text{Cp}(\text{CO})_2\text{Fe}=\text{CH}_2^+$ found an early transition state, no intermediates, and back side nucleophilic attack of the olefin on the carbene carbon. Metallocyclobutanes were found to be very high in energy [225]. Chromium–carbene complexes cyclopropanated dienoid esters exclusively at the γ,δ -double bond, in good yield with good diastereoselectivity [226]. α,β -Unsaturated imines formed cyclopropanes, not aziridines [227]. Titanium complexes effected some unusual cyclopropanations (Eq. (60)) [228] and (Eq. (61)) [229]. Sulfur ylides cyclopropanated chromium tricarbonyl complexed styrene [230].



A new chiral ligand for rhodium carbenoids, 4(*S*)-MEOX was very efficient for intramolecular CH insertion of sterically demanding diazoacetates [231]. Rhodium(II) complexes with 5(*S*)-MEPY or chiral bis-oxazolines as ligands catalyzed the insertion of carbenes into cinnanyl phenyl sulfide allylic C–H bonds with allylic transposition in up to 34% ee [232]. Rhodium(II) (*S*)-PTPA catalyzed intramolecular insertion into an aryl CH bond with up to 95% ee for α,α -diphenyl- α' -diazoketones [233]. Rhodium(II) octanoate catalyzed five-membered ring formation by insertion into an aliphatic methylene group [234]. The indanol ester of diazoacetic acid decomposed to give either indanone or what would be the Wittig product of indanol, depending on the rhodium catalyst [235]. More complex insertion reactions are shown in Eq. (62) [236] and Eq. (63) [237].



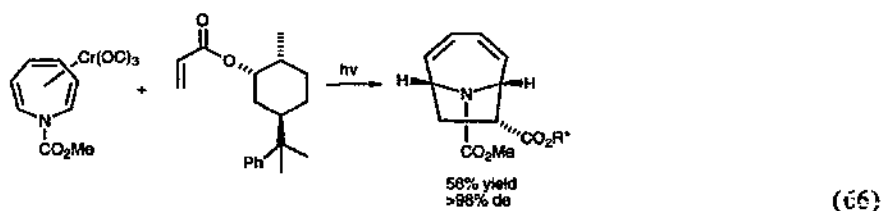
Rhodium catalyzed diazo decompositions that proceed via ylides are shown in Eq. (64) [238] and Eq. (65) [239,240].



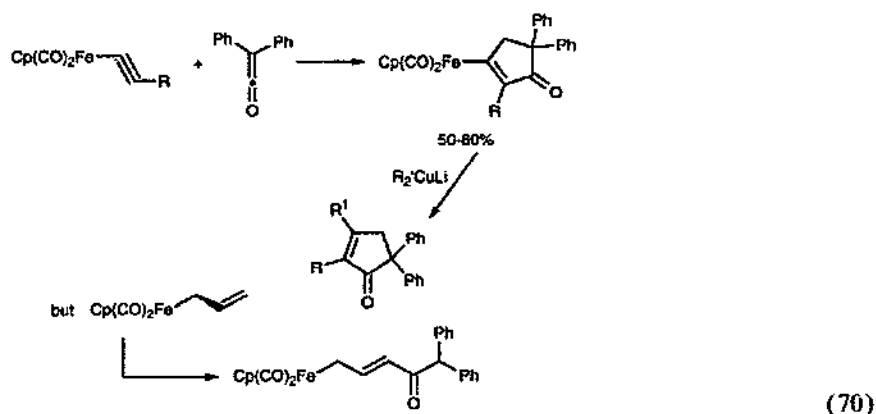
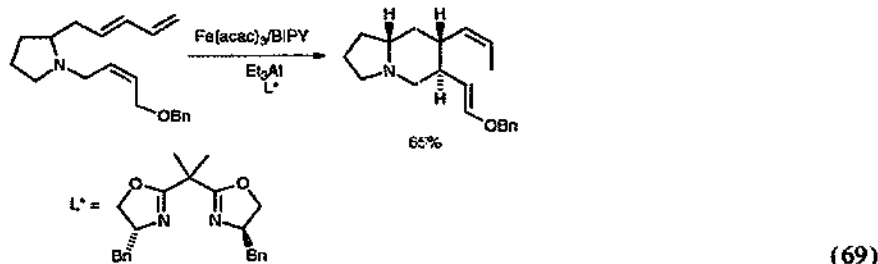
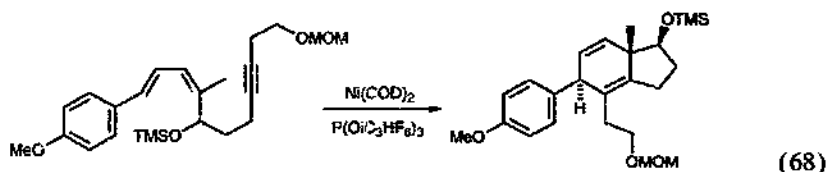
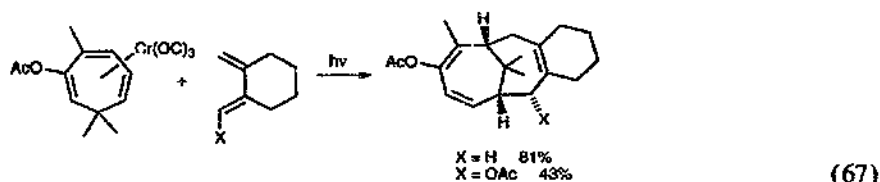
2.1.5. Cycloaddition

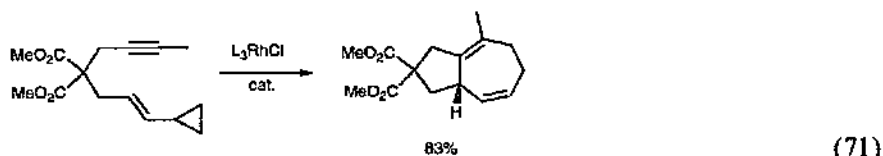
The full details of the cobalt-catalyzed 2 + 2 + 2 homo Diels Alder reaction between norbornadiene and alkynes [241] and activated alkenes [242] have been published, as have relatively simple applications of the alkyne cycloaddition to synthesis [243,244]. Asymmetry has been induced in the photochemical cycloaddition of activated alkenes to chromium tricarbonyl complexed cycloheptatrienes by selective facial complexation of chromium to the cycloheptatriene with a chiral auxiliary (e.g., (-)-8-phenylmenthol) on the sp^3 carbon [245] or by using chiral acrylate esters as substrates (Eq. (66)) [246]. This cycloaddition process was efficient with alkynes [247] as well as with chromium complexed cyclooctatrienes or tetraenes [248]. Complex systems could be quickly assembled in this manner (Eq. (67)) [249].

Other synthetically useful cycloadditions are shown in Eq. (68) [250] and Eq. (69)



[251]. Fp allyl and propargyl complexes cycloadded to ketenes (Eq. (70)) [252]. Rhodium(I) complexes catalyzed the cycloaddition of alkynes with vinylcyclopropanes (Eq. (71)) [253].

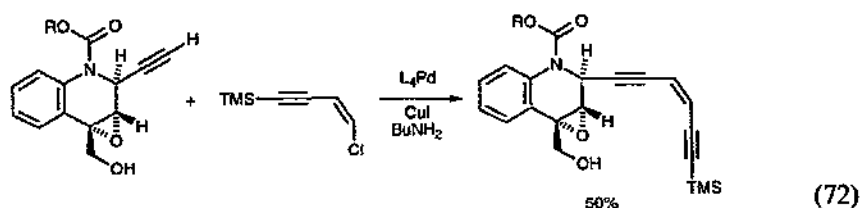




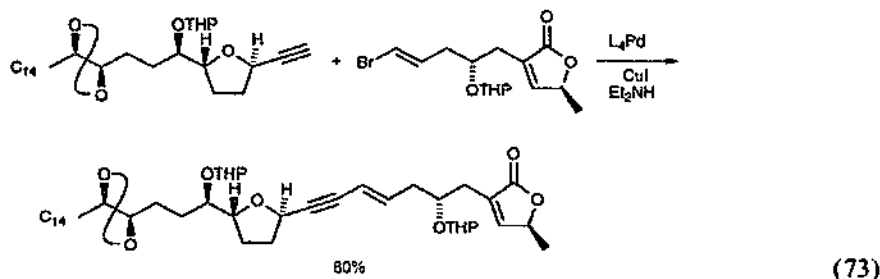
2.1.6. Alkylation of alkynes

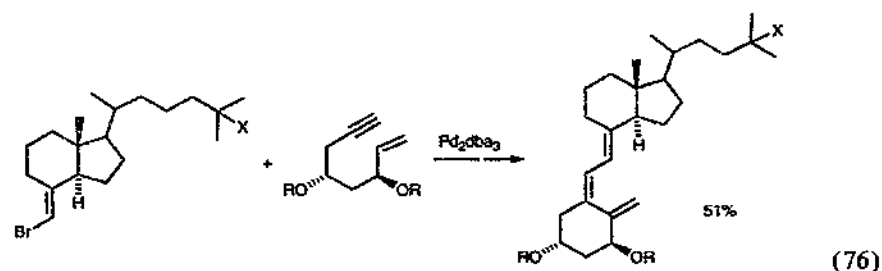
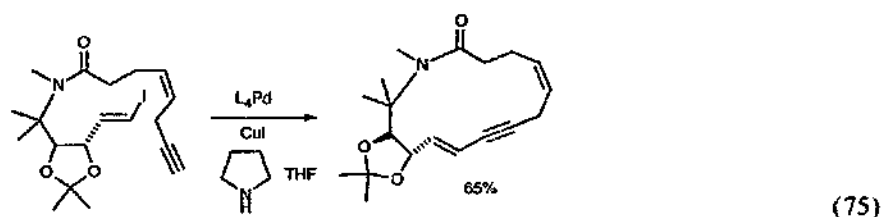
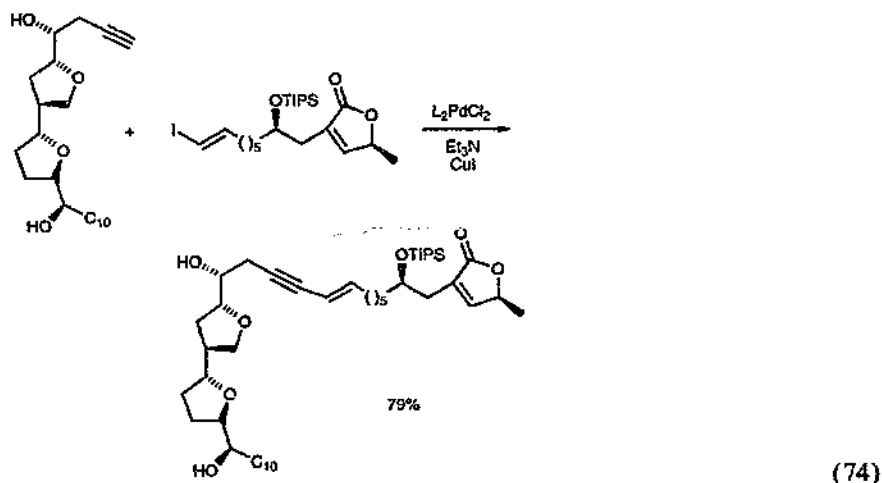
The total synthesis of ene-diyne drugs has involved extensive use of the palladium catalyzed alkylation of alkynes by vinyl halides. The design of ene-diyne products has been reviewed (57 references) [254]. 1,2-Dichloroethene was bis-alkynylated by simple alkynes as well as enynes to give polyenynes using palladium/copper iodide catalysts [255]. 1-Chloro-1-iodo alkenynes were bis-alkynylated stepwise, using palladium(0)/copper(I) catalysts [256]. As expected, the iodide reacted first. Similarly 1,2-dihaloalkenes were bis-alkynylated [257]. Alkenes tetrasubstituted with alkynes were prepared by the palladium/copper catalyzed coupling of alkynes to 1,1-dibromo-2,2-(bis)trimethylsilylethynyl ethene [258,259].

1-Bromo-1-trimethylsilyl ethene was coupled to a range of terminal alkynes using palladium(0)/copper(I) catalysts in the presence of butyl amine [260]. A related catalyst system was used to couple substituted bromocyclopentenes to *o*-ethynyl benzyl alcohol [261] and vinyl bromide to a functionalized propargyl ether bearing an aliphatic bromo group [262]. Ene-diyne were prepared by the palladium/copper/amine catalyzed coupling of alkynes to chloroenynes [263,264], Eq. (72) [265].



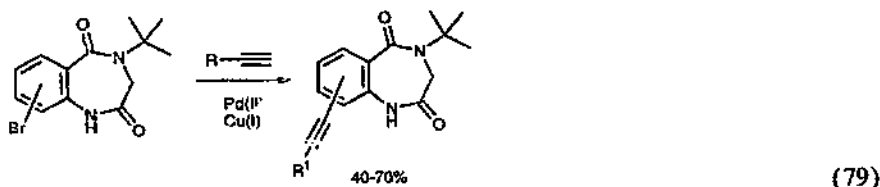
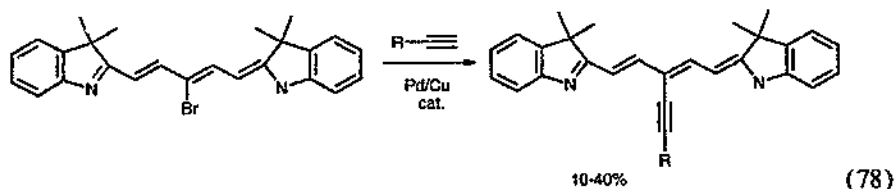
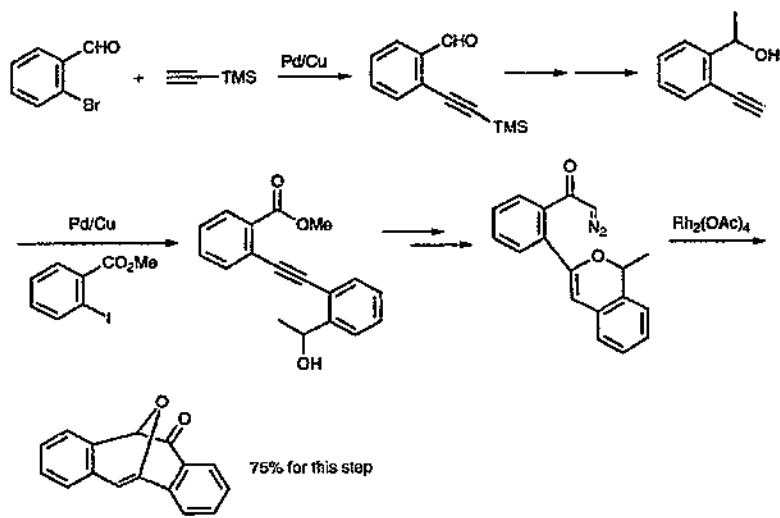
Other alkylations of alkynes by vinyl halides in more complex systems are shown in Eq. (73) [266], Eq. (74) [267], Eq. (75) [268] and Eq. (76) [269,270].





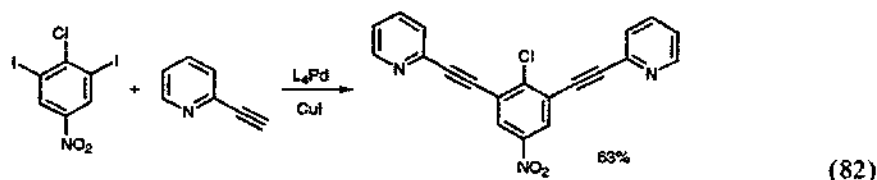
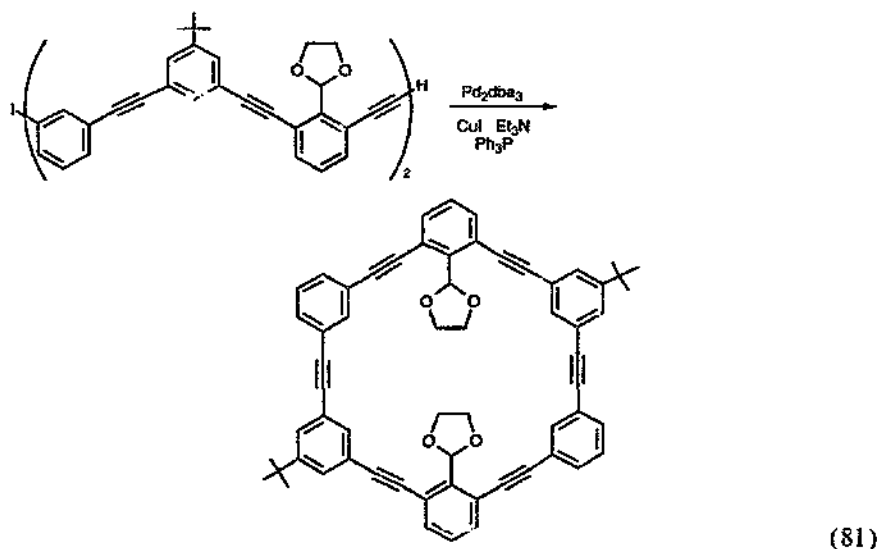
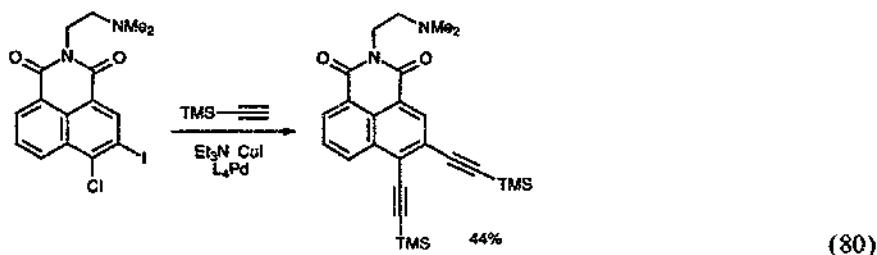
Palladium/copper/amine catalysts also promoted the arylation of alkynes by aryl halides. The system palladium on carbon/ Ph_3P / CuI / K_2CO_3 /DME/ H_2O at 80°C catalyzed this process efficiently with a wide range of aryl and heteroaryl halides [271]. Trimethyl silyl acetylene was arylated by 2,3-dimethoxy iodobenzene using $\text{L}_2\text{PdCl}_2/\text{CuI}$ /piperidine as catalyst [272], as was *m*-methoxyiodobenzene [273a]. Tetraphenyl porphyrins having one terminal alkyne substituted phenyl group coupled to tetraphenyl porphyrins have two iodophenyl groups to form dimers and trimers [273]b. Related reactions were used in the synthesis of more complex systems (Eq. (77)) [274], (Eq. (78)) [275], and (Eq. (79)) [276]. The acetyl group was the

best protecting group for the SH group of thiophenols when these are substrates for the palladium catalyzed alkylation of alkynes [277].



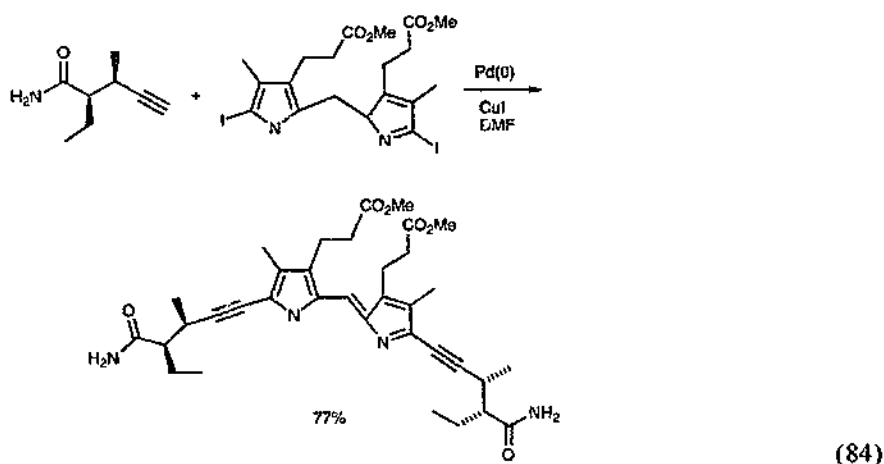
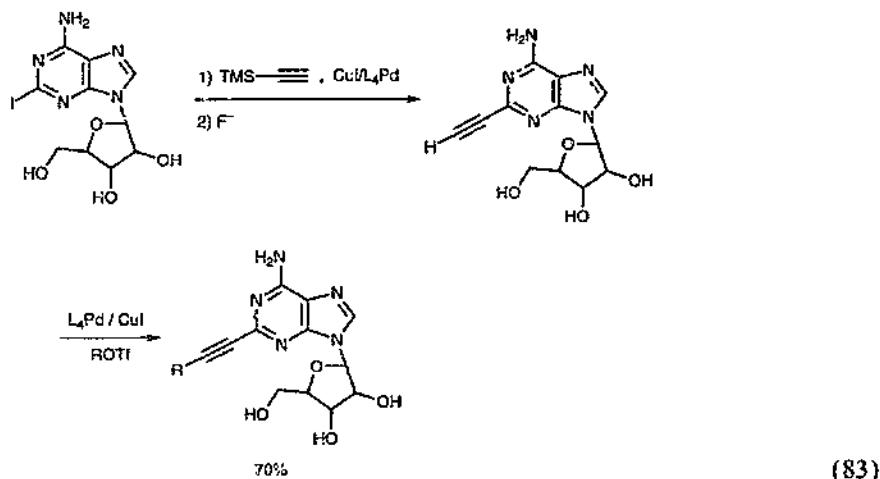
1,2-Diiodobenzenes were symmetrically [278] and unsymmetrically [279] bis-alkynylated using palladium/copper/amine catalysts. 1,3-Diiodoarenes reacted in a similar fashion [280a]. 1,3,5-Triiodobenzene, 1,2,4,6-tetraiodobenzene, and periodobenzene were polyalkynylated under similar conditions [280b]. A complex chloriodoarene was bis-alkynylated (Eq. (80)) [281]. Macrocyclic polyalkynes were prepared by systematic coupling of diiodoarenes with polyalkynes [282] (Eq. (81)) [283]. Chloro-diiodoarenes reacted only at the iodide sites (Eq. (82)) [284]. 2,7-Dibromofluorene was bis-alkynylated by acetylenic esters using $L_2PdCl_2/CuI/Et_3NH$ catalysts [285].

Aryl halides alkylated a range of propargyl alcohols under palladium/copper catalysis conditions [286]. Aryl groups could come from aryl phosphines used to



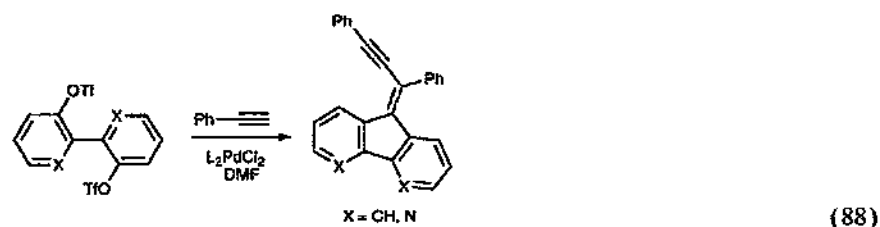
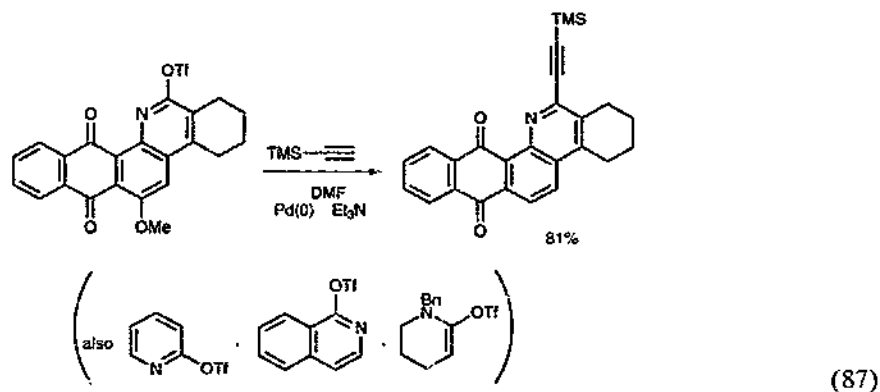
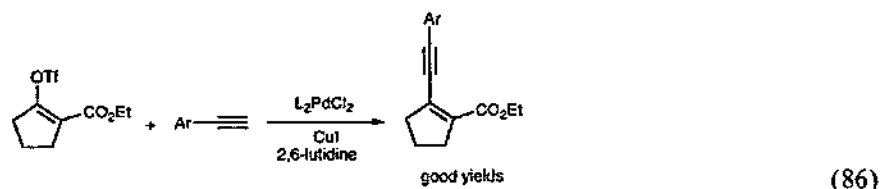
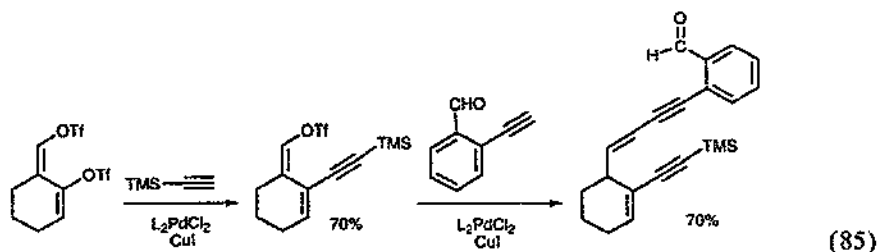
stabilize the catalysts [287]. Propargyl alcohols were arylated by *p*-iodobenzoic acid esters [288], 2-amino-5,6-difluoroiodobenzene [289], and 1,3-diiodo-4,5-dimethoxybenzene [290], while 3-butyn-1-ol was arylated by 3,4-dicyanoiodobenzene [291], all using palladium/copper type catalysts.

Alkynes were also arylated by heteroaromatic halides including iodonucleosides (Eq. (83)) [292], 3-iodo-1,2-pyrazines [293], 3,8-dibromo-1,10-phenanthrolines [294], 3,4-diiodopyrroles [295], as well as more complex iodopyrroles (Eq. (84)) [296,297].

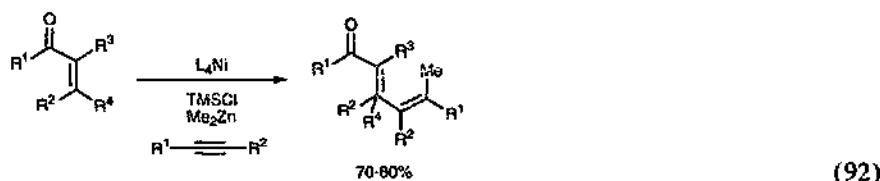
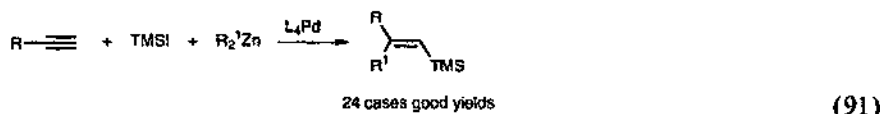
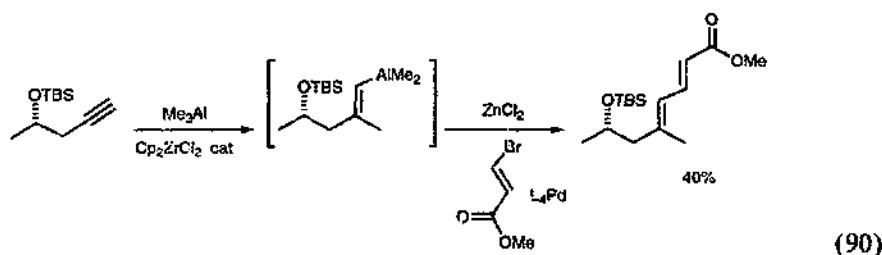
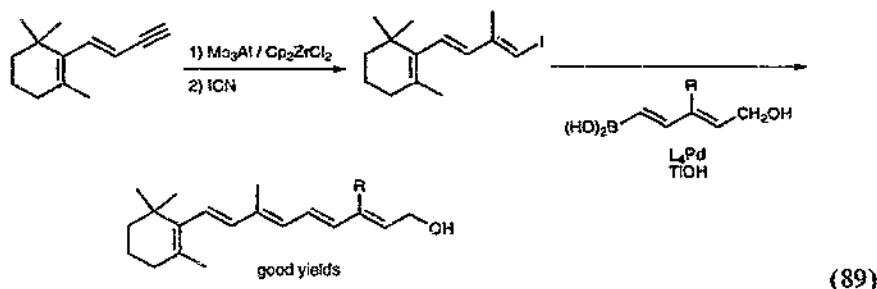


Vinyl triflates also alkylated alkynes (Eq. (85)) [298], (Eq. (86)) [299], (Eq. (87)) [300], and (Eq. (88)) [301].

The palladium/copper catalyzed coupling of iodoalkynes to alkynes was optimized and its mechanism studied [302]. Using water-soluble phosphine ligands it could be run in aqueous acetonitrile [303]. Kinetic studies demonstrated that although palladium(II) acetate was the catalyst precursor, palladium(0) was spontaneously produced. Zirconocene dichloride catalyzed the carboalumination of alkynes, which were then further alkylated (Eq. (89)) [304] and (Eq. (90)) [305]. Titanocene dichloride catalyzed the “hydrozincation” of alkynes to give vinyl zinc halides, which were coupled to aryl halides using palladium catalyst [306]. Other unusual reactions of alkynes are seen in Eq. (91) [307] and Eq. (92) [308].

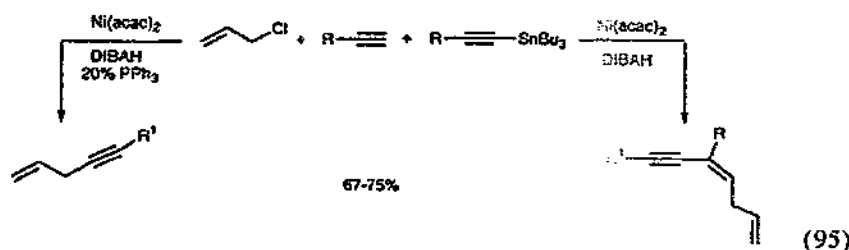
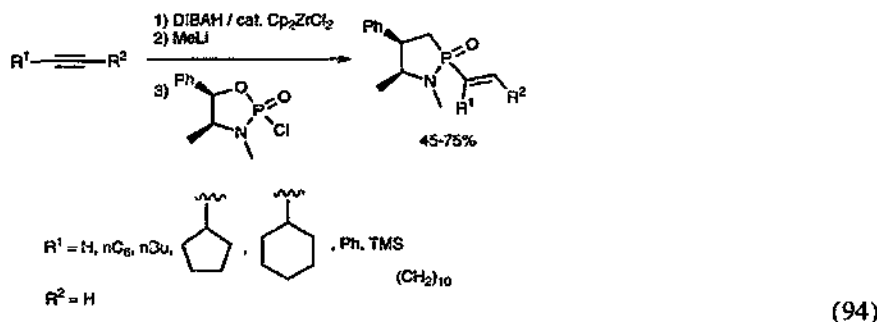
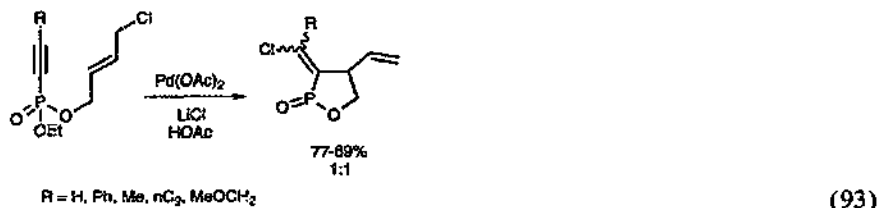


Perfluorophenyl copper added to perfluoro-2-butyne to give a vinyl cuprate which alkylated iodo-perfluoropropene [309]. β -Alkynulenones underwent cuprate additions at the γ -position followed by alkylation at the α -position to give allenes in good yield [310].

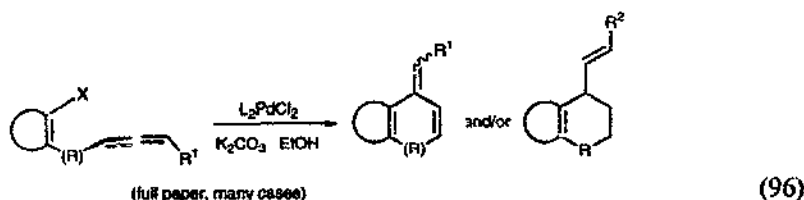


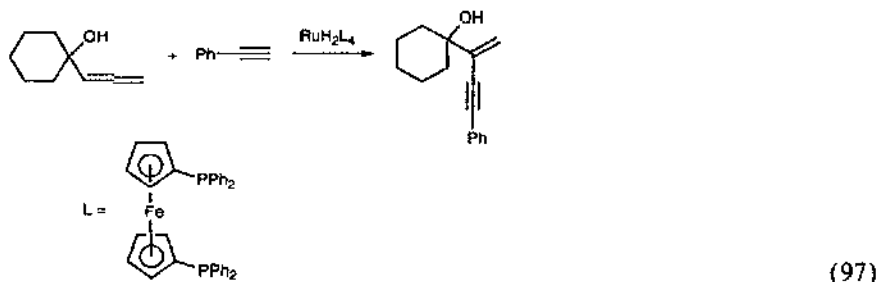
Aromatic ketones were vinylated by alkynes (formal aromatic C–H addition to an alkyne) *ortho* to the carbonyl group by $\text{RuH}_2\text{L}_3\text{CO}$ catalysts [311]. Phosphorous heterocycles were made by intramolecular additions to alkynes (Eq. (93)) [312] and (Eq. (94)) [313]. Reduced nickel complexes catalyzed the reactions of allyl chlorides with terminal alkynes and alkynyl tin reagents to give dienyynes (Eq. (95)) [314].

Alkynes were chloropalladated by PdCl_2 , and the resulting σ -vinylpalladium(II) complex coupled to allyl chloride to give 5-chloro-1,4-dienes [315]. Palladium(0)



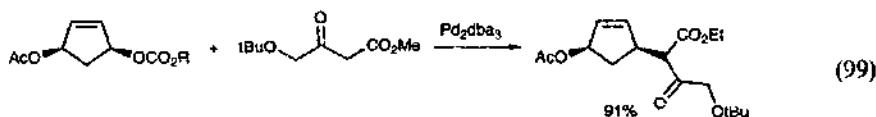
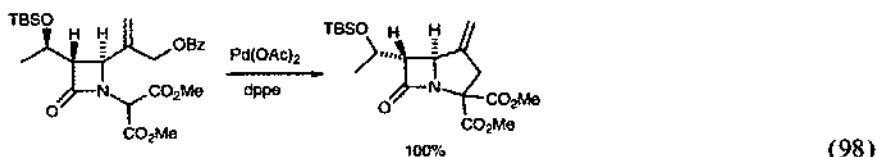
complexes catalyzed the alkylation of allenes by stabilized carbanions [316] to give mixtures of regioisomers as well as dialkylation. Terminal aryl allenes were alkylated by cyanide stabilized carbanions in the presence of palladium(0) catalysts. Attack occurred at the terminal position when the aryl group was electron donating, but at the internal position when it was electron withdrawing [317]. Sulfone stabilized carbanions alkylated allenes at the terminal position [318]. Other interesting reactions of allenes are shown in Eq. (96) [319] and Eq. (97) [320].



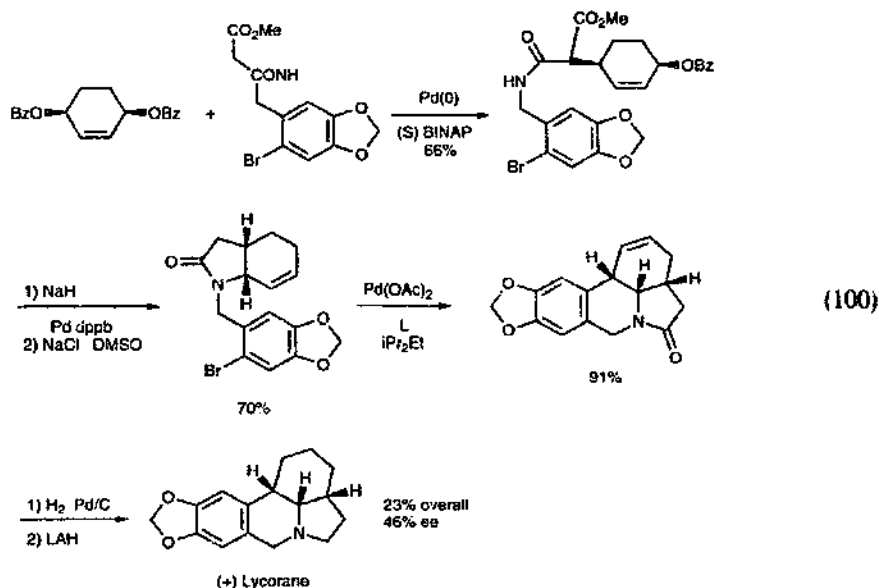


2.1.7. Alkylation of allyl, propargyl, and allenyl systems

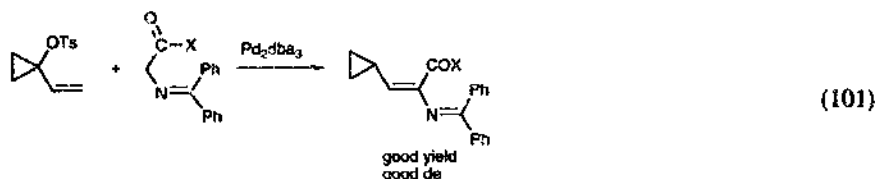
The palladium catalyzed alkylation of allylic substrates by stabilized nucleophiles continues to be extensively exploited. Allyl acetates bearing methoxy groups [321] or fluoro groups on the double bond [322] were alkylated to malonates. Allyl chlorides bearing vinyl tin groups [323] were alkylated by vinyl zinc or aluminum reagents under palladium catalysis, while allyl epoxides reacted with vinylstannanes predominately by an S_N2' pathway [324]. Palladium(0) catalyzed the alkylation of allyl stannanes by aryl halides and triflates [325] as well as by stabilized carbanions [326]. Vinyl silanes in the absence of fluoride [327], arylboronic esters [328] and 3-mercurioindoles [329] all alkylated allylic halides under palladium catalysts. Stabilized enolates alkylated benzyl carbonates in the presence of palladium(0) catalysts [330]. Synthetic applications of this type of reaction are seen in Eq. (98) [331], Eq. (99) [332], and Eq. (100) [333].



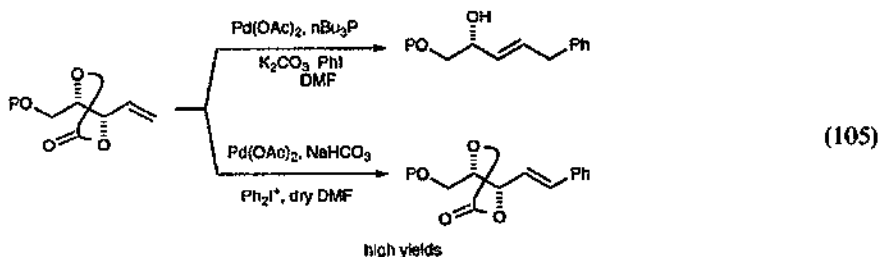
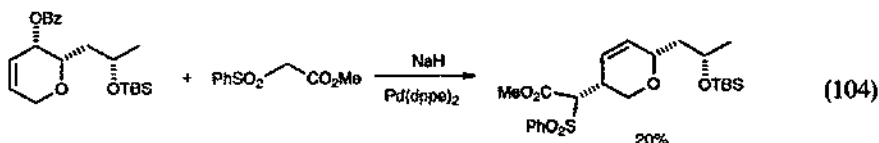
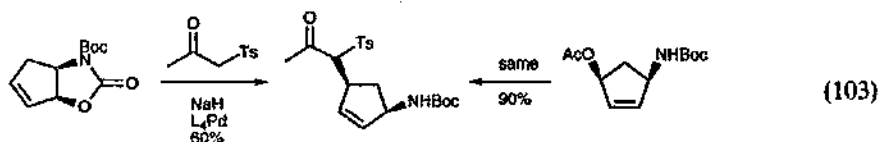
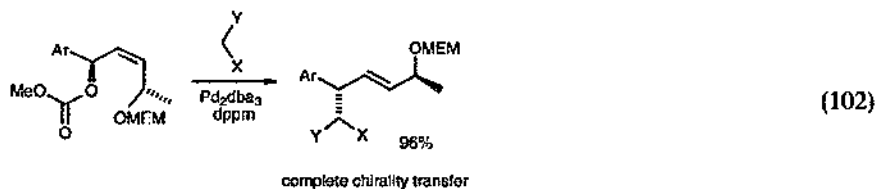
Asymmetric induction in the palladium-catalyzed alkylation of allylic acetates has become a growth industry, with the 1,3-diphenyl allyl system being the favored substrate. Chiral diamines (up to 95% ee) [334], bis-cyclic imines and oxazolines (X-ray of intermediates, 88–94% ee) [335], 2-(*o*-diphenylphosphino)phenyl oxazolines (nitromethane anion, 97% ee) [336], TADDOP (mechanism study) [337] and *ortho*-bis-chiral sulfoxide ligands (20–64% ee) [338] have all been used with varying degrees of success. A 1-acetoxy allyl acetate was alkylated in excellent ee using a chiral diamide-bis-phosphine catalyst [339]. 1,3,3-triphenyl allyl systems were also



efficiently asymmetrically alkylated using chiral phosphine-oxazoline ligands [340,341], even with the carbanion of α -iminophosphonic acid esters [342,343]. Terminal allyl phosphonates underwent alkylation at the internal position, using the same ligand in 86–95% ee [344]. Cyclic allyl acetates were alkylated by malonates [345] and phosphonate ester analogs of malonates using chiral β -diphenylphosphino carboxylic acids as ligands [345,346]. Benzyl acetates were asymmetrically alkylated by stabilized carbanions in the presence of palladium(0) (*S,S*)-BDPP ligands [347]. Chiral enolates utilizing Evans' chiral oxazolidinone auxiliary alkylated allyl acetates in modest yield but with good stereocontrol [348]. Chiral sultams alkylated cyclopropyl-allyl tosylates efficiently (Eq. (101)) [349]. Palladium catalyzed alkylation of allylic acetates having a chiral sulfoxide on the 2-position with 20–80% de [350]. Chiral allyl acetates were alkylated with complete chirality transfer (Eq. (102)) [351]. Other useful asymmetric transformations are shown in Eq. (103) [352,353], Eq. (104) [354] and Eq. (105) [355].



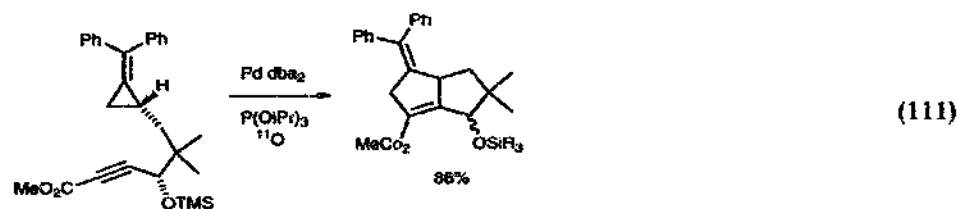
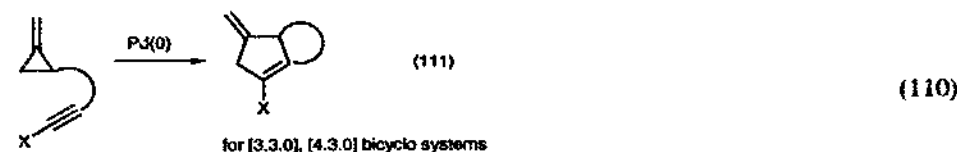
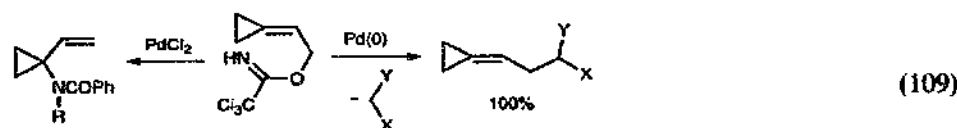
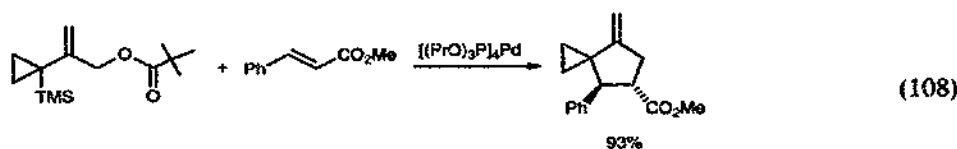
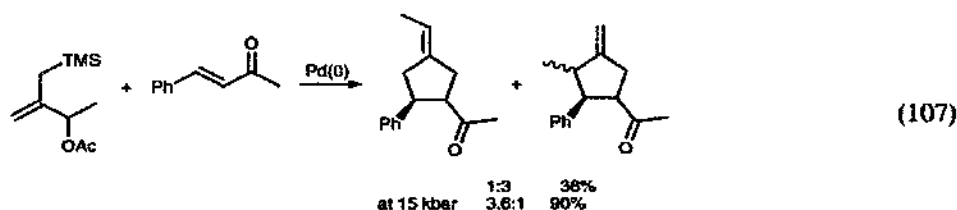
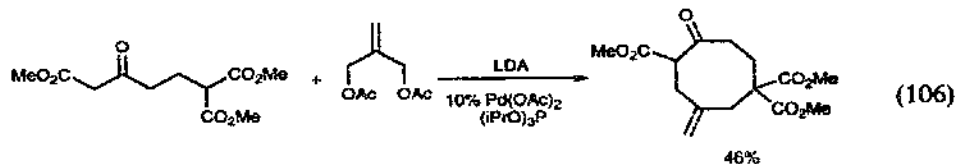
Palladium(II) chloride catalyzed the alkylation of allylic alcohols by enol ethers under acidic conditions [356], while chiral quaternary allylic ammonium salts were



asymmetrically alkylated in an $\text{S}_{\text{N}}2$ manner by stabilized carbanions [357]. α -Acetoxy- α -imino acids were alkylated by malonates in the presence of palladium catalysts [358].

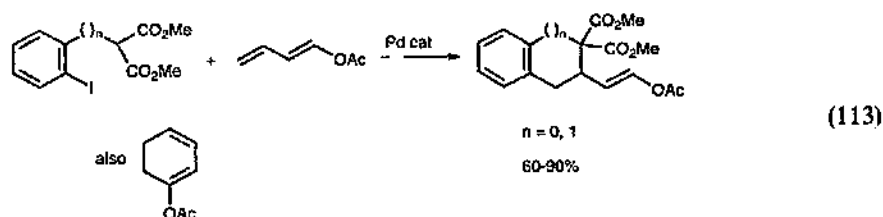
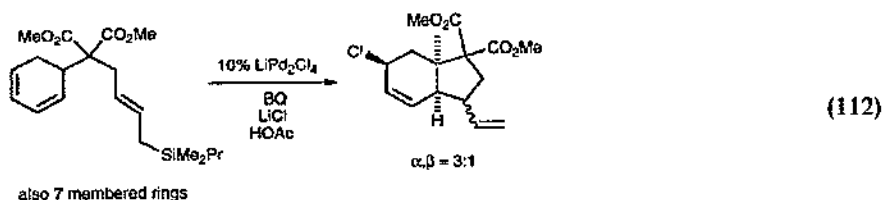
Bis-allylic halides were bis-alkylated by enolates of α -iminoesters in the presence of palladium(0) catalysts [359]. Bis-allylic systems were precursors to palladium-catalyzed trimethylenemethane cycloaddition reactions (Eq. (106)) [360] and (Eq. (107)) [361]. Vinyl cyclopropanes (Eq. (108)) [362] and methylene cyclopropanes (Eq. (109)) [363], (Eq. (110)) [364], and (Eq. (111)) [365] all underwent palladium catalyzed cyclization reactions.

Tungsten(0) phenanthroline-carbonyl complexes catalyzed the alkylation of allylic acetates by stabilized carbanions with retention of the olefin's original position

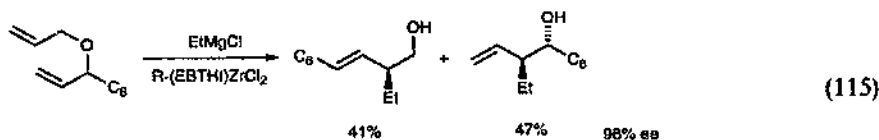
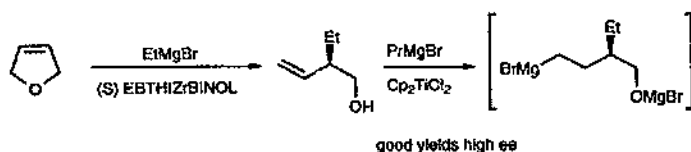
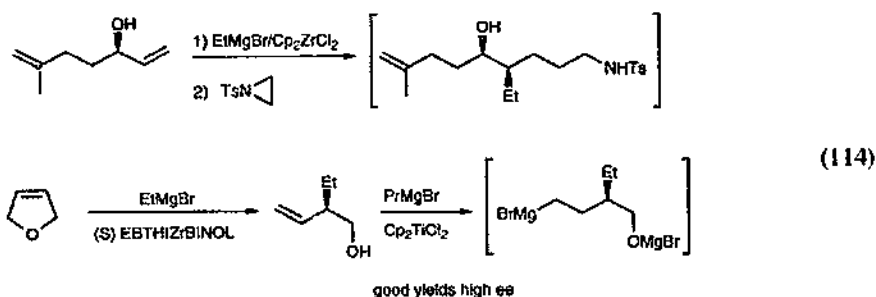


and geometry [366], as well as with clean retention of configuration with optically active substrates [367].

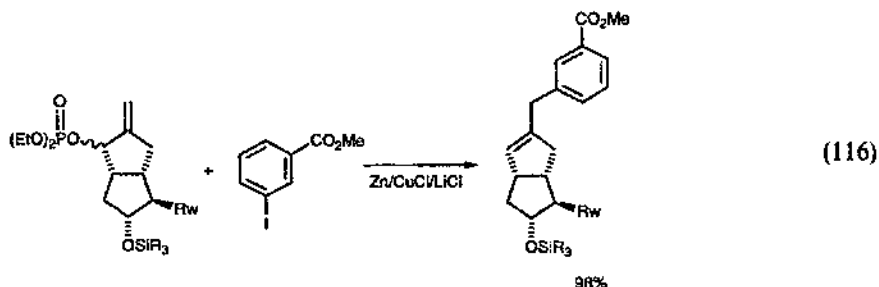
A detailed study of the (η^3 -cyclohexenyl)palladium system, from oxypalladation of cyclohexadienes, has appeared [368]. Other palladium catalyzed reactions of dienes are seen in Eq. (112) [369] and Eq. (113) [370].



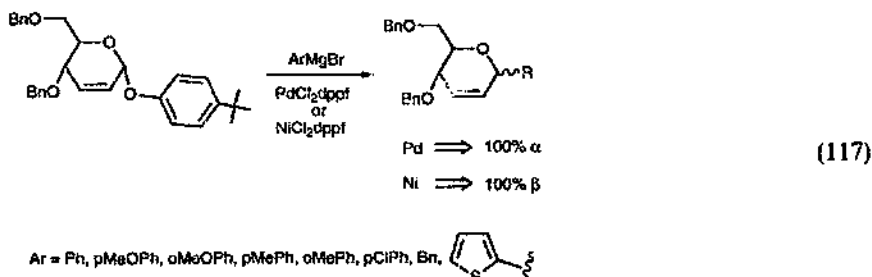
Zirconocene catalyzed addition of ethyl Grignard reagents to allylic alcohols, along with the zirconium catalyzed resolution of cyclic allylic ethers was used in synthesis (Eq. (114)) [371]. Bis-allylic ethers underwent highly stereoselective alkylation by ethyl Grignard reagents in the presence of optically active zirconocenes (Eq. (115)) [372].



Copper iodide in DMSO promoted the alkylation of allylic halides by a wide variety of functionalized vinylstannanes, mainly at the α -position [373]. The effect of solvent and copper(I) precursor on the regiochemical outcome of the reaction of preformed mono and dibutyl cuprates with allyl acetates [374] was studied. Reduced copper species promoted the alkylation of allyl phosphates with aryl iodides (Eq. (116)) [375].



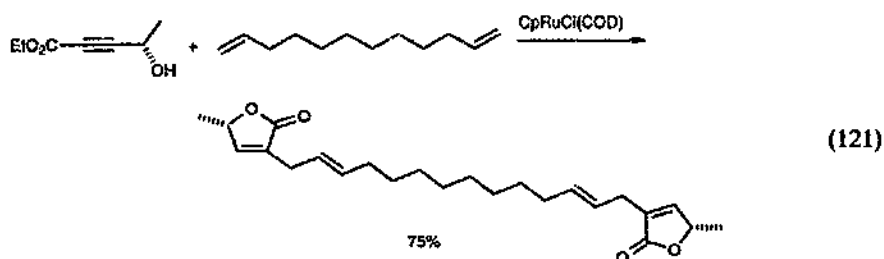
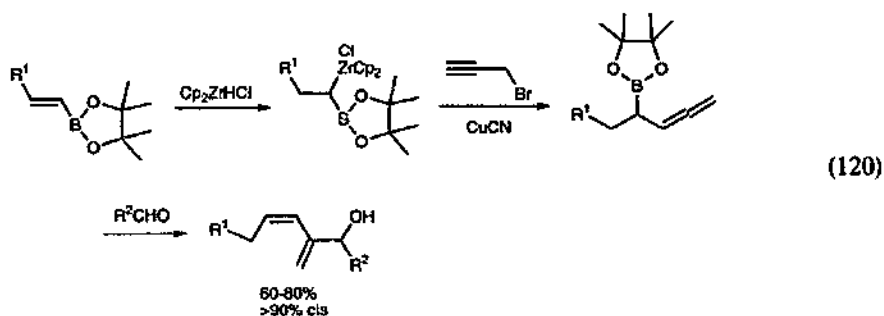
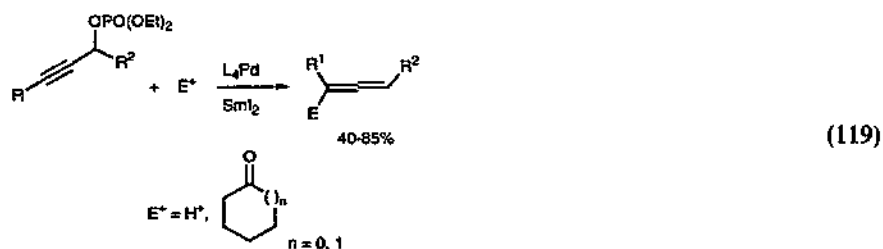
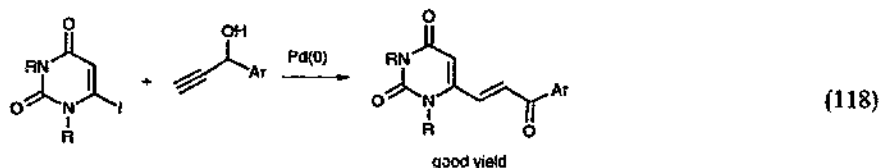
Nickel(0) complexes catalyzed the alkylation of allyl amines [376], allyl carbonates [377,378] and allyl epoxides by vinyl and aryl boronates or vinyl boronic acids. Nickel(II)-phosphine complexes catalyzed the reaction of Grignard reagents with allyl carbonates [379,380]. An interesting application is shown in Eq. (117) [381].



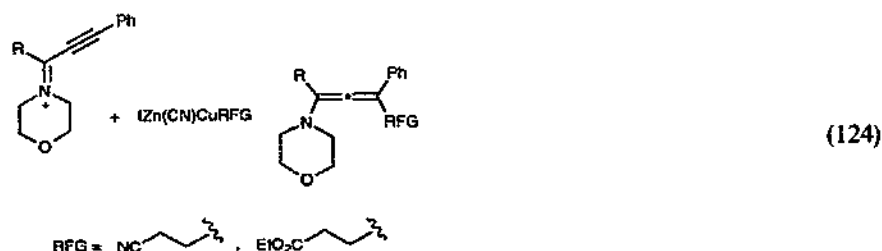
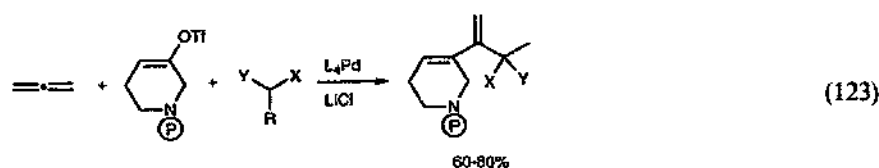
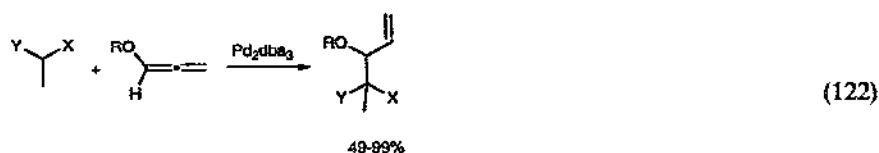
Palladium-catalyzed reactions of propargylic compounds in organic synthesis has been reviewed (> 80 references) [382]. Propargyl chlorides were alkylated by silylenol ethers *without* the production of allenes via the formation of $\text{Co}_2(\text{CO})_8$ stabilized propargyl cations (the Nicholas reaction) [383]. Propargyl ethers were similarly alkylated by silylenol ethers via their cobalt stabilized propargyl cations [384,385].

Palladium(0) catalyzed the arylation of propargyl alcohols by iodopurines (Eq. (118)) [386]. Propargyl phosphates were converted to functionalized allenes using palladium(0)/samarium(II) iodide (Eq. (119)) [387]. Hydrozirconation of vinyl boronates followed by copper(I) cyanide catalyzed coupling with propargyl bromide, followed by an aldehyde gave dienes (Eq. (120)) [388]. The full details of the ruthenium catalyzed reaction of olefins with propargyl alcohol propiolates (Eq. (121)) [389] have been published.

Palladium(0) catalyzed the addition of malonates to allenic ethers (Eq. (122))



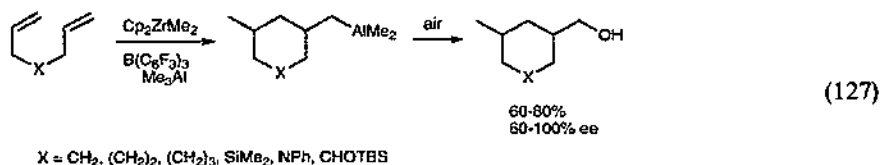
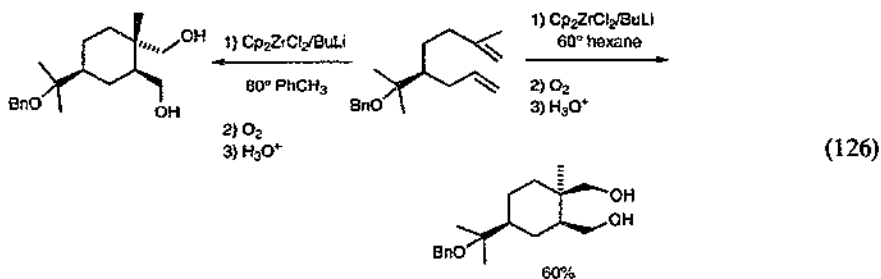
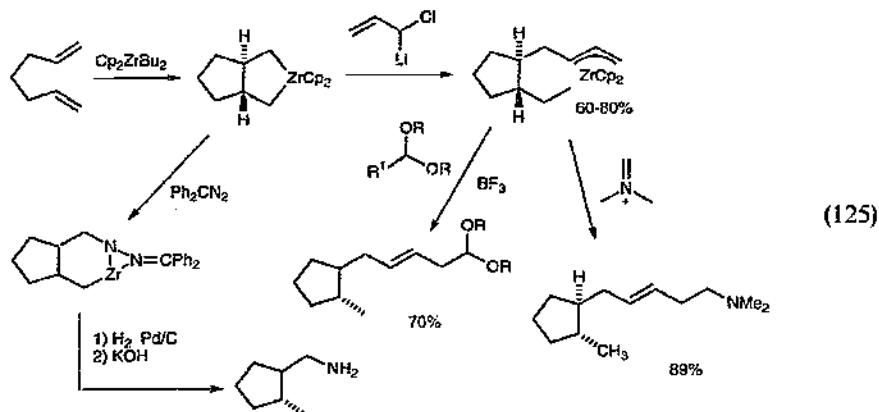
[390] and the alkylation of allenes by vinyl triflates and malonates (Eq. (123)) [391]. Functionalized zinc cuprates added to propargaldehyde iminium salts to give alleneimines (Eq. (124)) [392].



2.1.8. Coupling reactions

The formation of carbon–carbon bonds using a zirconocene–butene complex (“Cp₂Zr”) as a synthetic tool has been reviewed (29 references) [393]. The reagent reductively dimerized *o*-vinyl benzyl ethers in good yield [394]. Dienes cyclocoupled to give cyclic dialkylzirconium complexes which had rich reaction chemistry (Eq. (125)) [395,396]. Depending on the conditions, either *cis* or *trans* stereochemistry was observed (Eq. (126)) [397,398]. Dimethylzirconocene catalyzed the carbocationic polymerization of dienes to ultimately give cyclic alcohols (Eq. (127)) [399].

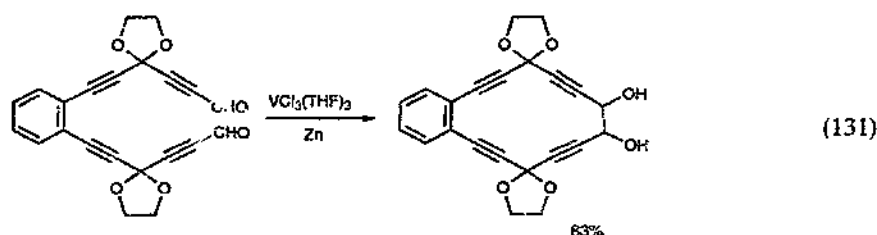
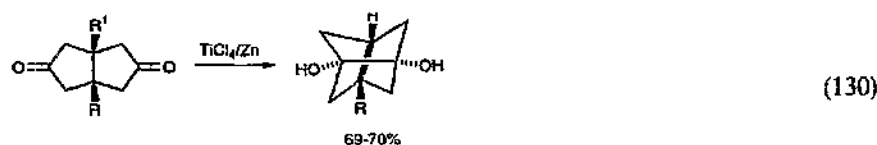
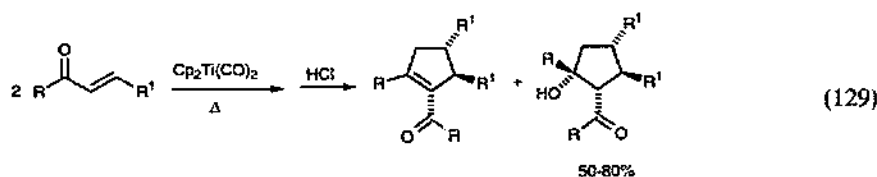
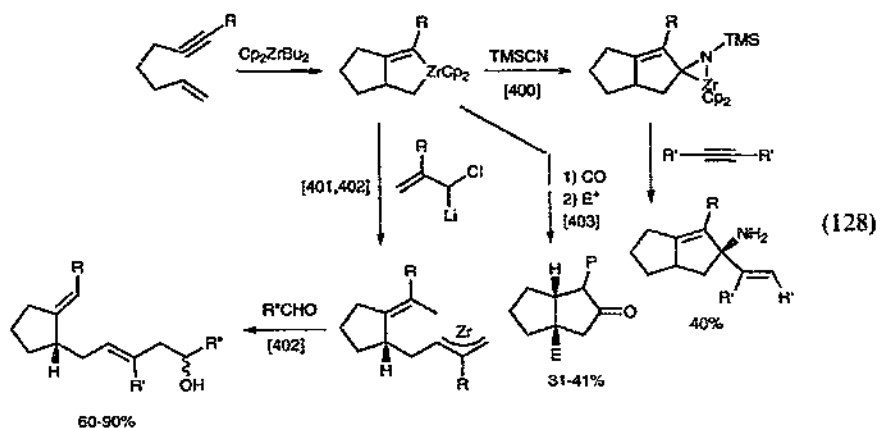
“Zirconocene” also reductively cyclized enynes to zirconacyclopentenes which also had very rich reaction chemistry (e.g., Eq. (128)) [400–404]. The same reagent intermolecularly coupled alkenes with enol ethers to give, after cleavage with iodine, 1-iodo-1,3-dienes [405], or 1,5-dienes when allyl ethers were used [406]. Diynes formed bis-zirconacyclopentenes when treated with zirconocene and ethyl Grignard reagents. These were carbonylated to give bis-cyclopentenones or hydrolyzed to give bis-alkenes [407]. Treatment of zirconacyclopentenes with alkynes led to extrusion of the alkene to give zirconacyclopentadienes which formed acyclic 1,3-dienes upon protolytic cleavage [408]. Zirconacyclopentadiene underwent reaction with alkynes in the presence of copper(I) chloride to give polysubstituted benzenes [409]. 4,4'-Bis-trimethylsilylphenylbiphenyl underwent reaction with zirconocene to give cyclic tris- and tetrakis-zirconacyclopentadiene linked by biphenyl groups [410]. Titanium



isopropoxide/isopropylmagnesium chloride cyclized 1,7-dienes to cyclopentenes, 1,7-enynes to methylcyclopentenes, and 1,7-diynes to bis-methylenecyclopentenes [411]. A similar reagent coupled 1,3-dienes with alkenes to give 1,4-dienes [412]. Enones were cyclocoupled by titanocene dicarbonyl (Eq. (129)) [413].

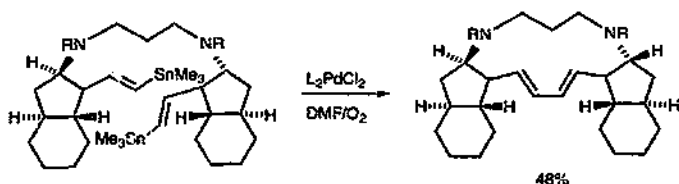
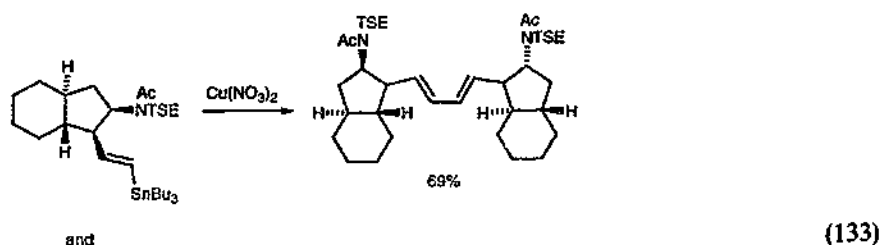
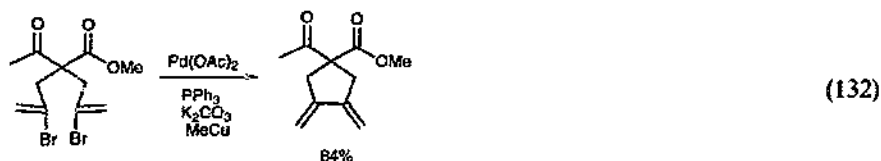
A comparative study of the McMurry coupling reaction with [HTiCl(THF)₅], TiCl₃(DME)_{1.5}/Zn/Cu, and TiCl₂ · LiCl as coupling agent has been carried out [414]. Highly distorted cone calix[4]arenes were made by McMurry coupling of opposing aryl aldehyde groups [415], as were other highly strained systems (Eq. (130)) [416]. α,β-Unsaturated aldehydes were coupled to trienes [417], while bis-propargaldehydes were coupled by related vanadium reagents (Eq. (131)) [418].

2-Metallated (zinc) oxazolines were coupled to give 2,2'-bis-oxazolines by treat-

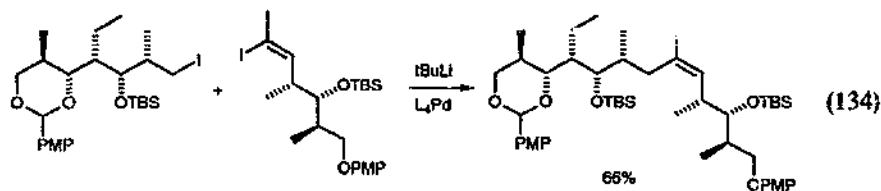


ment with palladium(0) complexes [419]. Bis-vinyl halides were coupled by treatment with palladium(II) acetate, in the presence of phosphine and methyl copper (Eq. (132)) [420]. Nickel chloride in the presence of zinc and triphenyl phosphine reductively coupled 2-bromo-polythiophenes to give thiophene oligomers [421] while 2-metallated-5-bromothiophene underwent exclusive head-to-tail dimerization when

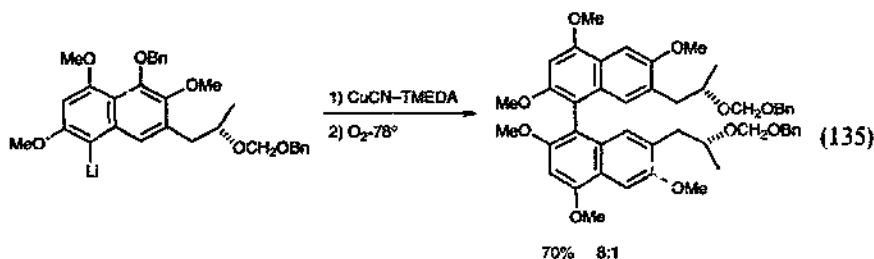
treated with nickel catalysts [422]. Nickel(0) complexes also coupled aryl tosylates [423] and mesylates [424] to give biaryls. Aryl halides were coupled to aryl triflates by treatment with hexamethylditin and palladium(0) catalysts [425]. Heterocyclic (*S*)-vinyl triflates coupled under similar conditions [426]. Vinyl stannanes were coupled with both copper(II) salts and palladium(II) complexes (Eq. (133)) [427].



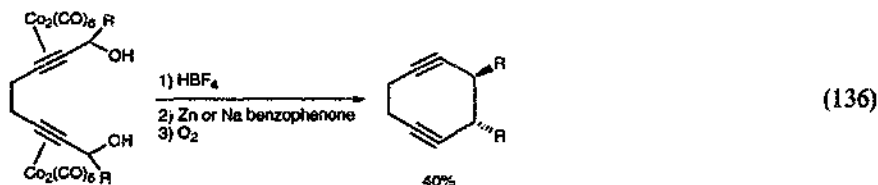
A vinyl iodide was coupled to an alkyl iodide by treatment with *t*-butyllithium in the presence of palladium(0) complexes (Eq. (134)) [428]. Organolithium reagents were coupled by treatment with nickel(II)–phosphine complexes [429], while copper powder in pyridine was used to couple 1-(chiral oxazoline)-8-bromonaphthalenes to give optically active BINAP systems [430]. BINAPs were also synthesized by copper-assisted coupling of naphthyllithium reagents (Eq. (135)) [431].



The full details of the ruthenium(II) catalyzed coupling of terminal alkynes with alkenes to give dienes have been published [432]. Internal alkynes were coupled to



allylic amines and alcohols to give 1,4-dienes by TaCl_5/Zn [433]. Reduced rhodium complexes coupled terminal alkynes to give enynes or dienyynes [434]. Stilbenes were produced by the palladium(0) catalyzed coupling of benzyldiene dibromides (ArCHBr_2) [435]. Palladium(0) complexes coupled propargyl acetates to diynes and allenynes [436]. Cyclic diynes were prepared by the oxidative coupling of cobalt complexed propargyl cations (Eq. (136)) [437].

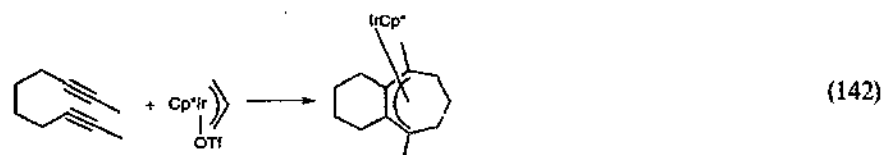
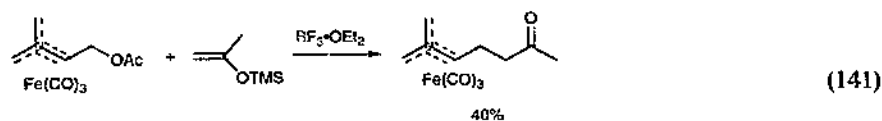
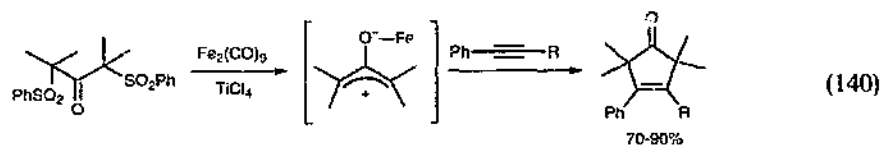
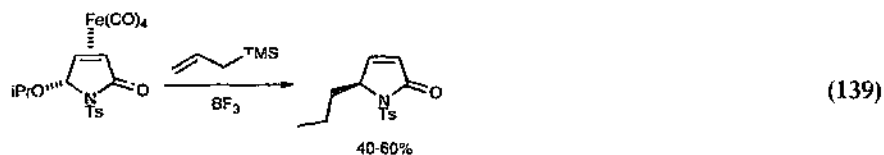
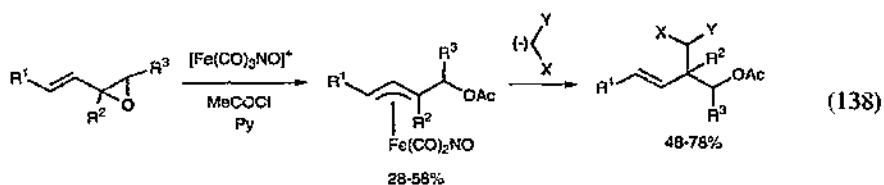
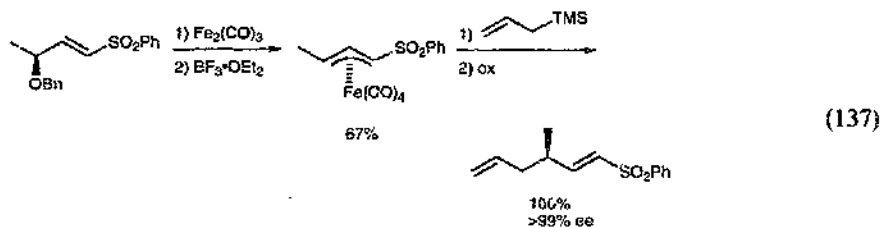


2.1.9. Alkylation of π -allyl complexes

A review (77 references) dealing with the stereochemistry of palladium catalyzed cyclization reactions — addition to π -allyl intermediates — has appeared [438]. η^3 -(2-Chloroallyl)palladium complexes were bis-alkylated to give 2,3-dialkyl propenes, when treated with stabilized carbanions in the presence of bipy or TMEDA [439]. Palladium catalyzed the reaction of vinyl halides, alkenes, and stabilized carbanions to produce alkylated allyl products [440]. The reaction proceeded through η^3 -allyl complex intermediates. α -Branched nucleophiles attacked the central carbon of η^3 -allylpalladium complexes to produce metallacyclobutanes (characterized by X-ray) which collapsed to cyclopropanes [441].

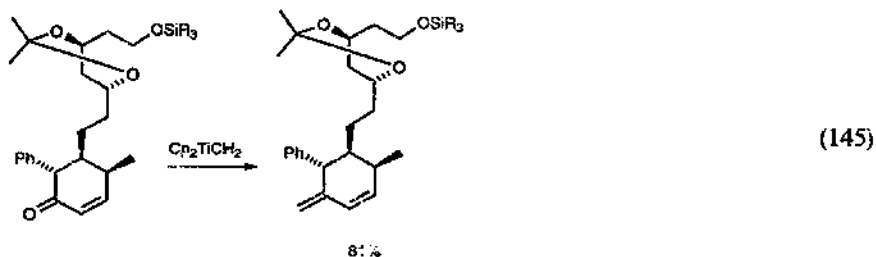
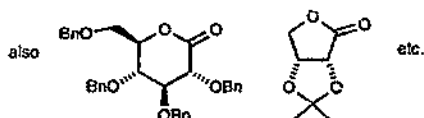
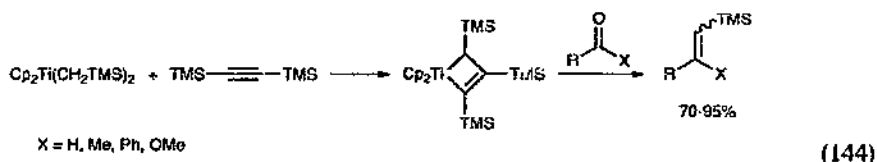
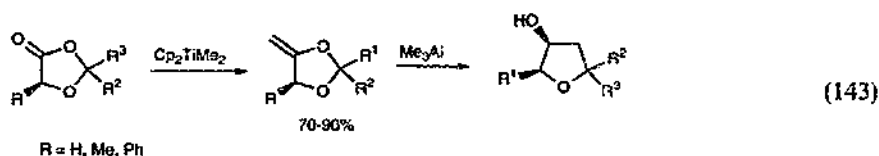
Chiral γ -benzyloxy- α,β -unsaturated sulfones were alkylated by allyl silanes with high enantioselectivity via η^3 -allyl iron complexes (Eq. (137)) [442–444]. Allyl epoxide rings opened to give η^3 -allyl iron complexes which were then alkylated by stabilized carbanions (Eq. (138)) [445]. Other reactions involving η^3 -allyliron complexes are shown in Eq. (139) [446,447], Eq. (140) [448] and Eq. (141) [449].

Preformed η^3 -allylmolybdenum complexes were used to synthesize polyhydroxylated polyenes [450]. η^3 -Allylrhodium complexes underwent reaction with a wide variety of organic substrates (amines, enolates, alkenes, oximes, ketones, aldehydes) to incorporate an allyl group [451]. Diynes underwent a $3+2+2$ cycloaddition with iridium η^3 -allyl complexes (Eq. (142)) [452].



2.1.10. Alkylation of carbonyl compounds

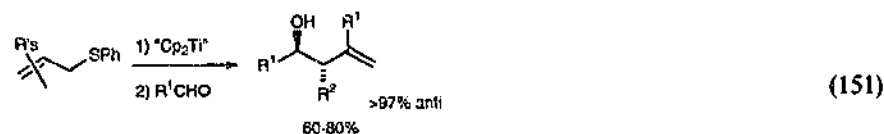
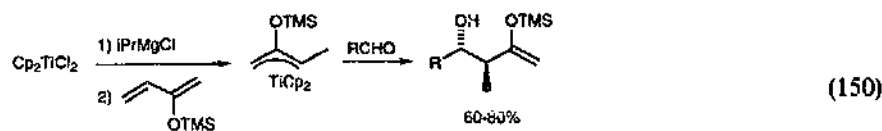
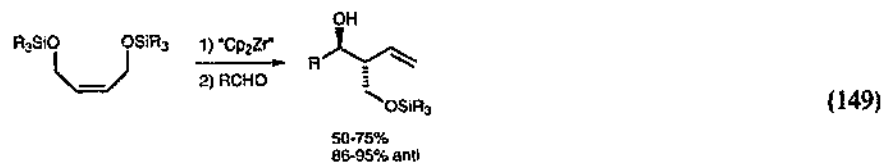
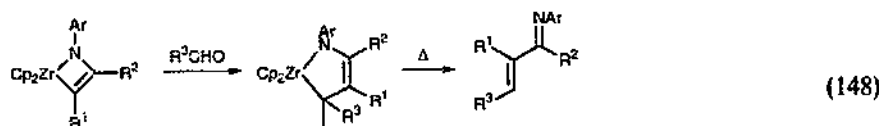
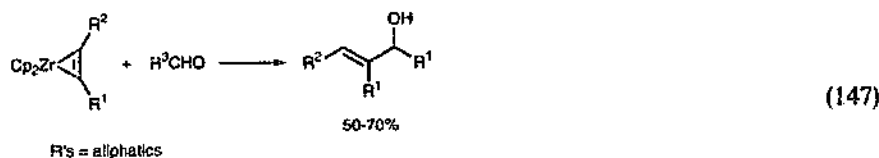
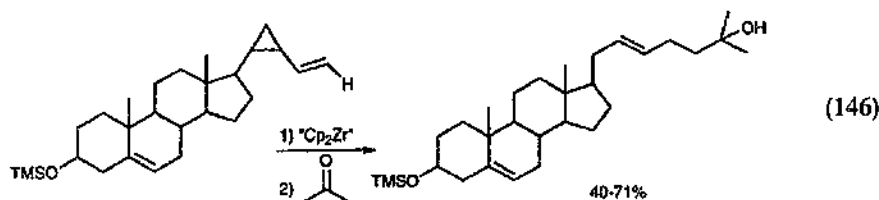
Petasis' reagent (Cp_2TiMe_2) efficiently methylenated a wide range of carbonyl compounds RCOX ($\text{X}=\text{H}, \text{R}^1, \text{Ar}, \text{OR}^1, \text{OSiR}_3, \text{OCOR}^1, \text{NR}_2, \text{NR}^1\text{COR}^1, \text{SR}^1, \text{SeR}^1, \text{TMS}$) [453] as well as cyclobutene diones [454] and lactones (Eq. (143)) [455]. A reagent which transfers the CHTMS group has also been developed (Eq. (144)) [456]. Tebbe's reagent methylenated sterically hindered cyclopentenones, while Wittig reagents just epimerized them [457]. It was also efficient with complex cyclohexenones (Eq. (145)) [458]. A reagent for transfer of the CHtBu group to ketones has been developed [459].



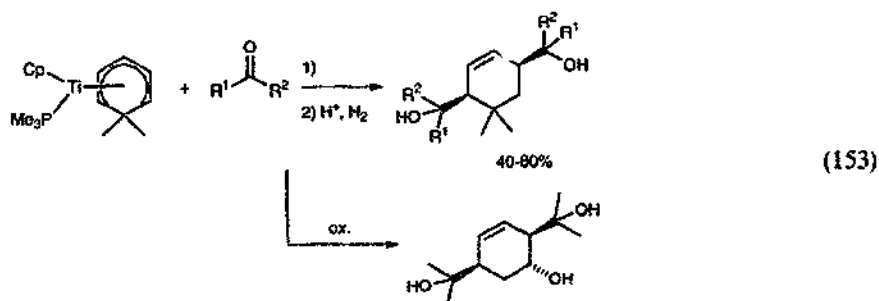
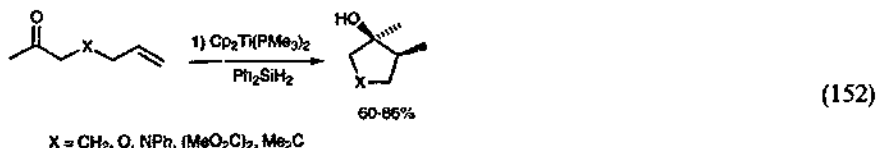
Various zirconocene derivatives alkylated ketones and aldehydes (Eq. (146)) [460], (Eq. (147)) [461], (Eq. (148)) [462] and (Eq. (149)) [463].

Aldehydes were alkylated by propargyl halides in the presence of isopropyl magnesium chloride and titanium(IV) isopropoxide [464]. Vinyl silylenol ethers (Eq. (150)) [465] and allylsulfides (Eq. (151)) [466] alkylated aldehydes in the presence of titanocene.

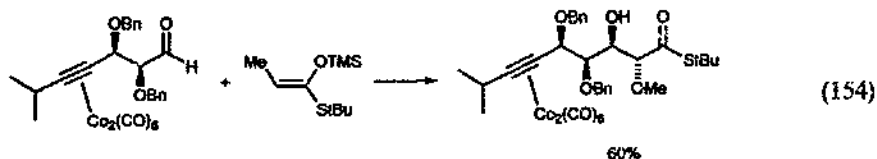
Titanocene-phosphine complexes catalyzed the hydrosilylation-cyclization of



olefinic ketones (Eq. (152)) [467,468]. Titanocene cyclohexadienyl complexes underwent reaction with two aldehydes (Eq. (153)) [469]. Alkynes were hydrotitanated (to give vinyl titanium species) then treated with ketones to produce allyl alcohols [470]. Manganese enolates were silylated to silylenolates [471].

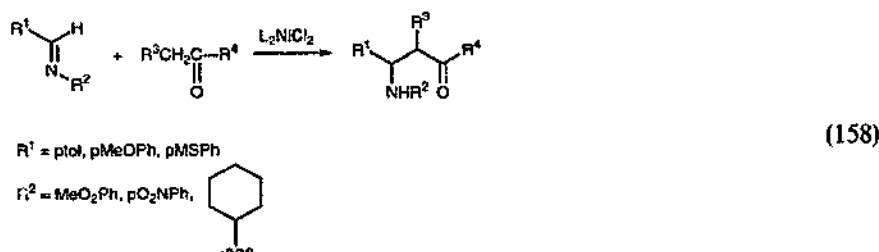
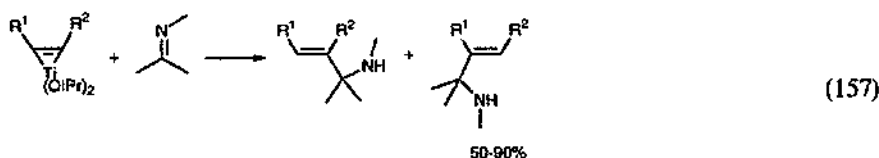
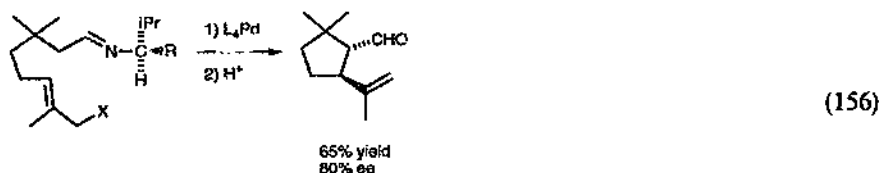
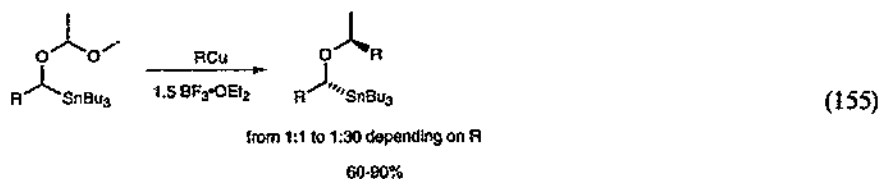


Palladium(0) complexes catalyzed the alkylation of aldehydes by allylstannanes [472], silylenol ethers [473] and cyclic allyl carbonates [474]. Cobalt complexed alkynyl aldehydes were alkylated by ketene silylacetals (Eq. (154)) [475], while cobalt stabilized enynes alkylated aldehydes in the presence of dimethylaluminum chloride [476].



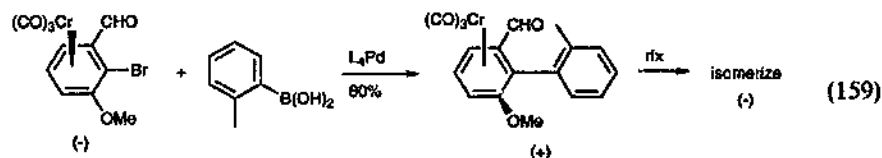
Ruthenium complexes (RuH_2L_4) catalyzed the alkylation of aldehydes by activated nitriles (malononitrile, phenylacetone nitriles) [477]. η^1 -Allyliron complexes alkylated aldehydes [478]. Ketones were dialkylated by Grignard and organic halides in the presence of $VCl_2(TMEDA)_2$ [479].

α -Stannyl ketals were alkylated by cuprates (Eq. (155)) [480]. Palladium catalyzed the cycloalkylation of chiral imines (Eq. (156)) [481]. Imines were alkylated by titanium-alkyne complexes (Eq. (157)) [482] and ketones in the presence of nickel catalysts (Eq. (158)) [483].

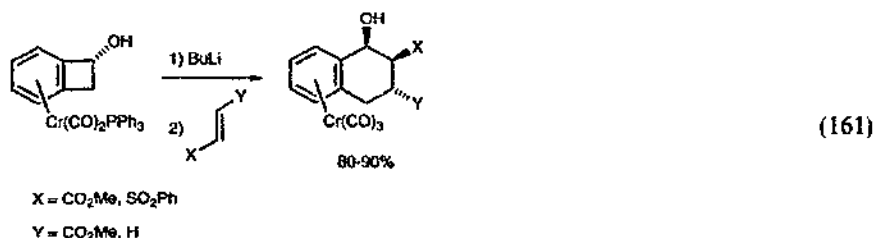
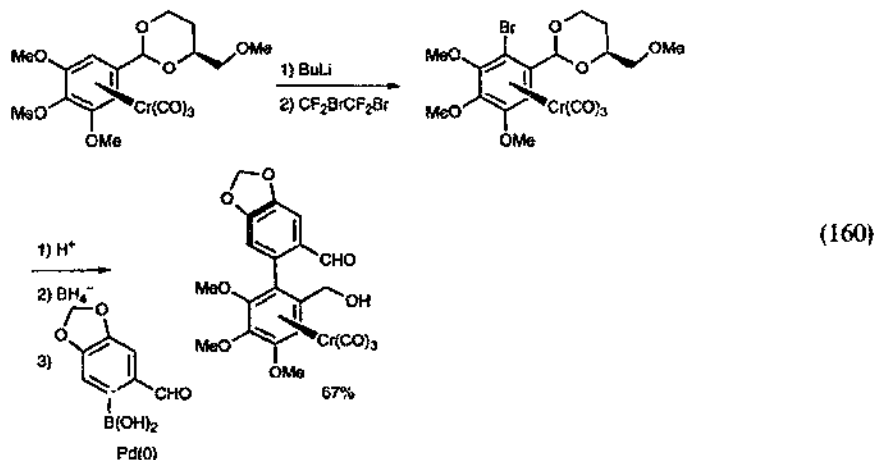


2.1.11. Alkylation of aromatic compounds

Chromium complexed aryltin reagents underwent palladium catalyzed Stille coupling to aryl halides [484], while optically active chromium aryl bromide complexes underwent Suzuki coupling (Eq. (159)) [485].



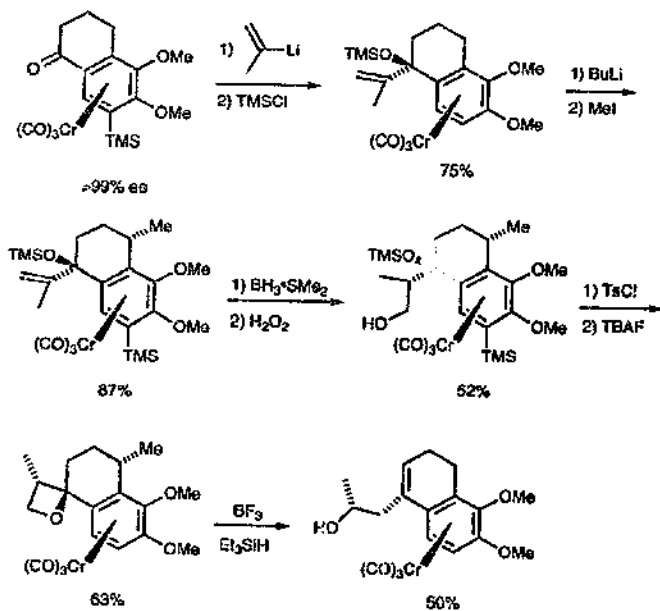
Chiral diaminoketals [486], ketals [487] (Eq. (160)), and amins [488] of benzaldehydes, and aryl sulfoxides complexed to chromium [489] were enantioselectively



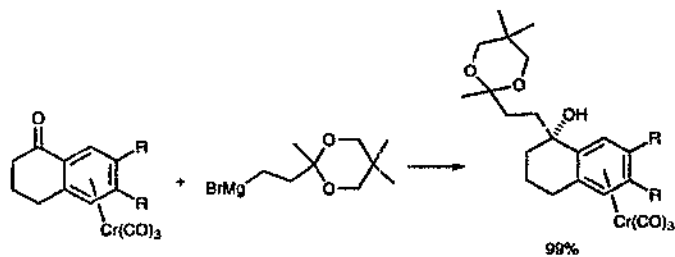
α -lithiated and reacted with electrophiles to give optically active *ortho* disubstituted arenechromium tricarbonyl complexes. Chromium complexed thiophene underwent α -lithiation alkylation [490]. A single enantiomer of the chromium tricarbonyl complex of *o*-methoxybenzaldehyde was converted to the iron carbene complex by treatment with Fp anion and TMSCl, followed by TMSOTf and styrene to give the optically active cyclopropane [491]. The same complex was methylenated with the Wittig reagent and then dialkylated by treatment with *t*-butyllithium followed by methyl iodide to give the optically active arene [492]. Other interesting chromium arene complex reactions are shown in Eq. (161) [493], Eq. (162) [494], and Eq. (163) [495].

Organolithium reagents added to substituted arenechromium tricarbonyl complexes with loss of a methoxy group (Eq. (164)) [496]. Addition of nucleophiles to cationic manganese complexes of C-2 symmetric anilines proceeded with varying degrees of stereoselectivity depending on the nucleophile [497]. Cationic manganese complexes of functionalized anisoles underwent nucleophilic attack, the regiochemistry of which depended on the face occupied by Mn (Eq. (165)) [498]. The regioselectivity of alkylation of cationic iron complexes of 2,4-dimethoxy-naphthalene depended on the nucleophile [499].

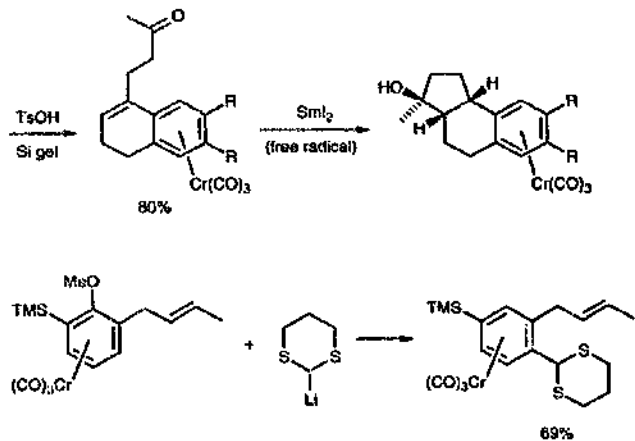
The ability of osmium +2 complexation to deconjugate aromatic systems was utilized to functionalize complexed pyrroles (Eq. (166)) [500,501] and furans (Eq. (167)) [502].



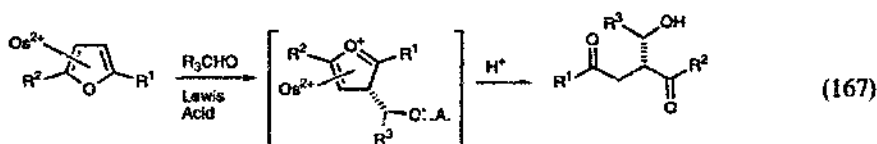
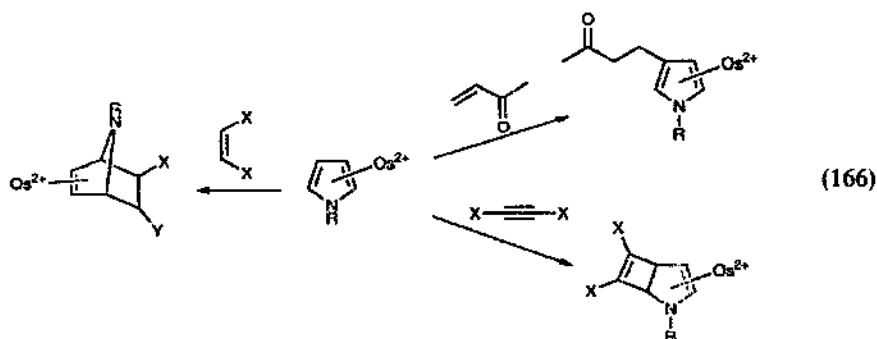
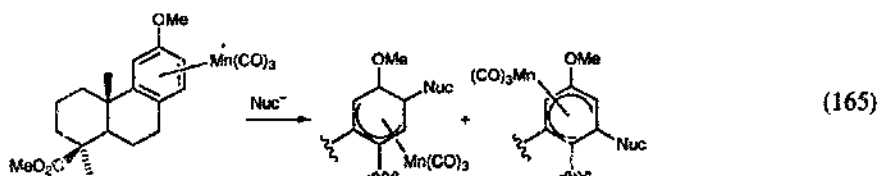
(162)



(163)

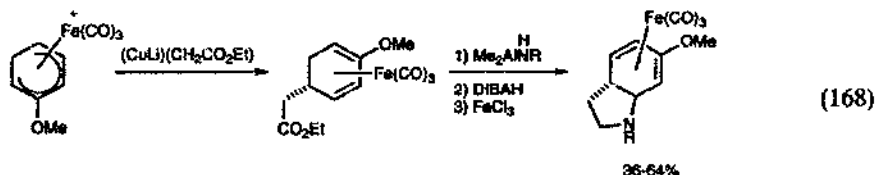


(164)

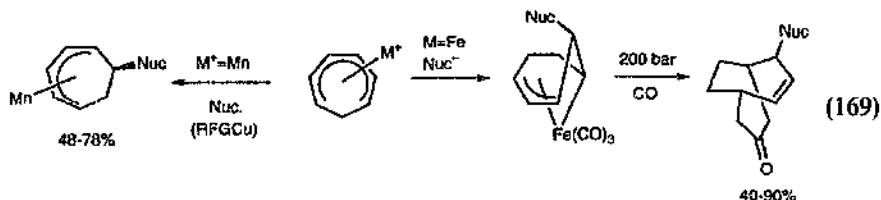


2.1.12. Alkylation of dienyl and diene complexes

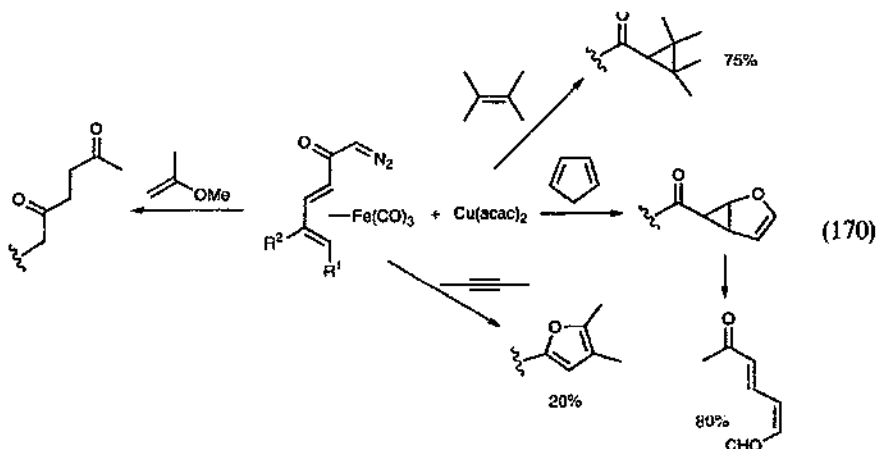
The cationic η^5 -hexadienyliron tricarbonyl complex underwent nucleophilic addition primarily at the internal position [503]. The reaction of substituted anilines with η^5 -cyclohexadienyliron tricarbonyl complexes was used yet again to make carbazoles [504–506]. Tetrahydroindoles could also be made from this type of complex (Eq. (168)) [507]. Cationic iron η^5 -cycloheptadienyl complexes underwent nucleophilic attack at an internal position [508], while the corresponding manganese complex underwent reaction at the terminal position [509] (Eq. (169)).



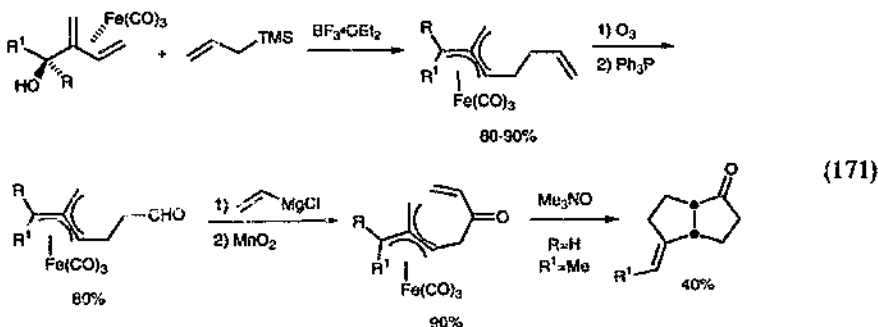
Racemic iron tricarbonyl complexes of dienoic acids were resolved via their chiral esters [510]. Sonication of α,β -unsaturated imines from chiral amino acid esters gave

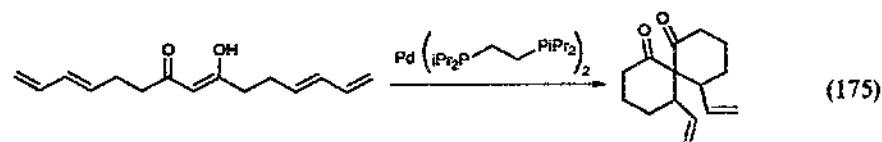
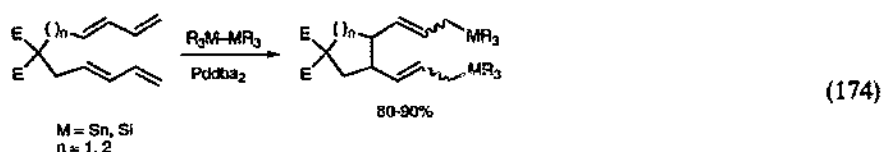
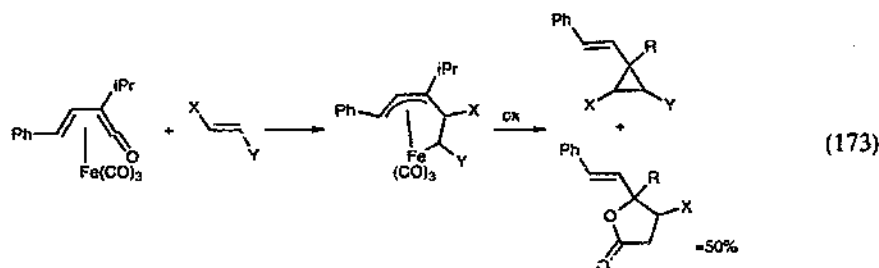
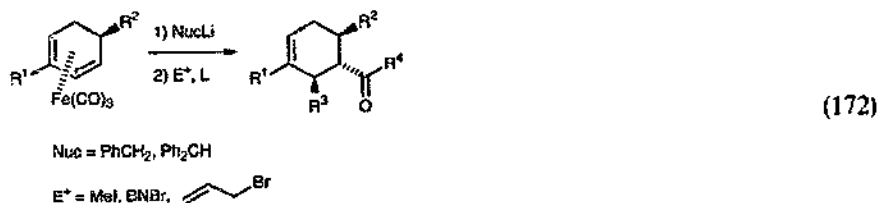


mixtures of diastereoisomeric complexes which could be equilibrated [511]. Iron complexed α -diazo-dienylketones underwent reactions typical of α -diazoketones (Eq. (170)) [512]. Iron complexed dienylketones were alkylated at the carbonyl by carbanions, and converted to trienes by $SOCl_2$ -assisted elimination of water [513].



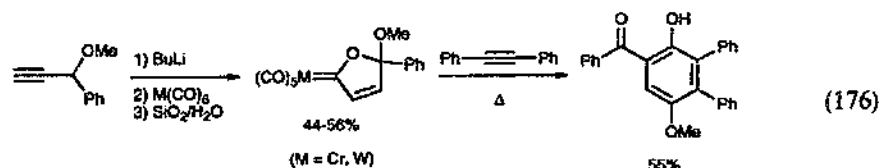
Other uses of iron diene complexes in synthesis are shown in Eq. (171) [514] and Eq. (172) [515]. Iron vinylketene complexes underwent interesting insertion chemistry (Eq. (173)) [516]. Palladium(0) catalyzed bis-metallation of tetraenes (Eq. (174)) [517], as well as their cyclization (Eq. (175)) [518].

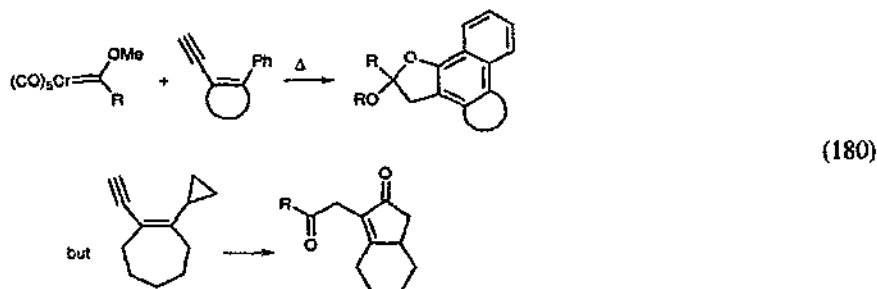




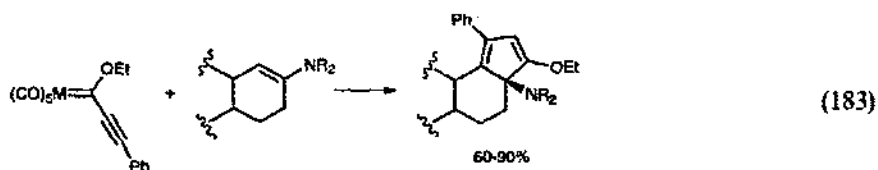
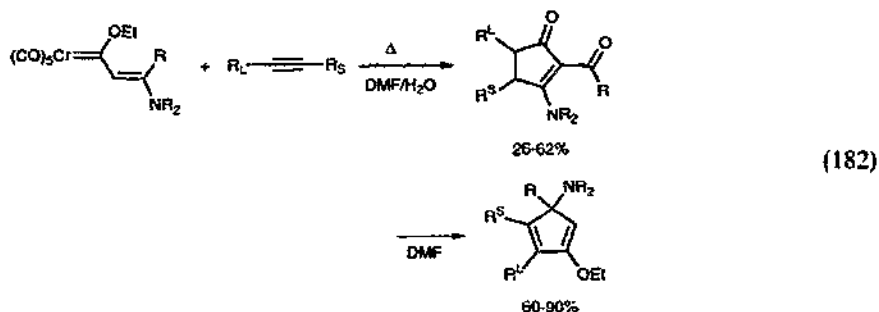
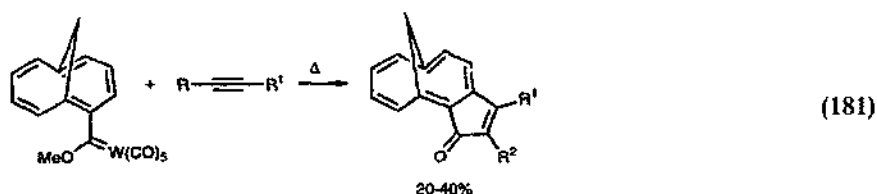
2.1.13. Metal carbene reactions

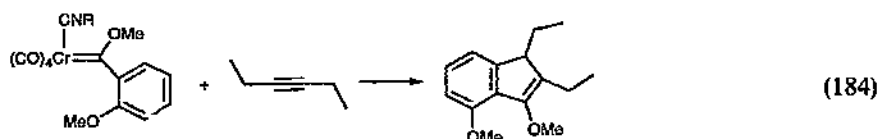
Fischer carbene complexes were synthesized directly from propargyl alcohols or ethers (Eq. (176)) [519], (Eq. (177)) [520] or by the reaction functionalized dialkylzinc reagents with $\text{Cr(CO)}_5(\text{THF})$ [521].



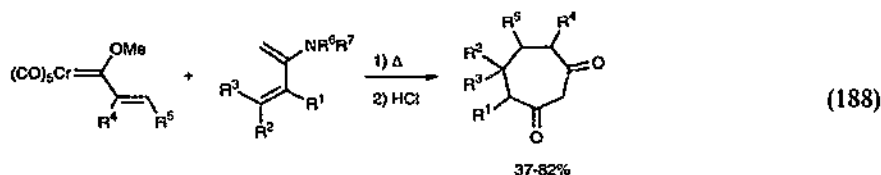
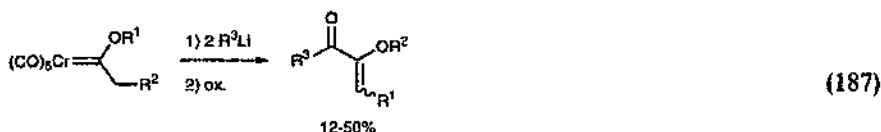
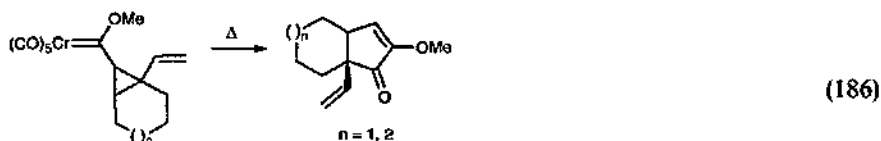
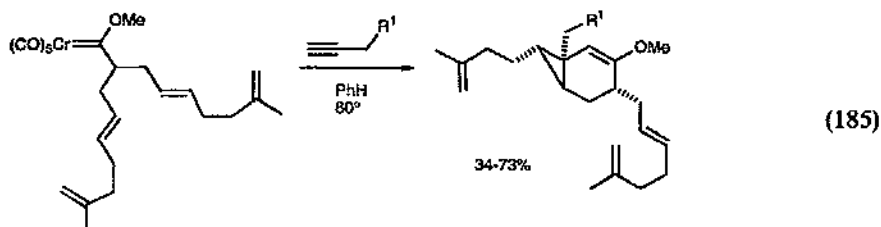


In the reaction of 1-hexyne with (phenyl)(methoxy) group 6 carbene complexes, molybdenum complexes gave indenenes as the major product with CO ligands, but naphthoquinones with phosphine ligands. Chromium complexes always favored quinones [536]. Other five-membered ring forming reactions are shown in Eq. (181) [537], Eq. (182) [538-540], Eq. (183) [541] and Eq. (184) [542].

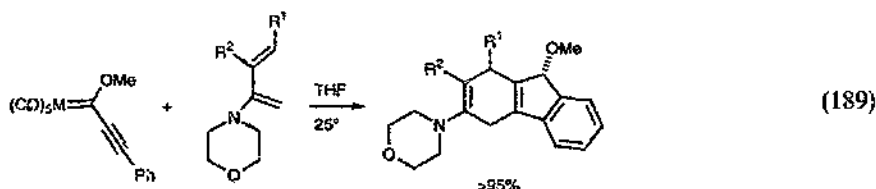




Other more exotic reactions of carbene complexes are shown in Eq. (185) [543], Eq. (186) [544], Eq. (187) [545], Eq. (188) [546] and Eq. (189) [547].



Carbene complexes having pyrrole as the heteroatom substituent behaved more like alkoxycarbene complexes than aminocarbene complexes, cyclopropanating olefins, forming quinones with alkynes, and cyclobutanones and β -lactams under photolytic conditions [548]. Unsaturated carbene complexes bearing a β -(2-pyrrole)



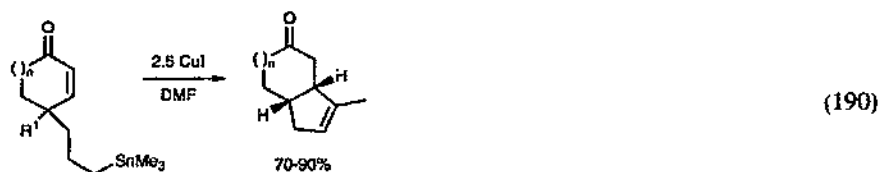
group gave cyclopentanes when reacted with activated olefins in cyclohexane, but cyclopropanes when acetonitrile was used as solvent [549]. Optically active 4,4-disubstituted butenolides were made by photolysis of chromium carbene complexes in the presence of optically active ene carbamates, followed by Baeyer-Villiger oxidation and elimination. Reactions of these butenolides were studied [550].

2.1.14. Alkylation of metal acyl enolates

There were no synthetically significant examples of this class of reaction this year.

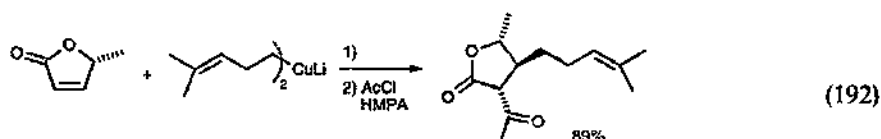
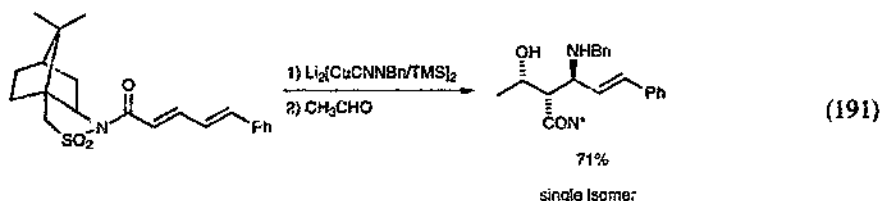
2.2. Conjugate addition

A new mechanism to rationalize the effects of added trimethylsilyl chloride on the conjugate additions of cuprates to enones has been advanced [551]. By appropriate choice of cuprate, either diastereoisomer could be made from the conjugate alkylation of allyl esters of acrylates, followed by Claisen rearrangement [552]. The conjugate addition of functionalized organozinc reagents to conjugated enones was catalyzed by $\text{Me}_2\text{CuCNLi}_2$ in the presence of excess TMSCl [553]. $(\text{Me}_3\text{SnCH}=\text{CH})\text{Cu}(\text{R})\text{CNLi}_2$ transferred the R group to enones, while $(\text{Me}_3\text{SnCHCH})_2\text{CuCNLi}_2$ transferred the vinyltin group [554]. Copper(I) salts promoted the conjugate addition of α -lithiated hydrazones to enones [555], as well as the conjugate addition of α -zirconated alkoxyboranes [556]. Copper(I) iodide promoted the intramolecular conjugate addition shown in Eq. (190) [557].

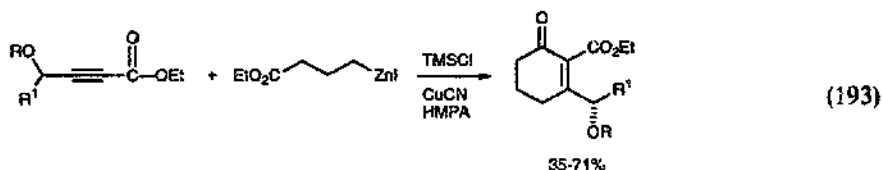


An NMR investigation of copper catalyzed conjugate addition in the presence of 4-phenyloxazolidinone ligands has been published [558]. Conjugate additions to cyclic enones in the presence of chiral diamines [559], and proline derived aminophosphine ligand [560,561] were efficient. In the latter case, the counterion reversed stereoselectivity. Conjugate addition of cuprates to chiral camphor sulfam derivatives of conjugated acids proved an efficient way to induce asymmetry [562–564].

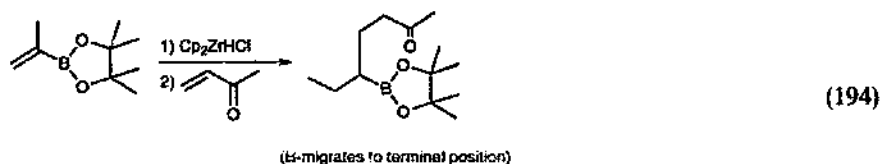
(Eq. (191)) [565]. Conjugate addition to chiral substrates was also efficient (Eq. (192)) [566,567].

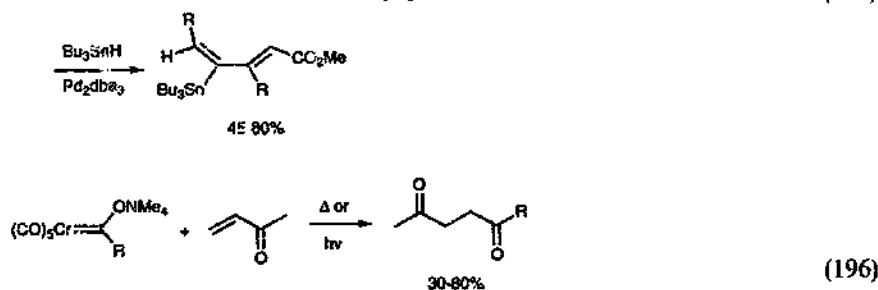
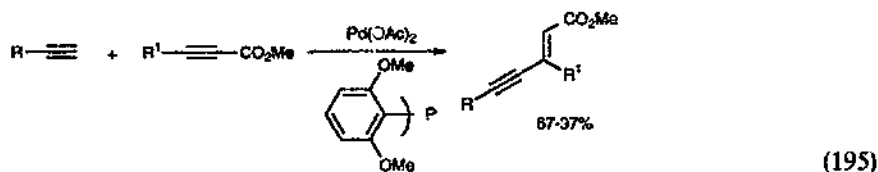


Cuprates Michael added to propiolate esters [568] (Eq. (193)) [569], and replaced the *cis* chloro group of β -dichloroenones [570]. Organozinc halides, including sterically hindered ones (e.g., adamantyl) Michael added to enones [571].



Nickel(II) acetylacetonate catalyzed the β -alkylation of sterically hindered enones [572] especially steroidal systems [573]. Other less common 1,4-additions are seen in Eq. (194) [574], Eq. (195) [575] and Eq. (196) [576]. Palladium(II) catalyzed the β -arylation of enones by tetraaryl borates [577].





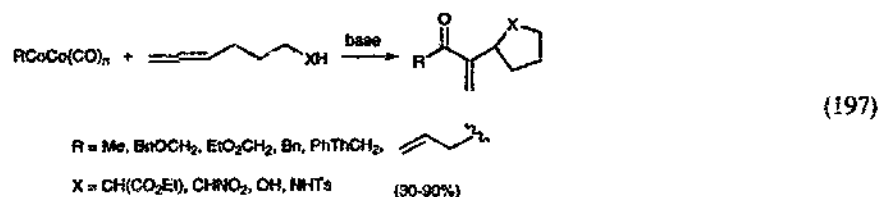
2.3. Acylation reactions (excluding hydroformylation)

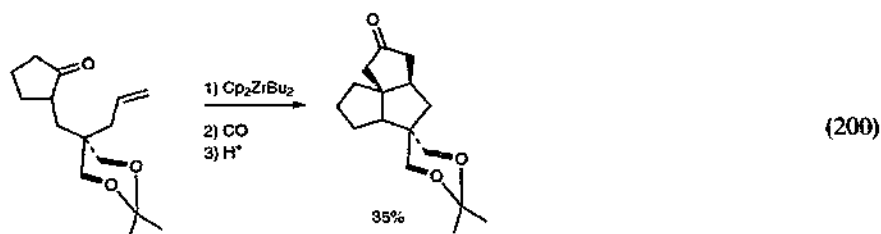
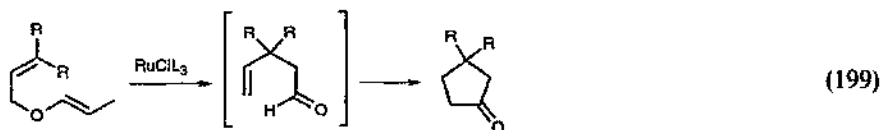
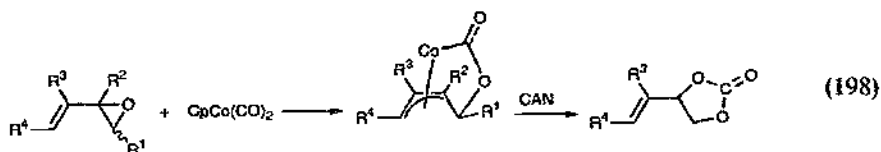
For carbonylations that form heterocycles, see Section 3.5.

2.3.1. Carbonylation of alkenes and arenes

Palladium(0) catalyzed the carbonylation of aryl and vinyl boronates [578], vinyl triflates [579], and vinylmercuric chlorides from carbomercuration of alkynes [580]. Dihydrofurans were aroylated by reaction with aryl halides, carbon monoxide and palladium(0) catalysts [581]. Aromatic hydrocarbons were directly carbonylated by palladium(II) under oxidizing conditions [582]. Anionic acylnickel carbonyl complexes acylated electron deficient olefins [583]. Acyl cobalt complexes cycloalkylcarbonylated allenes (Eq. (197)) [584] and allyl epoxides (Eq. (198)) [585]. Terminal alkenes were carbonylatatively dimerized to symmetrical ketones by reduced cobalt species [586]. Ene allenes cyclocarbonylated with rhodium(I) catalyst to give methylene cyclopentenone [587]. The full details of the rhodium(I) catalyzed α -acylation of styrenes by acid anhydrides have appeared [588]. Ruthenium(II) complexes catalyzed the Claisen rearrangement followed by hydroacylation (Eq. (199)) [589]. Zirconocene cyclocarbonylated olefinic ketones (Eq. (200)) [590].

Iron tricarbonyl complexed conjugated enones were converted to vinyl ketene





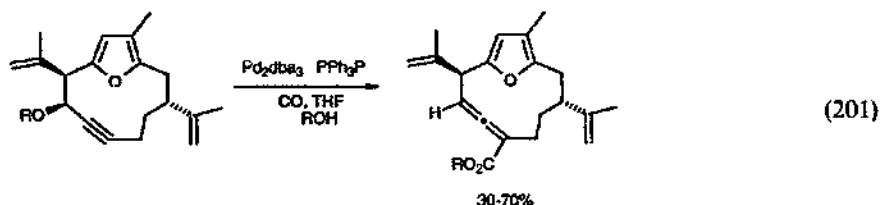
complexes by treatment with methyllithium and carbon monoxide [591]. α -Diazoiron dienes underwent thermal cyclocarbonylation [592]. Asymmetric hydroformylation of alkenes has been reviewed (101 references) [593], (77 references) [594].

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

Terminal alkynes were acylated at the terminal position by aryl halides and carbon monoxide in the presence of palladium(0) catalyst [595]. Carboxylic acids added across alkynes to give vinyl esters in the presence of $[\text{PdMo}_3\text{S}_4(\text{TaCN})_4\text{Cl}]\text{PF}_6$ [596]. Palladium(0) catalyzed the carbonylative thiolation of terminal alkynes by aryl thiols to give β -thio acroleins [597]. Palladium acetate catalyzed the hydrocarboxylation of 2-alkynyl naphthalenes to give high turnovers for branched unsaturated acids [598]. Rhodium(I) complexes catalyzed the hydroformylation of internal alkynes [599]. Palladium(0) catalyzed the alkyne carbonylation reaction shown in Eq. (201) [600].

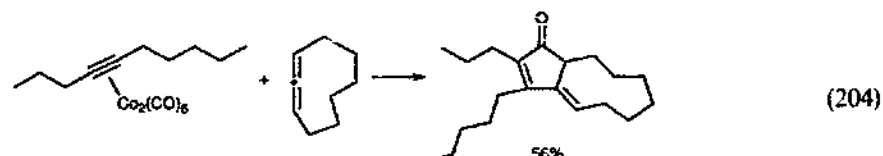
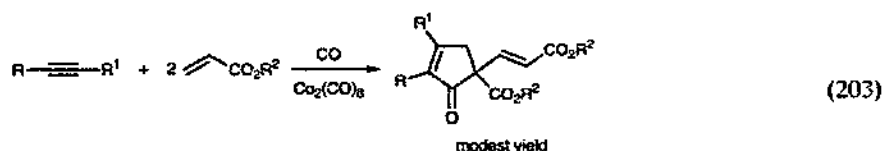
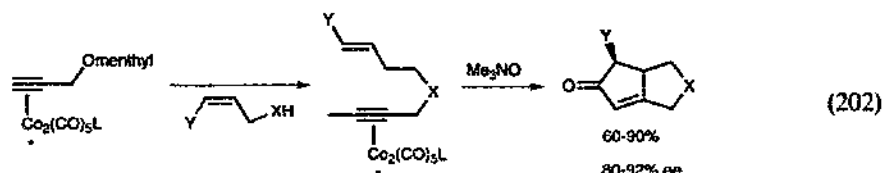
Iron carbonyl promoted the cyclocarbonylation of diynes to iron-complexed cyclopentadienones [601,602] while $\text{Rh}_4(\text{CO})_{12}$ cyclosilylcarbonylated diynes to α -silylcyclopentenones [603]. Simple terminal alkynes were silylformylated to give β -silyl unsaturated aldehydes [604]. This process occurred in an intramolecular fashion as well [605]. Both iron carbonyl and molybdenum carbonyl cyclocarbonylated yne allenes to give cyclopentenones [606,607].

The Pauson Khand reaction between norbornene and cobalt complexed ethylpro-

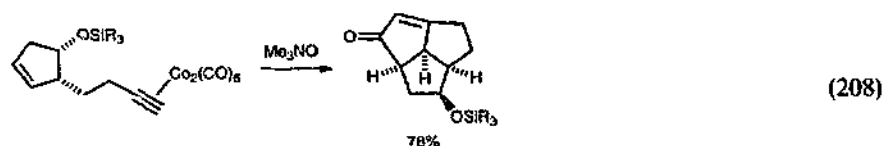
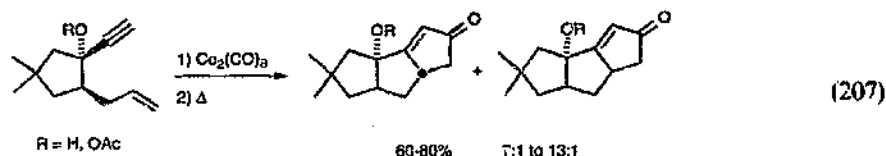
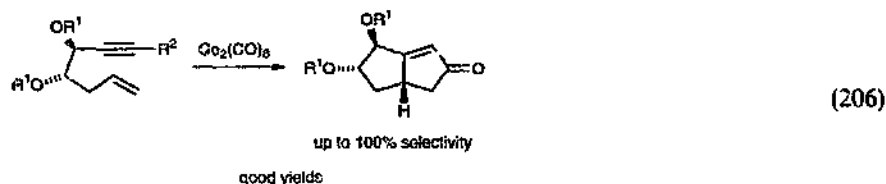
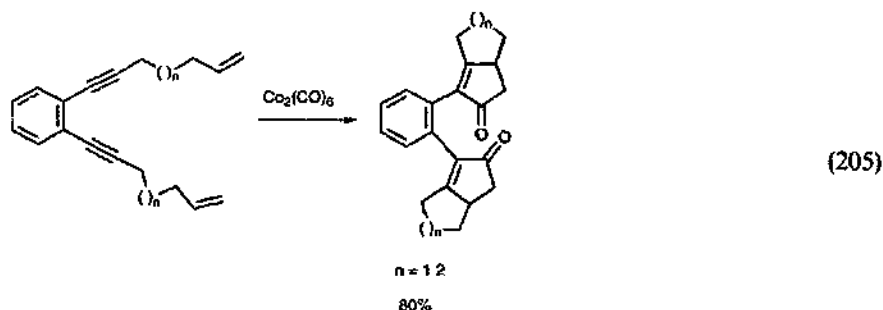


prionate [608] and propargyl alcohols [609] has been reported. Conditions for use of ethylene as the olefin in the Pauson Khand reaction was optimized [610].

Asymmetric induction into the Pauson Khand reaction has been achieved with modest selectivity by using chiral alkoxyalkynes [611] or alkynyl esters [612]. By making and separating diastereoisomeric cobalt complexes of propargyl menthyl esters, high de was obtained in the Pauson Khand reaction (Eq. (202)) [613]. Similarly, chiral phosphine complexes of cobalt alkynes resulted in good asymmetric induction in the Pauson Khand reaction [614]. Cobalt carbonyl promoted the cyclocarbonylation of alkynes with electron deficient olefins (Eq. (203)) [615] and allenes (Eq. (204)) [616].



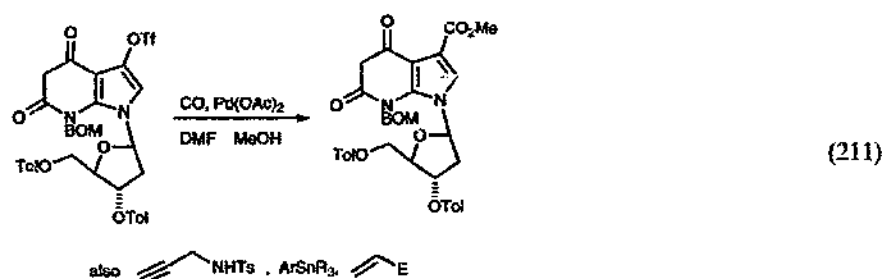
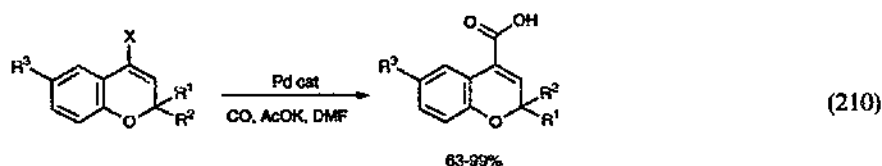
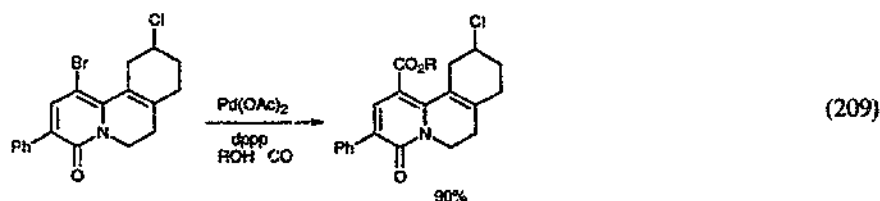
Molecular mechanics calculations for stereoselectivity with the intramolecular Pauson Khand reaction were carried out [617]. Useful intramolecular Pauson Khand reactions are shown in Eq. (205) [618], Eq. (206) [619], Eq. (207) [620], and Eq. (208) [621].



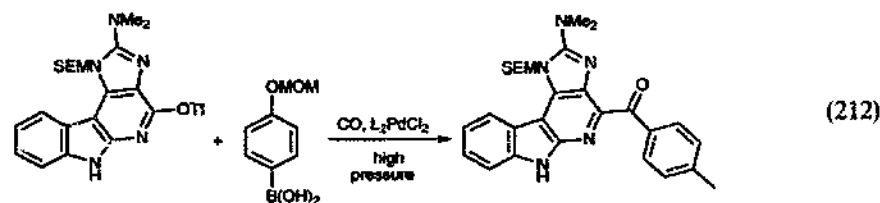
2.3.3. Carbonylation of halides and triflates

Palladium catalyzed double and single carbonylation of aryl halides and allylic compounds has been reviewed (>66 references) [622]. Palladium catalyzed the carbonylation of bromopyridones (Eq. (209)) [623], the acylation of halides by alkyltins using ^{11}CO [624] and the carboxylation of halogenated benzopyrans (Eq. (210)) [625]. Aryl halides, heteroaryl halides and vinyl halides were carbonylated to give esters by cobalt complex reducing agents [626]. Iodonucleosides were carbonylated by treatment with alkyltins, carbon monoxide and palladium catalyst [627].

Aryl triflates were converted to aryl methyl ketones by reaction with tetramethyltin, carbon monoxide, and a palladium catalyst [628]. Pyrrole triflates were alkoxy-carbonylated using palladium catalysts [629,630], (Eq. (211)) [631]. Heteroaryl



triflates were carbonylatively coupled to aryl boronates under palladium catalysis (Eq. (212)) [632]. Palladium(0) catalyzed the carbonylative coupling of allyl halides to functionalized organozinc reagents [633]. Aryl antimonates were carbonylated by palladium catalysts under oxidizing conditions [634].



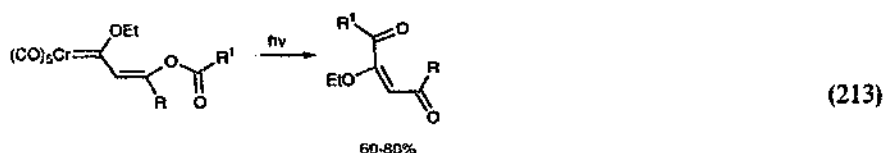
Cobalt bromide catalyzed the carbonylative coupling of functionalized organozinc reagents to give symmetrical ketones [635]. Nickel carbonyl carbonylated vinyl halides to give esters [636].

2.3.4. Carbonylation of nitrogen compounds

Amines were carbonylated to carbamates using palladium catalysts [637].

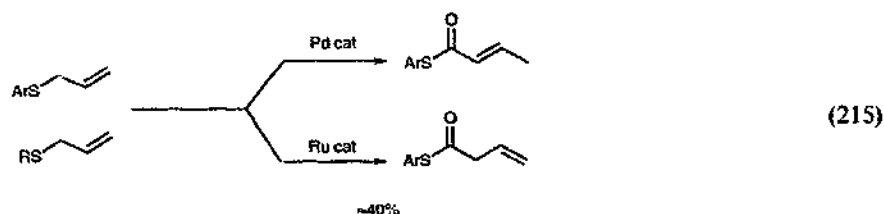
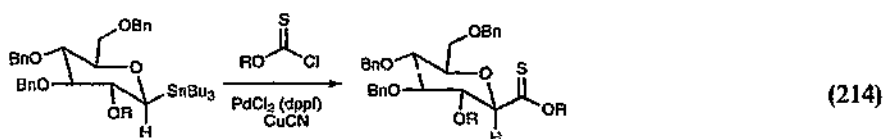
2.3.5. Carbonylation of oxygen compounds

Thioketals were converted σ -thioesters by treatment with high pressures of carbon monoxide in the presence of rhodium(I) catalysts [638]. β -Acetoxychromium carbene complexes were converted to diketones by photolysis (Eq. (213)) [639].



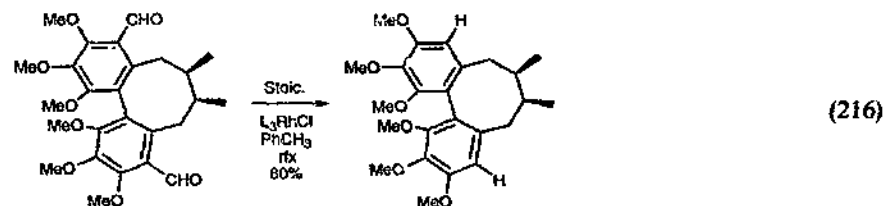
2.3.6. Miscellaneous carbonylations

Miscellaneous carbonylations are shown in Eq. (214) [640] and Eq. (215) [641].



2.3.7. Decarbonylations

The sole interesting decarbonylation is shown in Eq. (216) [642].



2.3.8. Reactions of carbon dioxide

“Utilization of carbon dioxide in organic synthesis and polymer synthesis by the transition metal catalyzed carbon dioxide fixation into unsaturated hydrocarbons” was the subject of a review (39 references) [643]. Nickel cyclams under electrochemical reduction converted epoxides into cyclic carbonates [644].

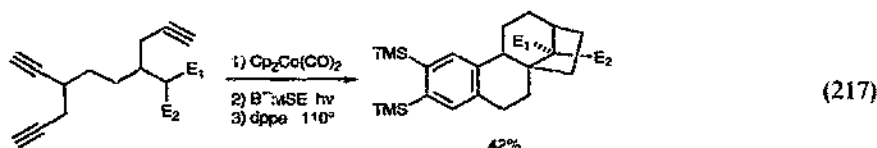
2.4. Oligomerization (including cyclotrimerization of alkynes and metathesis polymerization)

Papers dealing with the oligomerization of ethylene by cationic phenanthroline methylpalladium complexes [645], the roles of Lewis Acids in Ziegler-Natta alkene insertions — competition of Ti–C vs. Al–C alkene insertions [646], and a review entitled “Stereospecific olefin polymerization with chiral metallocene catalysts” (235 references) [647] have appeared. Cationic chelating diamine metal complexes catalyzed the dimerization of terminal alkenes to give predominantly branched polymers [648].

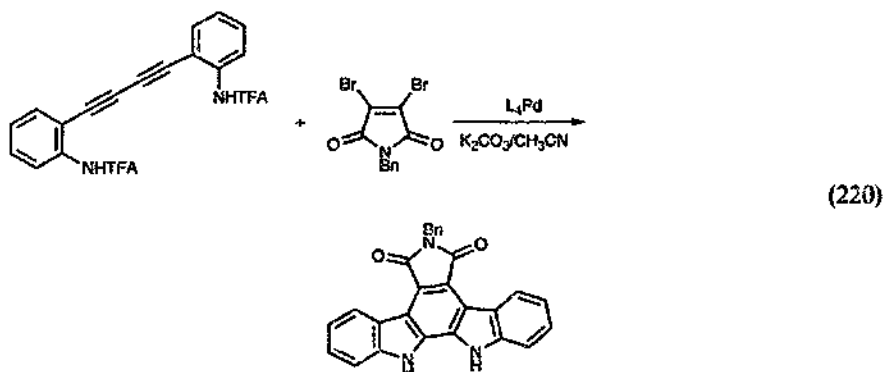
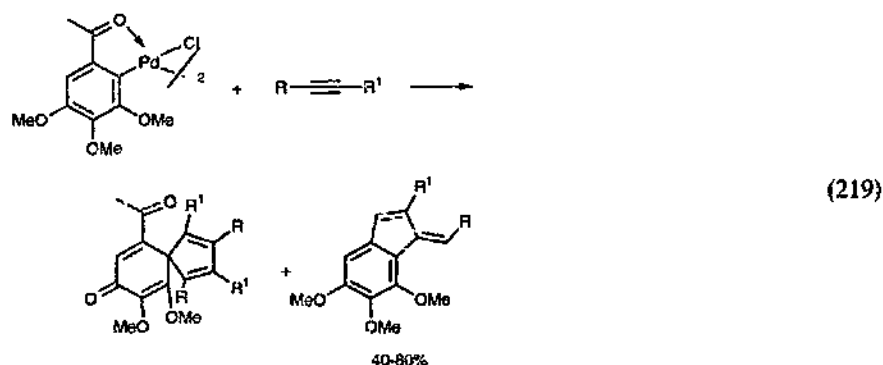
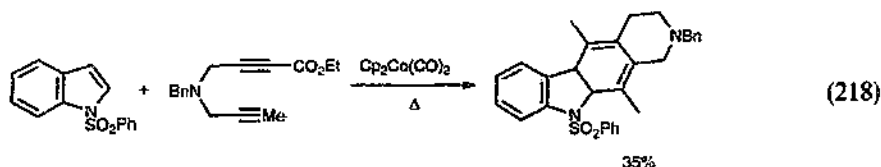
Papers dealing with palladium catalyzed alternating copolymerization of alkenes and carbon monoxide [649,650], and the chiral polymerization of terminal alkenes with carbon monoxide to give isotactic optically active 1,4- and 1,5-polyketones [651,652] have appeared.

A paper dealing with polymerizations of α -olefins and butadienes and cyclotrimerizations of 1-alkynes by chelating phenoxide titanium and zirconium species has appeared [653]. Palladium(0) complexes catalyzed the cyclodimerization of butadienes to 1-methylene-2-vinylcyclopentane [654]. Chiral nickel(0) complexes catalyzed the cyclodimerization of butadiene with methyl sorbate to give cyclooctadienes with low ee [655]. Palladium(0) catalyzed the silyl-dimerization of alkynes with trichlorosilane to give silylbutadienes [656].

Cobalt catalyzed cyclotrimerizations are shown in Eq. (217) [657] and Eq. (218) [658]. Cobalt cocyclotrimerized chiral nitriles with acetylene to give pyridines [659]. *Ortho* palladated acetophenones reacted with alkynes (Eq. (219)) [660]. Palladium(0) catalyzed the reaction of diynes with dibromophthalimides (Eq. (220)) [661]. Nickel aryne complexes reacted with alkynes to give naphthalenes [662]. Tantalum pentachloride cocyclized diynes with alkynes to give arenes under reductive conditions [663]. Zirconocene cyclooligomerized diynes (Eq. (221)) [664].



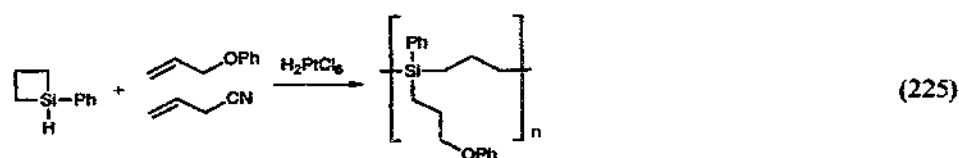
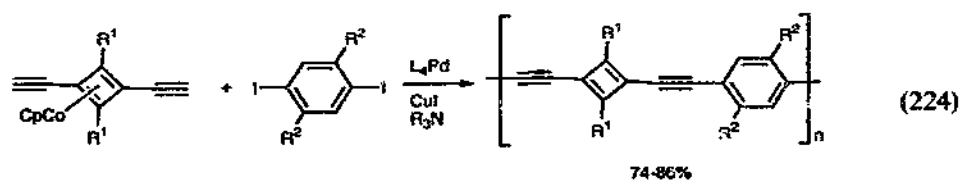
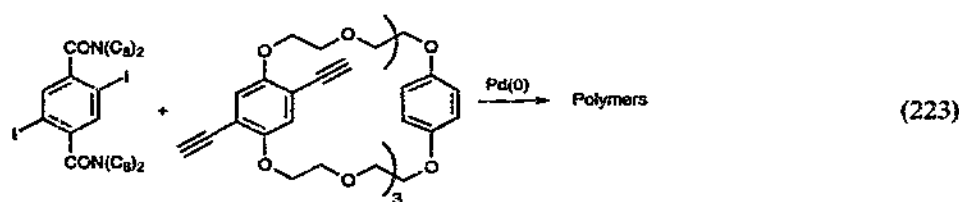
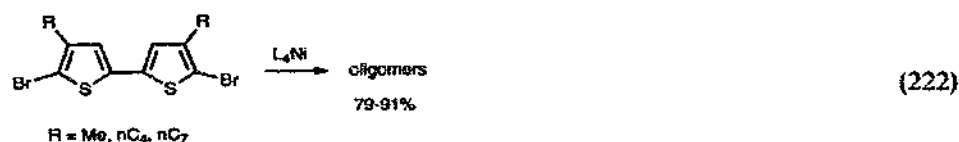
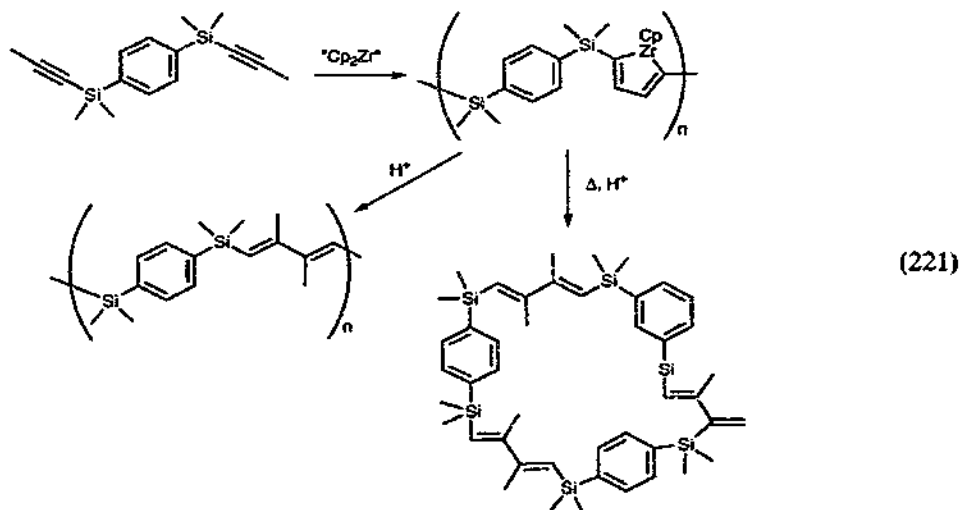
3-Substituted 2,5-bis-mercuriothiophenes were oligomerized (M_w 700–18 000) by treatment with palladium chloride and copper [665]. Nickel(0) complexes oligomerized dibromo-bis-heterocycles (Eq. (222)) [666]. 2,5-Bis-tributylstannyl thiophene cooligomerized with 1,4-diodo-2,5-bis-octanyl benzene in the presence of

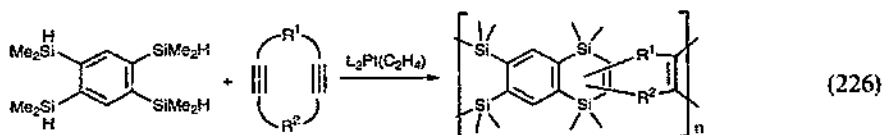


palladium(0) catalysts [667]. Other useful oligomerizations are seen in Eq. (223) [668] and Eq. (224) [669].

Treatment of areneRuCl₂ phosphine complexes with trimethylsilyldiazomethane gave a ROMP catalyst for norbornene and cyclooctene which tolerated esters and epoxides [670]. Chloroplatinic acid catalyzed oligomerization of silanes with unsaturated organics (Eq. (225)) [671] and (Eq. (226)) [672].

Transition metal synthesis of silicon polymers has been reviewed (53 references) [673], as has molecular trees: from synthesis towards applications (236 references) [674].

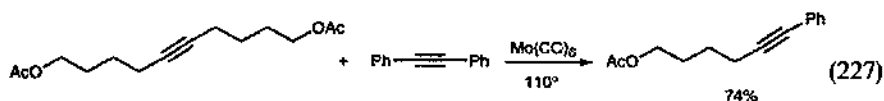




2.5. Rearrangements

2.5.1. Metathesis (metathesis processes which produce heterocycles are found in Section 3.5)

The complex $\text{RuCl}_2(\text{PCy}_3)_2(\text{CHAr})$ was an efficient metathesis catalyst for acyclic allenes [675]. Molybdenum alkylidene complexes catalyzed the cross-metathesis of terminal alkenes with acrylonitrile to give alkylated acrylonitriles [676]. Cyclobutenes underwent selective ring opening metathesis with terminal alkenes [677]. Unsaturated morpholinocarbene molybdenum complexes metathesized with electron poor olefins [678]. Dienes were cyclized by metathesis with $\text{W}(\text{O})(\text{ArO})_2\text{Cl}_2/\text{Et}_4\text{Pb}$ catalysts [679] (tolerates functional groups), $(\text{Cy}_3\text{P})_2\text{RuCl}_2(\text{CHCHCPh}_2)$ [680] and MeReO_3 [681]. *Para*-propynyl phenol metathesized to di-*p*-hydroxyphenyl acetylene in the presence of $\text{Mo}(\text{CO})_6$ [682]. The same catalyst cross-metathesized diphenyl acetylene with internal alkynes (Eq. (227)) [683].

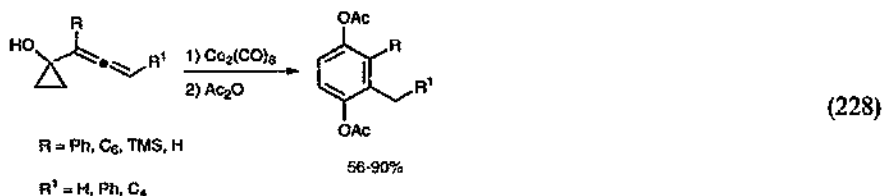


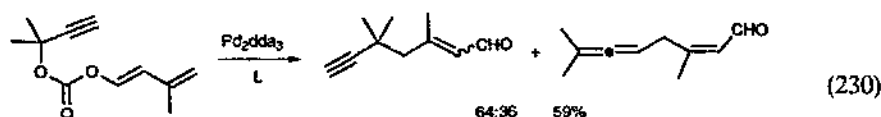
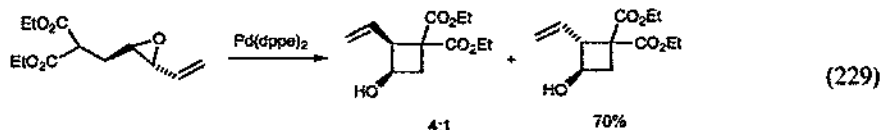
2.5.2. Olefin cycloisomerization

These are shown in Eq. (228) [684], Eq. (229) [685] and Eq. (230) [686].

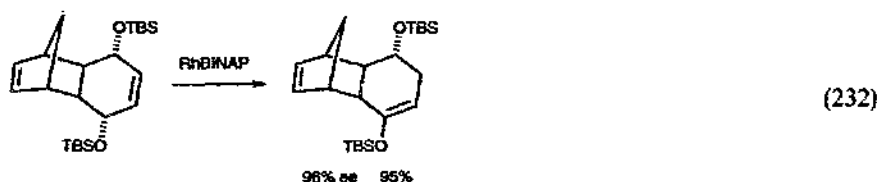
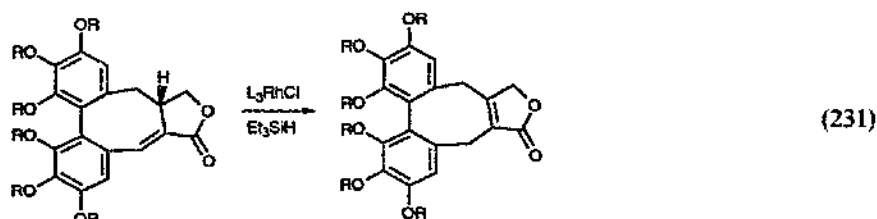
2.5.3. Rearrangement of allylic and propargylic systems

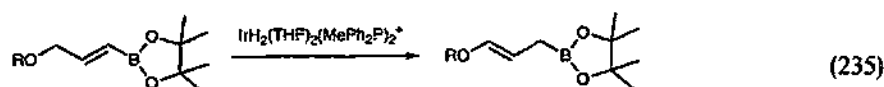
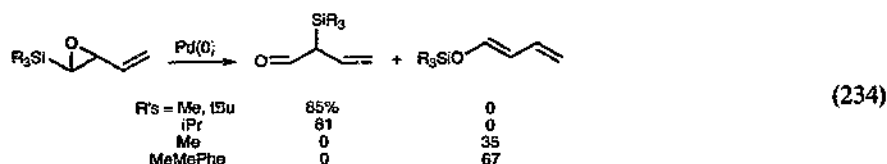
$\text{HCo}(\text{CO})_4$ isomerized allyl ethers to vinyl ethers [687]. Monoalkyl ketals of 1,4-dihydroxy-2-butene rearrange to the 1-butene when treated with nickel(II) phosphine complexes and isopropyl Grignard reagents [688]. Propargyl alcohols





rearranged to α,β -unsaturated esters when treated with rhodium(I) catalysts [689] or ruthenium(II) catalysts [690]. Palladium acetate rearranged 1,4-dihydropyrroles to 1,2-dihydropyrroles [691]. Wilkinson's catalyst was used to do the isomerization in Eq. (231) [692]. Palladium(0) catalyzed the rearrangement of alkynes to 1,3-dienes [693]. 1,4-Disiloxy-2-butenes were desymmetrized by RhBINAP catalysts (Eq. (232)) [694]. The stereochemical features of palladium(II) catalyzed Claisen rearrangement were studied [695]. Ruthenium(II) complexes catalyzed the allylic transposition of allyl alcohols (Eq. (233)) [696]. Other interesting isomerizations are seen in Eq. (234) [697] and Eq. (235) [698].



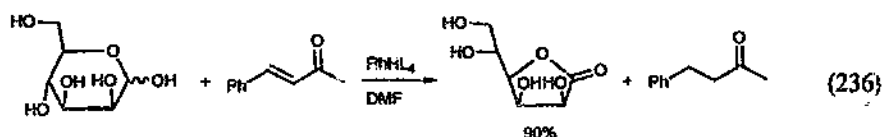


2.5.4. Skeletal rearrangements

There were none.

2.5.5. Miscellaneous rearrangements

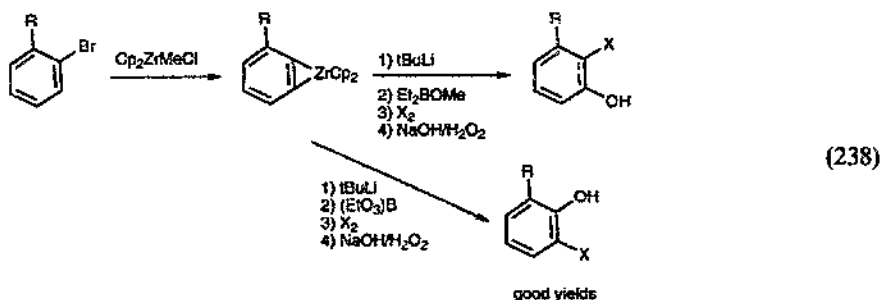
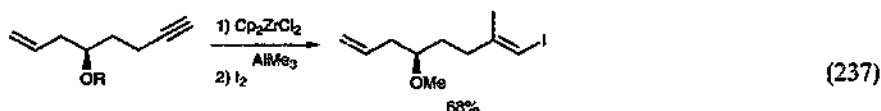
Terminal alkenes isomerized to internal alkenes when treated with $\text{Na}_2\text{Fe}(\text{CO})_4$ and copper(I) chloride or dibromoethane [699]. Rhodium hydride complexes catalyzed the contraction of glycopyranoses (Eq. (236)) [700]. Treatment of the chromium tricarbonyl complex of 2-methyl-5-trimethylsilylthiophene with *t*-butyllithium gave the complex of 2-methyl-3-trimethylsilylthiophene [701]. Palladium(0) complexes catalyzed the isomerization of methoxy groups along a polysilane backbone via silylenes [702].



3. Functional group preparation

3.1. Halides

Alkynes were converted into terminal vinyl iodides by zirconium catalyzed carboalumination followed by cleavage with iodine [703], (Eq. (237)) [704]. Hydrozirconation followed by NBS cleavage produced vinyl bromides [705]. Zirconabenzynes could be halogenated at either position (Eq. (238)) [706]. Vinyl triflates were converted to vinyl iodides by treatment with hexamethylditin in the presence of palladium catalysts, followed by *N*-iodosuccinimide [707].

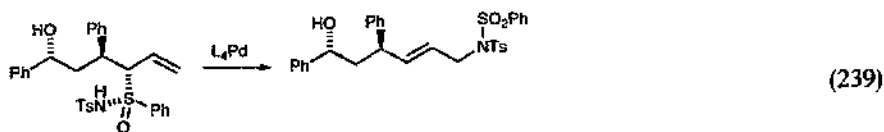


3.2. Amides, nitriles

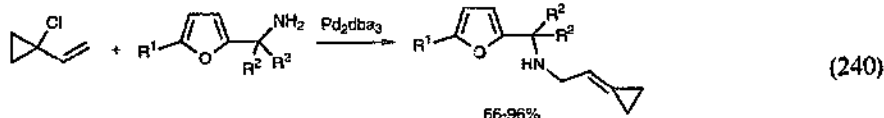
Treatment of *o*-allylcarbamates with palladium(0), tin hydride and acetic anhydride gave the free amine which was immediately acylated [708]. Cyclams were coordinated through three of their nitrogens to $\text{Mo}(\text{CO})_6$ and the fourth was cleanly acylated by acid halides [709]. $\text{Ru}_3(\text{CO})_{12}$ catalyzed the addition of terminal alkynes to acetanilides to give *N*-vinyl acetanilides [710]. α -Diazophosphonate esters inserted into the N–H bond of amides to give α -amidophosphonate esters [711]. Palladium catalyzed the reaction shown in Eq. (239) [712].

3.3. Amines, alcohols

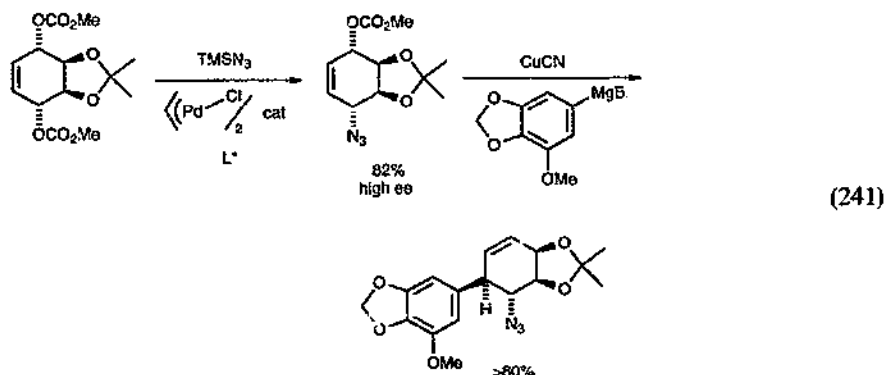
Palladium(0) complexes catalyzed the allylic amination of α -phosphonoallyl carbonates by hydroxylamines [713], the allylic amination of allyl alcohols in the presence of SnCl_2 [714], the asymmetric allylic amination of



1-acetoxy-1,3-diphenylpropene by phthalimide (up to 96% ee) [715], and by benzyl amine (66% ee) [716]. Carbocyclic nucleoside analogs were synthesized by palladium catalyzed amination of 3-acetoxycyclopentenones by pyrimidine bases (no yields reported) [717]. 1-Chloro-1-ethenylcyclopropane underwent allylic amination with transposition (Eq. (240)) [718]

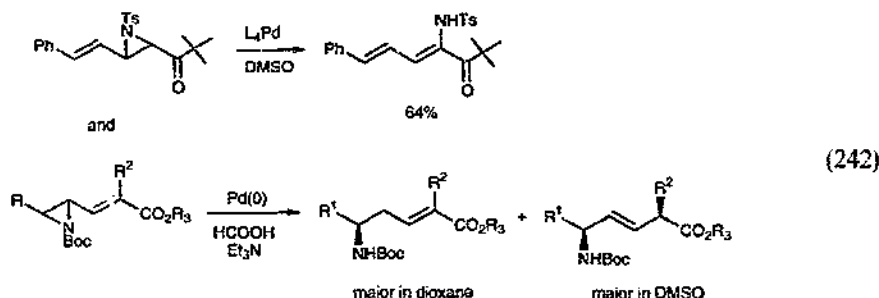


C-2-symmetric 1,4-acetoxy cyclohex-2-enes were azidated with high ee using chiral palladium catalysts [719], Eq. (241) [720]. Cyclic epoxides were asymmetrically opened with azide using chromium salen catalysts [721]. Diene monoaziridines were both oxidatively [722] and reductively [723] opened under palladium catalysis (Eq. (242)).

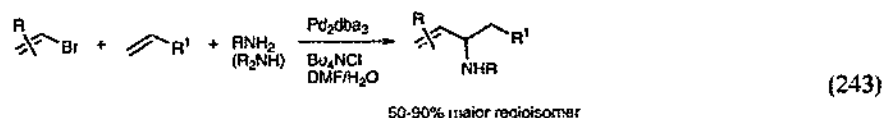


Palladium catalyzed amination of aryl halides was the subject of a short review (11 references) [724]. Efficient palladium catalyzed aminations of a very wide range of aryl bromides have been developed, one based on the use of *t*-butoxide [725] and one on the use of Li bis-trimethylsilyl amide [726]. Palladium(0) catalyzed the amination of iron-iodocyclobutadiene complexes [727].

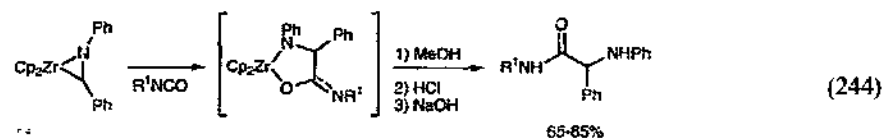
The full details on the reaction of alkenes, vinyl bromides and amines with



palladium(0) catalysts in aqueous DMF (water improved yield) to give allyl amines have been published (Eq. (243)) [728]. *t*-Butyl acetylene, 1,3-dichloro-1-propene and ethyl amine reacted with Pd/Cu catalysts to give amination of the allylic position and alkynylation of the vinyl position [729]. Terminal allenes reacted with secondary amines to give allyl amines plus 2-aminomethyl butadienes, incorporating two equivalents of the allene [730]. Diallylamines were deallylated in a stepwise fashion using palladium(0) catalyst and *o*-sulfhydryl benzoic acid [731,732].

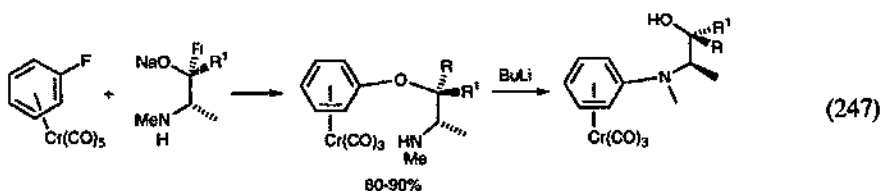
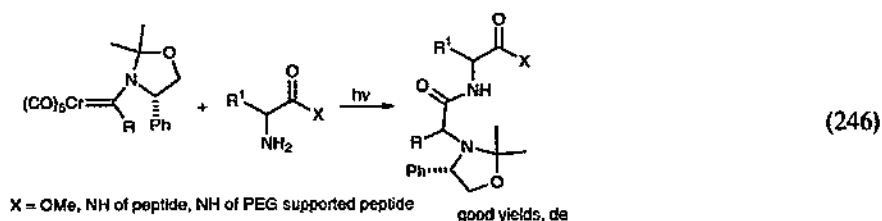
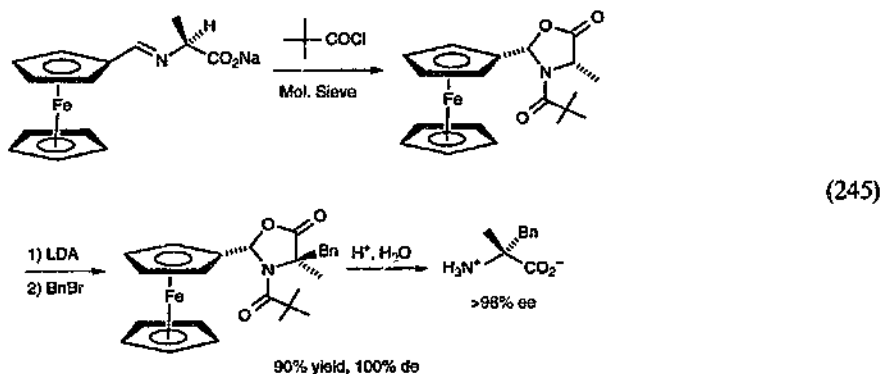


Cobalt complexed propargyl alcohols were converted to propargyl amines via cobalt stabilized propargyl cations [733]. Aryl azides, aroyl azides, and aryl sulfonyl azides were reduced to the corresponding NH_2 species by MoS_4^{2-} [734]. Imines were reduced to amines by Cp_2MoH_2 [735], and by iridium(I) BINAP catalyst systems with high ee [736]. Olefins were converted to terminal amines by hydrozirconation followed by cleavage with $\text{ArSO}_2\text{ONH}_2$ [737]. α -Aminoamides were synthesized from zirconium-imine complexes and isocyanates (Eq. (244)) [738]. Iron complexed vinyl ketenes were converted to complexed ketenimines by treatment with phosphoramides and butyllithium [739].



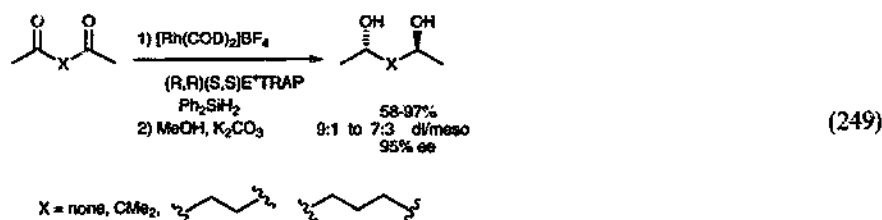
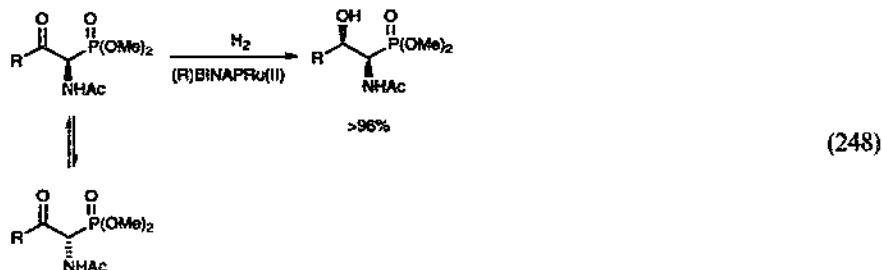
Chiral ferrocenyl aldehydes were used to synthesize optically active amino acids (Eq. (245)) [740]. Chromium aminocarbene complexes were used to introduce amino acid residues into peptides both on soluble PEG supports [741], and in solution

[742] (Eq. (246)). Fluorobenzene chromium tricarbonyl underwent reaction with deprotonated aminoalcohols to give complexed phenols which rearranged to complexed anilines when treated with butyllithium (Eq. (247)) [743].

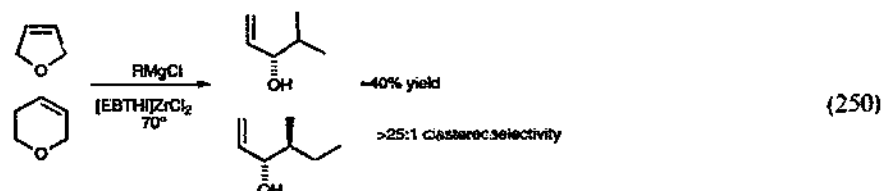


Asymmetric catalytic transfer reduction of ketones by isopropanol has been achieved in excellent ee using chiral ruthenium(II) catalysts [744,745]. Nickel(II) phosphine complexes catalyzed a similar reduction [746]. Styrene was asymmetrically hydroborated using rhodium(I) catalysts and chiral ferrocenylphosphine amines. Up to 98% ee was achieved, but with only a 68% yield and a 3:2 mixture of regioisomers [747]. Chiral aminophosphinoboranes catalyzed the asymmetric reduction of chromium complexed *p*-chlorobenzophenone with 90% yield and 92% ee. Cobalt complexed propargyl alcohols were asymmetrically reduced with similar efficiency [748]. Epoxides were reductively opened (attack at the less substituted position) by ammonium formate and Pd/C [749].

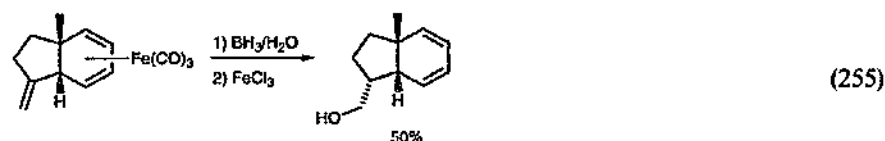
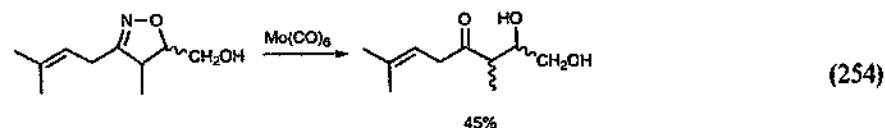
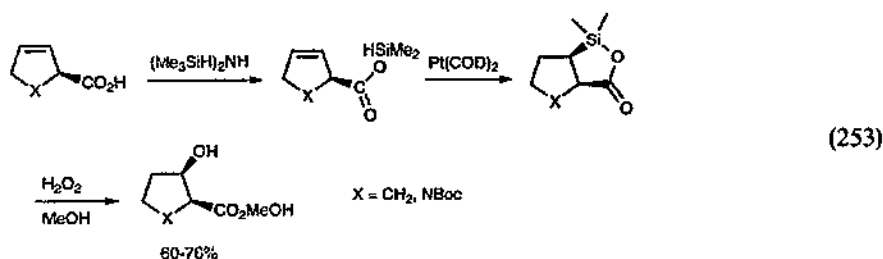
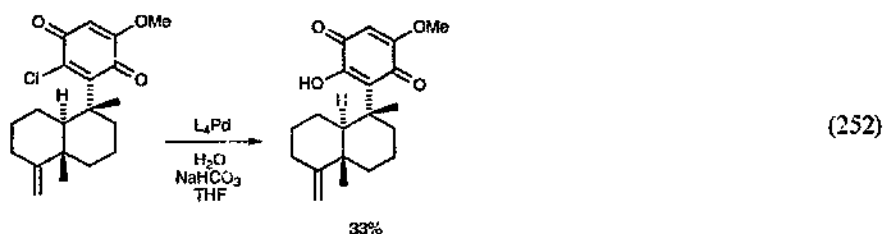
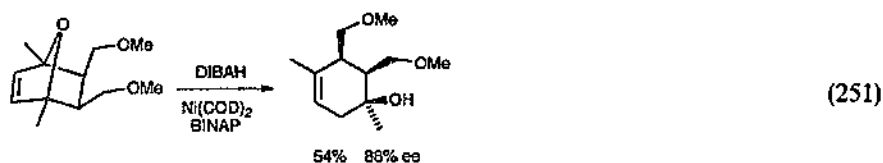
Chiral ruthenium(II) complexes catalyzed the reduction of 1,3-diketones to anti 1,3-diols in 70% yield and 100% ee [750]. α -Amino- β -ketophosphonates were asymmetrically reduced in high ee (Eq. (248)) [751], as were a variety of dicarbonyl compounds (Eq. (249)) [752]. β -Keto esters were asymmetrically reduced in high yield with high ee under mild conditions using (*R,R*)-(iPrBPE)RuBr₂ catalysts [753].



Chiral zirconocene complexes catalyzed the ring opening of dihydrofurans and pyrans (Eq. (250)) [754]. Nickel cyclooctadiene catalyzed the ring opening of bridged bicyclic ethers by DIBAL (Eq. (251)) [755]. Palladium(0) was required for the hydroxylation of chloroquinones (Eq. (252)) [756]. Allyl ethers were reductively cleaved to alcohols electrochemically in the presence of nickel(II) salts [757]. Terminal olefins were converted to secondary alcohols by palladium catalyzed hydrosilylation (HSiCl₃) followed by oxidation [758]. Adjacent carboxylate groups were used to direct this chemistry (Eq. (253)) [759]. Palladium(0) catalyzed the allylation of benzaldehyde (to give allyl alcohols) by allyl esters [760].



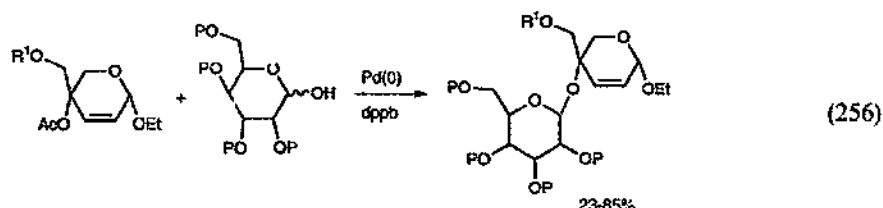
Molybdenum hexacarbonyl cleaved isoxazoles (Eq. (254)) [761]. Chiral zirconocene complexes catalyzed the asymmetric methalumination of terminal alkenes to



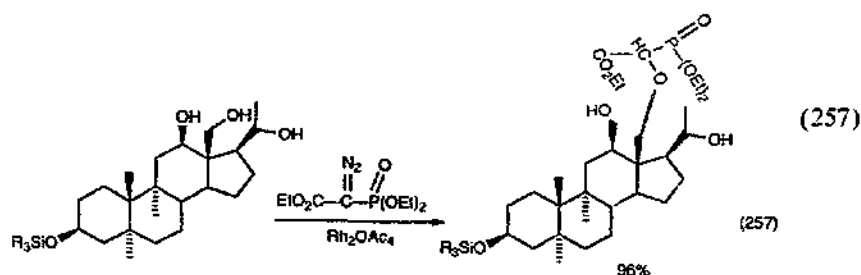
give α -methyl terminal alcohols in good yield and high ee, after oxidation [762]. Iron carbonyl complexation was used to protect a diene while a remote olefin was hydroborated (Eq. (255)) [763].

3.4. Ethers, esters, acids

The factors which influenced the stereo- and regioselectivity in the palladium catalyzed conversion of allyl carbonates to aryl phenyl ethers (phenoxide was the nucleophile) were studied [764]. Nickel(0) catalyzed the displacement of allyl acetates and carbonates by phenoxide [765]. Acetoxy glycals were converted to disaccharides by palladium catalyzed displacement by the anomeric OH of a sugar (Eq. (256)) [766]. Sugar OH groups were allylated by diallyl carbonate in the presence of palladium(0) catalyst [767]. Molybdenum(II) catalyzed the conversion of allyl acetals to allyl methyl ethers [768].

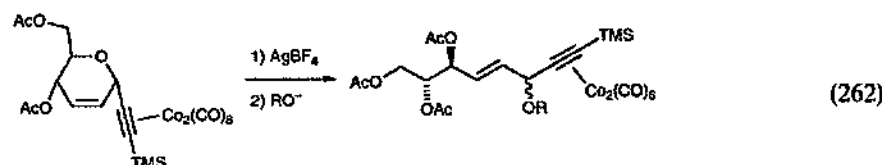
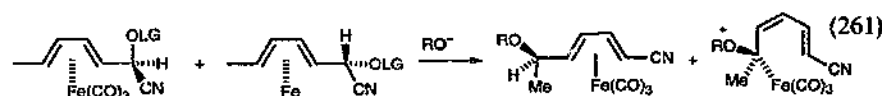
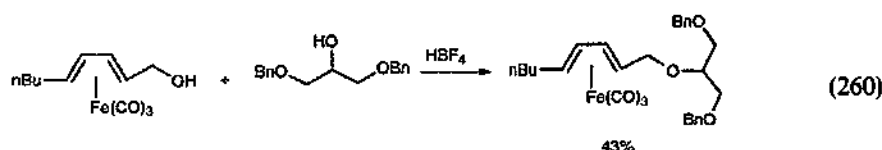
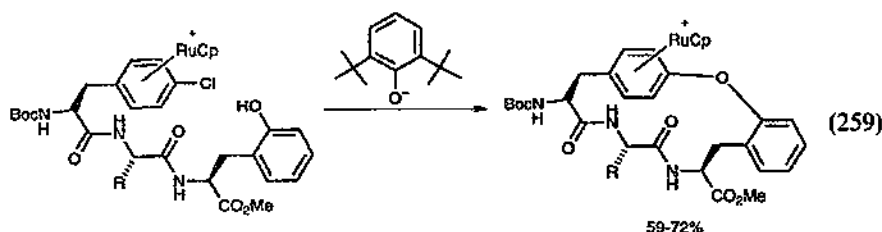
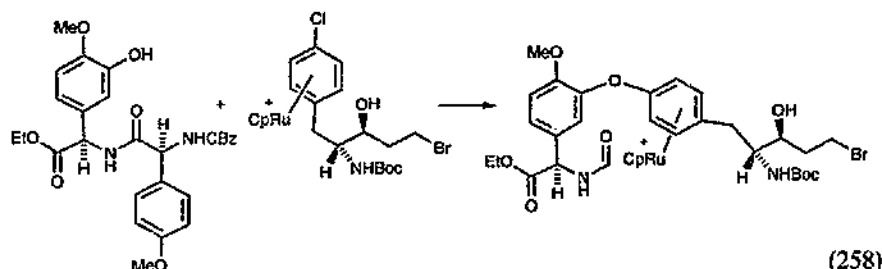


Rhodium(II) acetate catalyzed the decomposition of chiral α -diazo esters in alcohols to give α -alkoxy esters in good yield and up to 50% de [769]. Complex ethers could be made in this way (Eq. (257)) [770]. Fluorinated α -diazo esters [771], and diazo ketones [772] underwent similar insertions into alcohol OH bonds to give ethers.

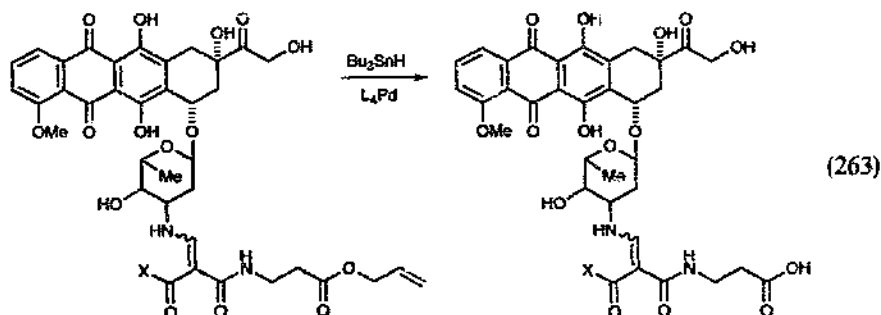


Diaryl ethers were made by the copper promoted coupling of aryl bromides with phenols [773]. Treatment of the cationic iron Cp complex of chlorobenzene with alkoxides under irradiation gave metal-free aryl ethers by displacement of the chloride [774]. The corresponding ruthenium complexes of polychlorinated arenes gave poly aryl ethers or thioethers [775]. This has been used to make quite complex systems (Eq. (258)) [776] and (Eq. (259)) [777].

Hydroxy groups allylic to iron coordinated dienes were replaced by alcohols to form ethers in the presence of Lewis acids (Eq. (260)) [778]. Eq. (261) shows an unusual displacement [779]. Esters were deoxygenated to ethers by treatment with manganese acyl catalysts and phenyl silane [780]. Copper iodide catalyzed the displacement of tertiary propargyl halides by phenoxide [781].



Ruthenium complexes catalyzed the addition of carboxylic acids to alkynes to give secondary [782] or primary [783] vinyl esters. Benzene was oxidized to cyclohexane-hexacetate by barium chlorite in the presence of OsO_4 catalysts [784]. Cobalt

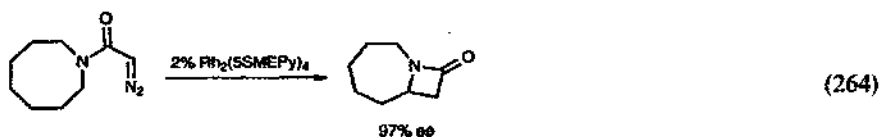


complexed propargyl glycols were ring opened by alkoxides (Eq. (262)) [785]. Palladium catalyzed the cleavage of an allyl ester to the acid in a complex system (Eq. (263)) [786].

3.5. Heterocycles

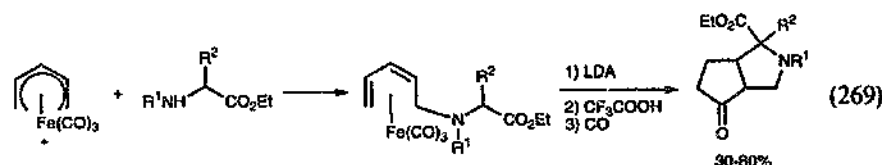
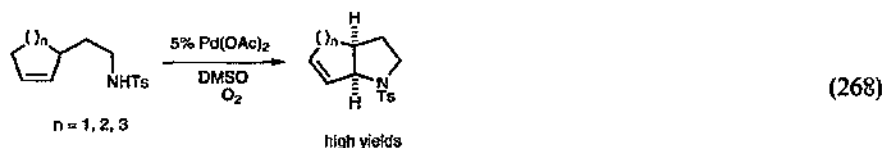
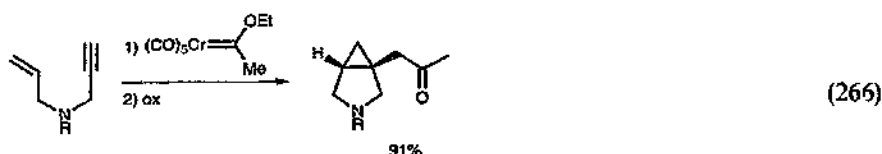
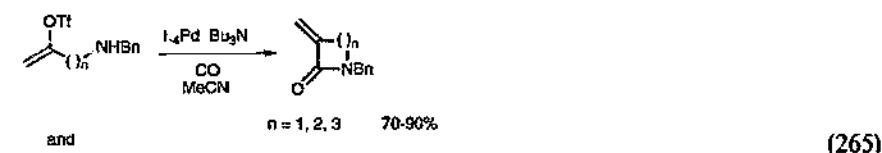
Aziridenes were made by the rhodium(II) [787], rhenium(VII) [788], and copper(II) catalyzed decomposition of ethyl diazoacetate in the presence of imines. By using chiral bis-oxazoline ligands, the copper(I) catalyzed decomposition gave good yields, >10:1 *trans* to *cis* and ee values from 40% to 67% in this process [789,790]. The mechanism of the copper(I) catalyzed aziridination of olefins by $\text{PhI}=\text{NTs}$ was studied [791].

β -Lactams were synthesized by rhodium(II) catalyzed H insertion processes (Eq. (264)) [792]. Palladium on carbon in the presence of copper(I) iodide catalyzed the oxidative carbonylative cyclization of propargyl amines to β -lactams [793]. Alkynes reacted with nitrones in the presence of copper(I) iodide to give β -lactams [794]. Vinyl triflates with pendant amino groups underwent carbonylative cyclization to β -lactams using palladium catalysts (Eq. (265)) [795].

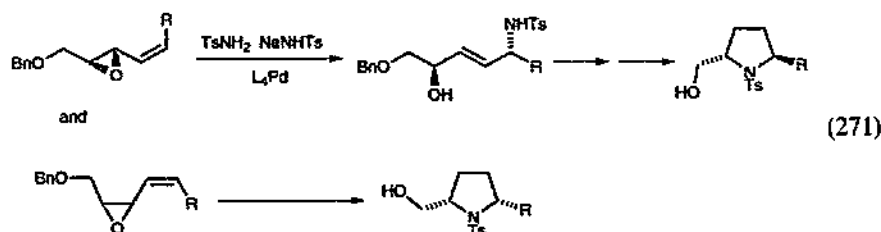
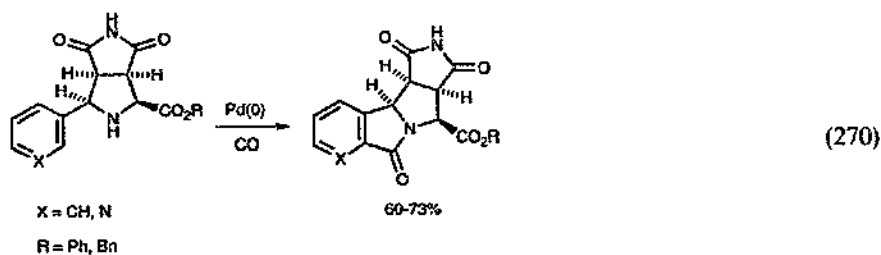


Pyrrolidines were made from allyl propargyl amines by chromium carbene mediated metathesis/cyclopropanations (Eq. (266)) [796], by palladium catalyzed cyclo-carbonylation (Eq. (267)) [797], by palladium(II) catalyzed amination of olefins (Eq. (268)) [798], and by the oxidation of amino-iron diene complexes (Eq. (269)) [799]. Two useful palladium catalyzed syntheses of this ring system are seen in Eq. (270) [800] and Eq. (271) [801].

γ -Nitroketones were reductively cyclized to five-membered imines by $\text{Ru}_3(\text{CO})_{12}$

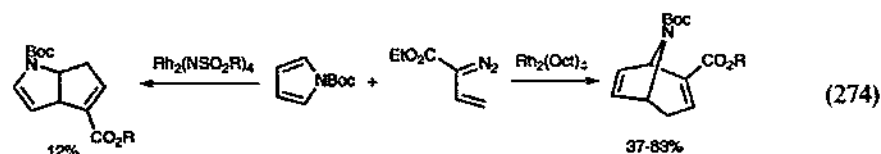
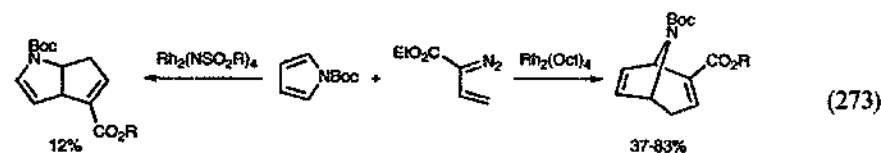
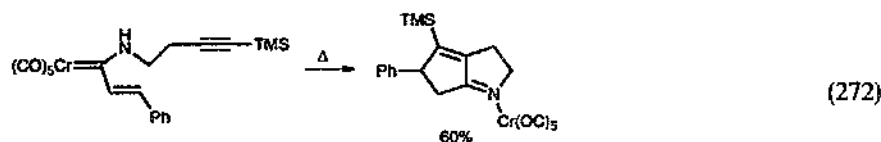


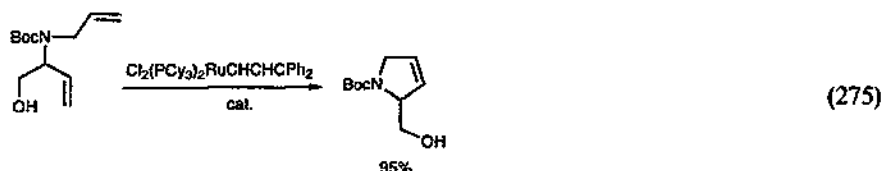
and 9,10-phenanthroline [802]. Complex aminocarbene complexes thermally produced cyclized five-membered imines (Eq. (272)) [803]. Rhodium(II) octanoate catalyzed the cycloaddition of α -diazo- β -vinyl esters to pyrroles (Eq. (273)) [804]. 2,5-Dihydropyrroles were made by chromium carbene complex promoted metathesis



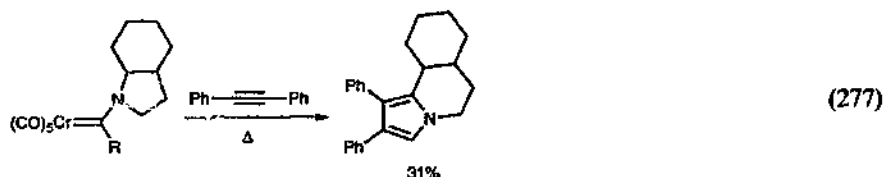
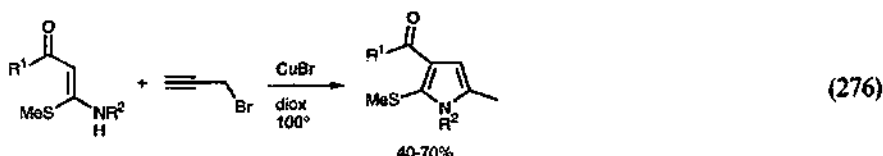
processes (Eq. (274)) [805] and ruthenium carbene complex metathesis (Eq. (275)) [806].

Pyrroles were synthesized by McMurry coupling of β -amido enones [807], the

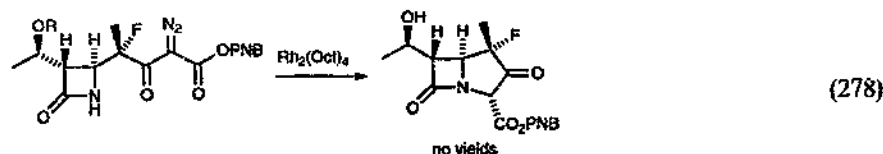




copper catalyzed reaction of propargyl bromide with β -thio- β -amino ketones (Eq. (276)) [808] and by the reaction of aminocarbene chromium complexes with alkynes (Eq. (277)) [809].

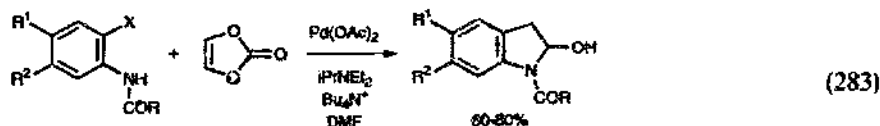
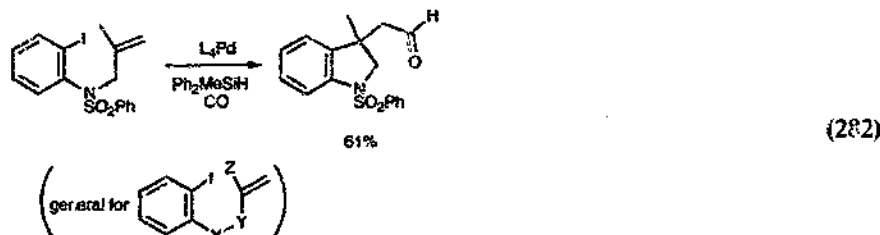
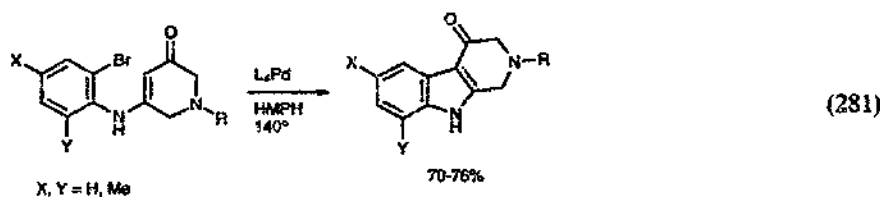
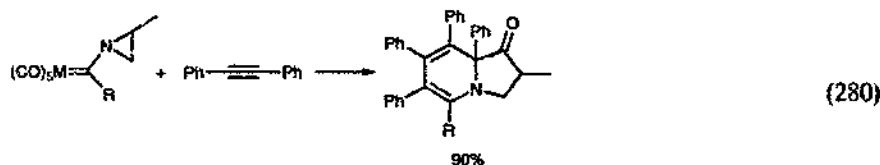
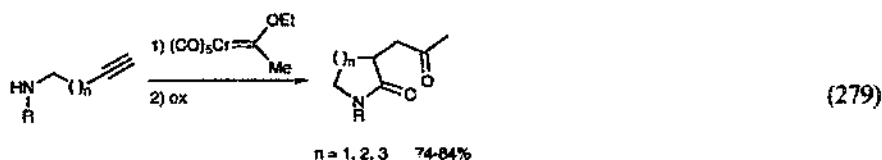


Five-membered lactams were made by rhodium(II) catalyzed C–H insertion processes of α -diazoamides [810], or N–H insertion (Eq. (278)) [811], by carbonylative cyclizations of *o*-aminophenylacetylene by $\text{Rh}_4(\text{CO})_{16}$ [812], the chromium carbene assisted cyclization of acetylenic amines (Eq. (279)) [813] and the reactions of aminocarbene complexes with alkynes (Eq. (280)) [814,815].



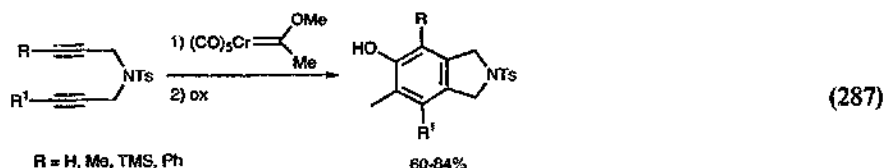
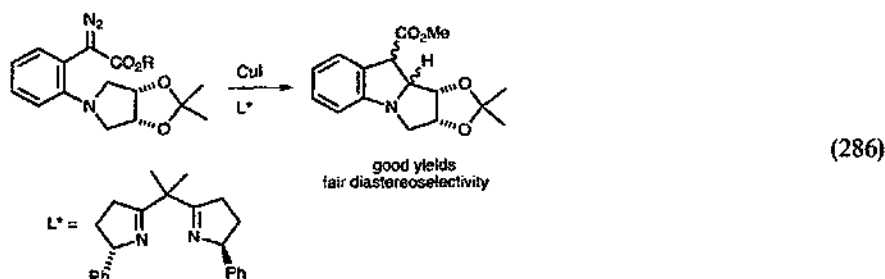
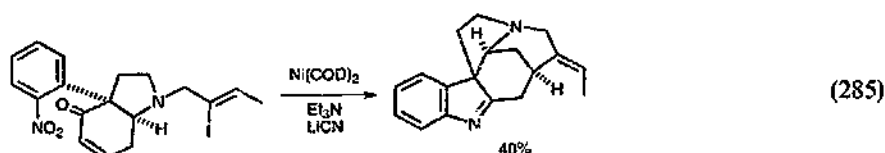
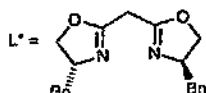
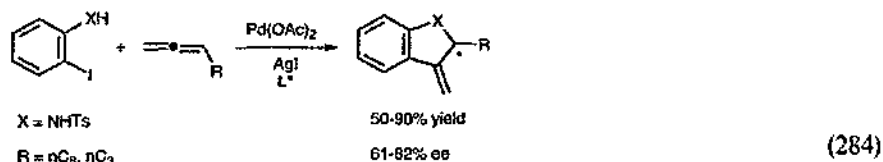
Indolines were synthesized in the many approaches shown in Eq. (281) [816], Eq. (282) [817], Eq. (283) [818], Eq. (284) [819], Eq. (285) [820], Eq. (286) [821], Eq. (287) [822], Eq. (288) [823] and Eq. (289) [824].

A catalytic version of McMurry coupling was used to synthesize indoles from the reductive coupling of the carbonyl groups of *o*-acyl anilides in good yields [825,826]. Palladium acetate catalyzed the oxidative coupling of *N*-aryl aminoquinones



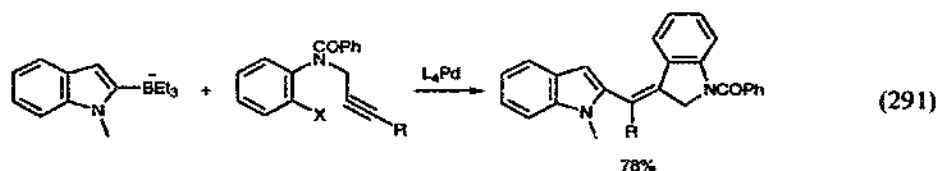
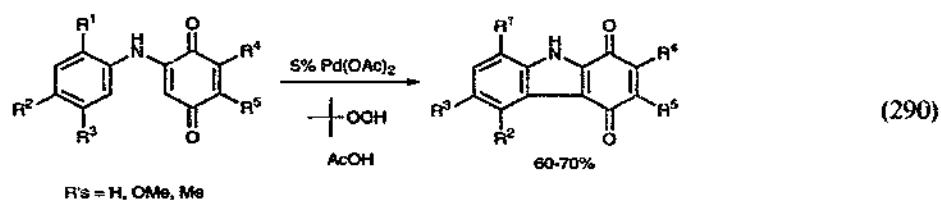
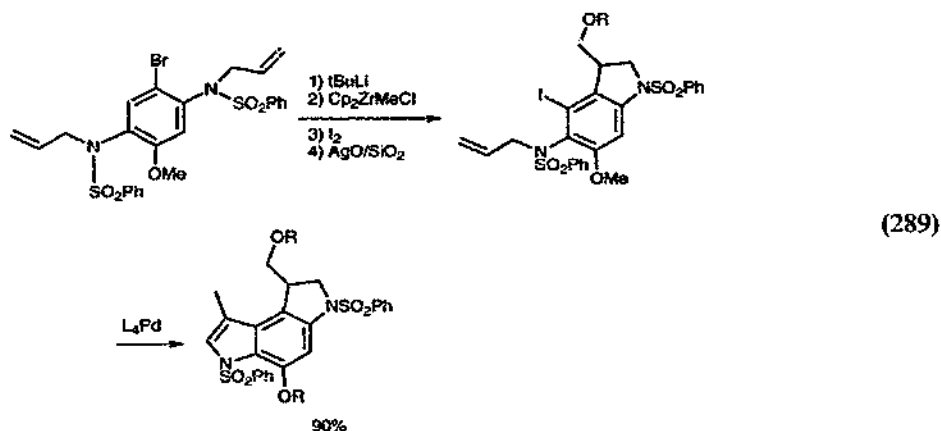
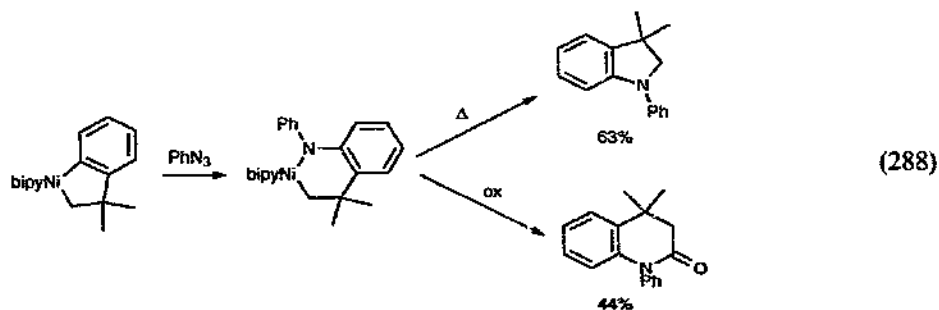
(Eq. (290)) [827]. Other indole syntheses are seen in Eq. (291) [828], Eq. (292) [829], and Eq. (293) [830].

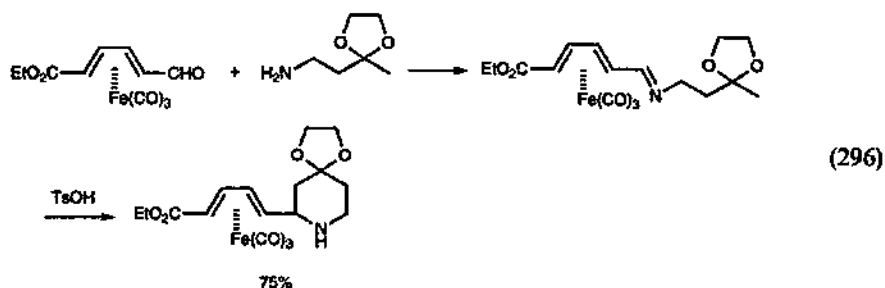
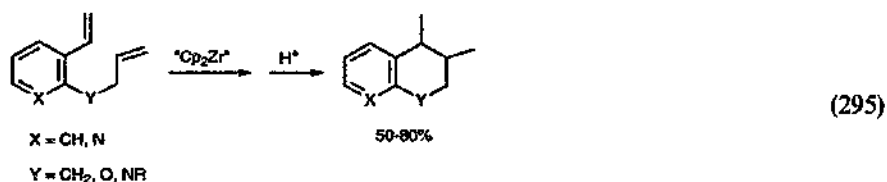
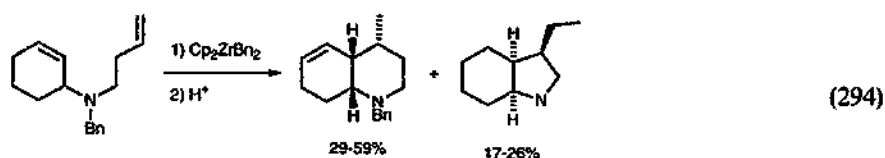
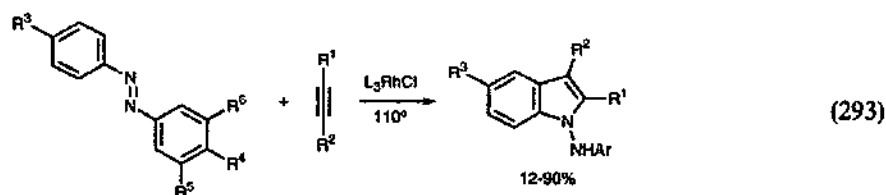
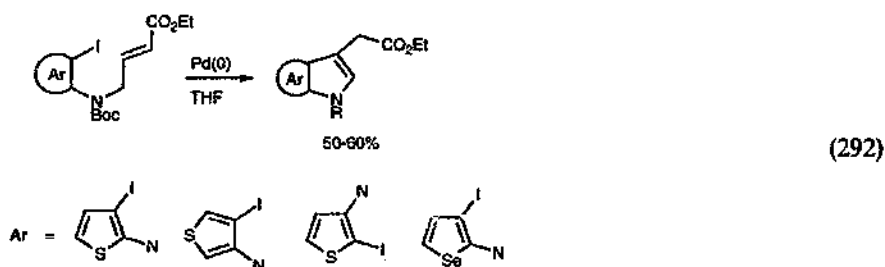
Zirconocene cyclized bis-olefinic amines to piperidines: Eq. (294) [831] and Eq. (295) [832]. Hydroformylation of allyl glycine esters gave piperidines or tetrahydropyridines [833]. Tetrahydroisoquinolines were synthesized by intramolecular

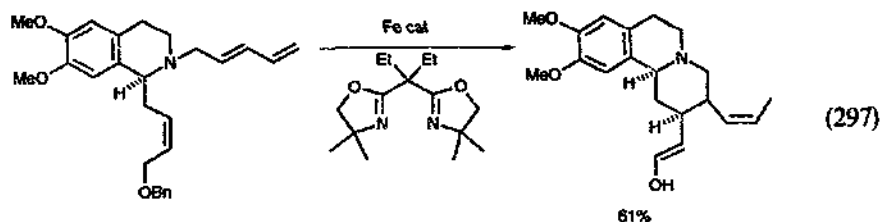


Heck cyclization of *o*-iodo-*N*-allylbenzylamines [834]. Palladium(0) catalyzed the reaction of optically active aminoalcohols with 1,4-diacetoxy-2-butene to form morpholines [835]. Iron catalyzed processes were used to make piperidines (Eq. (296)) [836] and (Eq. (297)) [837].

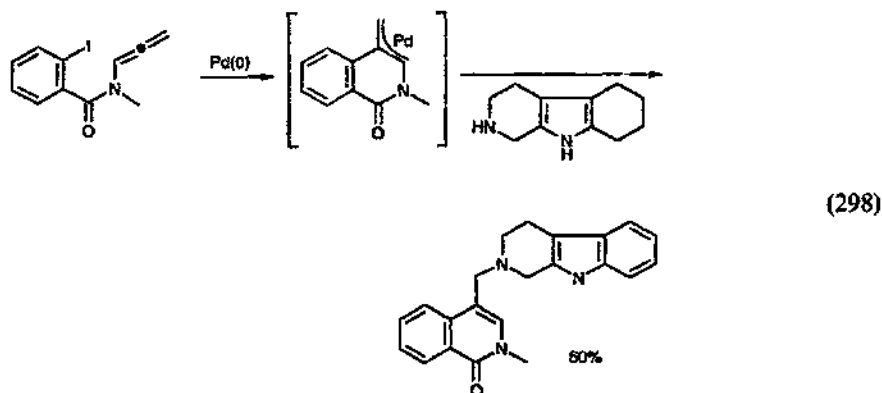
Dihydroisoquinolines were synthesized by palladium(0) catalyzed Heck cyclization of *o*-iodo-*N*-allyl benzamides [838,839]. The same type of cyclization with *N*-allenyl benzamides gave a π -allyl palladium complex which could be further functionalized (Eq. (298)) [840]. Quinolines were prepared from palladium(0) catalyzed cyclization of the vinyl triflate of β -keto esters with *o*-boronic acids of aniline







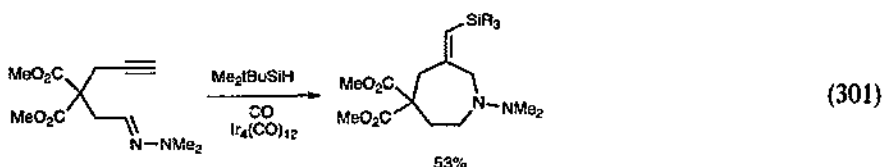
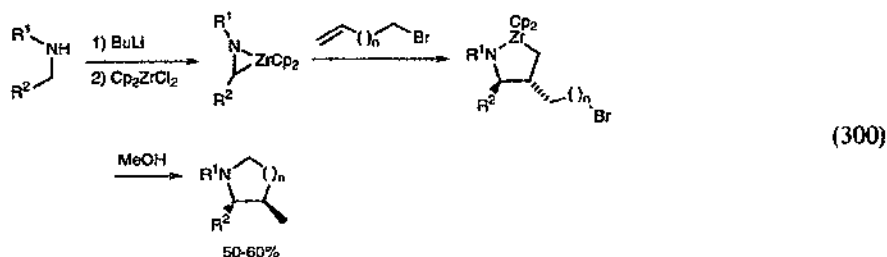
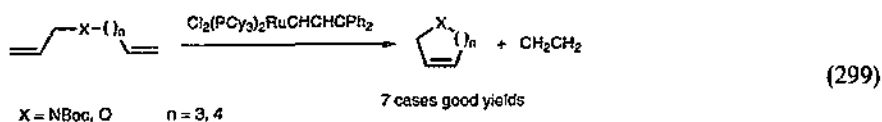
carbamates [841], and the carbonylative cyclization of amine containing iodoenynes [842]. *N*-Aryl-2-aminomethylindoles cyclized to give fused tetrahydroquinolines in low yield when treated with one equivalent of palladium acetate [843]. Iron complexed vinyl ketenes reacted thermally with imines, followed by oxidation to give dihydroquinolines [844]. Iron diene complexed imines of sorbaldehyde underwent 4+2 cycloaddition with Danishefsky's diene to give dihydro-4-pyridones [845].



Quinolines were prepared by the palladium catalyzed reaction of formamide with *o*-nitrophenyl ketones in 20–46% yield [846], while pyridines were produced from hydrazones of dialdehydes [847].

Larger ring nitrogen heterocycles were made by ruthenium carbene catalyzed metathesis processes [848], Eq. (299) [849]. Azirenes cycloadded to chromium complexed cycloheptatriene in a 2+6 cycloaddition to give 4.3.1-bicyclo nitrogen heterocycles [850]. Macrocyclic lactams were made by palladium(0) catalyzed cyclization of (alkylation) of long chain malonamides with remote allyl acetates [851]. Larger ring (7–10) benz fused nitrogen heterocycles were prepared by Heck cyclization of *o*-iodoarenes with enamides in the side chain [852]. Zirconium (Eq. (300)) [853] and iridium (Eq. (301)) [854] chemistry was used to make larger ring nitrogen heterocycles.

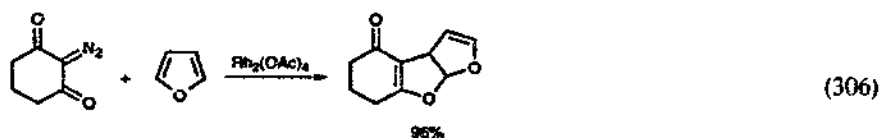
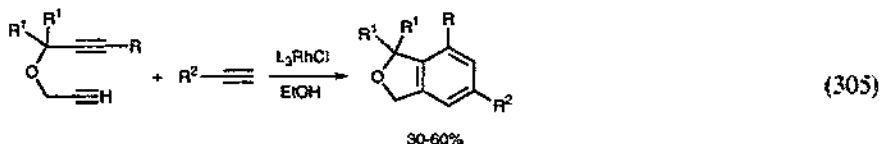
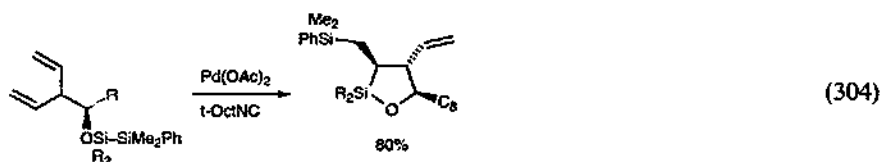
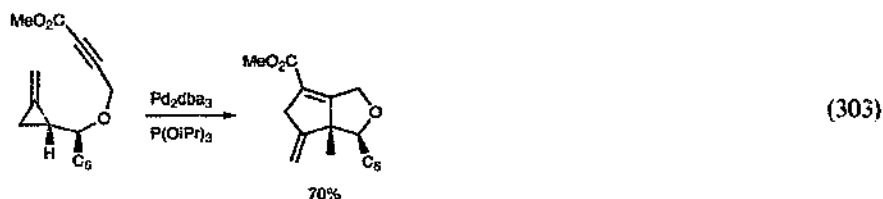
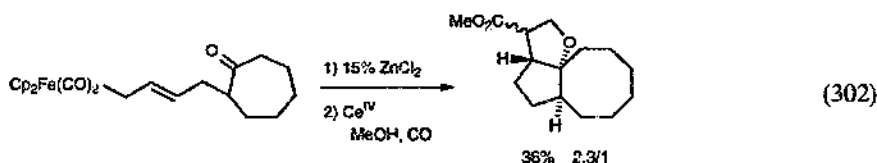
Manganese salen catalyzed asymmetric oxidation of simple olefins has been reviewed (36 references) [855], as has enantiomeric epoxidation via chemical or enzymic methods (in French, 190 references) [856]. Chiral manganese salen complexes continued to be refined as asymmetric epoxidation catalysts [857–859].



Aldehydes were converted to epoxides by rhodium(II) catalyzed reaction of diazoalkanes with thioethers to form sulfur ylides in situ [860]. Cationic platinum complexes catalyzed the epoxidation of enones by hydrogen peroxide in a process which required excess enone [861]. β -Lactones were synthesized by rhodium(II) catalyzed decomposition of diazomalonates (CH insertion into a carbon of the ester group) [862].

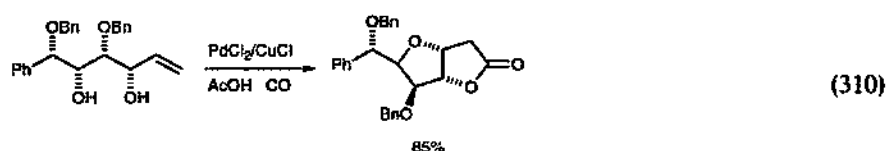
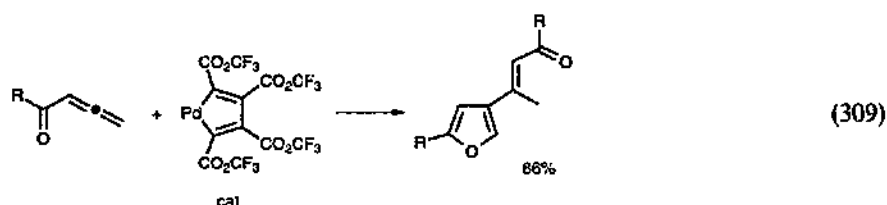
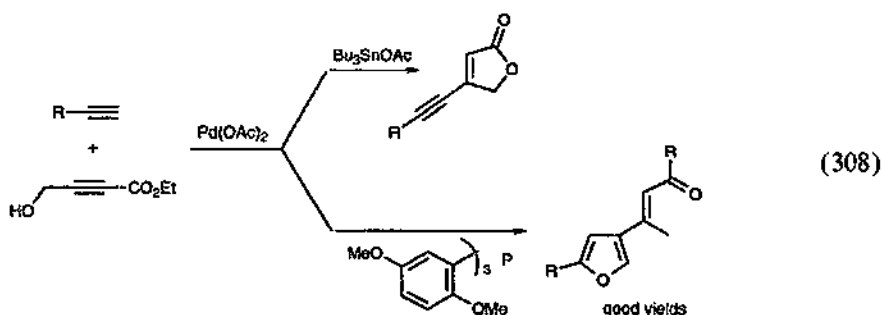
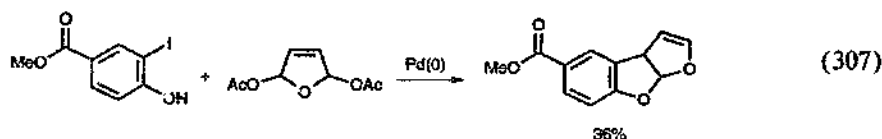
Tetrahydrofurans were synthesized by nickel(0) catalyzed cyclization of allyl ethers having a halide in the other arm [863,864], and by iron-assisted alkoxylation of complexed alkenes (Eq. (302)) [865]. Allenes having γ -hydroxy groups were cyclized to tetrahydrofurans by palladium catalyzed arylation at the central carbon followed by attack of OH on the thus-formed π -allyl palladium complex [866]. Other palladium catalyzed tetrahydrofuran forming reactions are shown in Eq. (303) [867] and Eq. (304) [868]. Homoallyl alcohols cyclized to tetrahydrofurans when treated with three equivalents of Re_2O_7 and H_5IO_6 [869]. These were also produced via rhodium(II) catalyzed decomposition of diazo esters (C–H insertion into β -o-ethyl group) [870].

Dihydrofurans were synthesized by $\text{Mo}(\text{CO})_6$ catalyzed cyclization of homopropargyl alcohols [871,872], by metathesis of diallyl ethers [873], by cocyclotrimerization of bis-propargyl ethers (Eq. (305)) [874] and by rhodium carbene chemistry (Eq. (306)) [875].



Areneruthenium complexes cyclized ynolols to furans [876]. Palladium catalyzed approaches to furans are shown in Eq. (307) [877], Eq. (308) [878] and Eq. (309) [879]. Dihydrofuranylmolybdenum carbene complexes decomposed to bis-furans linked at the 2-position [880]. Alkynes cyclized with *o*-iodophenols in the presence of palladium catalysts to give benzofurans [881]. Heck cyclization of *o*-bromo-*O*-allylphenols also gave benzofurans [881].

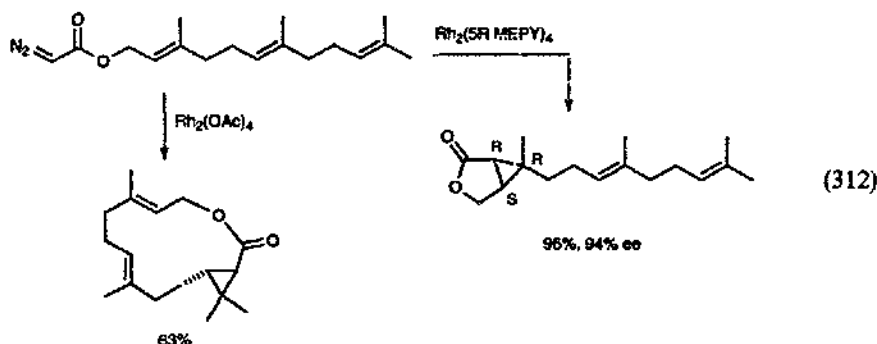
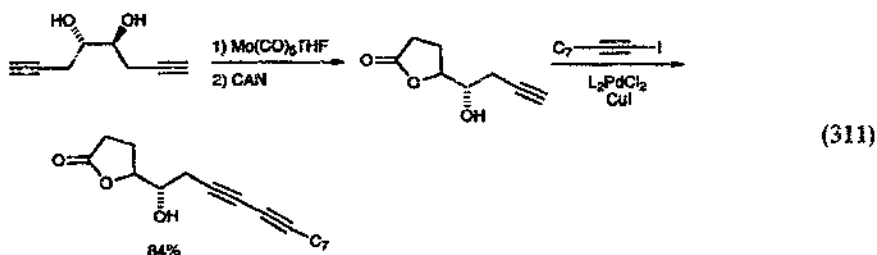
Butyrolactones were made by palladium(II) catalyzed alkoxycarbonylation of



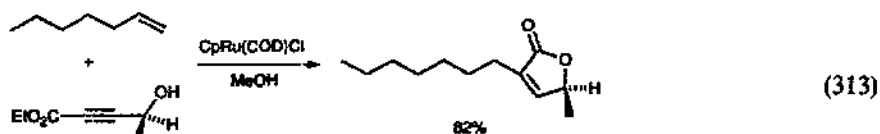
alkenes [882], (Eq. (310)) [883], palladium catalyzed oxidative cyclization of β -hydroxytrimethylsilylacetylenes [884], cobalt catalyzed cyclocarbonylation of allyl alcohols [885] and iron catalyzed cyclizations of unsaturated carboxylic acids [886]. Molybdenum carbonyl cyclized homopropargyl alcohols (Eq. (311)) [887].

Butyrolactones were made by rhodium(II) catalyzed cyclopropanation of alkenes by diazo esters (Eq. (312)) [888]. The site of cyclopropanation of polyolefins depended on the ligand on rhodium [889], as did the endo/exo ratio and ee [890,891]. Butyrolactones were also synthesized by rhodium catalyzed CH insertion of diazoalkanes, with effective catalysts for enantioselective insertion into 3° positions of alkylazo acetates [892,893]. 2-Oxo-tetrahydrofurans were made this way [894].

Butenolides were prepared by nickel catalyzed cyclocarbonylations of ynones

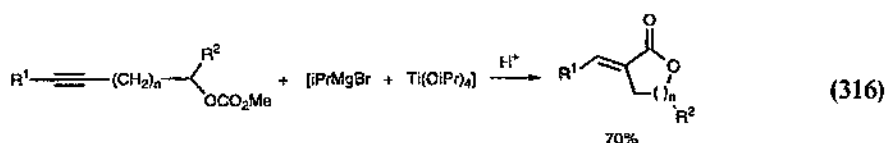
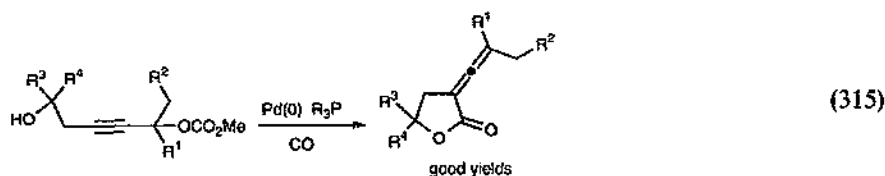
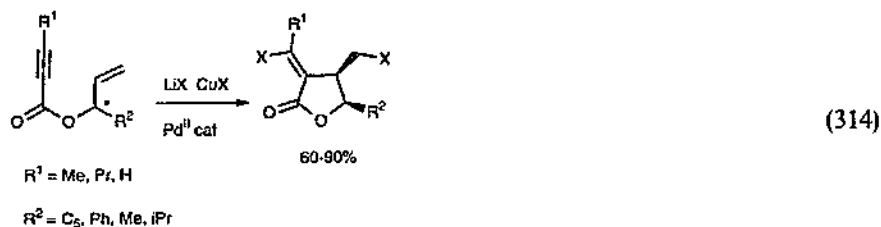


[895], palladium catalyzed cyclocarbonylation of β -iodoenones, [896], and the ruthenium catalyzed reaction between alkenes and acetylenic esters (Eq. (313)) [897].

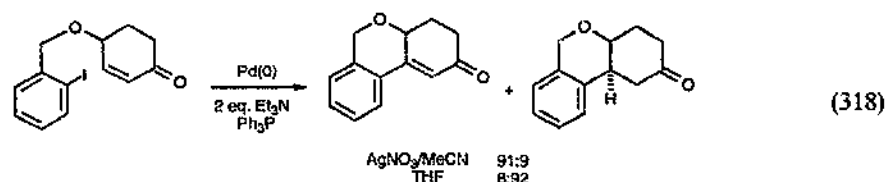
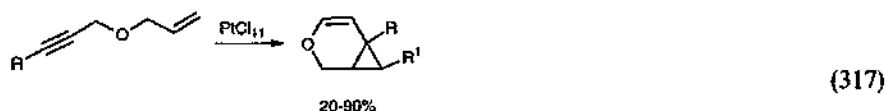


β -Halo- α -methylene lactones were synthesized by palladium catalyzed halocyclization of allyl esters of propionic esters (Eq. (314)) [898–904], the palladium catalyzed cyclocarbonylation of propargyl carbonates (Eq. (315)) [905] and the low valent titanium cyclization of alkyne containing carbonates (Eq. (316)) [906]. Bis-propargyl alcohols were cyclocarbonylated to succinic anhydride [907,908].

Palladium(II) catalyzed the alkoxy acetoxylation of cyclohexadiene by a $(\text{CH}_2)_3\text{OH}$ attached at the two position to give exomethylene pyrans. The stereochemistry depended on the presence or absence of added lithium chloride or acetate [909]. Propargyl allyl ethers cyclized to tetrahydropyrans in the presence of PtCl_4 (Eq. (317)) [910]. *Ortho* iodo benzyl allyl ethers cyclized to dihydrobenzopyrans under Heck reaction conditions (Eq. (318)) [911,912]. Similar processes were used

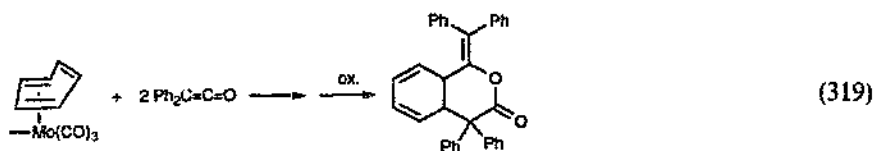


to cyclize into the double bond of dihydropyrans [913] and unsaturated nitro compounds [914] to make six-membered heterocycles.

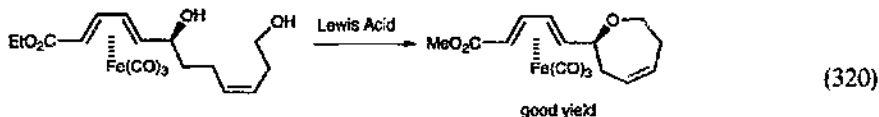


Ortho iodobenzoic acid and alkynes formed isocoumarins under palladium catalysts [915]. γ -Pyrones formed by CH insertion in a rhodium(II) catalyzed decomposition of α -diazoacetophenones having an *ortho* isopropyl group [916]. Diphenylketene

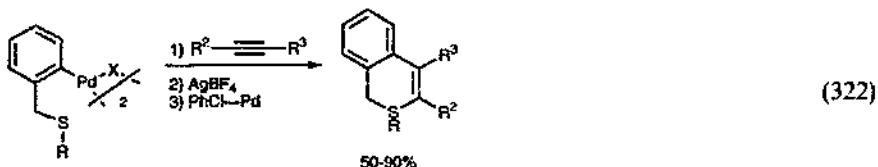
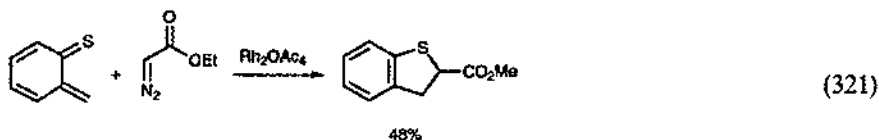
reacted twice with anionic diene molybdenum complexes to give α -methylene lactones (Eq. (319)) [917].



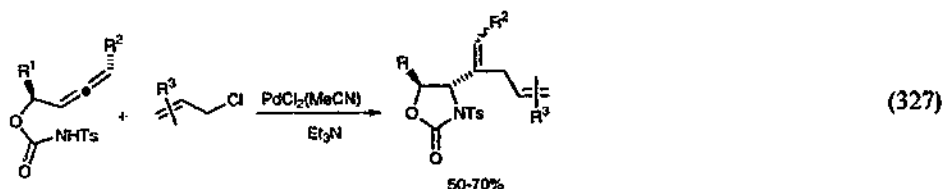
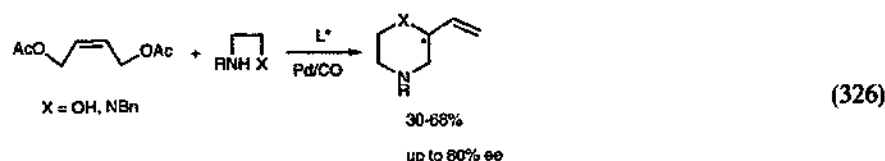
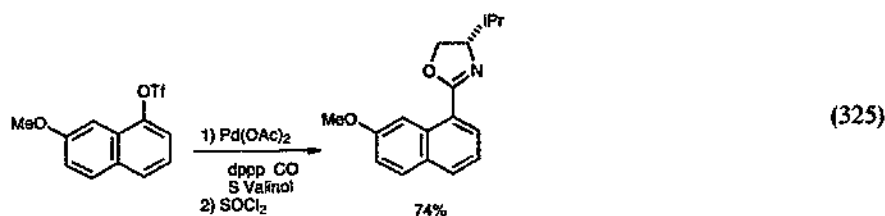
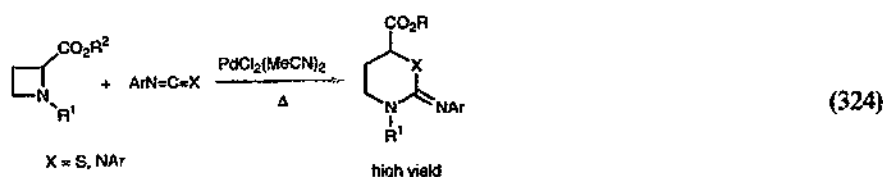
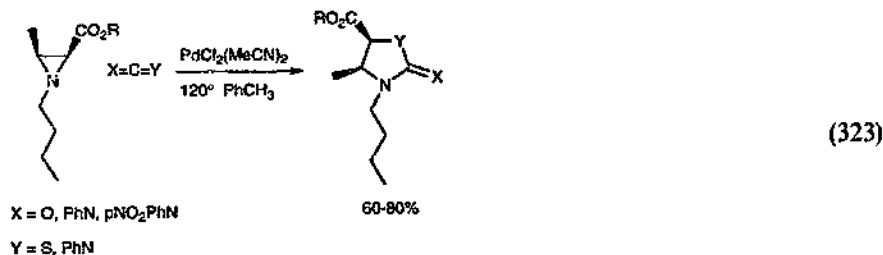
A seven-membered oxygen heterocycle containing a cobalt complexed alkyne was made by propargyl cation chemistry [918]. Eight-membered unsaturated cyclic ethers were made by palladium catalyzed intramolecular alkylation of allyl carbonates [919,920] and by cyclization on to an iron stabilized dienyl cation (Eq. (320)) [921]. Tungsten carbonyl cyclized γ -hydroxy alkynes to seven-membered α,β -unsaturated lactones [922]. Palladium(0) complexes catalyzed the cyclocarbonylation of ω -alkoxy terminal alkynes to cyclic alkynyl lactones in modest yield (ring sizes 16, 17 and 20) [923].



Dihydrobenzothiophenes and isothiophenes have been made (Eq. (321) [924], Eq. (322) [925]).

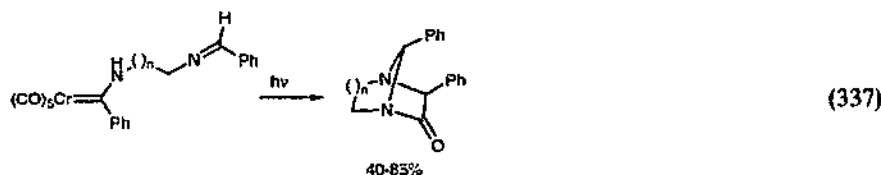
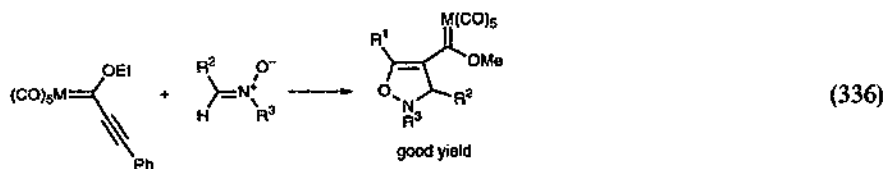
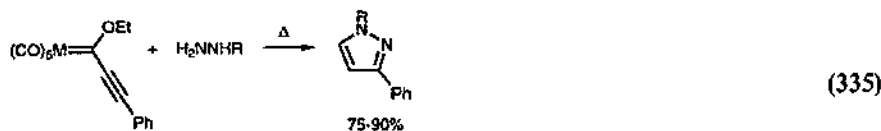
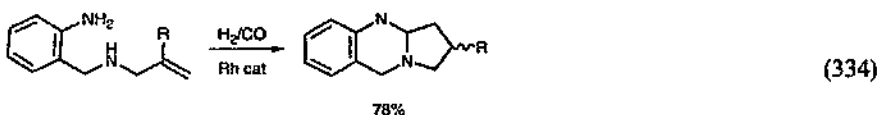
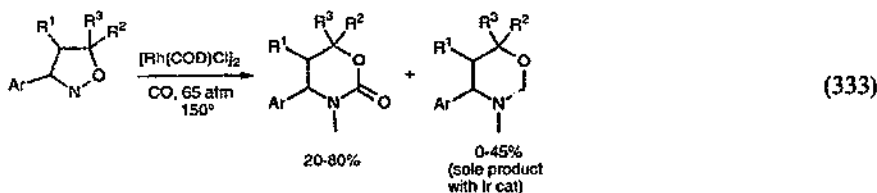


Palladium catalyzed syntheses of heterocycles containing more than one heteroatom are listed in Eq. (323) [926], Eq. (324) [927,928], Eq. (325) [929], Eq. (326) [930], Eq. (327) [931], Eq. (328) [932], and Eq. (329) [933].



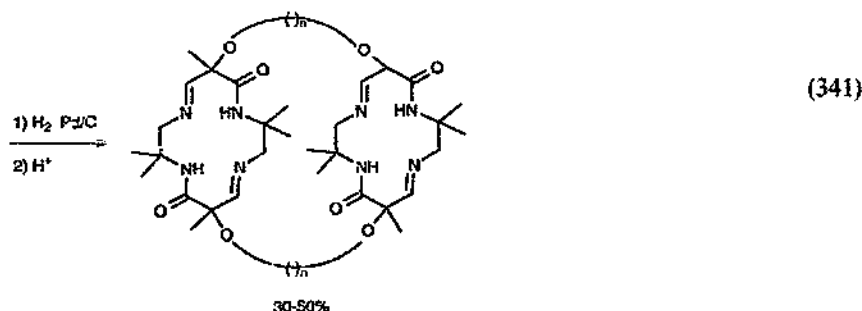
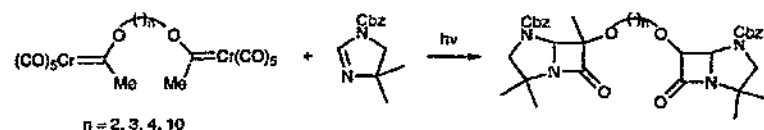
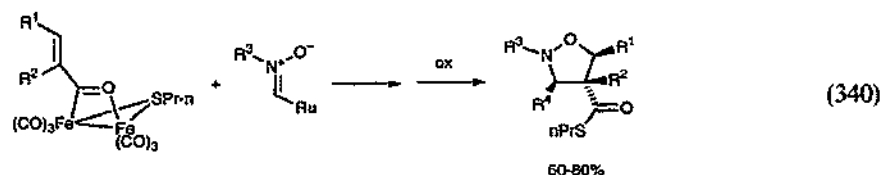
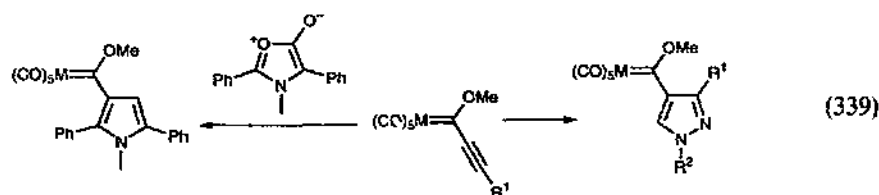
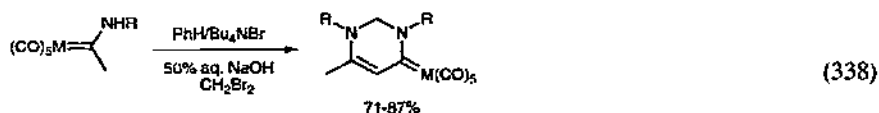
Rhodium catalyzed formation of heterocycles containing more than one heteroatom are seen in Eq. (330) [934], Eq. (331) [935], Eq. (332) [936], Eq. (333) [937], and Eq. (334) [938].

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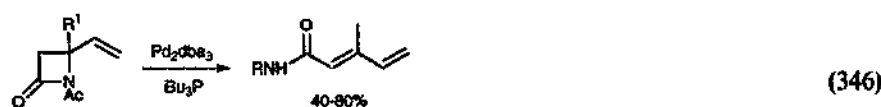
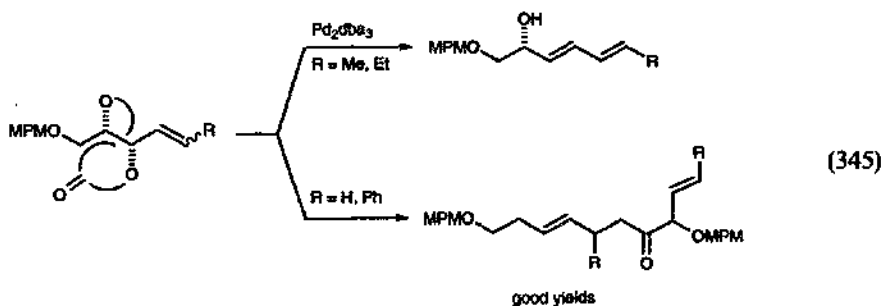
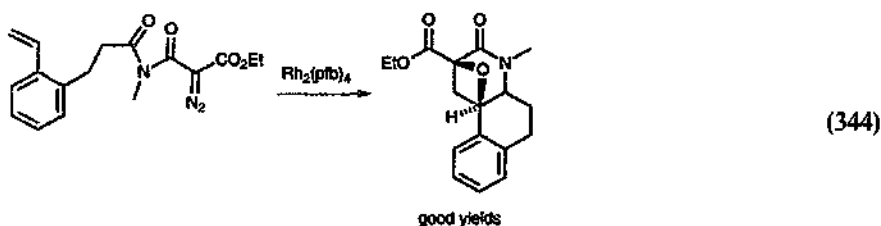
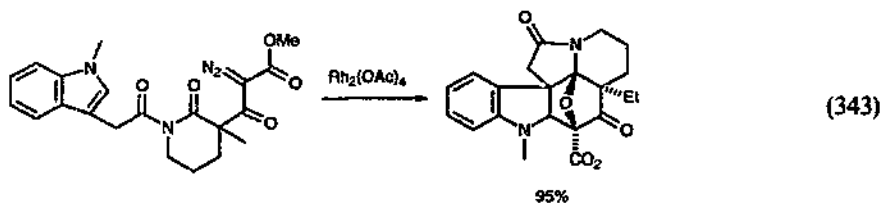
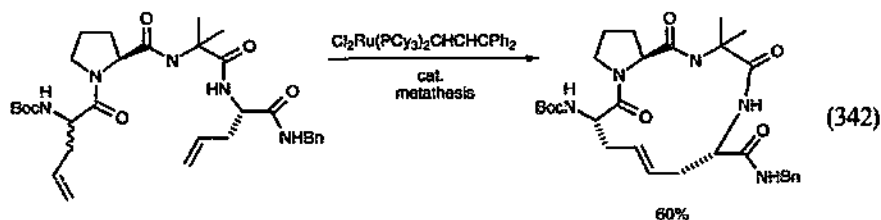
3.6. Alkenes, alkanes

Alkenes were produced by elimination of vicinal bromides by nickel(II)/ethyl Grignard mixtures [949]. Allyl acetates were eliminated to give dienes by palladium(0) catalysts [950,951], (Eq. (345)) [952] and (Eq. (346)) [953]. Allyl epoxides were eliminated to dienes via η^3 -allyliron species [954]. Dimethyl amine was oxida-

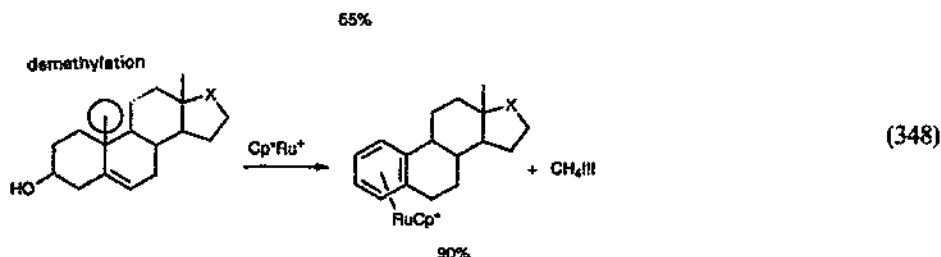
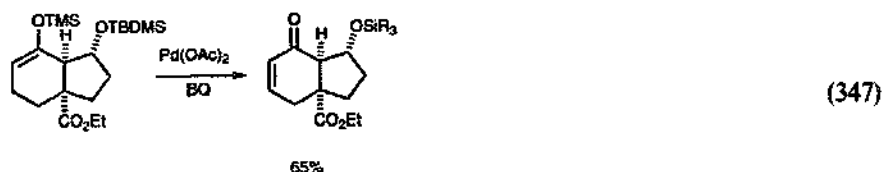


tively eliminated from chromium tricarbonyl benzylamine complexes to give complexed styrenes [955]. Glycols were made by elimination of bromide and acetate from the 1- and 2-positions of 1-bromosugars [956].

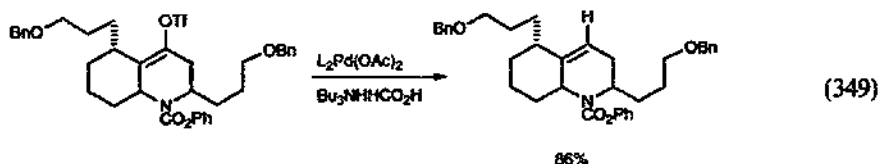
Silylenol ethers were oxidized to α,β -unsaturated enones *catalytically* using 10% Pd(OAc)₂ in DMSO with O₂ [957]. Stoichiometric versions of this were used to



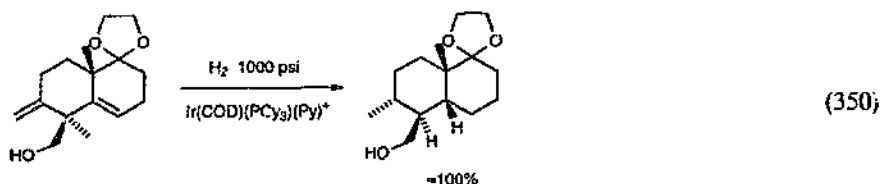
make 4-pyridones [958], to “unsaturate” steroidal D-ring cyclopentanones [959], and to make complex systems (Eq. (347)) [960]. The Cp* ruthenium fragment activated C–H, C–C, C–Cl, and C–S bonds (review, 42 references). An example is seen in Eq. (348) [961].



Aryl and vinyl triflates were reduced to arenes and olefins by ammonium formates in the presence of palladium catalysts [962,963]. Triethyl silane also served as a reducing agent under these conditions [964]. Allyl benzyl ethers were reduced to the alkene [965], but regular benzyl ethers survived (Eq. (349)) [966]. PhSiH₃ and *N*-trimethylsilyl-*N*-methyl trifluoroacetamides were new allyl group acceptors for the palladium catalyzed deallylation of allyl carbonates and ethers [967].

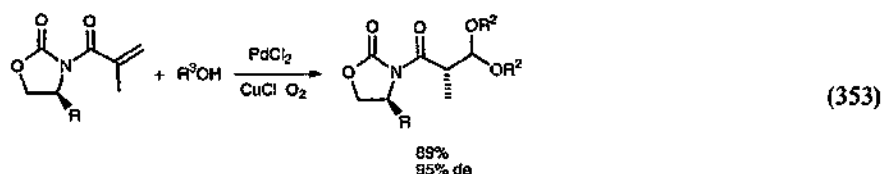
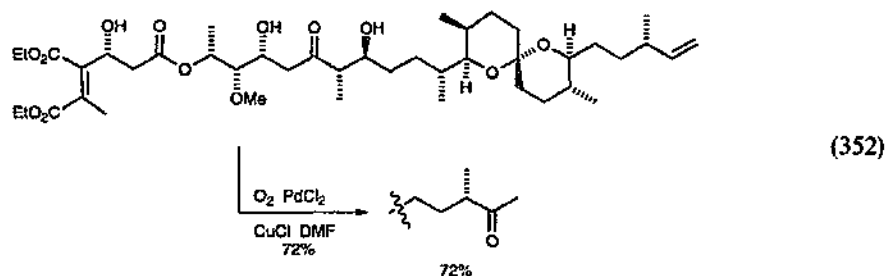
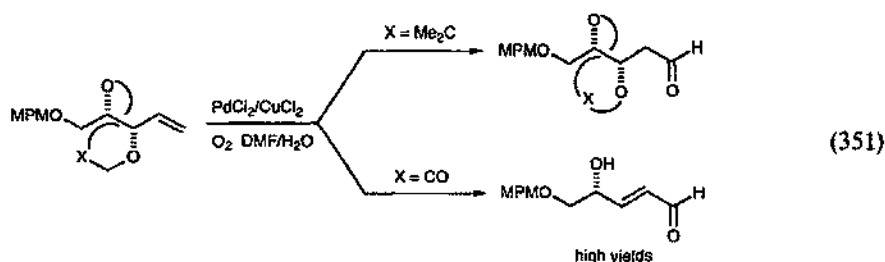


Alkynes were reduced all the way to alkanes by 2 equiv. of Cp(CO)₃WH in triflic acid [968]. β,β-Dialkyl-dehydroamino acids were reduced to β-dialkyl amino acids with high ee by Rh(I) (*R,R*)(*S,S*)-TRAP systems [969], or even more highly selectively by cationic rhodium(I) MeDuPhos or MeBPE catalyses [970]. α-Methylenebutyrolactones and cyclopentenones were reduced to the saturated carbonyl systems with high ee using ruthenium(II) BINAP catalysts [971]. Crabtree catalysts efficiently reduced olefins with high stereoselectivity (Eq. (350)) [972].



3.7. Ketones, aldehydes

Recent advances in oxypalladation have been reviewed (51 references) [973]. The Wacker oxidation of long chain alkenes to methyl ketones was accomplished with a complex catalysis system [974]. Synthetically significant Wacker oxidations are seen in Eq. (351) [975], Eq. (352) [976], and Eq. (353) [977]. Palladium chloride in DMSO oxidized diarylalkynes to the α -diketene [978]. β -Substituted styrenes were asymmetrically converted to benzyl alcohols by palladium catalyzed asymmetric hydrosilylation followed by oxidation of the silane [979].

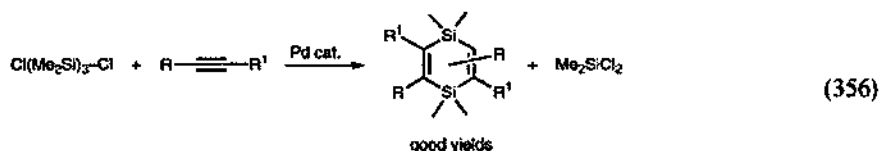
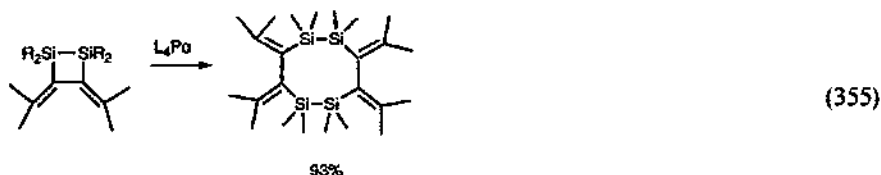


Secondary alcohols were oxidized to ketones by RuCl_3 , ROCO_2H [980], and palladium catalysts with Andogene 464 [981]. 1,2-Diols were oxidized to α -diketones by hydrogen peroxide, and peroxytungstophosphate [982].

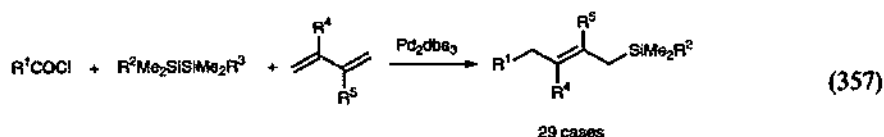
Ruthenium porphyrins catalyzed the oxidation of arenes to quinones by pyridine *N*-oxide [983]. Although isoindanone would not directly complex to chromium, its ketal would, which, after deprotection allowed a rich reaction chemistry of the complexed ketone [984]. The catalyst RuCl_2L_3 with ethylene diamine and KOH reduced aldehydes and ketones in preference to olefins [985]. Cyclic ene diynes having pendant hydroxymethyl groups were protected as their bis-dicobalt alkyne complex, permitting Swern oxidation of the hydroxymethyl group [986].

3.8. Organosilicon compounds

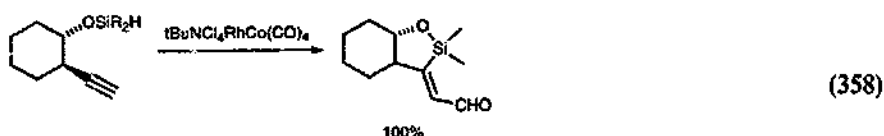
Dienes and alkenes were hydrosilylated with good ee using chiral palladium catalysts [987]. Optically active silyl allyl epoxides underwent palladium catalyzed ring opening with complete chirality transfer (Eq. (354)) [988]. Other palladium catalyzed reactions of silicon compounds are seen in Eq. (355) [989], Eq. (356) [990] and Eq. (357) [991].



Rhodium(I) complexes catalyzed the ring opening of vinylcyclopropanes to give regioisomer mixtures of unsaturated silanes [992]. Propargyl alcohols were converted

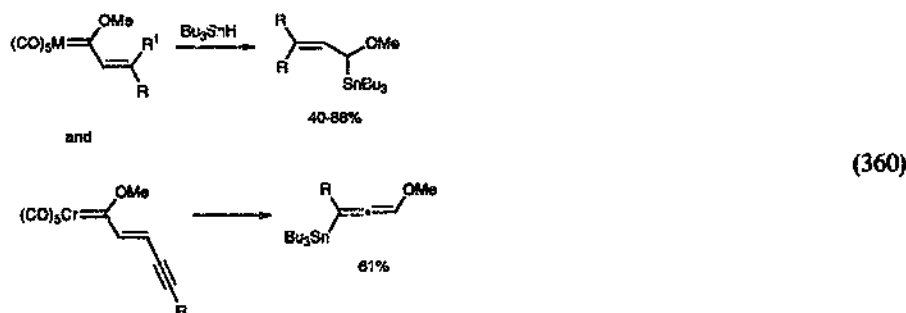
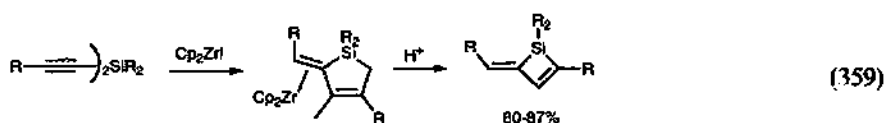


to β -silyl ketones by rhodium(I) catalyzed hydrosilylation [993]. Rhodium(II) acetate catalyzed the insertion of diazo ester derived carbenes into Si–H bonds [994]. Rhodium(I) complexes catalyzed the intramolecular silylformylation of alkynes (Eq. (358)) [995].



Chromium complexed anisole was *ortho* silylated with 88% ee by deprotonation with a C-2 symmetric chiral amide base [996]. Platinum(0) catalyzed the *ortho* silylation of benzaldehyde imines [997]. Eq. (359) shows a strange silylation reaction [998].

Palladium(0) catalyzed the *cis*-bis-stannylation of alkoxyalkynes [999] and acety-

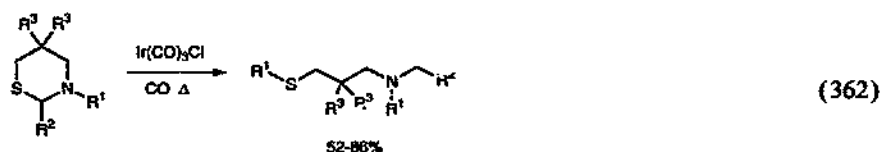
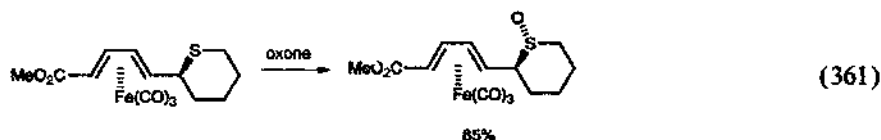


lenic esters by hexamethylditin [1000]. These rearrange to the *trans* on heating. Palladium(0) catalyzed the conversion of vinyl triflates into vinyl stannanes with hexamethylditin [1001,1002]. Terminal alkynes were converted to terminal vinyl stannanes by hydrazirconation/transmetalation [1003]. Vinyl chromium carbene complexes were converted to α -alkoxy allyl stannanes [1004] while acetylenic carbene complexes were converted to allenyl stannanes by treatment with tributyltin hydride (Eq. (360)).

3.9. Organophosphorous and sulfur compounds

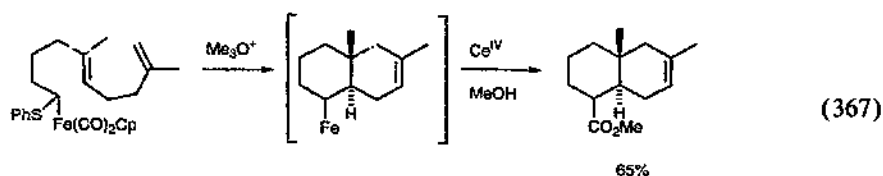
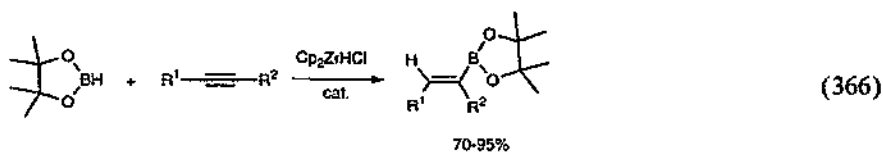
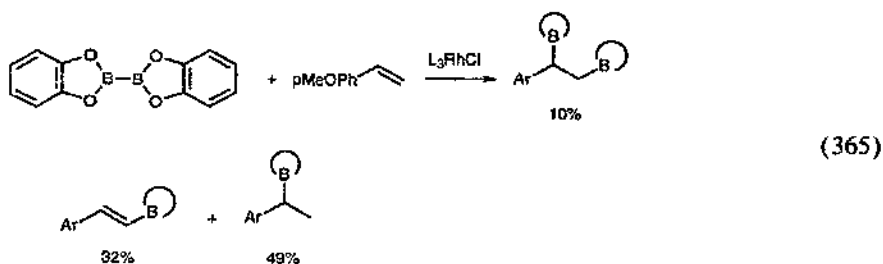
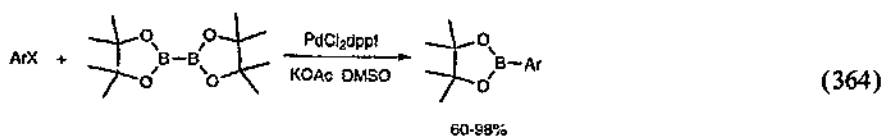
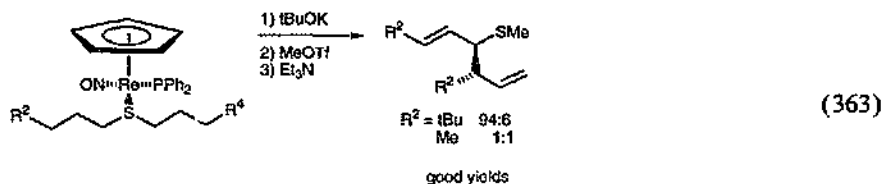
Tungsten complexes catalyzed the metathesis of allyl diphenyl phosphine to 1,4-bis(diphenylphosphino)-2-butene [1005]. Palladium(0) catalyzed the coupling of bromophosphabenzene with phospholes at P [1006]. Cationic iron hexadienyl complexes underwent attack at the less substituted terminus by triphenyl phosphine to give the alkylated phosphonium salt [1007]. Palladium(0) complexes were used to cleave allylphosphonate esters on nucleotide [1008].

Allyl carbonates [1009] acetates and chlorides [1010] were converted to allyl sulfonates by treatment with sodium toluene sulfinate and a palladium catalyst. Copper iodide catalyzed the arylations of aryl sulfonates by aryl halides [1011]. Protected serine was *S*-arylated by aryl halides and a palladium(0) catalyst [1012]. Chiral manganese salen complexes catalyzed the oxidation of aryl thio ethers to chiral sulfoxides in up to 70% ee [1013]. Other reactions of organosulfur compounds are shown below (Eq. (361)) [1014], (Eq. (362)) [1015], and (Eq. (363)) [1016].



3.10. Miscellaneous

Chiral manganese salen complexes were used to resolve racemic chromenes [1017]. Racemic chiral propargyl carbamates were resolved by complexation to $\text{Co}_2(\text{CO})_8$ [1018]. Unusual boronating systems are shown in Eq. (364) [1019], Eq. (365) [1020] and Eq. (366) [1021]. An iron promoted cationic cyclization is shown in Eq. (367) [1022].



4. Reviews

The following reviews have appeared:

Transition metal catalyzed synthesis of seven membered carbocyclic rings (18 references) [1023].

Synthetic aspects of metal-catalyzed oxidations of amines and related reactions (161 references) [1024].

Recent advances in the use of tandem reactions for organic synthesis (167 references) [1025].

The conquest of Taxol (30 references) [1026].

Synthetic applications of the OH insertion reactions of carbenes and carbenoids derived from diazocarbonyl and related diazo compounds (248 references) [1027].

Thermolysis, photolysis, and transition metal catalyzed 3,4-benzol-1,1,2,2-tetraethyl-1,2-disilacyclobut-3-ene (53 references) [1028].

High selectivity induced by neighboring group effects in C–C bond-forming reactions with organotransition metal reagents (53 references) [1029].

Regio- and stereochemical aspects of the palladium catalyzed reactions of silanes (163 references) [1030].

Activation of the Si–Si-bond by transition metal complexes (145 references) [1031].

Redox induced radical and radical ionic carbon–carbon bond forming reactions (284 references) [1032].

Ligand accelerated catalysis (89 references) [1033].

Stereoselective reactions mediated by functionalized diorganozincs (56 references) [1034].

Transition metals in organic synthesis. Annual survey 1993 (947 references) [1035].

Transition metals in organic synthesis. Hydroformylation, reduction, oxidation. Annual survey 1993 (708 references) [1036].

Atom economy — A challenge for organic synthesis: homogeneous catalysis leads the way (159 references) [1037].

α -Monohalo ethers in organic synthesis (231 references) [1038].

Preparation of heterocyclic and carbocyclic compounds by the use of an allylzinc and an allylpalladium in tandem (76 references) [1039].

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