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Transition metals in organic synthesis: highlights for the year 1998 (25th Anniversary)

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Contents

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1. General comments

This survey highlights the use of transition metals in organic synthesis for the year 1998. It offers a fairly comprehensive coverage of the literature, with extensive citations, but only unusual or significant transformations are presented in equation form. This format requires a little more effort from the user, and a lot less effort from the author, and allows thorough coverage in a minimum format. This is the 25th annual survey written by the author. The field, gratifyingly, has grown enormously and transition metals have been routinely incorporated into the tool box of the synthetic chemist. It is hoped this review continues to be useful.

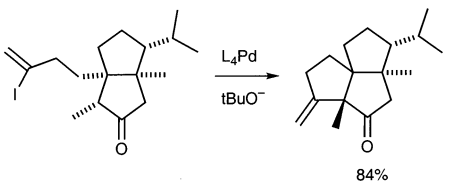
2. Carbon–carbon bond-forming reactions

2.1. Alkylations

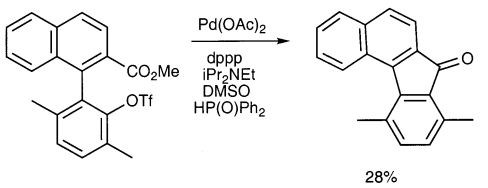
2.1.1. Alkylation of organic halides, tosylates, triflates, epoxides and aziridines

Dialkylcuprates alkylated N-protected aziridines at the less substituted position [1]. Enol triflates of conjugated ketones were alkylated by Grignard reagents in the presence of copper(I) iodide [2]. Palladium(0) catalyzed the alkylation of 2-iodo-

allylic alcohols by alkenyl Grignard reagents [3], while palladium(II) phosphine–amine complexes catalyzed the coupling of aryl triflates with alkynyl Grignard reagents [4]. Dibromocarbazoles were bis-alkylated by Grignard reagents in the presence of nickel catlaysts, and bis-carbonylated in the presence of palladium catalysts and carbon monoxide [5]. Nickel(0) phosphine–oxazoline complexes catalyze the coupling of cis β-bromostyrene to α-phenethyl Grignard reagents with low ee [6]. Palladium(0) complexes catalyzed the alkylation of brominated zinc prophyrins by malononitrile [7]. π-Allylpalladium complexes catalyzed the alkylation of aryl chlorides by a wide range of aryl, alkenyl and alkynyl dimethylfluorosilanes in the presence of TBAF [8]. Palladium(0) catalyzed the alkylation of alkenyl (Eq. (1)) [9] and aryl halides [10] by enolates, and the arylation of the methyl group of *p*-nitrotoluene by aryl bromides [11]. Intramolecular alkylation of aryl esters by aryl triflates (Eq. (2)) [12] was catalyzed by palladium(II) acetate, while a wide range of metallocenes (Fe, Co, Ni, $TiCl_2$, $ZrCl_2$) were converted to pentarylcyclopentadienes by reaction with aryl halides in the presence of palladium(II) acetate and copper(I) carbonate [13].



(1)

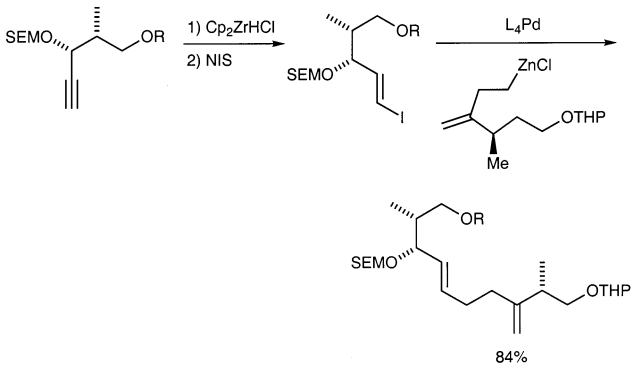


(2)

The role of dba in the reactivity of palladium(0) catalysis has been reviewed (98 references) [14]. Negishi coupling has found continued use in organic synthesis. Nickel and palladium complexes catalyzed the coupling of arylzinc halides with aryl chlorides [15], and the coupling of functionalized aryl halides with arylzinc or cadmium reagents [16]. Ortho iodo aryl triflates were converted to their bis zinc derivatives and coupled to aryl or aroyl halides by palladium(0) catalysts [17]. Quinones were synthesized by the bis-alkylation of 2,5-dibromo-1,4-dimethoxybenzene by arylzinc halides in the presence of palladium(0) catalyst [18]. Palladium(0) catalyzed the coupling of 2-halo or 2-metallo (ZnCl, $SnBu_3$) indoles with 5-halo or 5-metallo (ZnI) α-pyrones [19]. Palladium(0) complexes catalyzed the coupling of aryl and heteroaryl iodides to dimethoxypyridazene-zinc halides [20] and the coupling of α-bromopyridines to α-metallated (ZnBr) pyridines [21].

Palladium(0) complexes catalyzed the coupling of aryl and alkenyl triflates to (α-ZnCl)(β-methoxy) acrylates [22], organozinc halides to α,β-dibromoacrylates [23], isoprenylzinc bromide to β-bromobutenolides [24], and organozinc halides to 5-bromo-1,2-,3,4-dienals [25]. Long polyenes and polyunsaturated esters were synthesized by the palladium(0) catalyzed coupling of terminal bromopolyenes with polyenezinc halides [26]. γ-Methylenebutenolides were prepared by palladium(0) catalyzed coupling of alkynylzinc chlorides with β-bromoacrylates followed by lactone formation [27].

Palladium catalyzed coupling of alkylzinc halides with alkenyl halides has been used in complex synthetic procedures (Eq. (3)) [28]. Both nickel(0) and palladium(0) complexes catalyzed the coupling of cyclopropylzinc halides to aryl and benzyl iodides [29]. Nickel(II) acetylacetonate catalyzed the coupling of functionalized arylzinc halides with primary alkyl iodides in the presence of *para*-trifluoromethylstyrene as a promoter of the reductive elimination step [30].



(3)

Palladium(0) catalyzed the alkylation of dibromofuranoate esters by allyl, alkynyl, and aryl metals (ZnCl, $SnBu_3$, CuI) [31]. Bromomethylpyrimidines were coupled to aryl and alkenyl halides by conversion to the zinc halide derivative and subsequent palladium(0) catalyzed coupling [32]. Palladium(0) catalyzed the coupling of aryl halides and 2,2-difluoro-1-bromoethylzinc chloride [33], the conversion of β-nitrostyrene to β-alkyl styrenes by organozinc halides [34] and the synthesis of phenyl, homophenyl, and bishomophenyl alanines by the coupling of terminal iodozinc derivatives of protected aliphatic aminoacids with aryl halides [35]. α-Bromophenylzinc iodide was dialkylated by the palladium(0) catalyzed coupling with aryl, alkenyl, alkynyl, and aroyl halides, followed by coupling at the bromo position to arylzinc halides [36].

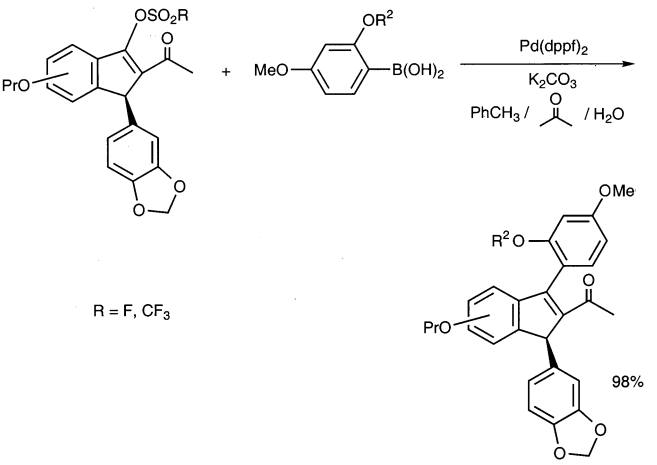
Suzuki coupling continued to be developed into a powerful synthetic tool. In a careful mechanistic study of $Pd(dba)_2$ catalyzed coupling process, the reactivity of various phosophine-palladium complexes was addressed [37]. The transmetallation step in Suzuki coupling occurs with retention [38,39]. Phosphine-free catalysts for

Suzuki coupling have been developed [40] as have orthopalladate triarylphosphite catalysts [41].

The development of solid supported Suzuki coupling advanced with the use of soluble PEG supports for biaryl synthesis [42], polystyrene bound aryl iodides for biaryl synthesis [43], polymer bound Suzuki coupling to make isoxazolinoisoquinolines [44], and the coupling of 4-bromopyridines to aryl boronates [45]. Arylsilane-based linkers have been developed for solid phase Suzuki biaryl coupling [46]. Silica supported palladium catalysts coupled organic halides to main group organometallics in HMPA [47].

The Suzuki coupling was used to synthesize chiral 1,1′-binaphthols [48], and to couple aryl bromides with 3,3′-bis-binolboronic acids [49]. Catalysts to couple aryl chlorides to arylboronic acids have been developed [50,51]. Palladium(0) catalyzed the arylation of 3,5-dimethoxyboronic acids [52], 2-(oxazolino)arylboronic acids [53], nitroaryl triflates by arylboronic acids [54], 2-naphthyltriflates by arylboronic acids [55], as well as 2-naphthyl boronic acids by aryl halides promoted by cesium fluoride [56]. 2,6-Ditertiarybutyl phenol formed biphenyls when treated with aryl bromides, triphenylborane, and palladium acetate [57].

Nickel(0) complexes catalyzed the coupling of aryl mesylates with aryl boronic acids [58] while palladium(0) complexes coupled aryl lead triacetate with aryl boronic acids [59]. Suzuki coupling was used to synthesize 2,5-diaryl-3,6-dimethoxyphenols in the synthesis of terprenin [60]. More complex biaryl couplings are shown in Eq. (4) [61] and Eq. (5) [62].



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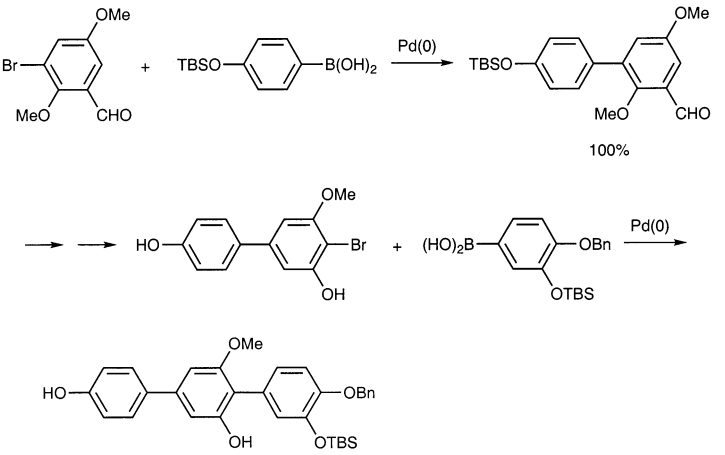
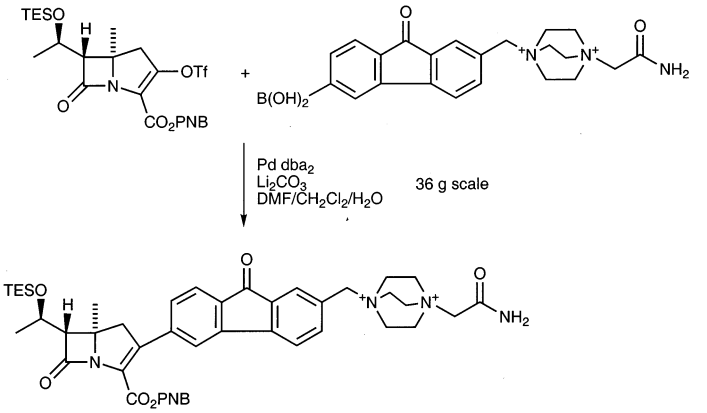


(5)

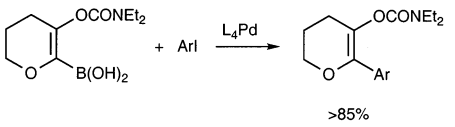
Palladium(0) catalyzed the coupling of protected 2-bromopyrroles to arylboronic acids [63], aryl and alkenyl halides and triflates to imidazole-4-boronic acids [64], as well as the arylation of vinyl triflates of thienamycin (Eq. (6)) [65].



(6)

Palladium(0) coupled α-bromopyridines to sterically hindered arylboronic acids [66], arylboronic acids to 2-pyridone-4-triflates [67], and aryl halides to 2-boronic acids of pyrroles and indoles [68]. Suzuki coupling was used to prepare 3-substituted [69] and 5-, 6-, and 7-substituted indoles [70]. Palladium(0) catalyzed the coupling of 4-iodoindoles to 2-(triethylborono)indole [71].

Palladium(0) catalyzed the coupling of aryl diazonium salts with vinyl trifluoroborates [72] as well as the couplings shown in Eq. (7) [73], Eq. (8) [74], and Eq. (9) [75], the coupling of α-silylvinylboranes with aryl halides [76], arylboronic acids with β-bromobutenolides [77], and the coupling of resin-bound vinyl halides with aryl- and alkylboronic acids (Eq. (10)) [78].



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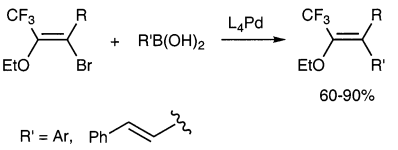

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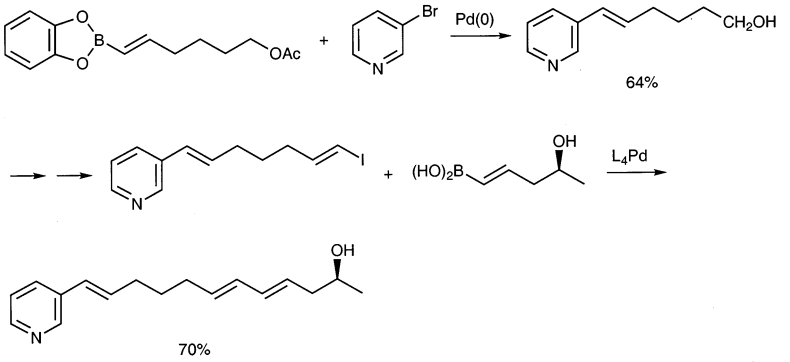

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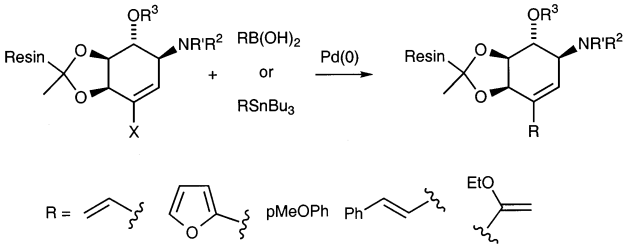
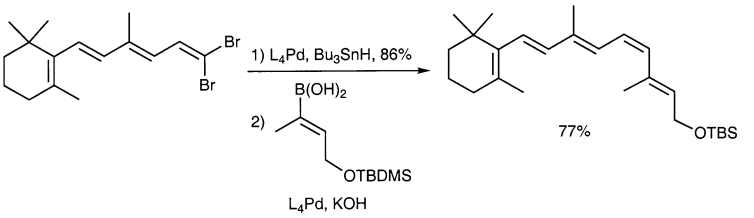


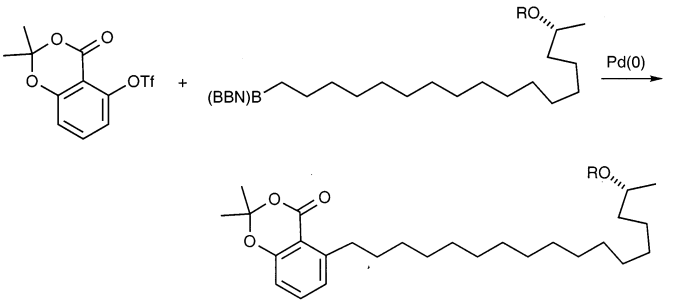
(10)

Nickel complexes catalyzed the coupling of sterically hindered alkenyl bromides with alkenylboronic acids [79], while palladium(0) complexes catalyzed the coupling of halopolyenes with alkenylboronic acids [80], (Eq. (11)) [81].



(11)

Chlorinated diarylmethanes were synthesized by the nickel(0) or palladium(0) catalyzed coupling of benzyl halides with arylboronic acids [82]. Palladium(0) complexes catalyzed the coupling of alkylboronic acids to aryl halides [83,84] and aryl triflates (Eq. (12)) [85]. Suzuki coupling was used to synthesize tetrazole aminoacids (Eq. (13)) [86].



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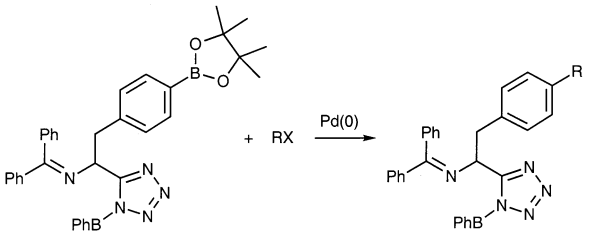
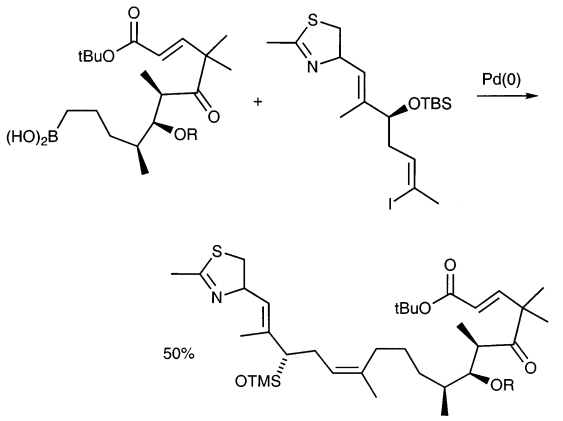


(13)

(β-Bromo)methyl methacrylate coupled to cyclopropylboronic acids under palladium(0) catalysis [87]. Suzuki coupling of alkylboronic acids was effective with highly functionalized systems (Eq. (14)) [88] and (Eq. (15)) [89].



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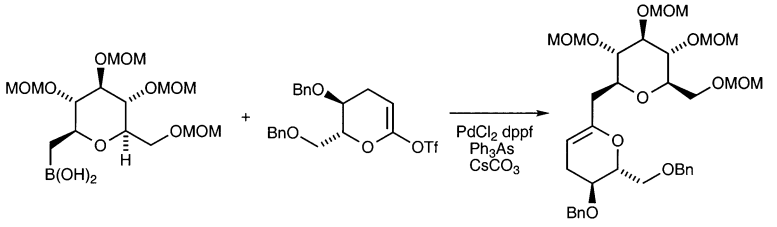
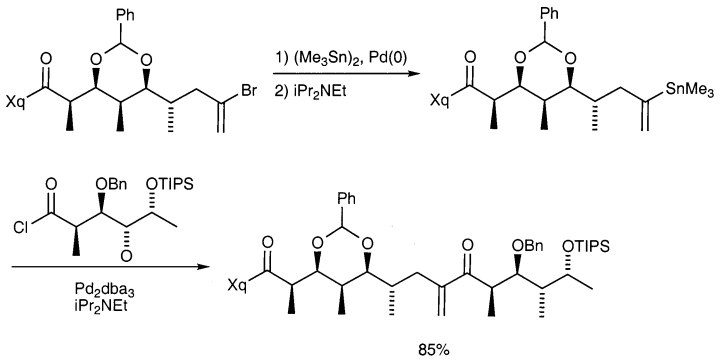


(15)

Palladium(0) catalyzed the coupling of allylboranes with aryl halides [90] and the methylation of long chain fatty ester boranes with ^{11}C labelled methyl iodide [91]. Suzuki coupling was used to append aryl groups at the five position for supramolecular assembly [92] and to surface functionalize polyether dendrimers [93].

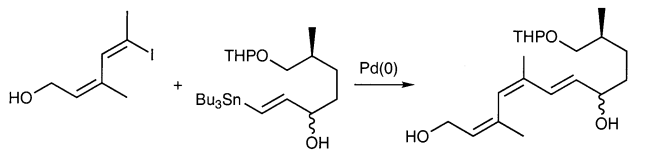
Stille coupling also continued to be used extensively in organic synthesis. The mechanism of transmetallation in the Stille coupling was thought to involve a 4-centered transfer of the alkyl group from Sn to Pd and the halide from Pd to Sn [94]. Low ligand to palladium ratios allowed the Stille coupling of very hindered stannanes with aryl iodides. The use of triflates resulted in cine substitution [95]. The use of the $CF_3(CF_2)_3SO_2$ group in place of triflate for Stille (and other Pd catalyzed processes) resulted in high yields and clean reactions [96]. Activated monoorganotin reagents $[RSn(X)CN[TMS)_2)_2]$ have been developed for Stille coupling [97]. The energetics of transmetallations of 2-furyltributyl tin to palladium(II) was assessed [98]. Copper(I) iodide catalyzed the alkylation of alkynyl iodides with stannanes [99]. Palladium(0) catalyzed the stannylation of alkenyl iodides followed by the alkylation of acid chlorides (Eq. (16)) [100].



(16)

Palladium complexes catalyzed the alkylation of β-iodo-β-trifluoromethyl acrylic esters by alkenyl and alkynyl tin reagents [101], the alkylation of 1-iodo allyl peroxyethers by alkenyl tin reagents (as well as Heck, Sonogashira, and carbonylation reactions) [102] and the coupling of alkenyl stannanes with β-iodoacrylic acid to give dienoic acids [103]. 1-Iodocyclohexa-1,3-diene-5,6-diol underwent Stille coupling with a wide range of organostannanes but in low yield [104].

Complex polyenes were made by Stille coupling (Eq. (17)) [105]; (Eq. (18)) [106]; (Eq. (19)) [107]; (Eq. (20)) [108]; (Eq. (21)) [109].



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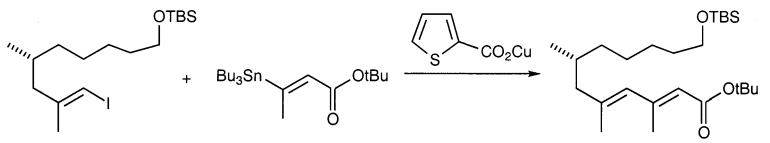

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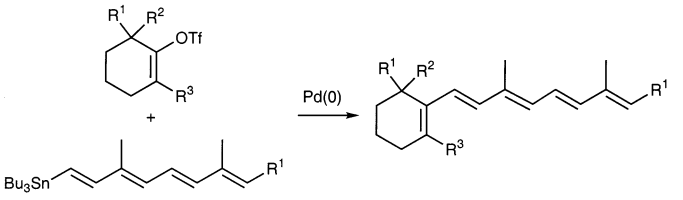

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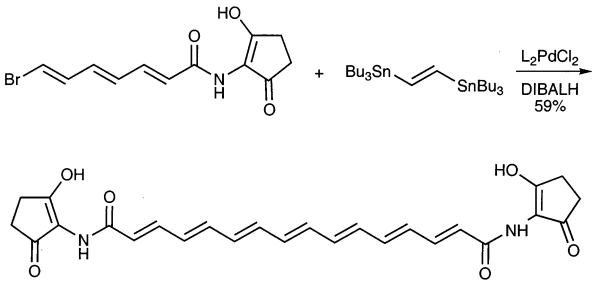

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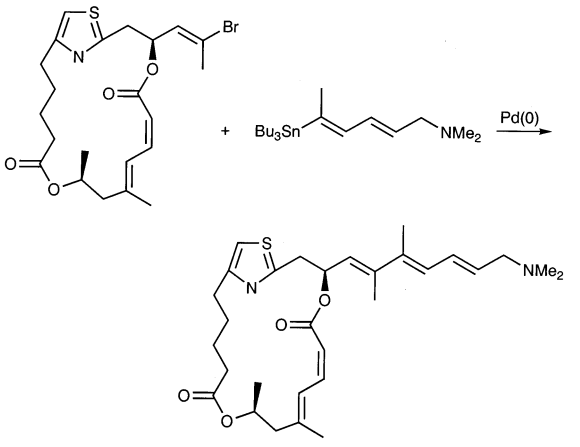
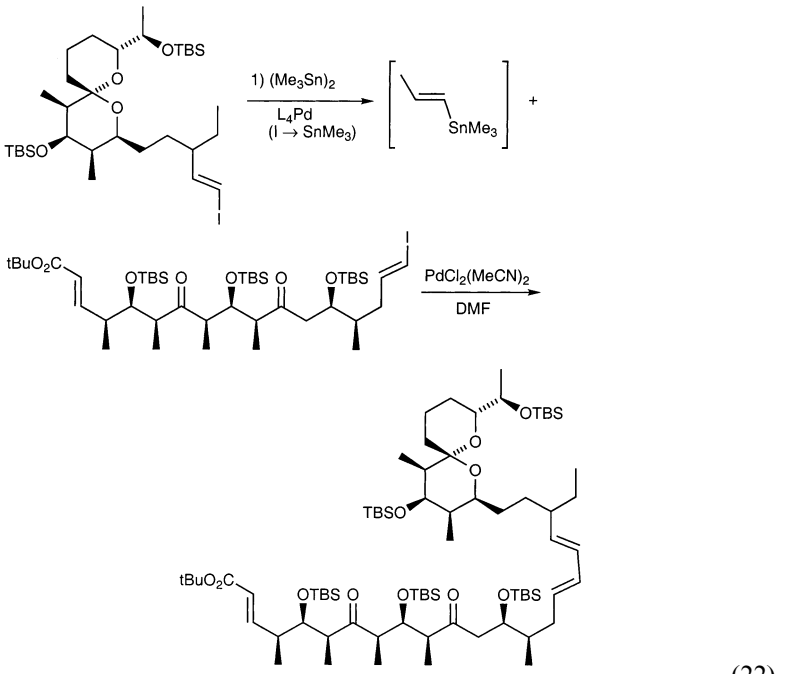


(21)

Stille coupling was used to couple alkenyl [110] and alkynyl stannanes [111] to vinyl triflates and halides of cephalosporin and thienamycin β-lactams. Complex inter- (Eq. (22)) [112] and intramolecular Stille reactions (Eq. (23)) [113] and (Eq. (24)) [114] are shown on the next page.



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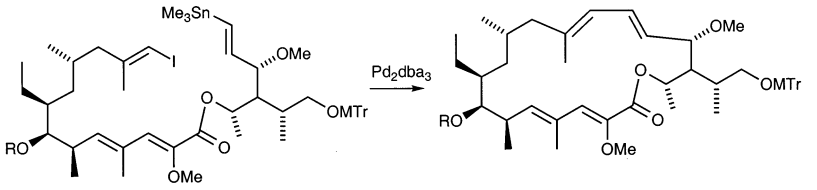

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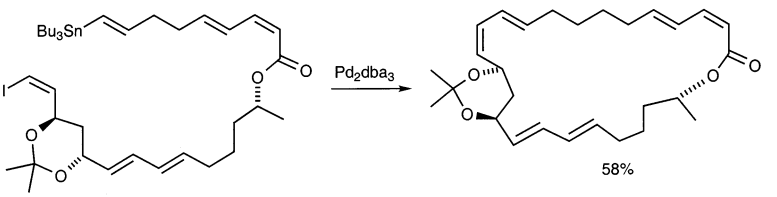
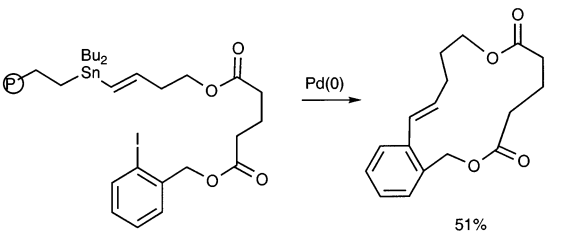


(24)

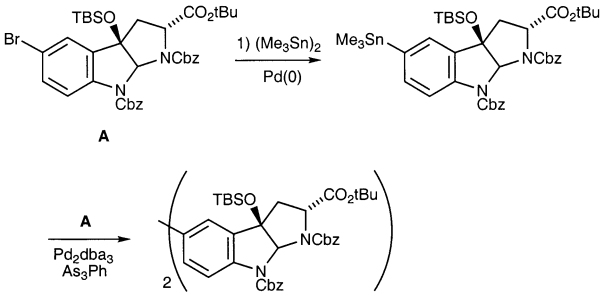
Palladium(0) catalyzed the coupling of F^{18} labelled para iodobenzene with aryl and vinyl stannanes [115] and the diarylation of *gem*-bis-stannylalkenes with aryl halides [116]. Aryl iodides coupled with aryl, alkenyl and alkynyl stannanes in the

presence of 10% copper(I) iodide/sodium chloride [117]. Stille coupling was used to convert aryl triflates into styrenes with vinylstannanes [118]. Intramolecular Stille coupling was used to both form a macrocycle and cleave it from a resin (Eq. (25)) [119].



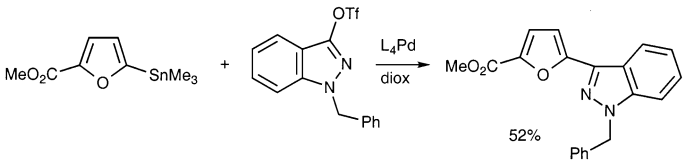
(25)

Palladium(0) complexes catalyzed the coupling of 2-chloropyridines with alkenylstannanes [120], 2-iodo-4-aminocyclohex-2-enone with 3-tributylstannylpyridines [121], aryl iodides with 2-stannyl benzofurans [122], and 4-bromo-2,2-dipyridines with polycyclic aromatic $-Sn(F_2)[(N(TMS)_2]_2$ stannates [123]. Palladium(0) catalyzed the dimerization of complex indolines (Eq. (26)) [124]. Nickel(0) complexes catalyzed the coupling of stannanes with aryl halides [125].



(26)

Bipyridines were made by the palladium(0) catalyzed coupling of 2-bromopyridines with 2-stannylpyridines [126]. Palladium(0) catalyzed the coupling of acid chlorides, aryl iodides and heteroaryl iodides with stannyl triazoles [127], and the coupling of 3-iodoindole with stannylated 2-thioimidazoles [128]. Other more complex heteroaryl Stille couplings are shown in Eq. (27) [129], Eq. (28) [130], and Eq. (29) [131]. Stille coupling was extensively used in the synthesis of the macrolactins (Fig. 1) [132]. Stille [133] and Suzuki [134] couplings of halobenzoates on polymer supports, and of alkenyl stannanes with polymer supported β-bromoacrylamides [135] were catalyzed by palladium(0).



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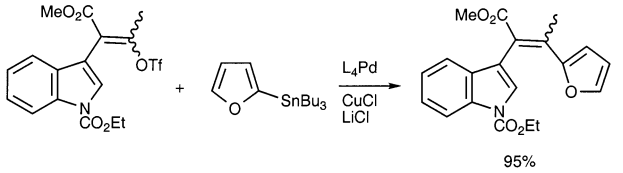

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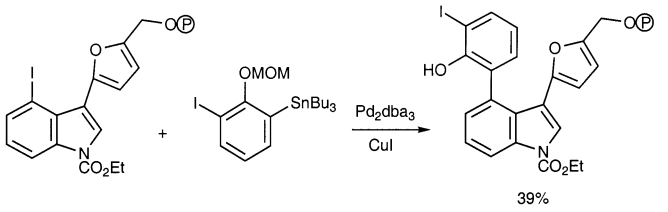


(29)

A review entitled 'Catalytic Cross-Coupling Reactions in Biaryl Synthesis' (311 references), covering Negishi, Stille, and Suzuki cross coupling has appeared [136]. Palladium catalyzed the coupling of a complex alkenyl iodide with an alkenylmercury complex (Eq. (30)) [137]. Zirconacyclopentadienes alkylated o-bisbromomethylarenes to give benzfused cyclooctadienes [138] in the presence of copper chloride. Cleavage with an aryl iodide in the presence of two equiv. of copper(I) chloride gave a 1-aryl-1,3-butadienyl-4-cuprate [139]. Palladium(0) complexes catalyzed the coupling of aryl, aroyl, allyl, and benzyl halides with acyl zirconocene complexes [140]. Epoxides were alkylated by alkylzirconocene chloride (Eq. (31)) [141]. Heteroaryl benzyl chlorides were alkylated by alkenylalanes in the presence of nickel(0) catalysts [142].

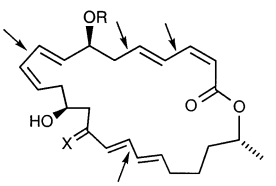
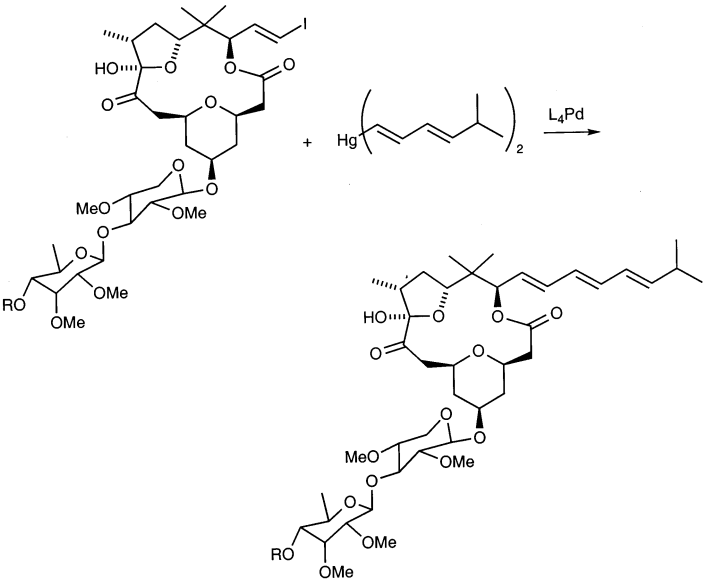


Fig. 1.



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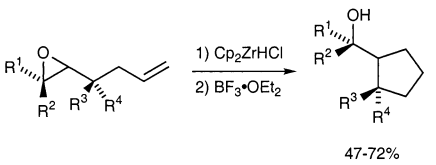
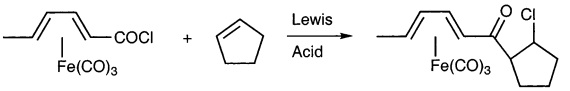


(31)

2.1.2. Alkylation of acid derivatives

Palladium complexes catalyzed the alkylation of chlorocarbamates by aryl and alkynylstannanes [143], and along with Zn/Cu couple the alkylation of acid halides by β-iodopropionates [144]. Benzylmanganese halides alkylated acyl halides [145]. Palladium(0) complexes catalyzed the alkylation of acid chlorides by heteroaryl zinc halides [146]. Iron complexed dienoyl chlorides were alkylated by alkenes (Eq. (32)) [147]. Rhodium(II) acetate catalyzed the addition of acid chlorides to diazodiketones (Eq. (33)) [148].



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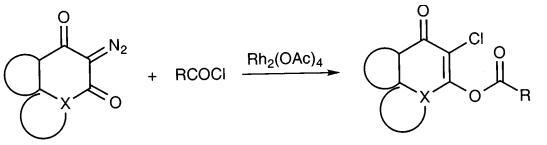


(33)

2.1.3. Alkylation of olefins

The Heck reaction is the most common method for the alkylation of olefins and its development and use continued unabated. 'Variations on a theme — Recent Developments on the Mechanism of the Heck Reaction and Their Implications for Synthesis' was the title of a review (32 references) [149]. A survey of reaction conditions of Heck and other palladium catalyzed reactions indicated that solvent choice may be more important than ligand choice [150].

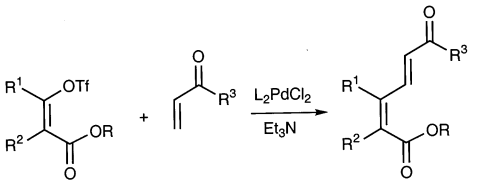
Many new conditions, ligands, and catalysts, have been developed for use in Heck reactions. These include the use of phosphites as ligands [151], the use of $Pd(OAc)_2$ or $PdCl_2(MeCN)_2$/*N*,*N*-dimethylglycine as ligandless Heck catalysts with aryl bromides [152], the use of preformed (but not in situ formed) chelating diphosphine catalysts [153], and the use of biphasic (ethylene glycol/toluene) conditions with sulfonated phosphine ligands [154].

A new catalyst for unreactive Heck systems (e.g. PhCl) consisted of 0.05% $Pd(MeCN)_2Cl_2{\cdot}6Ph_4PCl$ plus *N*,*N*-dimethylglycine [155]. Orthopalladated tris(2,4-ditertiarybutyl)phosphine was a highly active Heck catalyst [156]. Aroyl anhydrides [157] and organolead triacetates [158] served as sources of R groups for Heck reactions. Supercritical CO_2 was a good solvent for Heck [159,160] as well as Stille [160] couplings. Aryl diazonium salts were more reactive in the Heck reaction than were aryl iodides and these groups could be sequentially replaced [161]. Aryl dimethylsilanols were effective aryl group providers in the Heck arylation of alkenes in the presence of palladium(II) acetate, copper(II) acetate and lithium acetate [162] as were aryl iodonium salts [163].

The mechanism of palladium catalyzed carbopalladation/functionalization of allenes has been studied [164]. Supported Heck catalysts involving palladium grafted onto molecular sieve [165] and Heck reactions of Wang resin-bound *p*-iodophenols have been developed [166].

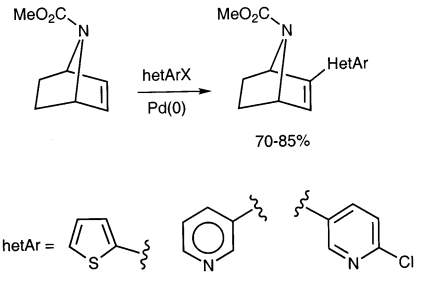
Palladium on carbon catalyzed the Heck alkylation of alkenes by bromoadamanatane [167]. Aryl triflates added to the internal carbon of allyl silanes under Heck conditions [168], whereas terminal alkylation of 2-trifluoromethyl allyl alcohols by aryl halides occurred under Heck conditions [169]. Ortho iodoaniline arylated homoallylic alcohols [170], as well as protected allylic diols [171] at the terminal position in the presence of palladium. Heck arylation of 2-carbomethoxy allyl alcohols produced arylated β-ketoesters by β-elimination towards the OH group to give the enol [172]. Orthogonally protected bis-armed amino acids were prepared by sequential Heck arylation of two different protected dehydroaminoacid esters with iodobromoarenes [173]. Vinyl glycines were β-arylated to give styryl glycines under Heck conditions without epimerization of the α-position [174].

Palladium(0) catalyzed arylation of silylketene acetals in the presence of tributyltin fluoride gave aryl acetic acids [175]. 5,5′-Diiododibenzofuran was bis olefinated using Heck olefination chemistry [176]. Palladium(0) complexes catalyzed the olefination of vinylogous triflates (Eq. (34)) [177].



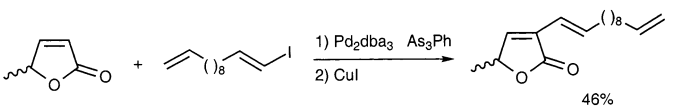
(34)

Unsaturated esters were arylated by 4-iodo- [178] and 2-thio-5-iodoimidazoles under Heck conditions [179]. Palladium(0) catalyzed olefination of α-bromocoumarins by conjugated esters, dihydrofurans, allyl and propargyl alcohols was efficient [180]. Heck arylation of [2.2.1]-bicyclo alkaloids occurred from the exo-face (Eq. (35)) [181].



(35)

Heck arylation of 2,3-dihydrofuran with aryl iodides gave a 1:1 mixture of the arylated 2,3- and the 2,4-dihydrofuran in the presence of most bases, but in the presence of tetrabutylammonium acetate a 3:97 mixture was obtained [182]. Palladium(0)/copper(I) catalyst systems were used to olefinate butenolides with unexpected regiochemistry (Eq. (36)) [183]. α-Hydroxy- and α-alkoxy cyclohexenones were β-arylated under Heck reaction conditions [184]. Other Heck reactions of heterocycles are shown in (Eq. (37)) [185] and (Eq. (38)) [186].



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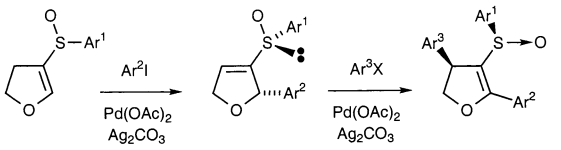
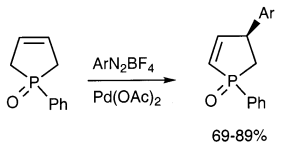
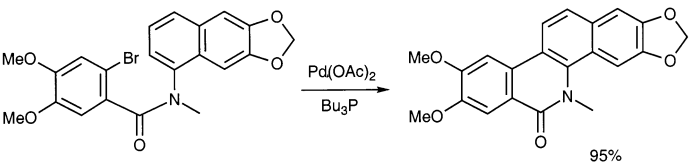


(37)



(38)

Even aromatic systems can undergo Heck type arylation (Eq. (39)) [187]; (Eq. (40)) [188]; (Eq. (41)) [189], and (Eq. (42)) [190].



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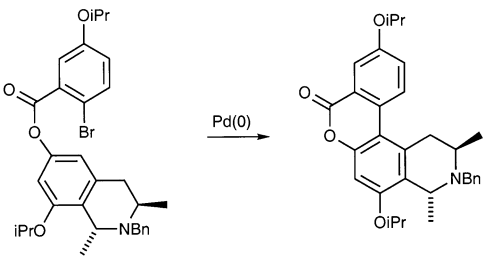
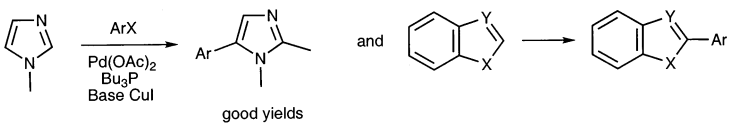
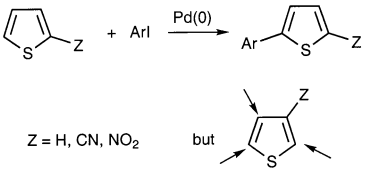


(40)

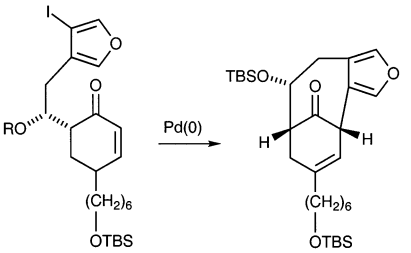


(41)

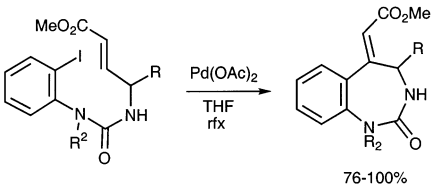


(42)

Intramolecular Heck reactions continued to be extensively used in synthesis (Eq. (43)) [191]; (Eq. (44)) [192]; (Eq. (45)) [193]; (Eq. (46)) [194]; (Eq. (47)) [195]; (Eq. (48)) [196]; (Eq. (49)) [197,198]; (Eq. (50)) [199], (Eq. (51)) [200], and (Eq. (52)) [201].



(43)



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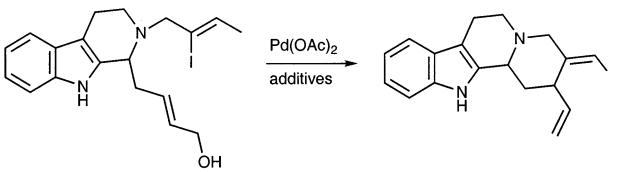

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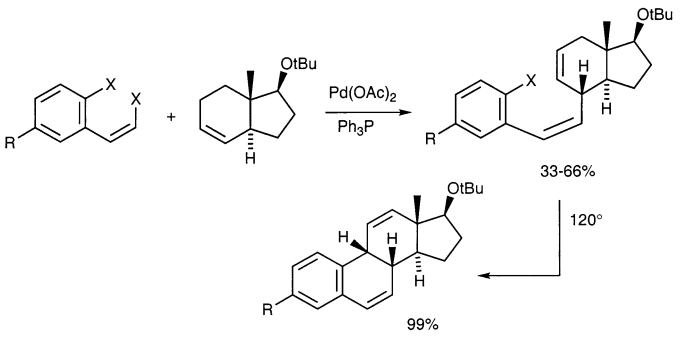
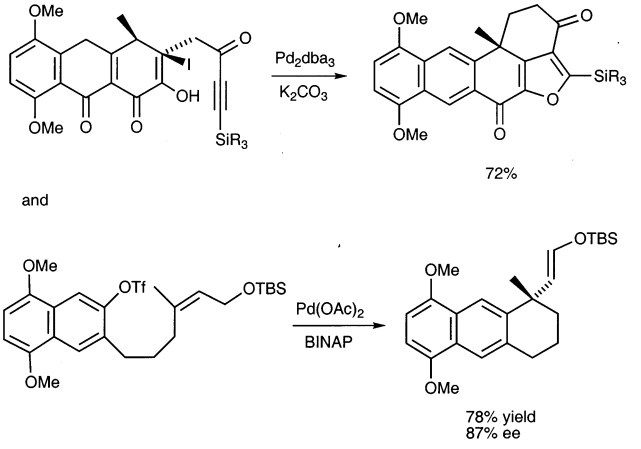
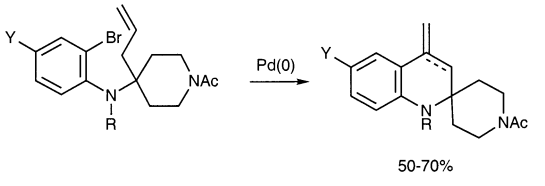

OtBu
X
X
R
+
OtBu
H
Pd(OAc)2
Ph3P
OtBu
X
H
H
R
33-66%
OtBu
120°
H
H
H
R
99%

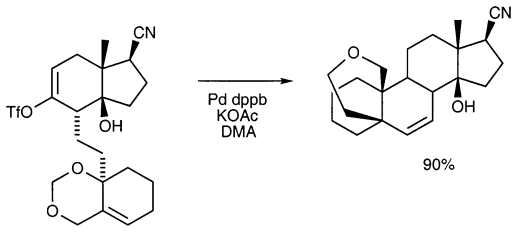
(46)


OMe
I
O
OH
OMe
O
O
SiR3
Pd2dba3
K2CO3
OMe
O
SiR3
O
OMe
O
72%
and
OMe
OTf
OTBS
OMe
Pd(OAc)2
BINAP
OTBS
OMe
OMe
78% yield
87% ee

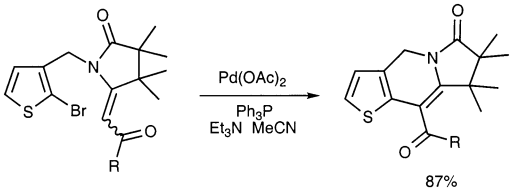
(47)


Y
Br
NAc
N
R
Pd(0)
Y
N
R
NAc
50-70%

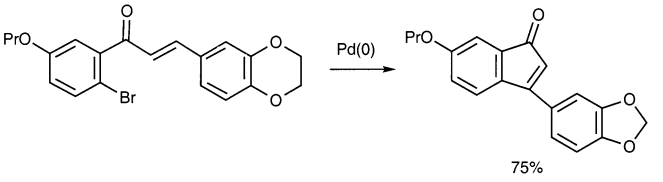
(48)



(49)



(50)



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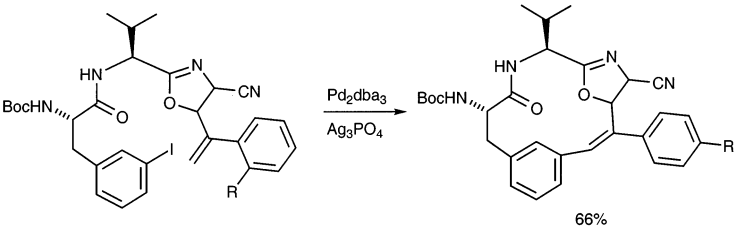
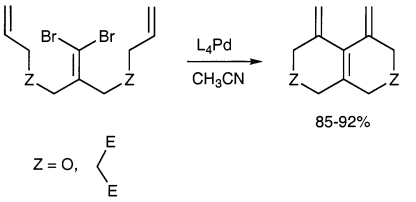


(52)

Vicinal dihaloalkenes underwent bis-Heck olefination [202] (Eq. (53)) [203]. Double alkene insertions could also be effected both intra- (Eq. (54)) [204] and intermolecularly [205]. A related transmetallation-insertion process went with low asymmetric induction (Eq. (55)) [206].



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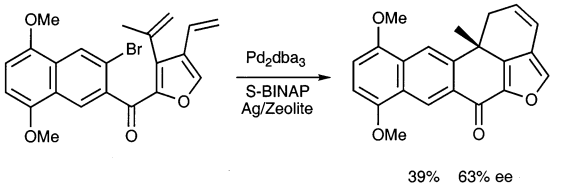

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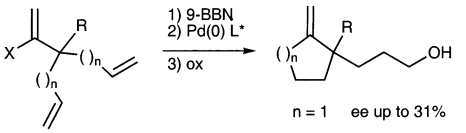
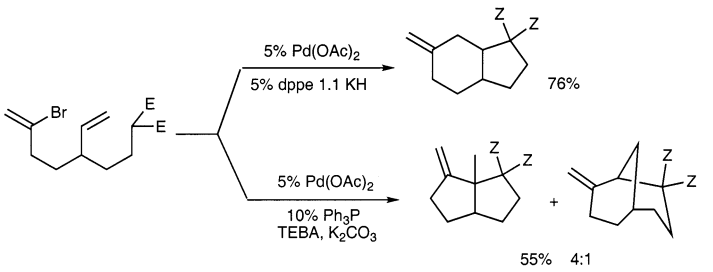
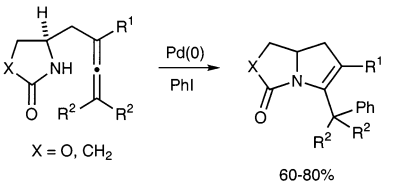


(55)

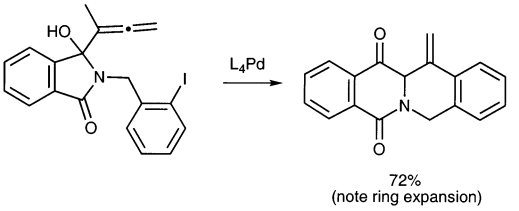
Norbornene [207] and norbornadiene [208] were dialkylated by combined Heck type alkylation of the alkene followed by trapping the σ-alkylpalladium insertion product by transmetallation from tin. Heck type olefination could also be intercepted by nucleophiles both intra- (Eq. (56)) [209,210] and intermolecularly [211]. Allenes also participated in similar reactions (Eq. (57)) [212,213] and (Eq. (58)) [214].



(56)



(57)

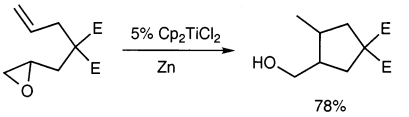


(58)

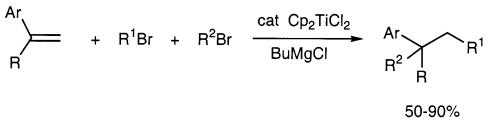
Palladium complexes catalyzed the alkylation of alkenyl bromides by enol esters [215]. α,β-Unsaturated aldehydes and ketones underwent γ-arylation when treated with aryl bromides, palladium acetate and cesium carbonate [216]. Ring closure of enynes by chloropalladation of the alkyne followed by insertion of the alkene has been reviewed (39 references) [217].

Ruthenium complexes catalyzed the alkylation (addition) of alkenes by the CH_2 group of *N*-benzylamines [218] and the β CH group of an α,β-unsaturated enone [219]. π-Allylnickel halides catalyzed the addition of ethylene to styrenes to give 3-aryl-1-butenes [220]. Nickel bromide/Zn catalyzed the alkylation of 2,3-dimethyl butadiene by β-iodoenones, to give 1,4-disubstituted butadienes [221].

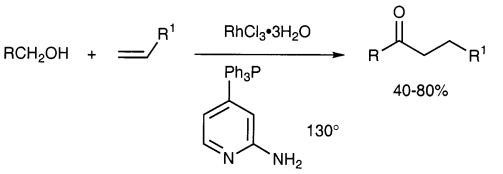
Chiral zirconocene dichloride catalyzed the addition of ethyl Grignard reagents to terminal alkenes to give optically active 2-ethyl-terminal Grignard reagents in up to 79% ee [222]. Chiral dimethylzirconocenes catalyzed the carboalumination of allyl benzene. Oxidation of the resulting terminal organoaluminum complex led to 2-methyl-3-phenyl propanol in 63% ee [223]. Unusual olefin alkylations are shown in Eq. (59) [224], Eq. (60) [225], and Eq. (61) [226].



(59)



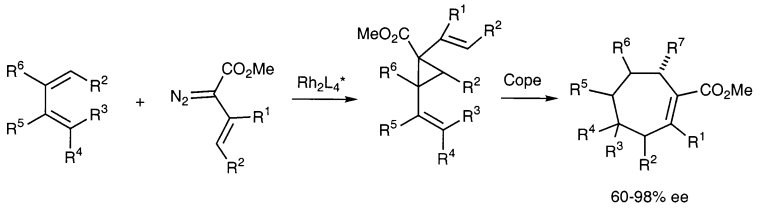
(60)



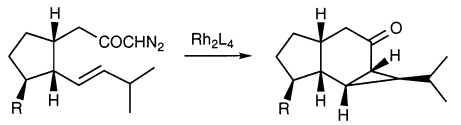
(61)

2.1.4. Metal-catalyzed diazo decomposition and other cyclopropanations

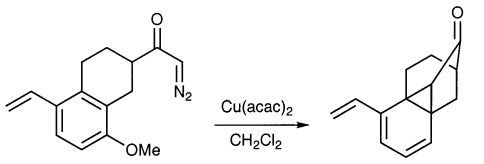
New aspects of catalytic (Rh) asymmetric cyclopropanation has been reviewed (111 references) [227]. The de of the cyclopropanation of styrene by α-diazophenylacetic acid was not dependent on the ligands on rhodium [228]. Copper(I) triflate catalyzed the asymmetric cyclopropanation of alkenes by ethyl diazoacetate in the presence of planar chiral C-2 symmetric bis-azaferrocene ligands [229]. Chiral rhodium(II) complexes catalyzed the cyclopropanation of enol ethers by α-diazo-β,γ-unsaturated esters with high ee [230,231]. With dienes, a tandem cyclopropanation-Cope rearrangement was developed (Eq. (62)) [232]. The diethylketal of propargaldehyde was cyclopropenated by ethyl diazoacetate with high ee in the presence of chiral rhodium(II) catalyst [233]. Other cyclopropanations are shown in Eq. (63) [234], Eq. (64) [235], and Eq. (65) [236], along with two unusual cycloadditions (Eq. (66)) [237] and (Eq. (67)) [238].



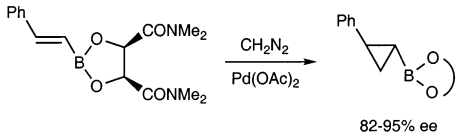
(62)



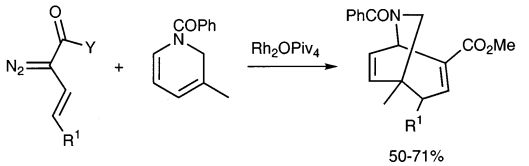
(63)



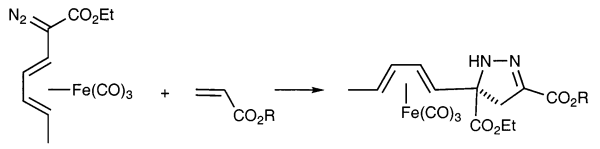
(64)



(65)

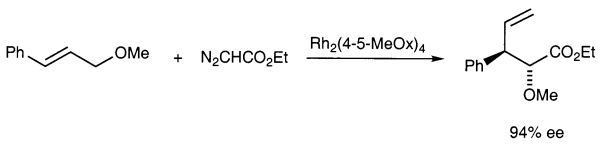


(66)

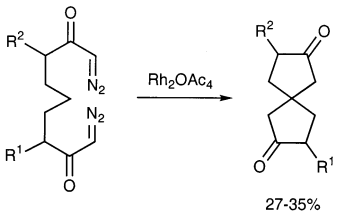


(67)

The electronic effects of rhodium(II) mediated carbenoid intramolecular CH insertion reactions were studied [239]. With allyl ethers, allylic transposition occurred (Eq. (68)) [240]. Rhodium catalyzed C–H insertion was particularly effective for making five membered rings [241–244]; (Eq. (69)) [245,246].

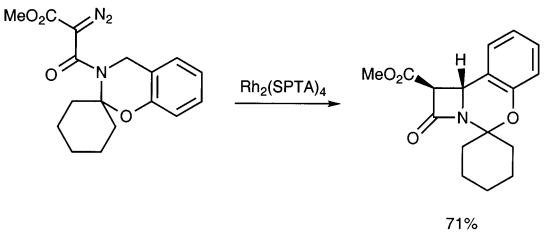


(68)



(69)

Chiral copper(I) complexes catalyzed the carbenoid insertions into Si–H–bonds with high de [247]. Other useful processes are shown in Eq. (70) [248] and Eq. (71) [249].



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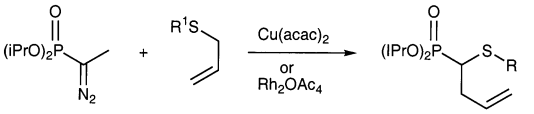
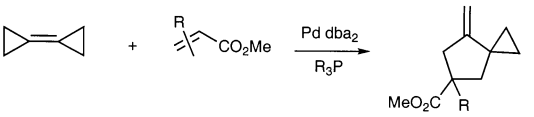


(71)

2.1.5. Cycloadditions

Palladium(II) complexes catalyzed the 1,3-dipolar cycloadditions of nitrones to enol ethers [250], as well as Diels-Alder reactions [251]. In the presence of BINAP high ee was obtained. Palladium(0) catalyzed the cycloaddition in Eq. (72) [252]. Reduced cobalt species catalyzed [2 + 2 + 4] cycloadditions (Eq. (73)) [253]. Higher order photocycloadditions of 1,3-dienes to chromium complexed cyclohexatriene continued to be exploited [254,255] (Eq. (74)) [256]. Cycloadditions of cyclobuta-

diene were achieved by oxidation of the corresponding iron complexes (Eq. (75)) [257].



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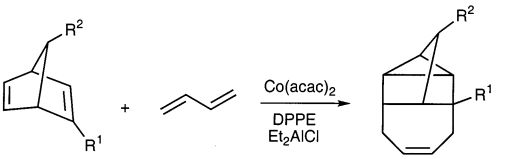

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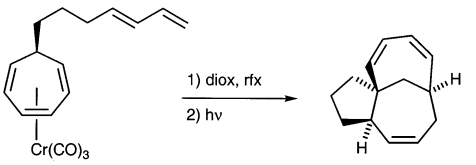
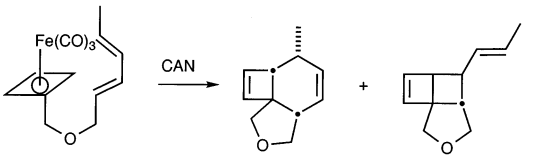


82%

(74)



75% 1/2.6

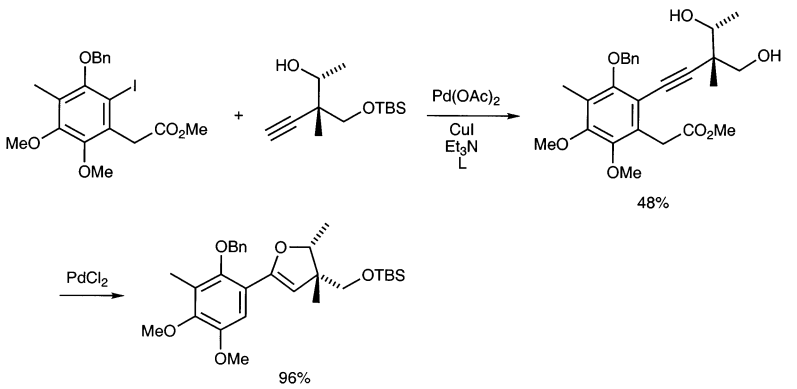
(75)

2.1.6. Alkylation of alkynes

The palladium/copper catalyzed alkylation of alkynes by aryl and alkenyl halides (Sonogashira coupling) continues to be extensively exploited in complex systems. Improved conditions involving in situ liberation of free alkynes from silylalkynes in the coupling of Zn/porphyrin complexes have been developed [258]. The use of THF as solvents in place of amines improved the Sonogashira coupling

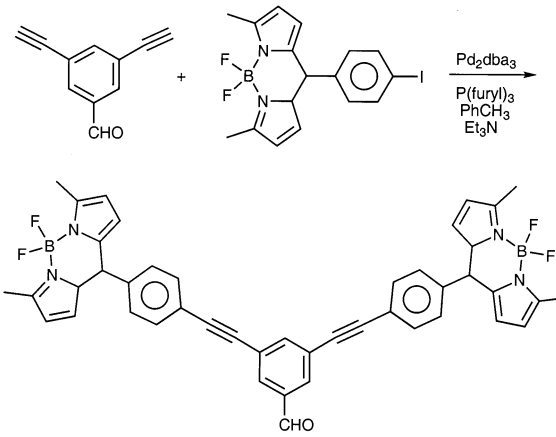
[259]. Biocompatible aqueous conditions for this coupling have been developed [260] and it has been used to introduce alkynes and cyanide into nucleoside bases [261]. Copper-free conditions ($L_4Pd/ZnCl_2/NaI/DMSO/DBU/Et_3N$) have been developed [262]. Acetone has been used to protect alkynes as the acetylene adduct (1,1-dimethylprop-3-yn ol) [263].

Sonogashira coupling has been used to couple aryl iodides to propiolate esters [264], alkynes to bromoporphyrins [265], to oligomerize bis-alkynyl porphyrins with 1,4-diiodoarenes [266], and to form molecular squares of bis-alkyne porphyrins and bis aryl halide containing porphyrins [267]. 4-Iodophenyl alanine was coupled to 2-ethynyl polyhydroxypyranes using palladium(0) catalysis [268]. A more complex example is shown in Eq. (76) [269].



(76)

Terminal alkynes were coupled to heteroaryl halides using 10% palladium on carbon, along with copper(I) iodide [270]. Sonogashira coupling was used extensively to couple alkynes to iodonucleosides [271] including solid-supported couplings [272]. Metal containing oligonucleotides with luminescent properties were prepared by coupling ethynyl nucleosides with brominated bipyridyl complexes of ruthenium [273]. Halogenated phenanthrolines underwent similar coupling to terminal alkynes [274,275]. 4,5-Dibromofurfural was coupled to alkyne terminated fatty acid esters exclusively at the 5-position [276]. Multiple Sonogashira couplings are shown in Eq. (77) [277] and Eq. (78) [278].



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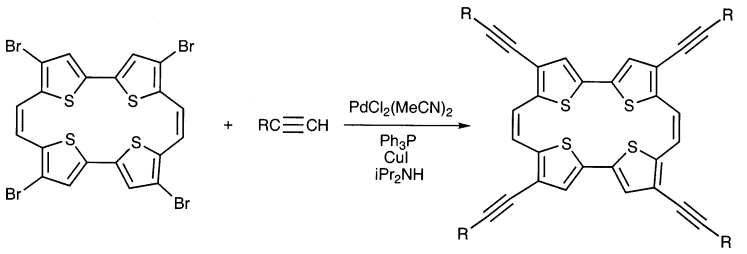
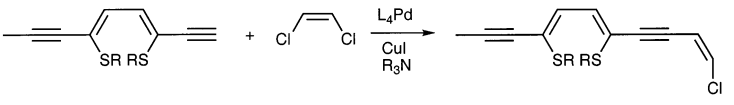


(78)

Liquid crystalline enynes were prepared by the palladium/copper catalyzed coupling of ethynylbenzenes with β-bromostyrenes having long chain aliphatic ethers at the para position [279]. Similar conditions coupled the enol triflate of 4-*t*-butylcyclohexanone to terminal alkynes [280], the diethylketal of β-iodoacrolein with trimethylsilylacetylene [281], and geminal vinyl bromothiols and selenols with terminal alkynes [282]. More elaborate couplings are shown in Eq. (79) [283], Eq. (80) [284], and Eq. (81) [285].



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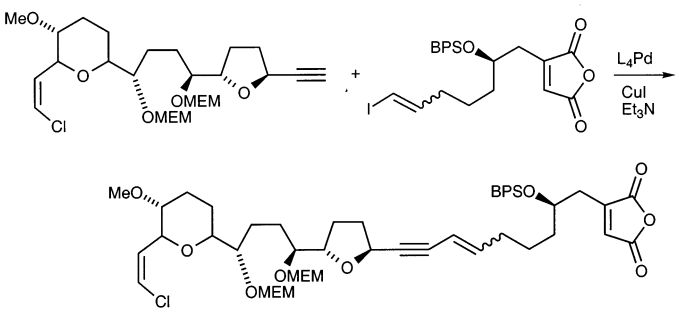

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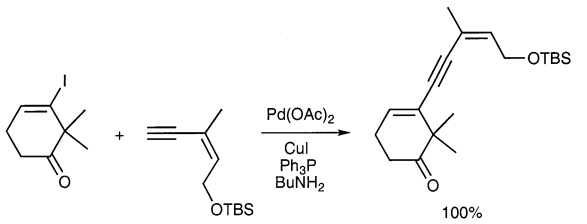
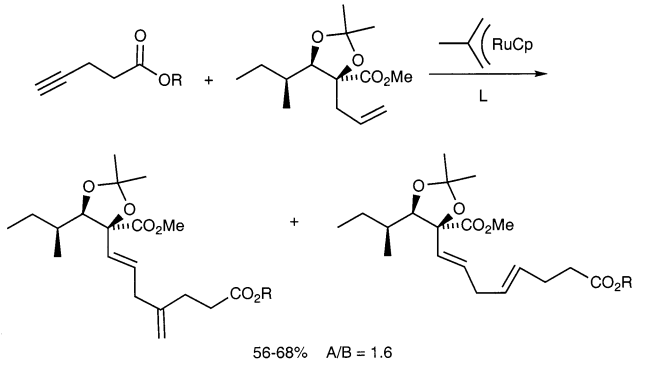


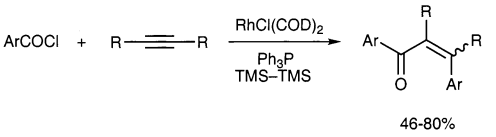
(81)

Palladium(0) in the presence of ammonium formates catalyzed the Heck type β-arylation of propiolamides to produce β-aryl acrylamides [286]. Palladium(0) catalyzed the reaction between 2-bromobiphenyl and alkynes to give phenanthrenes, probably involving insertion of an aryl bond into the initially formed σ-alkenylpalladium complex [287]. Palladium(0) in the presence of acetic acid catalyzed the nucleophilic attack of alkynes at the propargylic position to produce *allyl* products [288]. π-Allylruthenium complexes catalyzed an Alder-ene reaction (Eq. (82)) [289].



(82)

Unusual alkyne alkylations are shown in Eq. (83) [290], Eq. (84) [291], Eq. (85) [292], Eq. (86) [293], and Eq. (87) [294].



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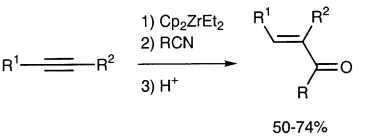
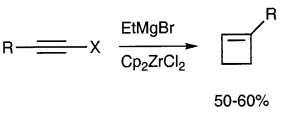
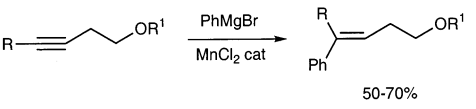


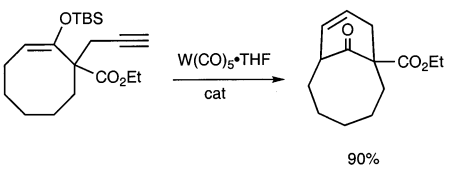
(84)



(85)



(86)



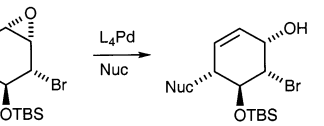
(87)

2.1.7. Alkylation of allyl, propargyl and allenyl systems

Palladium catalysis remained the method of choice for allylic alkylations. Silica supported aqueous phase palladium catalysts for allylic alkylation have been

developed [295]. Palladium catalyzed allylic alkylations in fluorous biphasic systems for ease in recycling catalysts have been developed [296]. A library of phosphino oxazoline ligands for palladium catlayzed allylic alkylation has been synthesized [297]. Appropriately situated ligands (phosphine) in palladium catalyzed allylic alkylation can cause π-allyl complex formation to occur with retention [298]. Addition of lithium iodide to the palladium catalyzed allylic alkylation of secondary allyl acetates results in exclusive alkylation at the primary position (S_N2') [299]. The effect of counterion on the regiochemistry of palladium catalyzed allylic alkylation of dienyl acetates has been probed [300].

Palladium(0) complexes catalyzed the alkylation of allyl carbonates by β-triesters [301], allylic acetates by β-diesters in the absence of base [302], and allyl epoxides by ester enolates [303] and malonates (Eq. (88)) [304]. α-Methylenemalonates were good alkylation agents for allyl carbonates [305,306]. Palladium(0) catalyzed the alkylation of acrolein ketals [307] or ketal esters (Eq. (89)) [308] by stabilized carbanions.



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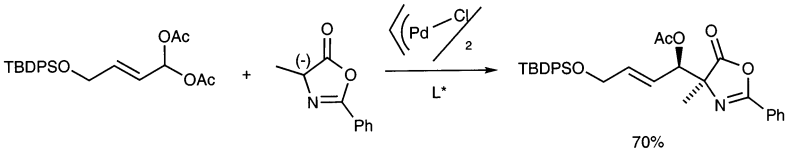
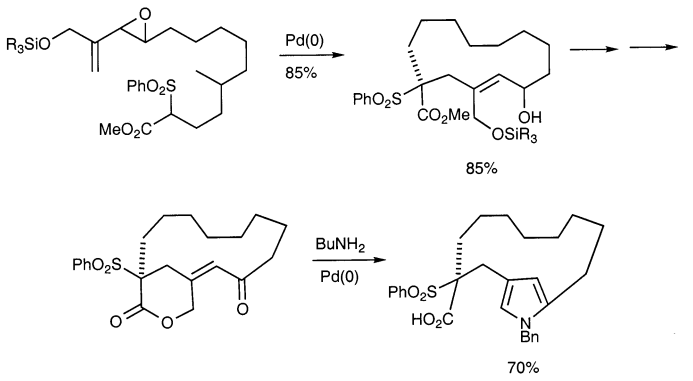


(89)

The stereochemistry of intramolecular allylic alkylation depended on the E,Z stereochemistry of the starting material, the ligand and the reaction time [309]. The regiochemistry depended on the substitution about the double bond [310]. Synthetic applications are shown in Eq. (90) [311] and Eq. (91) [312].



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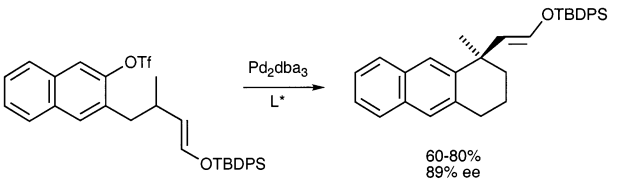
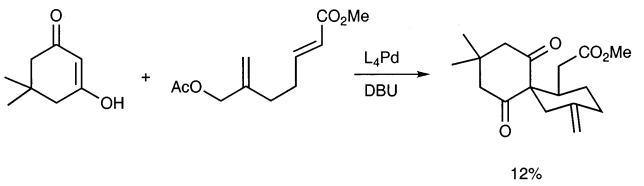


(91)

Palladium(0) catalyzed the reaction (double alkylation) between 1,4-dichloro-2-butene and the carbanion of *N*-cyanomethyl imines to give vinyl cyclopropanes [313,314]. Palladium(0) also catalyzed allylic alkylation/olefin insertion cascade reactions (Eq. (92)) [315]; (Eq. (93)) [316], and (Eq. (94)) [317].



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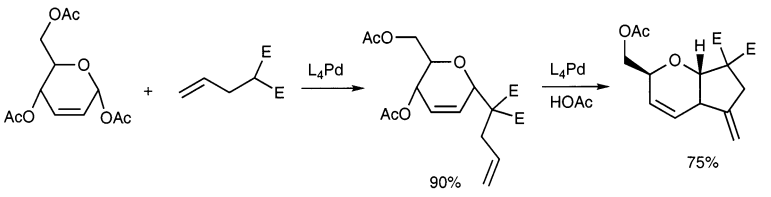
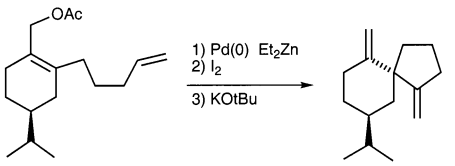
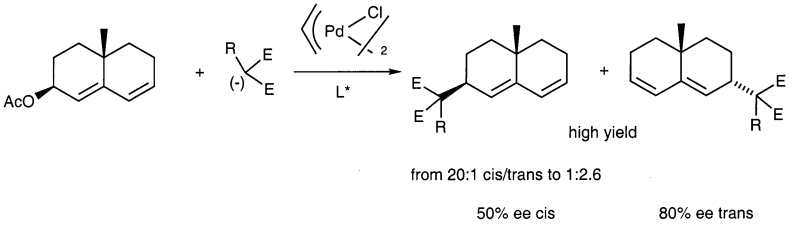


(93)



(94)

The search for chiral ligands to induce asymmetry into palladium(0) catalyzed allylic alkylation continues unabated [318–325]. Unfortunately most of these studies utilized the easiest substrate in which to induce asymmetry (1,3-diphenyl allyl acetate) and for which excellent asymmetric induction has already been achieved. These included the use of MOP ligands on ambiphilic resins in aqueous solution [326]. P–N chelate ligands for asymmetric alkylation of cyclic allylic acetates have been developed [327]. Use of sterically hindered MOP ligands for the allylic alkylation of unsymmetric allyl acetates resulted in attack at the original position of the acetate [328]. Binaphthol/oxazoline phosphite ligands favored internal alkylation of cinnamyl acetate [329], as did the use of molybdenum carbonyl catalysts [330]. In the kinetic resolution of cyclic allylic carbonates by malonates, added chloride accelerated the reaction and made it less selective [331]. Synthetic applications of asymmetric allylic alkylation are seen in Eq. (95) [332], Eq. (96) [333], Eq. (97) [334], and Eq. (98) [335].



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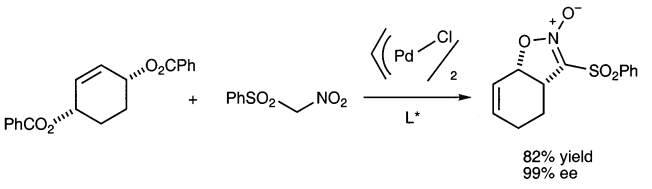

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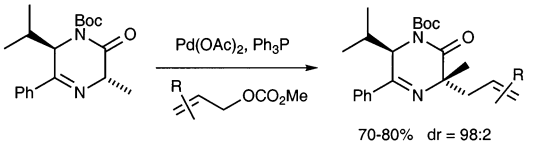

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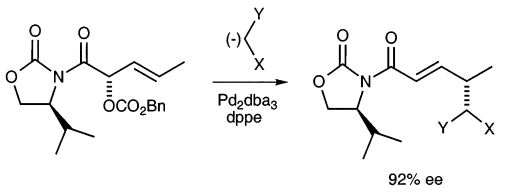
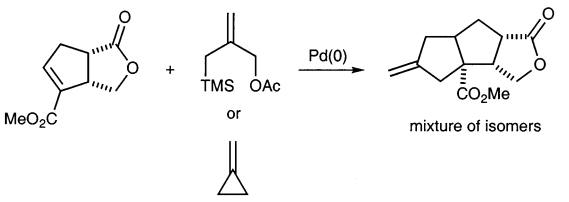
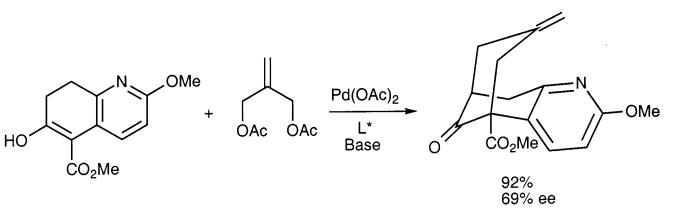


(98)

Related palladium catalyzed trimethylenemethane reactions (Eq. (99)) [336], and (Eq. (100) [337,338] were also useful. Palladium(0) bipyridyl complexes catalyzed the alkylation of allyl acetates by branched ester enolates at the central carbon to give cyclopropanes [339]. An unusual process in shown in Eq. (101) [340].



(99)



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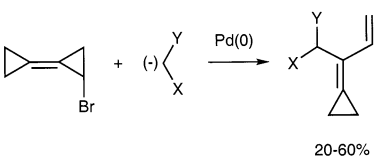
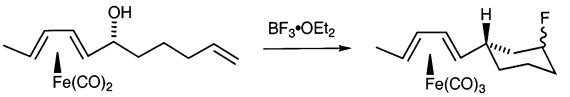


(101)

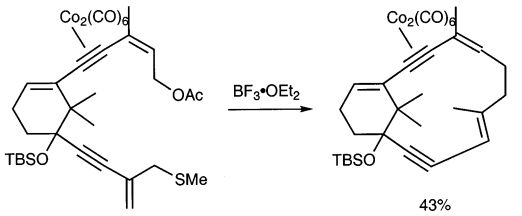
Nickel complexes catalyzed the alkylation of glycal phenyl ethers by Grignard reagents at the 1-position with inversion [341] while palladium catalysts resulted in retention [342]. Chiral nickel complexes catalyzed asymmetric alkylation of ketals of cyclic enones with S_N2' regiochemistry to produce enol ethers [343]. Diaryl- [344] and allylboronates [345] alkylated allyl acetates with inversion in the presence of nickel(II) catalysts. Rhodium(I) complexes catalyzed the alkylation of allyl acetates

at the *more* hindered terminus [346,347] with retention of configuration [348]. Iridium(I) complexes were similar in their regioselectivity [349]. Iron-complexed dienyl alcohols underwent alkylation by alkenes (Eq. (102)) [350].

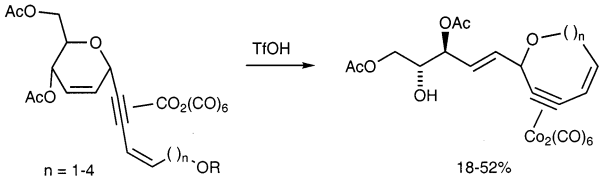


(102)

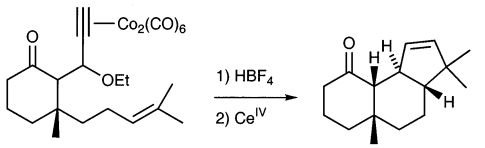
Alkylation of cobalt-stabilized propargyl cations has found diverse application in synthesis. Its use in stereocontrolled synthesis and reactions of sugar acetylenes has been reviewed (33 references) [351]. Examples are seen in Eq. (103) [352], Eq. (104) [353], and Eq. (105) [354]. Cobalt complexed propargyl chlorides were alkylated by allylstannanes [355]. The electrophilicity of cobalt catalyzed propargyl cations was assessed [356]. Replacement of one CO by a phosphine reduced the electrophilicity by 10^5. Palladium(0) catalyzed the S_N2' alkylation of propargyl carbonates by indole-2-borates to give 2-allenyl substituted indoles [357].



(103)



(104)

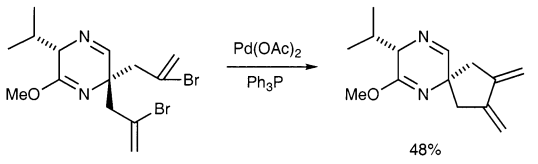


(105)

2.1.8. Coupling reactions

Copper(I) chloride in DMF couple alkenylstannanes to give 1,3-dienes [358]. Aryl bromides were coupled to symmetrical biaryls by palladium acetate/tetrabutyl

ammonium bromide [359]. A similar system coupled alkenyl bromides (Eq. (106)) [360], while palladium(0) complexes coupled enol triflates to aryl bromides (Eq. (107)) [361]. Palladium(II) acetate in acetic acid coupled indoles at the 2-position (Eq. (108)) [362,363].



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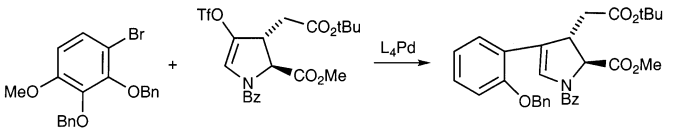

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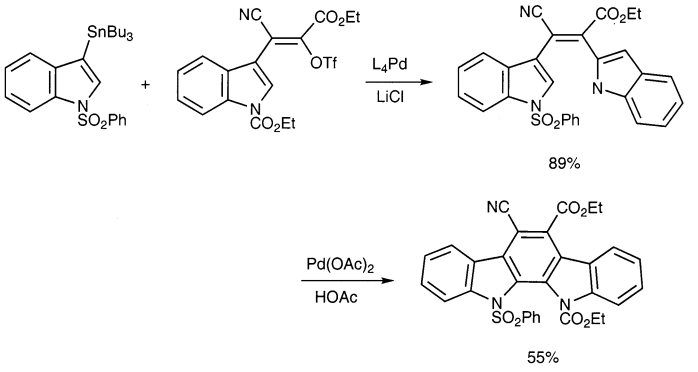
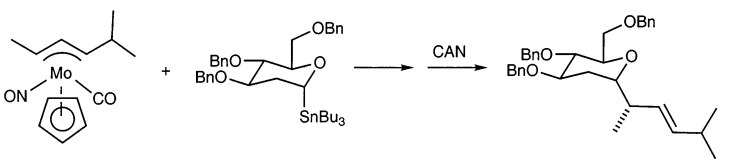


(108)

2.1.9. Alkylation of π-allyl complexes

Alkylations of various π-allyl complexes are shown in Eq. (109) [364], Eq. (110) [365], Eq. (111) [366], and Eq. (112) [367]. Tetracarbonyliron-mediated alkylations of 5-(R)-isopropoxy-3-pyrrolin-2-ones have been reviewed [368].



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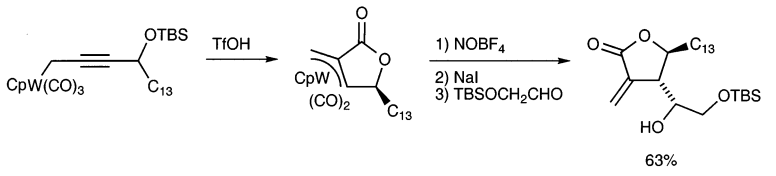

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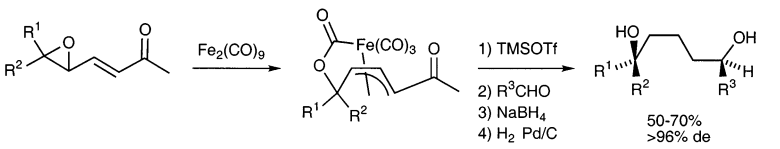

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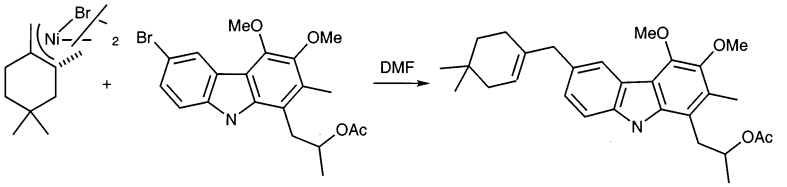
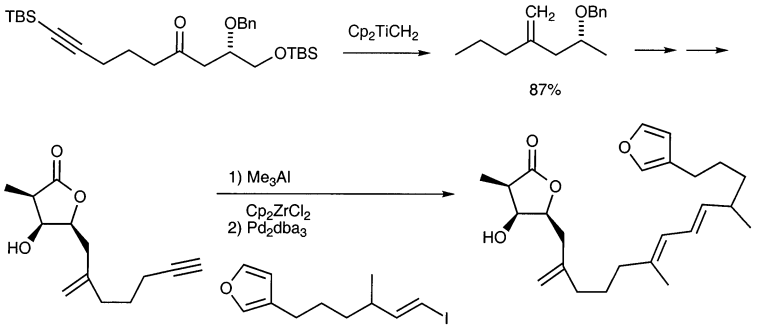


(112)

2.1.10. Alkylation of carbonyl compounds

Polymer supported esters were methylenated to produce enol ethers using Tebbe's reagent Cp_2TiCH_2 [369]. Petasis reagent, Cp_2TiMe_2 methylenated β-lactones [370]. Treatment of cyclobutanone dithioketal with excess $Cp_2Ti[P(OEt)_3]_2$ gave the cyclobutyl Tebbe type reagent which methylenated ketones to give methylenecyclobutanes [371]. Uses of Tebbe type methylenation in synthesis are shown in Eq. (113) [372] and Eq. (114) [373].



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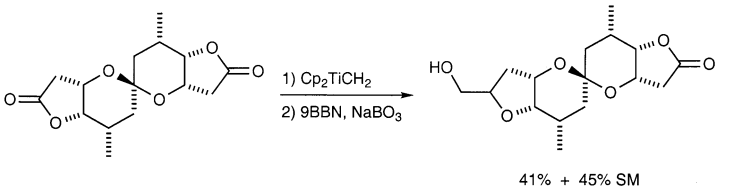
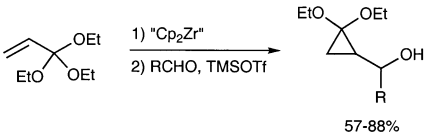


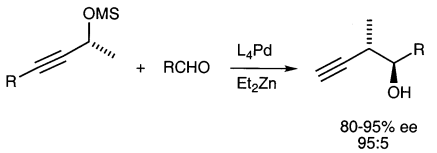
(114)

Titanocene/Me_3Al converted vinyl bromides into allyltitanium species (replacement of Br by $TiCH_2$) which then alkylated aldehydes [374]. Acylzirconocenes alkylated conjugated ketones both 1,2 and 1,4 [375]. An unusual aldehyde alkylation is seen in Eq. (115) [376].



(115)

Treatment of π-allylpalladium complexes with diethyl glycine converted the allyl group from an electrophile to a nucleophile which could alkylate aldehydes [377,378]. Palladium(II) complexes catalyzed the conversion of thioesters to ketones by alkylzinc halides [379]. Palladium(0) complexes catalyzed the alkylation of aldehydes by chiral propargyl mesylates in the presence of diethylzinc to give anti stereochemistry with high ee (Eq. (116)) [380]. Chiral palladium(0) complexes catalyzed the alkylation of imines by silylenol ethers [381] and allyl stannanes [382] with good ee. Nickel(0) catalyzed the cyclization shown in Eq. (117) [383].



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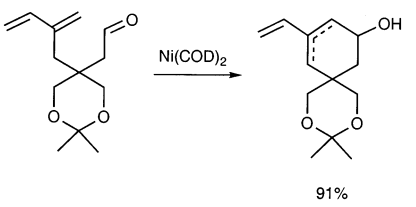
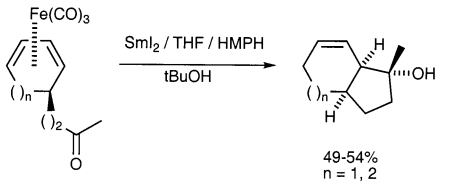


(117)

Diene dialdehydes complexed to iron underwent alkylation by dialkylzinc reagents in the presence of optically active amino alcohols with very high ee [384]. Iron complexed dienyl aldehydes having a chiral sulfoxide on one diene terminus were allylated by allylstannanes with high de [385]. Iron tricarbonyl diene complexes with pendent ketones cyclized in the presence of samarium iodide (Eq. (118)) [386]. Iron complexed dienals were homologated using alkylation of the aldehyde (Eq. (119)) [387]. π-Allyl iron-trimethylsilyl enolates alkylated aldehydes with high de (Eq. (120)) [388].



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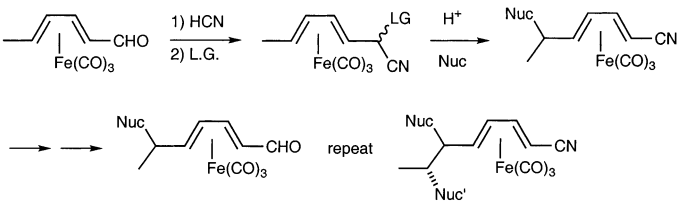

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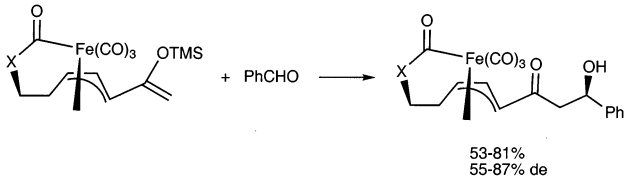
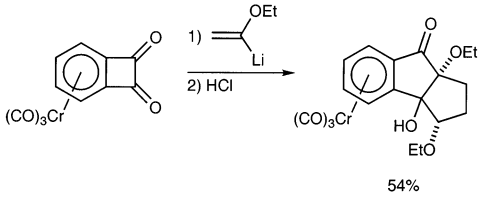


(120)

The α-anion of chromium(methoxy)(methyl) carbene complexes alkylated aldehydes in the presence of stannic chloride. The resulting β-hydroxy carbene complex lost water when treated with triethyl amine and mesyl chloride [389]. The mechanism of the Cp*Co(allylsilane) catalyzed alkylation of aldehydes by vinyl silanes was studied [390]. The S(+) chromium tricarbonyl complex of o-methoxybenzaldehyde underwent reaction with $CpFe(CO)_2^-$ to give the α-hydroxalkyl iron complex. This cyclopropanated isobutene with high ee [391]. Other interesting reactions of chromium complexes of aryl carbonyl compounds are shown in Eq. (121) [392], Eq. (122) [393], and Eq. (123) [394].



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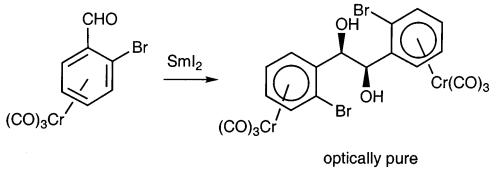

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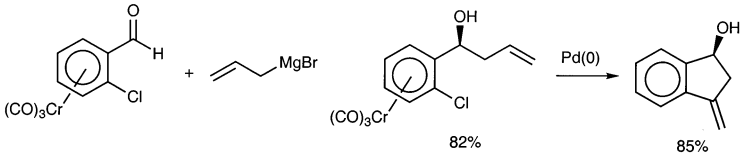
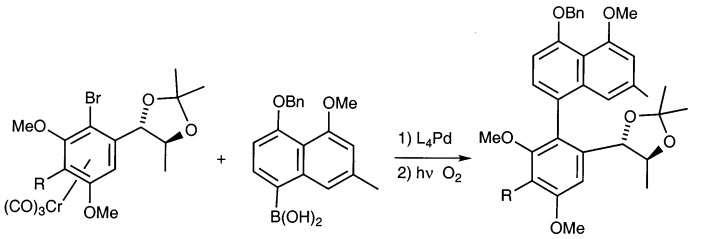


(123)

2.1.11. Alkylation of aromatic compounds

A paper dealing with mixed-ligand arene chromium tricarbonyl complexes as electronic modulators has appeared [395]. A review on indirect nucleophilic substitution on η^6-arene tricarbonyl chromium and η^5-cyclohexadienyl tricarbonyl manganese complexes has appeared (119 references) [396]. Photolysis of benzene-chromium tricarbonyl with methyl acrylate led to replacement of one CO by the acrylate. This labilized the arene group towards exchange with other arenes, dienes or CO [397]. Chromium complexed aryl halides underwent Suzuki coupling (Eq. (124)) [398] as well as Sonogashira coupling (Eq. (125)) [399]. Ortho-lithiation of chiral arene chromium tricarbonyl followed by alkylation was stereoselective (Eq. (126)) [400].



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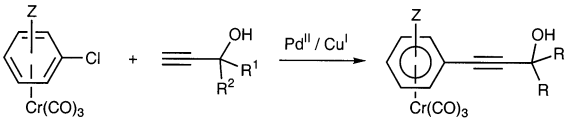

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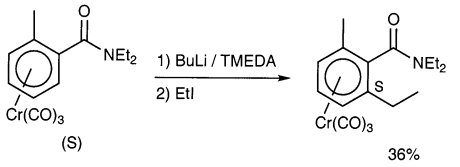
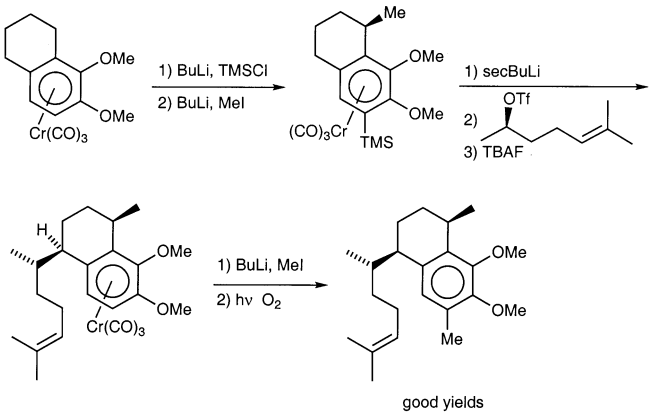


(126)

Regioselectivity in the benzylic deprotonation of η^6-arenechromium tricarbonyl derivatives was claimed to be controlled by eclipsing with CO of the $Cr(CO)_3$ triad [401]. Benzylic deprotonation of chromium complexed arenes has been used in complex syntheses (Eq. (127)) [402] and (Eq. (128)) [403]. A dipropylamino group at the homobenzyl position directed alkylation at the benzyl position to the opposite face regardless of the position of the chromium tricarbonyl fragment [404]. Asymmetric benzylic deprotonation using chiral amide bases resulted in benzylic alkylation with high ee [405]. Chromium complexation also permitted the production of chiral benzyl cations (Eq. (129)) [406].



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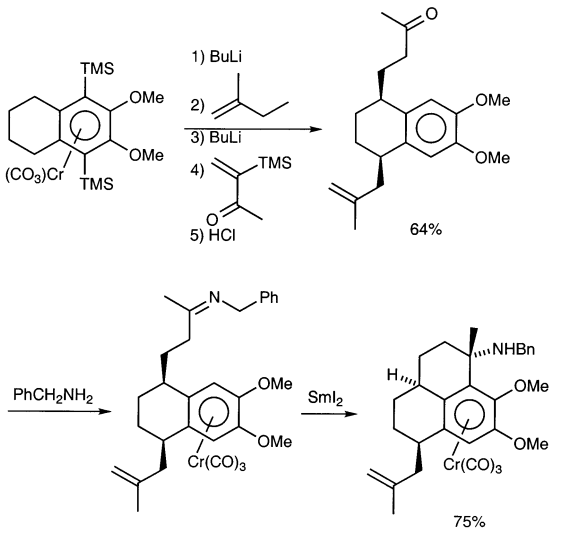

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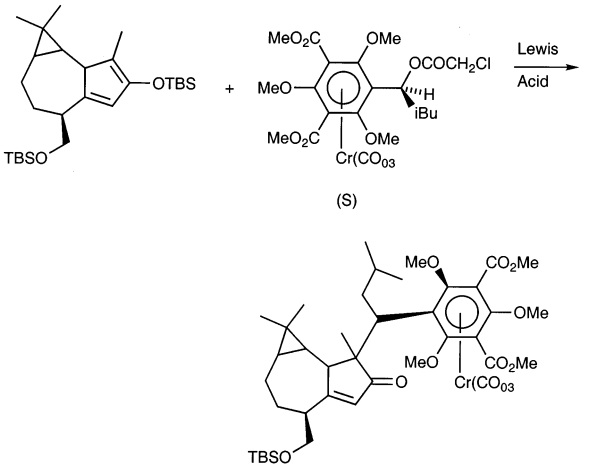
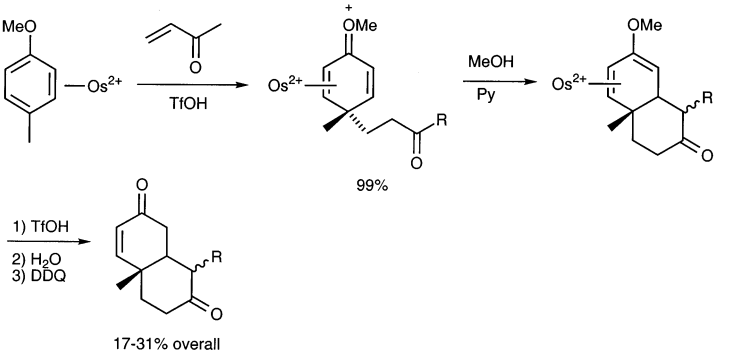


(129)

The manganese tricarbonyl complex of *N*-phenyl morpholine underwent *meta* phenylation with phenylmagnesium chloride. Treatment with NO^+, followed by sodium borohydride followed by hydrolysis gave 5-phenyl cyclohex-2-enone [407]. Ortholithiation of arenes having directing groups, followed by treatment with zirconocene dichloride produced a zirconabenzyne complex. Treatment with nitriles resulted in meta acylation of the arene [408].

η^2-Osmium complexes of arenes participate in some unusual reaction chemistry by 'deconjugating' arenes and allowing them to act as dienes or enol ethers. Naphthalene underwent electrophilic attack at the 1 position and nucleophilic attack at the 4 position to give 1,4-dihydroxnaphthalenes by sequential complexation to Os^{2+}, treatment with an electrophile, a nucleophile, the oxidative removal of osmium [409]. Interesting cyclization reactions were promoted this way (Eq. (130)) [410], Eq. (131)) [411], and (Eq. (132)) [412]. Anisoles were converted to 5-substituted cyclohexenones by related chemistry [413]. Osmium(II) complexed furans underwent complex reactions (Eq. (133)) [414].



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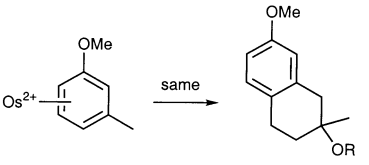

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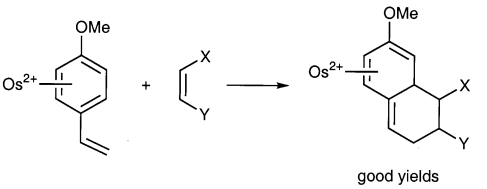

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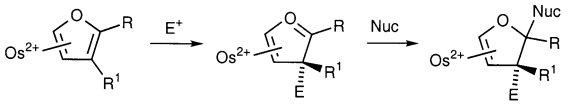
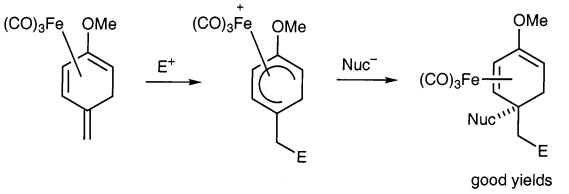


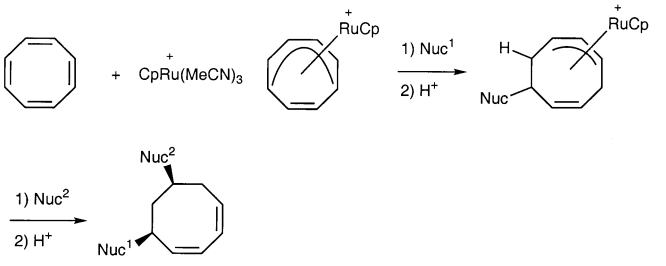
(133)

2.1.12. Alkylation of diene and dienyl compounds

Replacement of a CO by a phosphine on the iron dienyl complex of 1-methoxy-4-methyl cyclohexadiene directed nucleophilic attack to the quaternary center [415]. Diene complexes with an additional exocyclic double bond underwent electrophilic attack there (Eq. (134)) [416]. Cationic tungsten [417] and molybdenum diene complexes [418] underwent nucleophilic attack at a terminal position to give neutral π-allyl complexes. Cyclooctadiene was 1,3-dialkylated via the ruthenium η^6-trienyl complex (Eq. (135)) [419]. Ruthenium(II) complexes catalyzed the hydroacylation of 1,3-dienes, introducing the acyl group at the internal position [420].



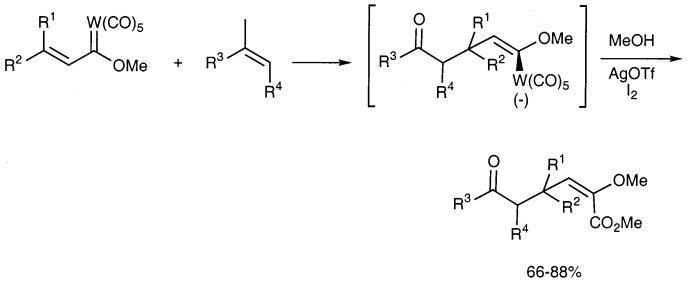
(134)



(135)

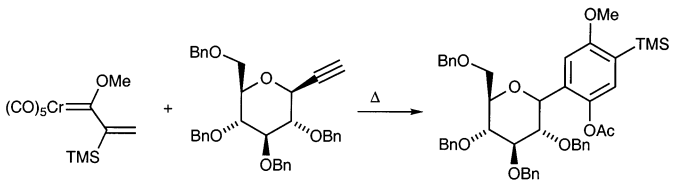
2.1.13. Metal carbene reactions

Treatment of tungsten alkoxycarbene complexes with dihydropyridine delivered a hydride to the carbene carbon. This anionic complex cyclopropanated enamines [421,422]. The thermodynamic acidity of Fisher carbene complexes in acetonitrile has been assessed [423]. Asymmetric synthesis (α-alkylation and aldol chemistry) with imidazolidinone and oxazolidinone Fisher carbene complexes has been reviewed [424]. C-2 symmetric morpholinocarbene complexes were α-deprotonated, then Michael-added to β-nitrostyrenes with reasonable de's [425]. Chromium alkoxycarbene complexes were α-acylated with acid halides, then decomposed to β-acyl enol ethers by base [426]. Ketone enolates Michael added to α,β-unsaturated tungsten carbene complexes (Eq. (136)) [427]. Treatment of chromium alkoxycarbene complexes with LDA followed by dibromomethane produced bromomethyl ketones [428].

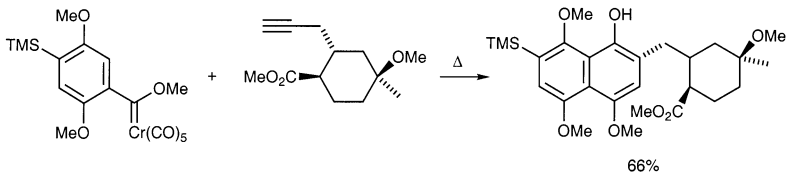


(136)

Calculations dealing with the Dötz benzannulation in the gas phase showed that CO loss is not required for the first step [429]. Aryl glycosides (Eq. (137)) [430], naphthoquinones (Eq. (138)) [431,432], and bis-naphthoquinones (Eq. (139)) [433] were made by the Dötz reactions. In this process electron-rich alkynes favored phenol formation, while electron poor ones favored lactone formation [434].



(137)



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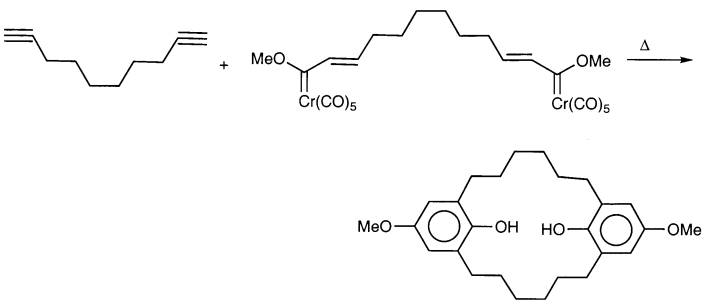
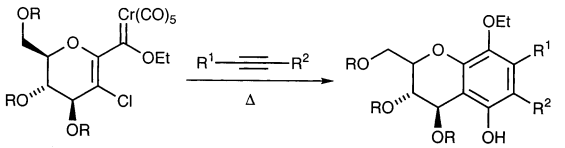
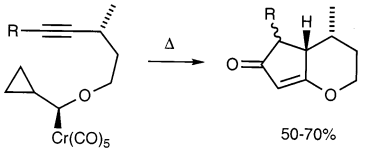


(139)

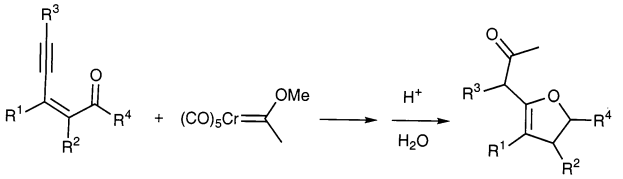
β-Chloro-α,β-unsaturated carbene complexes underwent the Dötz reaction cleanly (Eq. (140)) [435,436], while cyclopropyl carbene complexes gave cyclopentenones (Eq. (141)) [437] and enynones gave furans (Eq. (142)) [438]. Aldol chemistry, followed by Dötz chemistry was also effective (Eq. (143)) [439,440]. Intramolecular Dötz reactions of aminocarbene complexes gave lactams (Eq. (144)) [441].



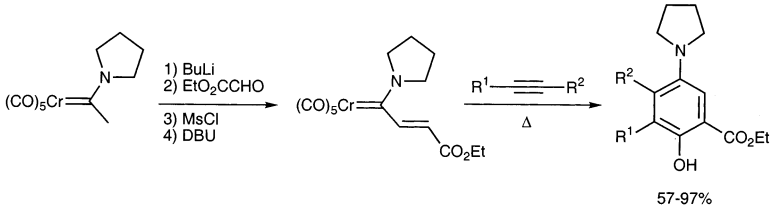
28-48% (140)



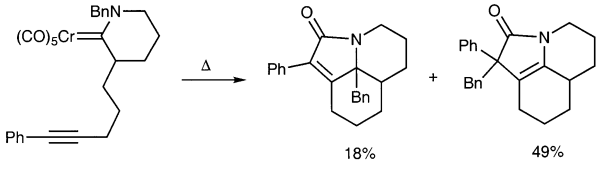
50-70% (141)



66-84% (142)

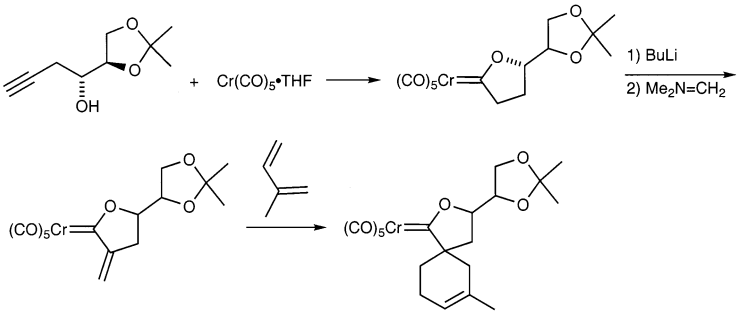


57-97% (143)

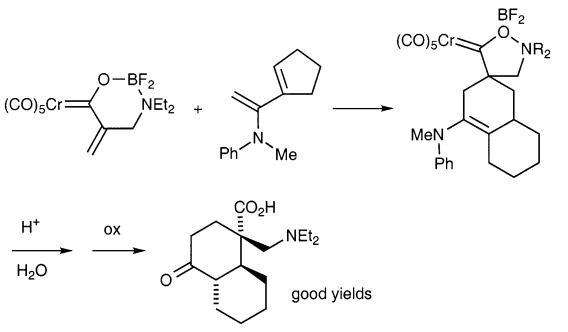


18% 49% (144)

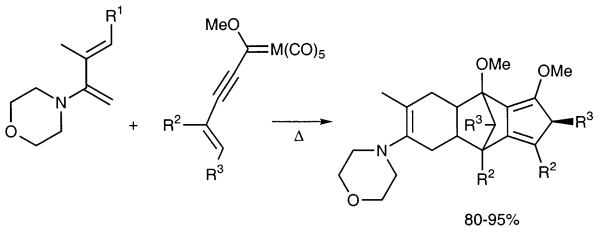
Kinetic and theoretical studies of 3 + 2 cycloadditions of nitrones to alkynyl carbene complexes have been carried out [442]. Cycloadditions of carbene complexes are shown in Eq. (145) [443], Eq. (146) [444], and Eq. (147) [445]. An unusual polycyclization is shown in Eq. (148) [446].



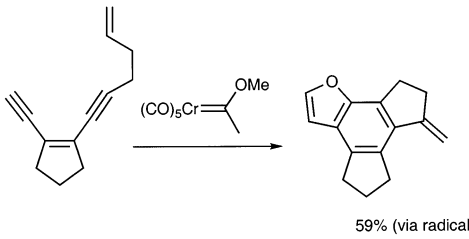
(145)



(146)

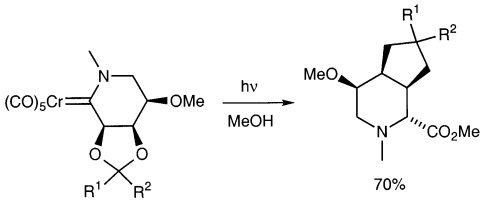


(147)



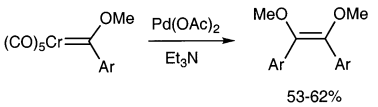
(148)

Photolysis of chromium carbene complexes generated species which have ketene-like reactivity. Photolysis in the presence of optically active ene carbamates produced optically active cyclobutanones which were further functionalized [447], photochemically ring-expanded to tetrahydrofurans [448], used to synthesize optically active cyclobut-A [449] and optically active spiroketals [450]. Photolysis of chromium carbene complexes in the presence of electron-rich arenes resulted in Friedel-Crafts acylation [451], while photolysis in the presence of methanol gave esters (Eq. (149)) [452].

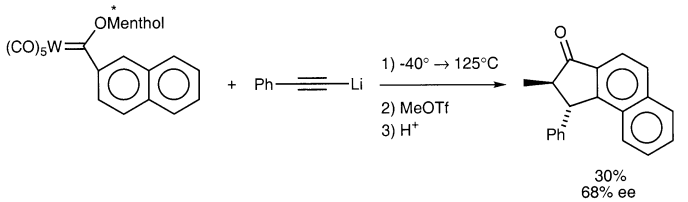


(149)

Unusual reactions of carbene complexes are seen in Eq. (150) [453], Eq. (151) [454], and Eq. (152) [455].



(150)



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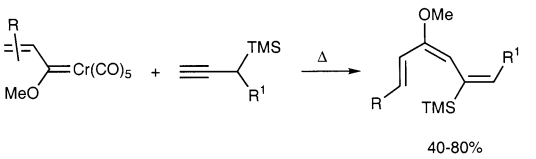
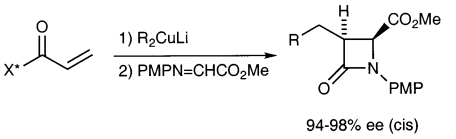


(152)

2.2. Conjugate addition

Rules for diastereofacial selectivity in the conjugate addition of cuprates to acyclic enones have been devised [456]. Cuprates catalyzed the conjugate addition of (phenyl)dimethylsilylzinc compounds to enonenes including aldehydes in the presence of 5% scandium triflate [457]. Copper(I) salts catalyzed the asymmetric Michael addition of aryl Grignard reagents to unsaturated chiral *N*-acyl oxazolidinones (Evans chiral auxilliary) [458]. Conjugate addition to Oppolzer's chiral sultam containing enones was used to synthesize β-lactams (Eq. (153)) [459].



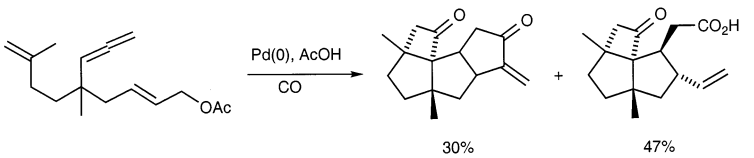
(153)

Rhodium(I)/(S)BINAP complexes catalyzed the conjugate addition of alkenyl catechol boranes to enones with $>95\%$ ee [460,461]. Rhodium(I) complexes also catalyzed conjugate additions of aryl stannanes to enones [462].

2.3. Acylation reactions excluding most hydroformylations

2.3.1. Carbonylation of alkenes and arenes

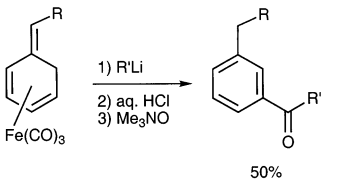
Water soluble dicationic palladium catalysts for biphasic hydroxycarbonylation of alkenes have been developed [463]. Alkenes were 1,2-bis carboxylated by treatment with palladium chloride catalyst in the presence of copper(I) chloride/oxygen and carbon monoxide [464]. Terminal alkenes were carbonylated to terminal α,β-unsaturated thioesters by palladium catalyzed carbonylation in the presence of thiophenol [465]. α,ω-Dienes were α-arylated and ω-carbonylated by treatment with perfluorophenylpalladium(II) bromide, followed by carbon monoxide and methanol. This occurred by insertion of a terminal alkene into the palladium-aryl σ-bond, followed by migration of Pd to the remote terminus for carbonylation [466]. Palladium(0) complexes catalyzed cascade carbonylation (Eq. (154)) [467].



(154)

$Ru_3(CO)_{12}$ catalyzed the *ortho* position (CH activation) carbonylation of arenes having donor ligands (N) either directly on the ring or in a benzylic position

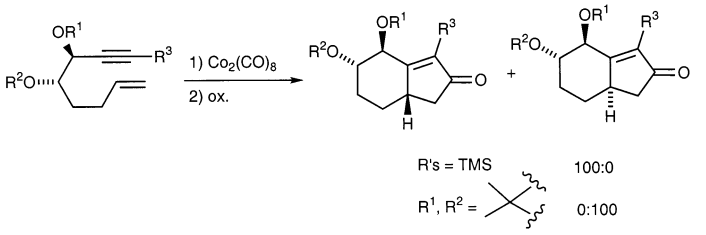
[468,469]. Iron diene complexes were acylated by reaction with organolithium reagents followed by oxidation (Eq. (155)) [470].



(155)

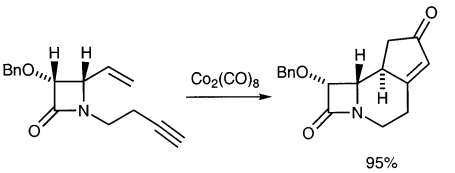
2.3.2. Carbonylation of alkynes (including the Pauson–Khand reaction)

New developments in the Pauson–Khand reaction have been reviewed (38 references) [471]. Catalysts for the Pauson–Khand reaction include $Co_2(CO)_6$–alkyne complexes [472], high purity $Co_2(CO)_8$ at 60–70°C [473], $Co_3(CO)_9$ μ^3CH_3 [474], and 1% $Co_2(CO)_8$ at 7 atm. CO 120° in DME with a small amount of water [475]. Amines promoted the Pauson–Khand reaction [476]. Ethylene diamine in THF decomplexed $Co_2(CO)_6$ from alkynes [477]. $Co_4(CO)_{12}$ was an extremely efficient catalyst (> 200 TON) for the Pauson–Khand reaction of terminal alkynes with norborene and norbornadiene [478]. Cyclopropyl propargyl alcohols (from acetylides and cyclopropanones) were converted to cyclopentenones by reaction with $Co_2(CO)_8$ [479]. Chiral centers in the substrate could induce asymmetry in the Pauson–Khand reaction [480], (Eq. (156)) [481,482].

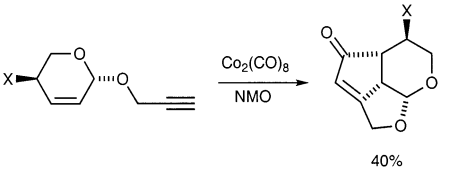


(156)

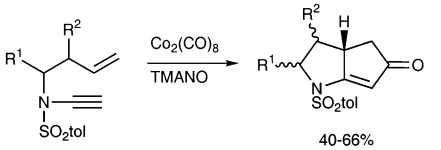
Synthetically interesting Pauson–Khand reactions are seen in Eq. (157) [483], Eq. (158) [484], Eq. (159) [485], Eq. (160) [486], Eq. (161) [487], and Eq. (162) [488].



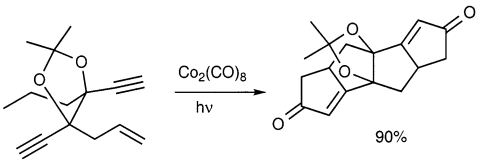
(157)



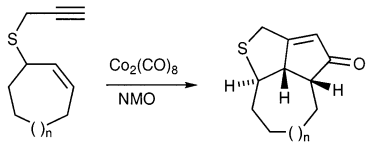
(158)



40-66% (159)

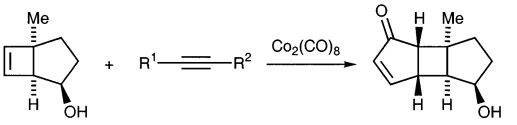


90% (160)



n = 1, 2

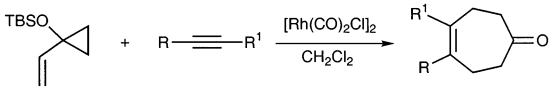
68-85% (161)



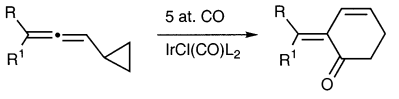
73% (162)

Treatment of $Co_2(CO)_6$ complexes of silylacetylenes with silylacetylenes gave siylated cyclopentadienones [489]. Under some conditions, enynes were cycloisomerized to dienes rather than carbonylated [490].

Molybdenum hexacarbonyl catalyzed Pauson–Khand cyclizations of alleneynes to give α methylenecyclopentanones [491,492]. The ring size depended on the substitution pattern. Rhodium(I) carbonyl complexes also catalyzed Pauson–Khand cyclizations of enynes [493,494], as well as the carbonylative cyclizations of vinylcyclopropenes with alkynes (Eq. (163)) [495], while iridium(I) complexes formed similar products from allenylcyclopropanes (Eq. (164)) [496].

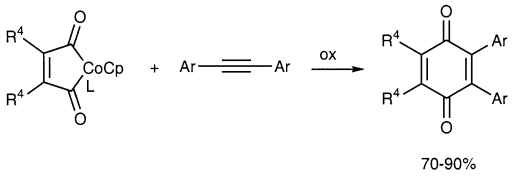


65-97% (163)

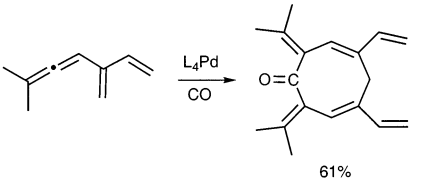


28-83% (164)

Titanocene ('Cp_2Ti') and zirconocene ('Cp_2Zr') produced cyclopentenones in two steps from enynes, via reductive cyclization CO insertion processes [497,498]. Quinones were produced by the reaction of alkynes with cobaltacycles (Eq. (165)) [499] and by the $Ru_3(CO)_{12}$ catalyzed cycloaddition of alkynes, 2CO, and norbornene [500]. Palladium(0) complexes catalyzed the carbonylative cyclodimerization of allenes (Eq. (166)) [501] as well as the carbonylation of alkynes to give acrylates [502]. Methylmanganese pentacarbonyl reductively carbonatively dimerized alkynes to give cyclopentadienols (Eq. (167)) [503].



(165)



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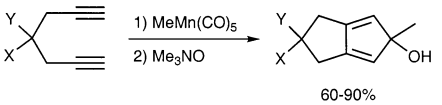
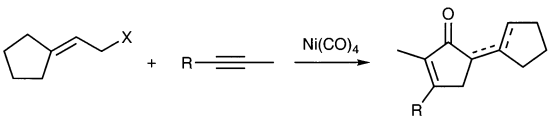


(167)

Hydrozirconation of terminal alkynes followed by CO insertion gave α,β-unsaturated acylzirconium species. These alkylated aldehydes to give α-α′-hydroxyenones [504]. Alkynes were converted to cyclobutene diones by treatment with $Fe(CO)_5$ and sodium borohydride [505]. Nickel carbonyl carbonylatively cyclized alkynes with allyl halides (Eq. (168)) [506]. $Ru_3(CO)_{12}$ cyclized ynals to butenolides (Eq. (169)) [507].



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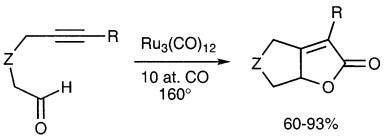
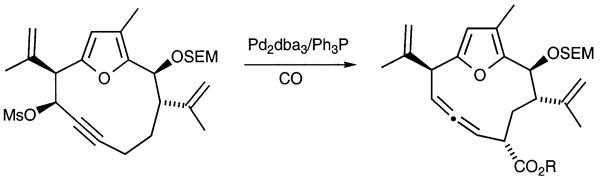


(169)

2.3.3. Carbonylation of halides and triflates

Palladium(0) complexes catalyzed the carbonylation of aryl tosylates to give benzoates [508], of 2-iodoindole to give the indole 2-carboxamide [509], aryl and alkenyl halides to give acrylamides using bis-trimethylsilylamine as the nucleophile [510], the polycarbonylation of polybrominated bipyridines and oligopyridines [511], tetra-bromophenyl zinc porphyrins [512], and 2-iodo-1,3-butadienes to give the cross conjugated esters [513]. Palladium(0) catalyzed the carbonylative coupling (to give ketones) of aryl and alkenyl halides with organostannes [514] and arylboronic acids [515] and of diaryiodonium salts with arylboronic esters [516]. Palladium(0) catalyzed the carbonylation of propargyl mesylates to give allenic esters (Eq. (170)) [517]. Benzyl alcohols were converted to α-methyl arylacetic acids when treated with $PdCl_2/CuCl_2/CO/MeOH$ [518].



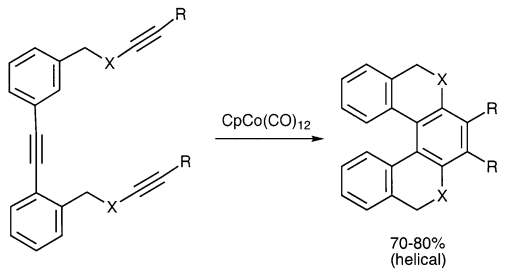
(170)

Palladium(0) complexes catalyzed the carboxylation (to give carboxylic acids) of aryl and alkenyl triflates by CO_2 under reducing conditions [519].

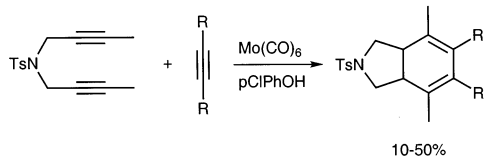
2.4. Oligomerization (including cyclotrimerization and metathesis polymerization)

Ruthenium complexes catalyzed the head-to-head dimerization of terminal alkynes to give enynes [520], and the addition of terminal alkynes to internal alkynes to give enynes [521]. Palladium acetate catalyzed the Michael addition of terminal alkynes to propriolate esters to give β ynenones [522]. Palladium(0) catalyzed the dimerization of allenes [523] while ruthenium(II) complexes catalyzed the head-to-head cross dimerization of alkenes with alkynes to give dienes [524]. Ruthenocyclopentadiene complexes catalyzed the cooligomerization of acetylene with acrylonitrile to give 1-cyanohexa-1,3,5-triene [525].

Water soluble cobalt-alkyne cyclotrimerization catalysts have been developed [526]. $CpCo(CO)_2$ catalyzed the cyclotrimerization of bis-[(diarylamino)aryl]alkynes to the hexa-paradiarylaminophenyl benzenes [527]. Intramolecular (Eq. (171)) [528,529] and intermolecular (Eq. (172)) [530] cyclotrimerizations were catalyzed by the same complex. Three different alkynes were stepwise cocyclotrimerized utilizing zirconium chemistry [531]. Diynes cyclotrimerized with ω-cyanoalkynes to give dipyridines (Eq. (173)) [532].



(171)



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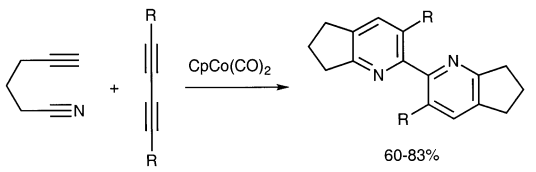
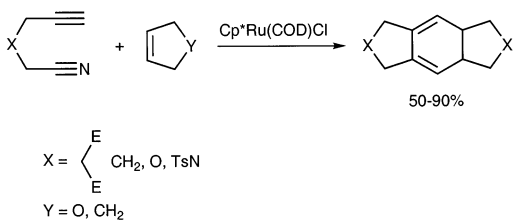


(173)

Palladium(0) cyclotrimerized 1,2-dibromoarenes and -alkenes to arenes via benzynes or alkynes [533,534]. Diyne-ene cocyclotrimerizations are shown in Eq. (174) [535], Eq. (175) [536], Eq. (176) [537], Eq. (177) [538], Eq. (178) [539], and Eq. (179) [540]. Other alkene alkyne reactions are shown in Eq. (180) [541] and Eq. (181) [542].



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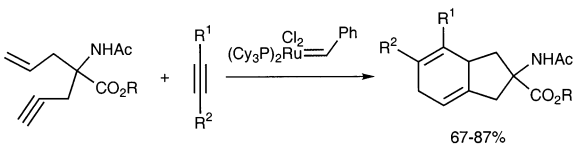

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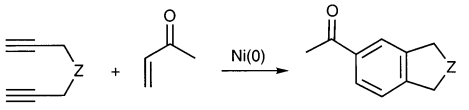

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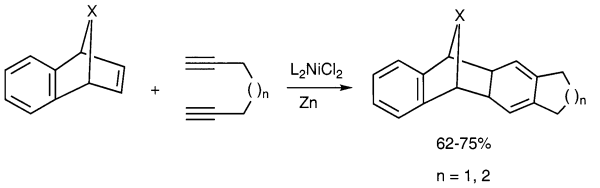

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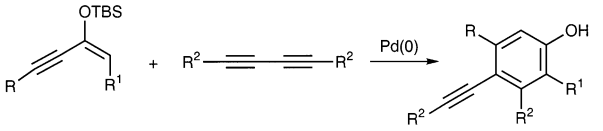
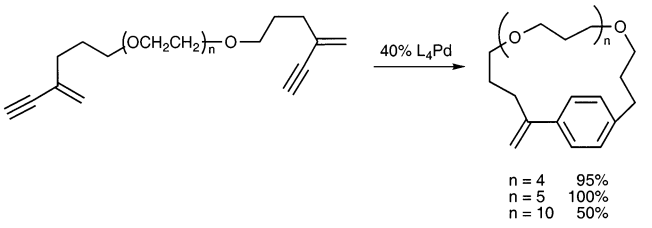
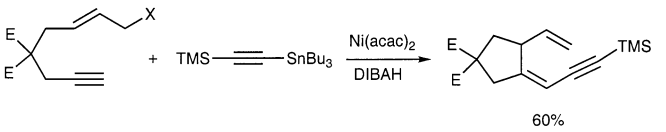


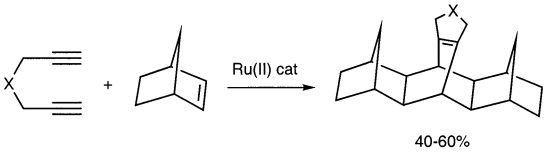
(178)



(179)



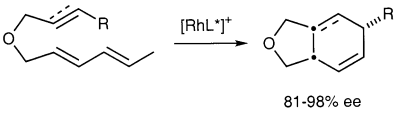
(180)



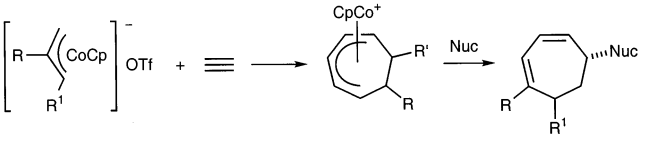
(181)

Triallylmanganese magnesium chloride cyclodimerized enynes and dienes, but incorporated an allyl group in the cyclodimerization of diynes [543]. Palladium

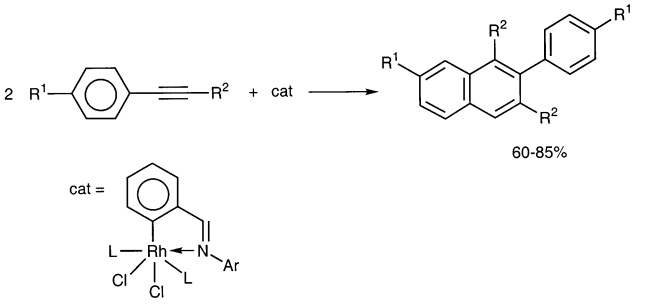
catalyzed the dimerization of conjugated enynes to give styrenes [544], while rhodium(I) complexes catalyzed the cocyclotrimerization of terminal alkynes with conjugated eneallenes to give arenes [545]. Chiral rhodium(I) complexes cyclotrimerized trienes and dienynes with high ee (Eq. (182)) [546]. Other unusual oligomerizations are shown in Eq. (183) [547], Eq. (184) [548], Eq. (185) [549], Eq. (186) [550], and Eq. (187) [551].



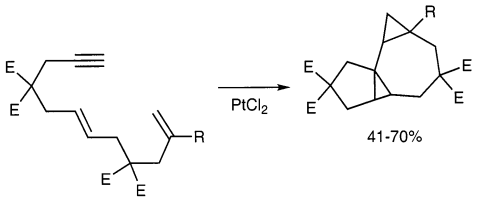
(182)



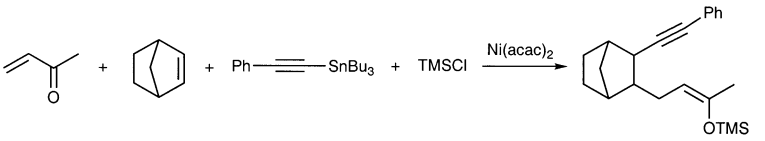
(183)



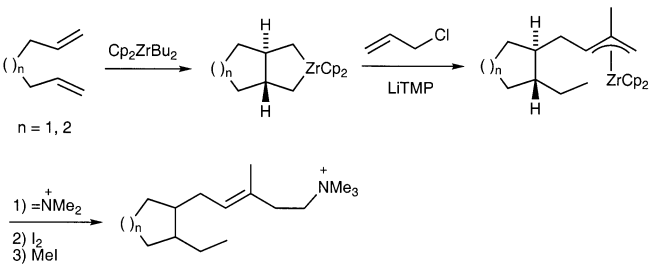
(184)



(185)



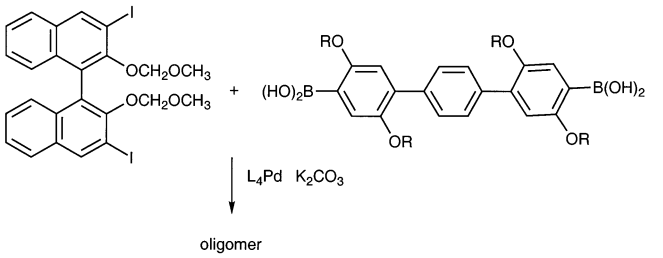
(186)



(187)

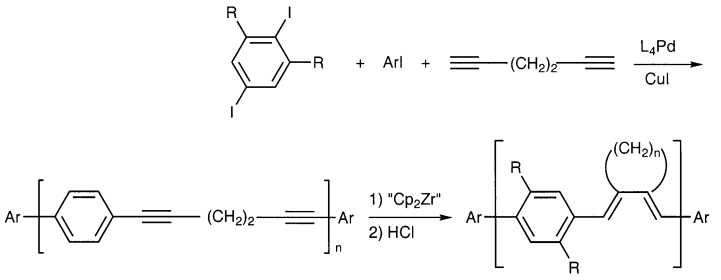
Many of the classic metal catalyzed cross-coupling processes were put to use to make oligomers. The use of palladium-catalyzed cross coupling for the synthesis of polymers incorporating vinylene and ethynylene groups has been reviewed (134 references) [552]. Stille cross coupling was used to make oligothiophenes on solid supports [553], polythiophenes for incorporation into triblock hybrid dendrimers [554], polythiophenes having alternating donor–acceptor units [555], and 1,2-binaphthol oligomers with 3,3′-alkyne spacers [556]. Palladium(0) catalyzed the oligomerization of 2,5-dibromosilacyclopentadienes with $Bu_3Sn{-}{\equiv}{-}X{-}{\equiv}{-}SnBu_3$ [557] and the oligomerization of 2,5-dibromopyridines with 2,5-distannyl pyridines to give precursors to ladder polypyridines [558].

Polyorthophenols were made by Suzuki coupling of o-haloboronic acids [559]. Suzuki coupling was used to bis couple thiophene-2-boronic acid with 2,6-dibromopyridine [560], to make dendritic nanorods by coupling polybrominated polyaryl ethers with 2,5-bis alkyl-1,4-bis phenylboronic acid [561], and to effect the coupling shown in Eq. (188) [562].



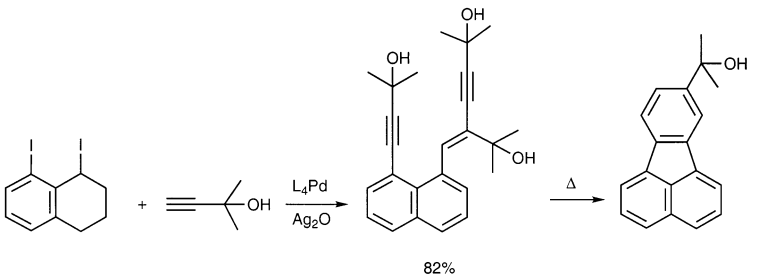
(188)

Sonogashira coupling of alkynes with aryl halides was used to synthesize conjugated helical acetylene-bridged polymers and cyclophanes [563], for molecular scale electronic systems [564], for dendrimer hosts having hydrogen bonding cores [565] and for fluorescent porous polymer films [566]. Palladium complexes catalyzed the oligomerization of acetylene with 3,5-diiodobenzoic acid in aqueous solution [567], the co-cyclotrimerization of o-diiodoarenes with acetylene [568], and the oligomerization shown in Eq. (189) [569].



(189)

Low Tg polymers were prepared by Heck reaction of divinyl benzenes [570]. Nickel(0) complexes catalyzed the oligomerization of 4,4′-dibromostilbenes [571] while molybdenum hexacarbonyl oligomerized 1,4-dialkynylbenzenes by alkynic metathesis [572]. The mechanism of palladium-catalyzed copolymerization of methyl acrylate with ethylene has been studied [573]. Palladium catalyzed the synthesis of monodispersed controlled length functionalized oligoanilines [574], and living alternating copolymers between carbon monoxide and norbornadiene diesters [575]. Ruthenium carbynes catalyzed the ROMP polymerization of cyclopentene [576]. An unusual cyclooligomerization is shown in Eq. (190) [577].



(190)

2.5. Rearrangements

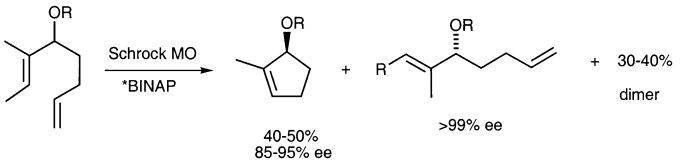
2.5.1. Metathesis

With the development of the Grubbs and Schrock catalysts for olefin metathesis, this process has been utilized very extensively in synthesis. Ring closing metathesis in organic synthesis (90 references) [578] and recent advances and applications of metathesis in synthesis have been reviewed (41 references) [579]. Libraries of olefins for combinatorial chemistry have been produced by metathesis [580,581]. Ring closing metathesis has been carried out in water [582], has been used to cross-link peptide helices [583], and has been carried out asymmetrically [584]. Sequential ring closing metathesis/Heck reaction chemistry has been developed [585]. The rates of olefin metathesis with the Grubbs Ru=CHPh catalyst was bulky < non-

bulky:trans < cis [586]. New, easy to synthesize, efficient ruthenium based olefin metathesis catalysts have been made [587]. C-2 symmetric diols were made by ring-closing metathesis of silyl-linked allyl alcohols [588].

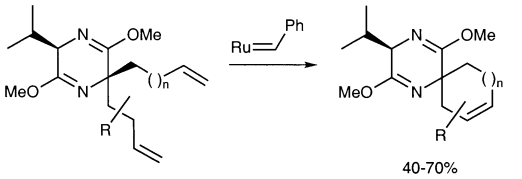
Procedures for efficient cross metathesis of terminal alkenes with internal alkenes has been developed [589]. Metathesis of cyclopropenone ketals with terminal olefins produced divinyl ketals [590]. Metathesis of functionalized alkynes with polymer bound allyl silanes produced dienyl silanes [591].

Allyl ethers of the anomeric carbon of acetoxylated sugars were dimerized with loss of ethylene using metathesis [592]. Metathesis of allyl alanine with terminal alkenes gave 40–60% methylene group interchange and 18–40% metathesis coupling of two allyl alanines with loss of ethylene [593]. Metathesis of polymer bound allyl esters was efficient with the Grubbs catalyst [594]. 4-Amino-1,7-octadiene ring closed metathesized to 4-aminocyclohex-1-enes [595]. Cyclohexenylboronates were made by ring closing metathesis [596]. The use of chiral ligands allowed asymmetric induction in the ring closing metathesis (Eq. (191)) [597].



(191)

Gem-bisalkenyl-bis lactim ethers metathesized easily (Eq. (192)) [598]. Constrained homoserines were made this way [599,600]. Ring closing metathesis was important in carbohydrate annulation (Eq. (193)) [601], in forming bridged tricycles (Eq. (194)) [602] and in synthesizing cyclic alkenes (Eq. (195)) [603,604].



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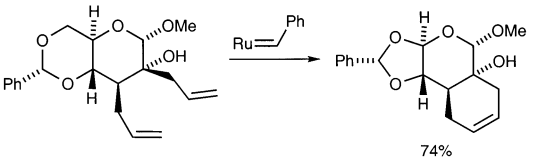

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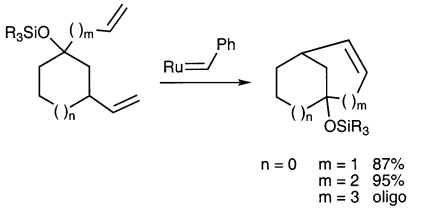

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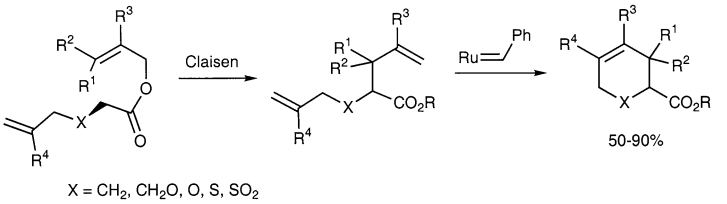
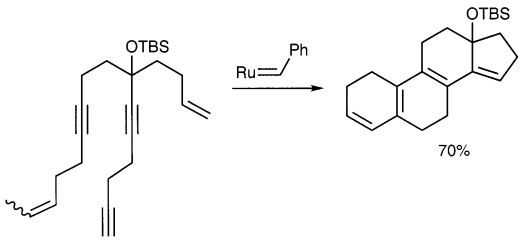


(195)

Ring closing metathesis was used to convert acyclic polyynes into polycyclic material (Eq. (196)) [605]. Tungsten carbynes ring closed metathesized diynes to cyclic alkynes [606]. The example in Eq. (197) involves ring closing, ring opening, and cross metathesis in the same system [607].



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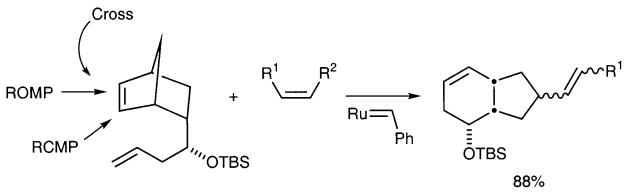
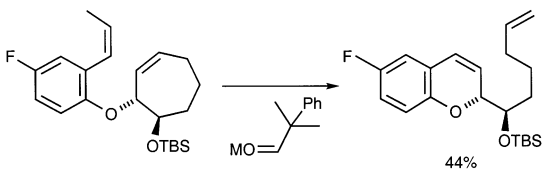


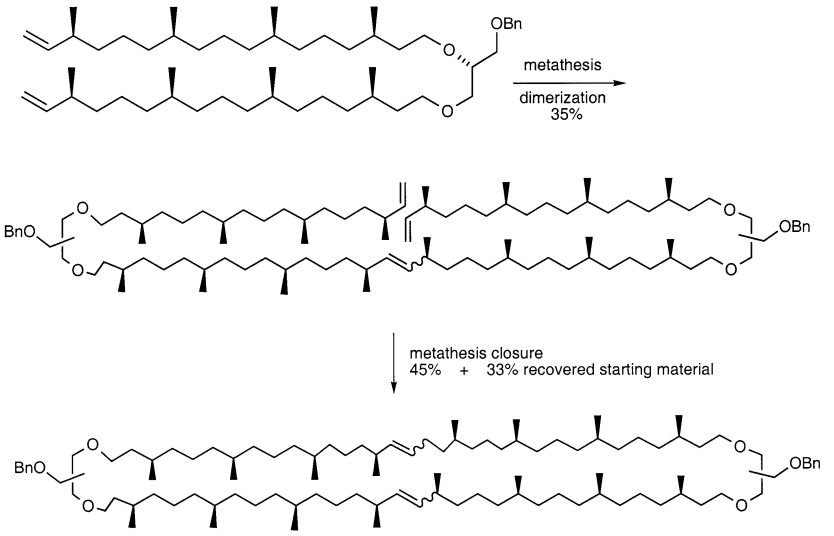
(197)

Ring closing metathesis has been used extensively in the synthesis of cyclic ethers including spirocyclic diethers [608], dihydropyrans [609], benzofurans [610], and benzopyrans (Eq. (198)) [611]. It has also been used for the kinetic resolution of racemic diallyl ethers [612] (see Eq. (191)).



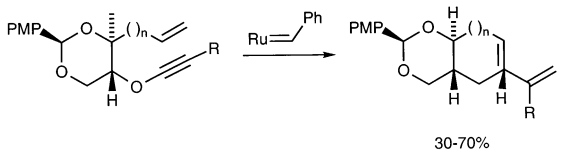
(198)

Ring closing metathesis of the allyl ketal of 1-vinyl sugars led to spirocyclic sugar-2,5-dihydrofurans [613]. An impressive metathesis reaction is seen in Eq. (199) [614].



(199)

Ring closing metathesis was used to make unsaturated seven-membered ring ethers [615], unsaturated ethers fused to β-lactams [616], the cyclic ethers in brevitoxin [617], 7-10 membered polyethers [618], (±)-helianane [619], and unsaturated cyclic ketals [620]. Enyne ring closing metathesis was also efficient (Eq. (200)) [621] as was polycyclization metathesis (Eq. (201)) [622].



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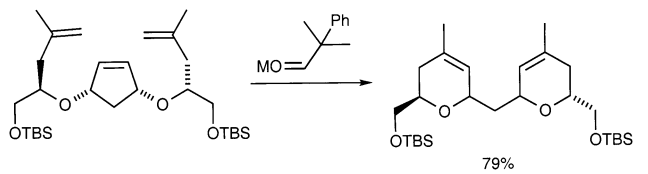
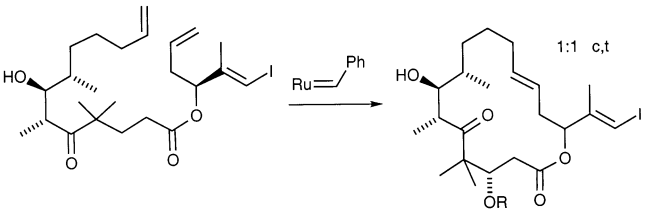


(201)

Ring-closing metathesis was used to make unsaturated lactones including six membered lactones [623], unsaturated γ, and δ-lactones as hydroxyethylene isosteres for protease inhibitors [624], and macrocyclic lactones [625–628], (Eq. (202)) [629], and (Eq. (203)) [630].



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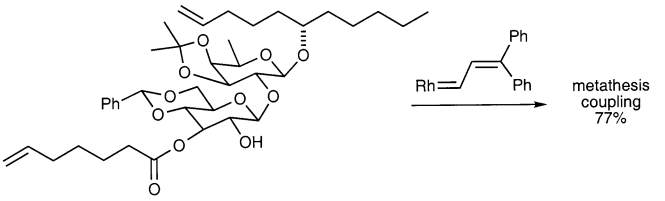
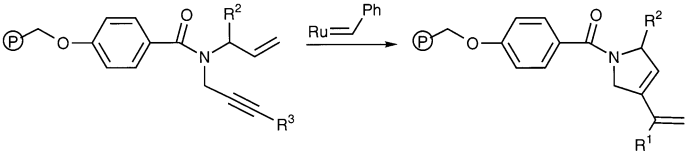


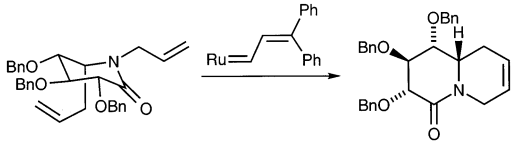
(203)

Ring closing metathesis was used to make unsaturated cyclic six membered amines [631], eight membered ring amines [632], and larger cyclic amines [633]. Grubbs catalyst promoted enyne metathesis on a solid support (Eq. (204)) [634] and in solution [635].



(204)

Ring closing metathesis was used to make unsaturated six membered lactams [636,637]; (Eq. (205)) [638], seven membered lactams [639], eight membered lactams [640], and macrocyclic lactams [641,642]; (Eq. (206)) [643]. Phosphorous heterocycles were also made by ring closing metathesis (Eq. (207)) [644].



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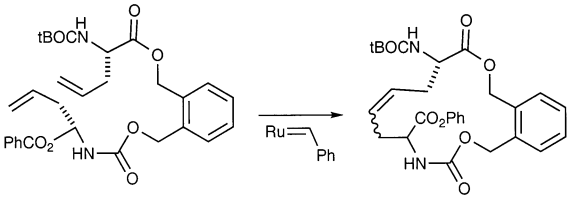

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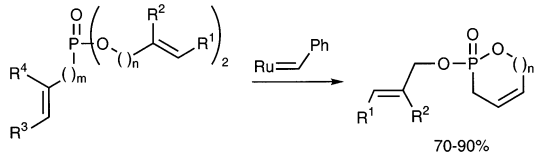
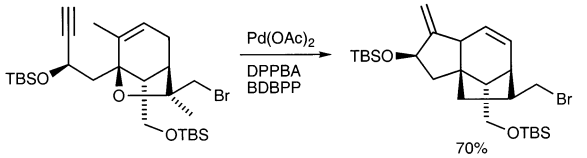


(207)

2.5.2. Olefin isomerization including cycloisomerization

Transition metal catalyzed cycloisomerization has been reviewed (93 references) [645]. Palladium complexes cycloisomerized *N*-tosyl diallyl amines to 3-methyl-4-methylenepyrrolidines [646]. Enynes (Eq. (208)) [647], dienes [648], tetraenes [649], dienynes [650], and enediynes (Eq. (209)) [651] all cycloisomerized. Allyl acetates underwent a related process (Eq. (210)) [652].



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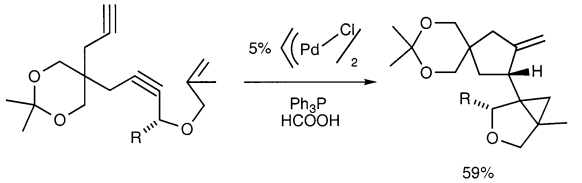

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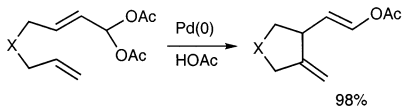
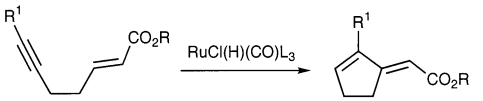


(210)

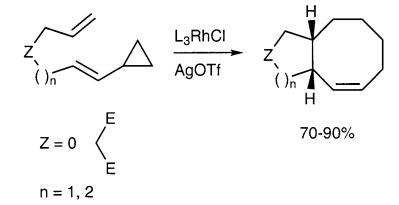
π-Allylnickel halides cycloisomerized diallyl amines, diallyl ethers, and diallylmalonates to five membered rings with methylene groups at the 3 position [653]. Low valent titanium complexes in the presence of *i*-propyl Grignard reagents effected similar cyclizations of alleneynes [654] and eneynes [655]. Ruthenium(II) hydrides cyclized enynes (Eq. (211)) [656] and dienes [657].



(211)

Wilkinson's catalyst cyclized vinylcyclopropanes with remote olefins [658] (Eq. (212)) [659] and alkynes [660]. Similar rhodium(I) complexes catalyzed the formal

4 + 2 cycloadditions of dienes to appended alkynes [661] and alkenes (Eq. (213)) [662].



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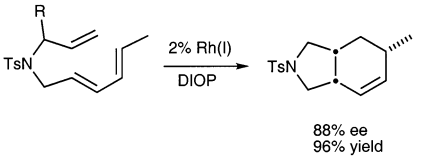
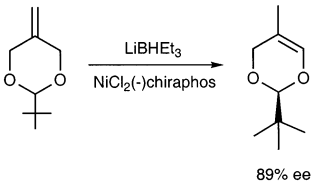


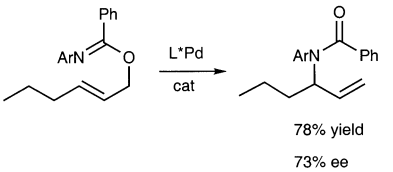
(213)

2.5.3. Rearrangements of allylic and propargylic compounds

Rhodium(I) and iridium(I) complexes equilibrated activated 2,3-*cis* and *trans* 3-alkyl-2-vinylaziridines via π-allyl intermediates [663]. Reduced nickel complexes in the presence of (−) chiraphos isomerized ketals of meso allylic diols with high ee (Eq. (214)) [664]. The same compounds isomerized allyl ethers to vinyl ethers [665]. Iridium(I) [666] and $Fe(CO)_5$ in the presence of base [667] catalyzed the same process. Chiral palladium complexes catalyzed the rearrangement of *o*-allyl imidates to allyl amides (Eq. (215)) [668].



(214)

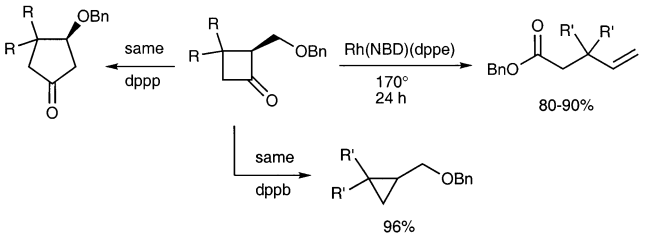


(215)

2.5.4. Skeletal and miscellaneous rearrangements

Rhodium(I) complexes catalyzed the rearrangement of allenylcyclopropanes to methylenecyclopentenes [669]. Under high temperatures rhodium(I) complexes catalyzed a number of skeletal rearrangements of cyclobutanones (Eq. (216)) [670]. Rhodium(I) complexes catalyzed the cyclization of allyl vinyl ethers to cyclopen-

tanones by combined oxyCope rearrangement/olefin hydroacylation [671]. Indenyl ruthenium(II) complexes racemized optically active 2° alcohols [672]. The mechanism of the palladium-catalyzed rearrangement of epoxides to ketones was studied [673].



(216)

3. Functional group preparation

3.1. Halides

Terminal olefins were converted to optically active chlorohydrins by reaction with $PdCl_4^{2-}/CuCl_2$ in water in the presence of chiral ligands [674]. Rhodium(I) complexes catalyzed the addition of chloroformates to terminal alkynes to give β-chloro unsaturated esters [675]. Ethyl chloroformate converted the chromium tricarbonyl complex of *N*,*N*-dimethyl α-phenethyl amine to the corresponding benzyl chloride complex (replacement of NMe_2 by Cl] [676]. Zirconium(IV) alkoxides in the presence of chiral amino triols catalyzed the asymmetric ring opening of epoxides to trans TMS protected iodohydrins with high ee [677].

3.2. Amides, nitriles, azides, imines

Palladium complexes catalyzed the aminocyclization of allenes to give substituted acrylamides [678], and the reaction of aryl aldehydes with acetamide to give *N*-acetyl arylglycines [679]. Palladium(0) complexes in the presence of copper [680] or zinc [681–684] converted aryl halides and triflates to aryl nitriles. Palladium(0) catalyzed the conversion of allyl acetates to allyl nitriles with overall inversion [685]. Jacobsen's chiral salen/aluminum complexes catalyzed the asymmetric hydrocyanation of *N*-allyl imines [686]. Palladium(0) catalyzed the conversion of allylcyclopropyl mesylate to azides with overall retention [687]. Palladium(0) complexes catalyzed the amination of aryl bromides by sulfoximines [688] and imines of benzophenones [689].

3.3. Amines, alcohols

Palladium catalyzed amination of aryl halides has become a growth industry, with every subtle variation in conditions or substrates being the occasion for

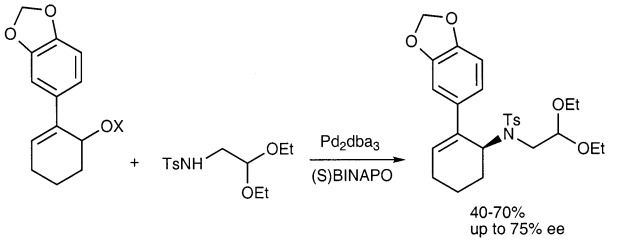
another publication. The impulse to run this reaction then report it seems irresistable, and many papers on the subject appeared this year.

Rational development of practical catalysts for aromatic carbon–nitrogen bond formation has been reviewed [690]. Electron rich, moderately hindered chelating diphosphines with bite angles of less than 90° led to the best selectivity [691], and a wide variety of these sorts of ligands have been studied for aryl bromide amination [692–696]. *N*-alkyl aryl amines aminated aryl bromides in aqueous medium [697] and diaryl amines were converted to triaryl amines [698]. End-functionalized oligoanilines were prepared by coupling diamino arenes to aryl bromides [699] while orthobromobenzamides were coupled to anilines [700]. Allyl amines could be used as ammonia surrogates [701].

Aryl piperazines were made by coupling monoprotected piperazines [702] as well as unprotected piperazines to aryl bromides [703]. Ketone hydrazones [704], amino crown ethers [705], and primary amines in the presence of 2° amines in polyamines [706] all aminated aromatic halides in the presence of palladium catalysts. Poly aryl bromides were polyaminated by morpholine [707], and β- [708] as well as α-amino acids were *N*-arylated [709].

Nickel(0) complexes catalyzed the amination of aryl chlorides [710] while palladium(II)/copper(II) complexes catalyzed the *N*-arylation of benzotriazines [711]. Copper acetate (one equivalent) promoted the amination of arylboronic acids [712–714].

Allylic amination has been reviewed (165 references) [715]. Palladium(0) catalyzed the allylic amination of allyl carbonates by sulfonamides [716]. This was used in the synthesis of large macrocycles [717]. In the amination of chiral allyl carbonates with diphenyl amine, diphosphine ligands resulted in retention while monophosphines allowed equilibration [718]. *N*-tosyl α-amino acid esters were allylated by allyl carbonates in the presence of palladium catalysts [719]. In the reaction of allyl acetate with α-amino acid esters in the presence of chiral ligands, the chirality resident on the amino acid did not determine the stereochemistry of the newly formed center but rather the chiral ligand was responsible [720]. A useful chiral allyl amination is shown in Eq. (217) [721]. π-Allyl palladium complexes catalyzed the amination of methylenecyclopropanes [722].



(217)

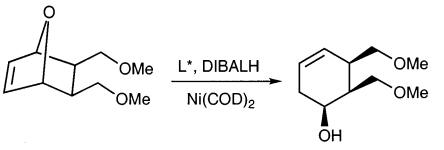
Palladium(0) catalyzed allylic amination of cyclopentenyl acetates was used in the synthesis of carbocyclic nucleoside analogs [723–725]. Allyl bromides were aminated via π-allyliron chemistry [726], while $[CpFe(CO)_2]_2$ promoted the reaction of alkenes with nitroarenes to give *N*-allyl aryl amines and CO_2 [727].

Metal-initiated amination of alkenes and alkynes has been reviewed (239 references) [728]. Rhodium(I) complexes catalyzed the hydroamination of dienes by morpholine [729]. π-Allyl palladium complexes catalyzed the 1,4-diamination of conjugated enynes [730]. Iridium complexes catalyzed the dimerization of nitriles having α-methylene groups to give β-amino acrylonitriles [731].

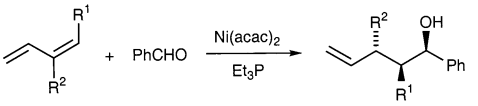
Chiral titanocene complexes catalyzed the asymmetric reduction of imines with high ee [732]. Rhodium(I) hydrides catalyzed the very mild reduction of tertiary amides to amines by diphenyl silane [733]. Epoxides were stable to the reduction. The palladium(0) catalyzed cleavage of alloc protecting groups from oligonucleotides was optimized [734]. *N*-Allyl amines were deallylated by reduced nickel complexes [735]. Iron-complexed imines of chalcone were isomerized to iron complexes of 2-aminodienes by strong base [736]. Chiral chromium complexed benzyl ethers were converted to benzyl amines with retention by reaction of *N*-hydroxycarbamates and HBF_4 [737]. Aryl halides were converted to aryl amines by reaction with TiX_4, TMSCl, Li metal and gaseous dinitrogen [738].

Ruthenium-BINAP catalyzed reduction of diketones to diols was dramatically accelerated by the addition of a strong acid (HCl) [739]. Chromium carbonyl complexed tetralone was reduced from the face of the metal by the Corey chiral aminoborane [740]. In this case, the reagent, not the metal, controlled the stereochemistry. Chromium tricarbonyl benzaldehyde complexes were reduced to the complexed benzyl alcohol by horse liver alcohol dehydrogenase with varying ee [741].

'Homogeneous Oxidation of Alkanes by Electrophilic Late Transition Metals' has been reviewed (100 references) [742]. Norbornene was converted to optically active norbornanol by palladium(0) catalyzed asymmetric hydrosilylation/oxidation [743]. Improved conditions for the nickel catalyzed ring opening of bicyclic ethers have been developed (Eq. (218)) [744]. Nickel complexes catalyzed the reductive alkylation of aldehydes by dienes (Eq. (219)) [745]. Palladium complexes catalyzed the ring opening of allyl epoxides to homoallylic alcohols [746]. Rhodium(I) complexes catalyzed the alkylation of aldehydes by arylboronic acids [747]. Reduced nickel catalysts deprotected allyl ethers without touching benzyl ethers [748]. Other interesting alcohol forming reactions are shown in Eq. (220) [749] and Eq. (221) [750].



(218)



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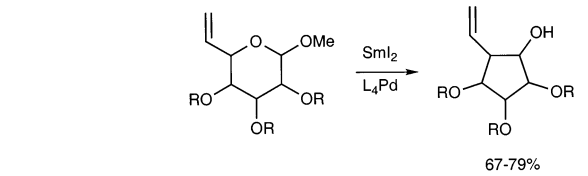

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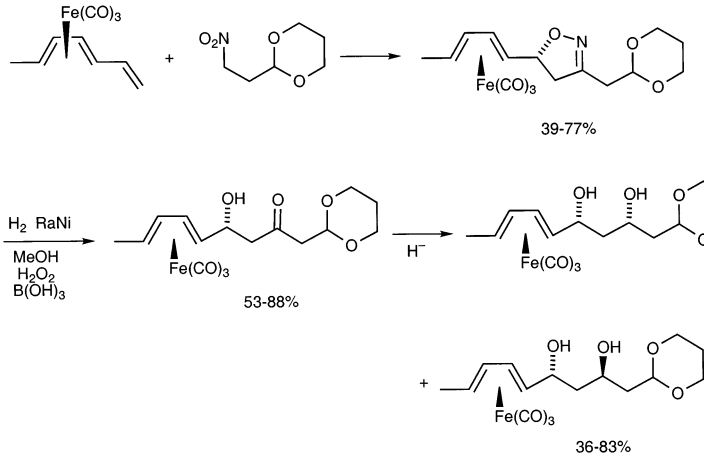
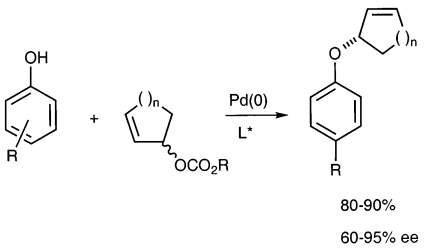


(221)

3.4. Ethers, esters, acids

Carbon heteroatom bond forming reductive elimination of amines, ethers, and sulfides has been reviewed (58 references) [751]. Copper salts catalyzed the coupling of aryl iodides [752] and arylboronic acids [753] with phenols to give diarylethers. Palladium(0) catalyzed the reaction of allyl carbonates with phenols (Eq. (222) [754,755] and other alcohols [756]. The mechanism of the attack of phenoxide on 1- and 2-bromo-π-allylpalladium complexes was studied [757]. Palladium(0) catalyzed the ring opening of chiral epoxides with high ee (Eq. (223)) [758]. Palladium acetate catalyzed the 1,4-diethoxylation of cyclohexadienes with modest ee [759]. Palladium and iron complexes catalyzed ring openings to give ethers (Eq. (224)) [760].



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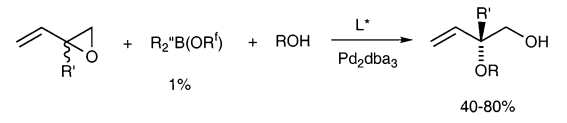

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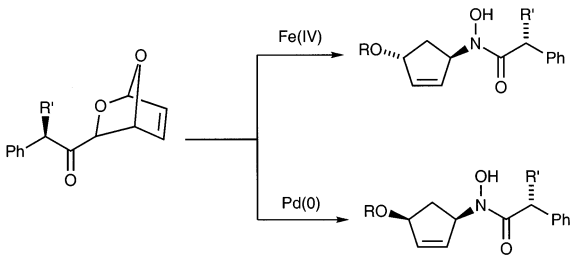
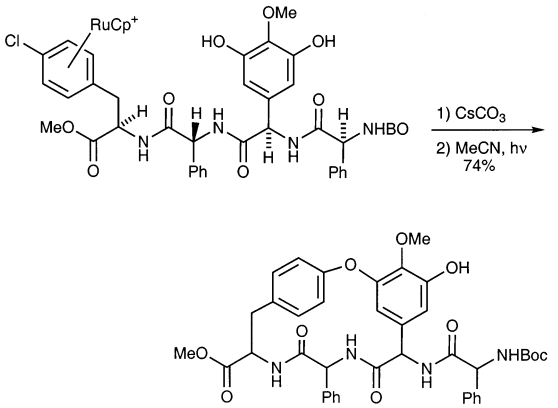


(224)

Attack of phenols on complexed aryl halides has become an important synthetic procedure (Eq. (225)) [761], (Eq. (226)) [762], and (Eq. (227)) [763]. Platinum(II) complexes catalyzed the insertion of diazo compounds into OH bonds to produce ethers [764]. Platinum catalyzed the ring opening of cyclopropanes by alcohols (Eq. (228)) [765].



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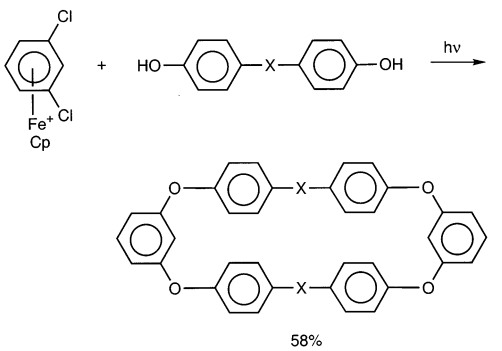

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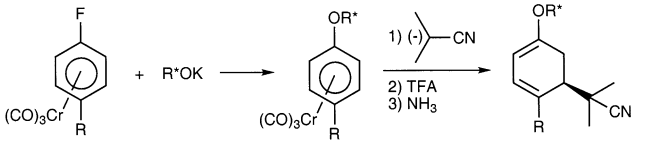

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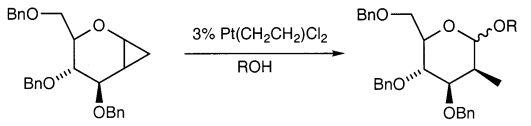
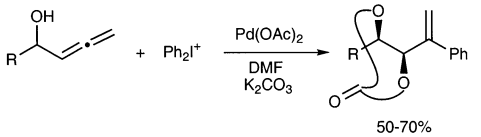


(228)

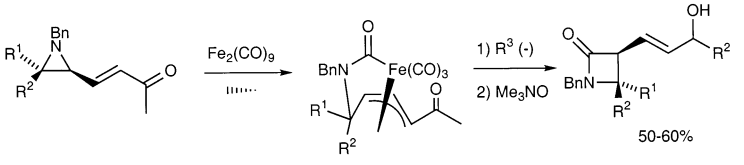
Palladium(0) catalyzed the addition of carboxylic acids to allenes to give allyl esters [766]. Palladium(II) complexes catalyzed the 1,4 alkylation/acetoxylation [767], and diacetoxylation of cyclohexadienes [768–770]. The stereochemistry depended on the presence of additives. Copper acetate catalyzed the OH insertion of diazoalkanes into carboxylic acids to give esters [771]. Ruthenium(II) complexes catalyzed the addition of carboxylic acids to propargyl ethers to give enol esters [772]. The regiochemistry depended on the catalyst. Allenic alcohols were converted to cyclic carbonates under palladium catalysts (Eq. (229)) [773]. Palladium(II)/copper(II) catalyst systems promoted aminocarboxylation of alkenes to give α-amino esters [774].



(229)

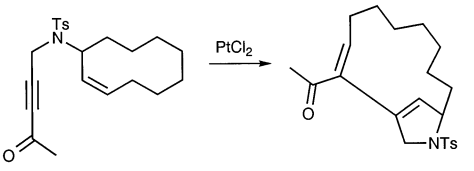
3.5. Heterocycles

Aziridines were made by the rhodium(II) catalyzed reaction of PhI=NNS with olefins [775], and the iron catalyzed addition of diazocompounds to imines [776]. Palladium(0) catalyzed the trans to cis isomerization of vinylaziridines [777]. β-Lactams were made by the $Fe_2(CO)_9$ catalyzed reaction of imines with trichloroacetic esters [778], rhodium(II) catalyzed C–H insertions of α-diazoamides of piperidines [779] and from iron-allyl chemistry (Eq. (230)) [780].

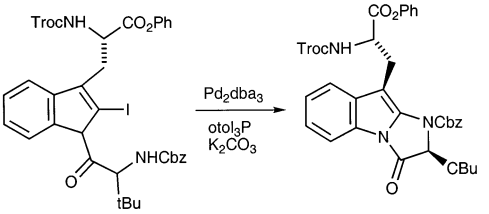


(230)

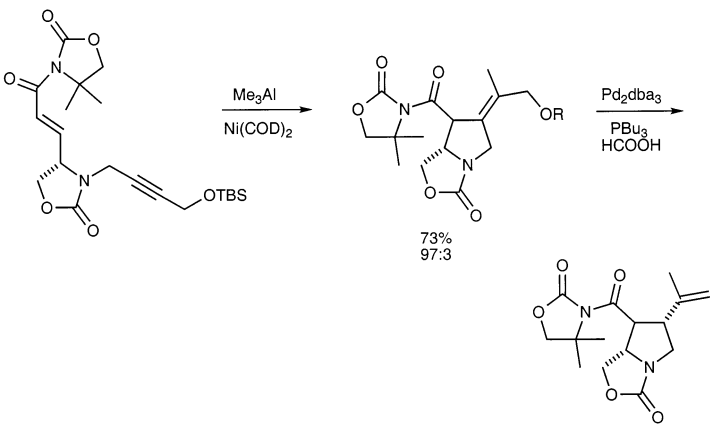
2-Vinylpyrrolidines were made by the palladium(II) catalyzed intramolecular amination of allenes [781]. 2,5-Dihydropyrroles were made using platinum(II) chemistry (Eq. (231)) [782]. Pyrrole-fused prolines [783] and 2-pyrrolidinones [784] were made by rhodium(II) catalyzed diazo C–H insertion. Rhodium(I) complexes catalyzed the production of 2-pyrrolidinones from alkynes, amines and carbon monoxide [785]. Palladium allylic alkylation chemistry [786] tungsten carbene chemistry [787] and palladium catalyzed amination of aryl iodides (Eq. (232)Eq. (232)) [788] made similar ring systems. Nickel catalyzed enyne cyclization (Eq. (233)) [789] and palladium catalyzed aryl halide-allene chemistry (Eq. (234)) [790] made five membered nitrogen heterocycles.



42% (231)



48% (232)



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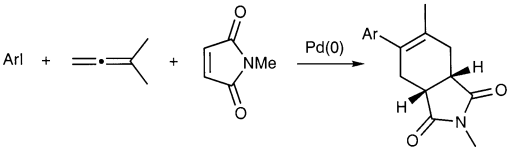
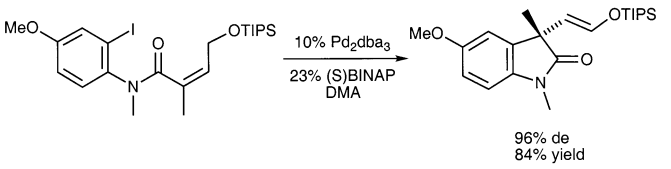


70-80% (234)

2-Vinylindolines were made by the palladium(0) catalyzed reactions of *o*-iodoanilines with dienes [791,792], the palladium(II) catalyzed cyclization of *o*-aminostyrenes in the presence of alkenyl halides [793], the palladium(0) catalyzed cyclization of 2-*iodo*-*N*-allyl anilines [794] and the palladium catalyzed cyclization of the *N*-benzyl enamines of iodoaniline [795,796]. Oxindoles were made by the palladium(0) catalyzed cyclization of *N*-acryloyl-*o*-iodoanilines (Eq. (235)) [797–799], and of *o*-bromo-*N*-acetamidoanilines (Eq. (236)) [800]. Palladium(0) catalyzed amination of 2-(bromoethyl)bromobenzene gave indolines [801].



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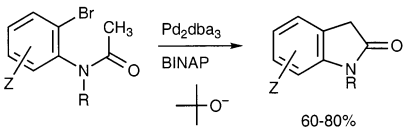
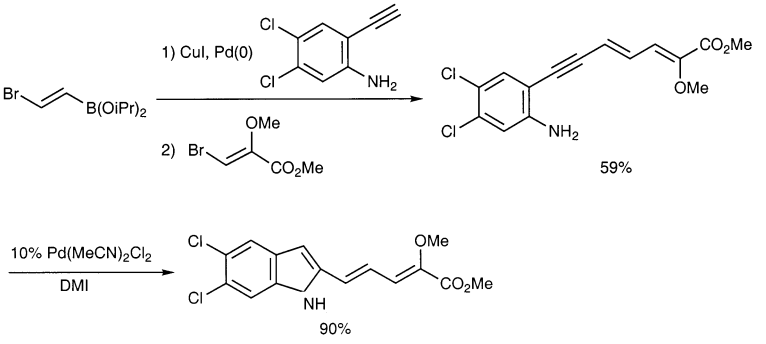


(236)

Indoles were synthesized by the palladium(0) catalyzed reactions of alkynes with o-iodoanilines [802,803], on solid supports [804], and from the palladium(0) catalyzed cyclization of o-aminophenyl acetylenes [805,806] (Eq. (237)) [807]. *o*-Iodo-*N*-propargyl anilines cyclized and captured alkenes under palladium(0) catalysts (Eq. (238)) [808], while palladium(II)/copper(II) systems oxidatively coupled *N*-aryl aminoquinones to indole-fused quinones [809]. Carbazoles were formed by the reaction of anilines with cyclohexadienyliron complexes (Eq. (239)) [810]. Ruthenium(III) chloride catalyzed the condensation of anilines with aminoalcohols to give indoles [811]. Rhodium(II) acetate catalyzed N–H insertion of anilines with α-diazoketo esters, the resulting product cyclized to indoles under acidic conditions [812].



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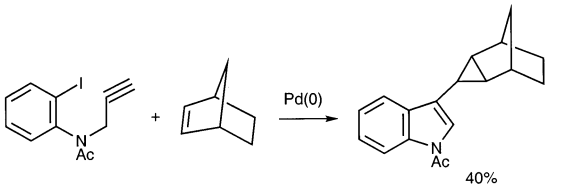

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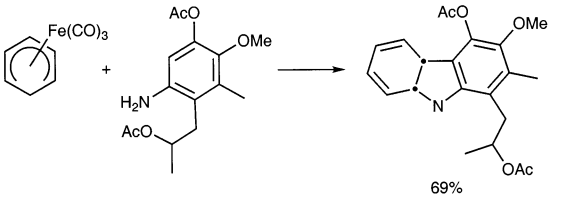
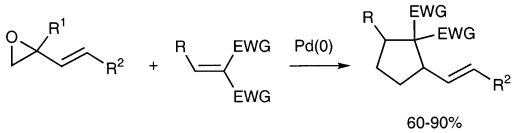


(239)

2-Vinyltetrahydrofurans were made by palladium(0) catalyzed cyclization of allyl carbonates [813] and the palladium(II) catalyzed cyclization of ω-olefinic alcohols [814,815]. 3-Vinyltetrahydrofurans were made by the palladium(0) catalyzed reactions of vinylidene malonate with allyl epoxides (Eq. (240)) [816]. Fused tetrahydrofurans were made by palladium(II) catalyzed alkoxy carbonylation of alkenes (Eq. (241)) [817], while five membered aminals resulted from the reaction of a zirconium azacyclopentene with ketones [818].



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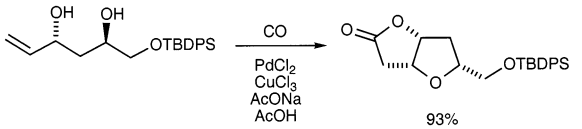
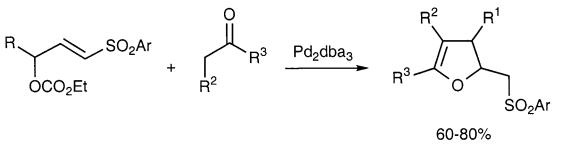


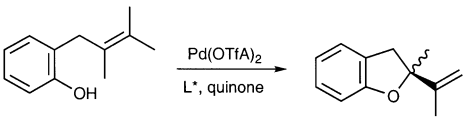
(241)

2,3-Dihydrofurans were synthesized by the palladium(0) catalyzed reaction of alkynes with γ-bromoallyl alcohols [819], of vinyl halides with allyl acetylacetonates [820], and of allyl carbonates with enolates (Eq. (242)) [821]. Rhodium(II) catalyzed decomposition α-diazo cyclohexane dione in the presence of acrylate esters gave 2,3-dihydrofurans [822], while ring closing metathesis of allyl propargyl ethers gave 2,5-dihydrofurans [823]. Tetrahydrofurans were prepared by palladium(II)/copper(I) catalyzed cyclization of γ-hydroxy-trimethylsilyl alkynes [824].



(242)

Dihydrobenzofurans were prepared by palladium(II) catalyzed cyclization of *o*-allylphenols (Eq. (243)) [825], the palladium(0) catalyzed cyclization of *o*-iodo allyl ethers of phenols under reducing (HCOOH) conditions [826], the palladium(0) catalyzed alkylation/cyclization of o-allylphenols (Eq. (244)) [827] and the ruthenium(II) catalyzed cyclization of *o*-allyl phenols [828].



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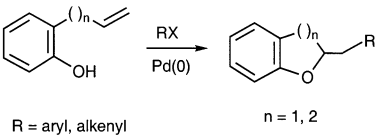
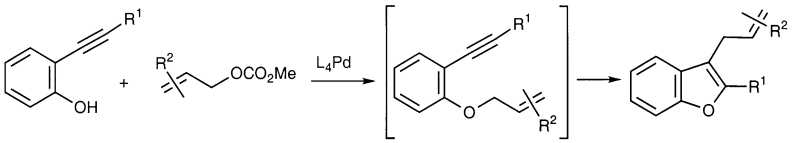


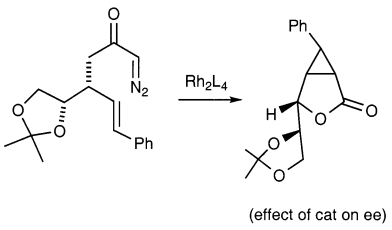
(244)

Palladium(II) complexes catalyzed the cyclization of o-allylphenols to benzofurans [829]. Palladium(0) complexes catalyzed the reaction of o-alkynyl phenols with allyl carbonates (Eq. (245)) [830,831] or propargyl carbonates [832] to give benzofurans, as well as the cyclization of propargyl ether of *o*-alkynyl phenols to give benzofurans [833]. Ortho iodo allenic ethers of phenols cyclized to 3-azidomethylbenzofurans when treated with sodium azide in the presence of palladium(0) catalysts [834].



(245)

Butyrolactones were synthesized by rhodium(II) catalyzed CH insertion reactions of α-diazoesters [835,836], intramolecular cyclopropanation (Eq. (246)) [837] and copper catalyzed Stevens rearrangements of α-diazoketone (Eq. (247)) [838]. Butyrolactones were also prepared by palladium(0) catalyzed akylative cyclizations of β-acetylenic carboxylic acids [839] and the palladium(II) catalyzed cyclization of β-allenic carboxylic acids [840]. Samarium iodide reduction of chromium complexed indanone in the presence of methyl acrylate gave the spiro fused butyrolactone from addition of indanone ketyl to the acrylate [841]. Pentitols and hexitols underwent reaction with rhodium(I) hydrides and chalcone to give glycono-1,4-lactones [842].



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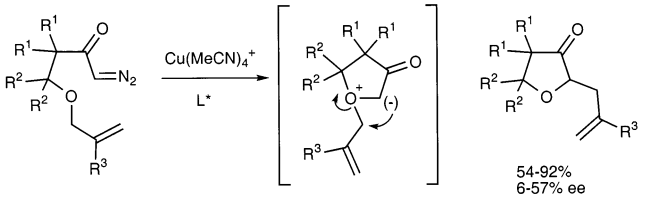
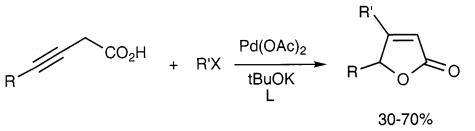
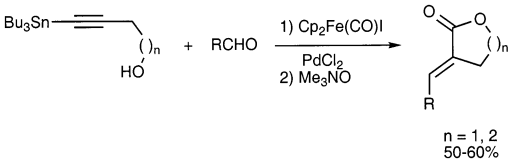


(247)

Butenolides were prepared by the palladium(0) catalyzed alkylative cyclization of allenic [843] and propargylic carboxylic acids (Eq. (248)) [844]. α-Methylene lactones were produced as in Eq. (249) [845] and Eq. (250) [846].



(248)



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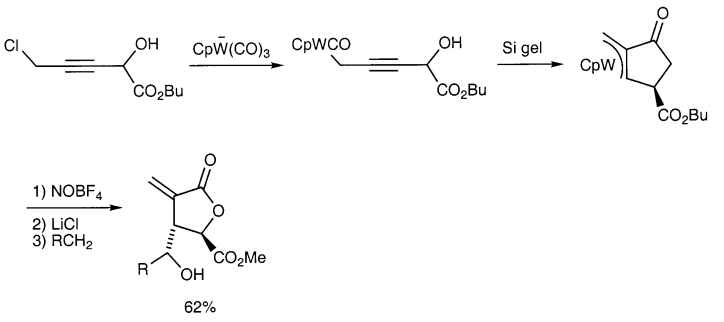
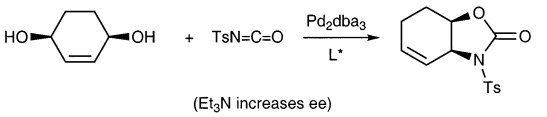


(250)

Palladium(0) catalyzed the formation of oxazolines from 4-*N*-benzoyl allyl acetates [847] and *N*-benzoyl oxazolidinones [848]. Rhodium(II) complexes catalyzed the formation of oxazolines from α-diazo-β-diketones and nitriles [849]. Chiral oxazolidinones were prepared by the palladium(0) catalyzed reaction of 1,4-dihydroxycyclohex-2-ene with tosyl isocyanante (Eq. (251)) [850] and (Eq. (252)) [851]. Rhodium catalyzed decomposition of α-diazoketones in the presence of α-amino esters gave oxazoles [852].



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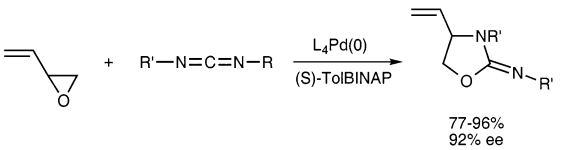
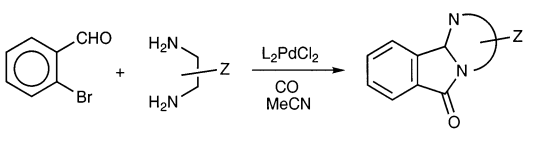
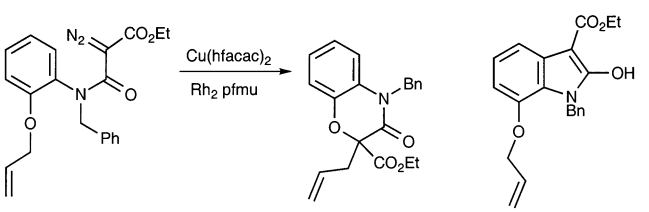


(252)

Δ-2-pyrazolines were made from chromium carbene complexes and chlorohydrazones [853] and oxadiazoles were made from the palladium(0) catalyzed reaction of aryl iodides and *N*-hydroxyamidines in the presence of CO [854]. Ureas reacted with chromium alkynylcarbene complexes to give pyrimidines after oxidation [855]. Palladium(0) catalyzed the bis amination of indole-2-carboxylate-3-triflates by orthophenylenediamine [856], and the alkylative cyclization of o-hydroxy-*N*-propargyl anilines with alkynes to give benzomorpholines [857]. Other polyheteroatom cyclization reactions are shown in Eq. (253) [858] and Eq. (254) [859].

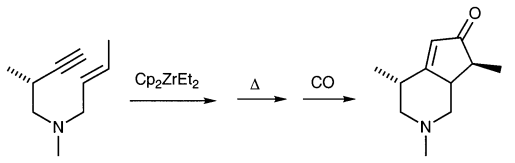


(253)



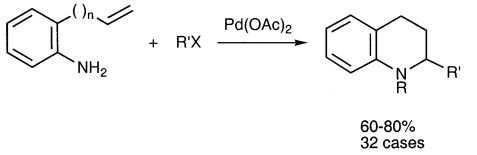
(254)

'Cp_2Zr' mediated the synthesis of symmetric 3,4-disubstituted piperidines by reductive cyclization of unsaturated amines [860] (Eq. (255)) [861]. Piperidines were also synthesized by palladium(II) catalyzed intramolecular amination of allyl alcohols [862], cobalt carbonyl assisted intramolecular amination of propargyl epoxides [863], rhodium(I) catalyzed cyclization of γ-aminoalkyenes (cyclic imine) [864] and the rhodium(I) catalyzed carbonylative cyclization of 4-amino-1,6-heptadienes [865].



(255)

Palladium catalyzed syntheses of tetrahydroquinolines (Eq. (256)) [866]; (Eq. (257)) [867], dihydroisoquinolines (Eq. (258)) [868], (Eq. (259)) [869], quinolines (Eq. (260)) [870]; (Eq. (261)) [871]; (Eq. (262)) [872], and isoquinolines (Eq. (263)) [873] are shown below.



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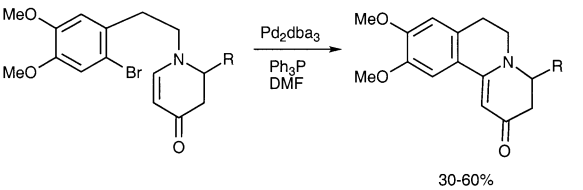

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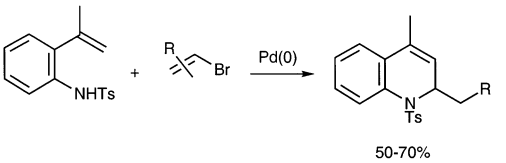

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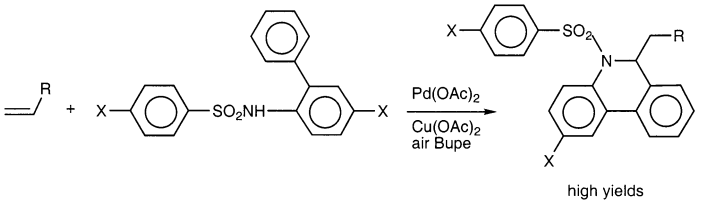

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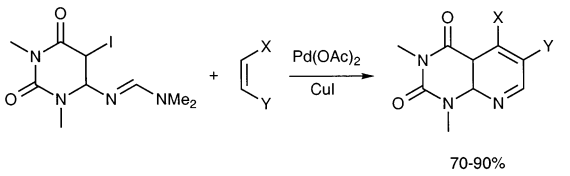

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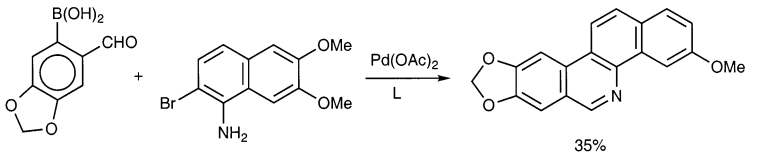

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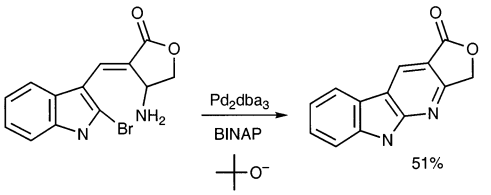

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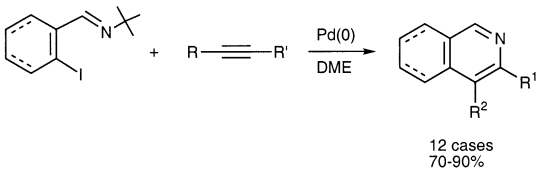
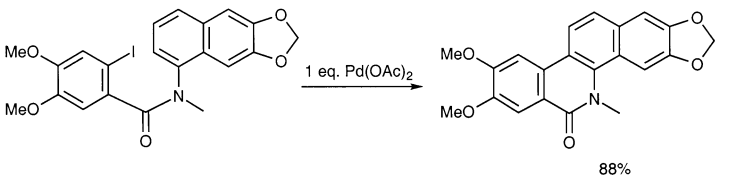


(263)

A variety of quinolines were made by Heck-type chemistry (Eq. (264)) [874–876] and palladium catalyzed allylic aminations (Eq. (265)) [877].



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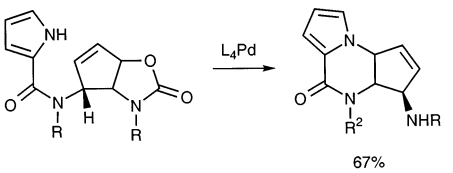
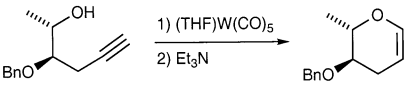


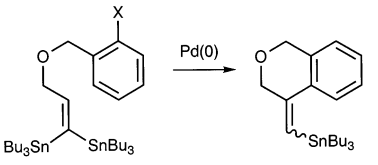
(265)

Tetrahydropyrans were synthesized by the cobalt carbonyl assisted cyclization of ω-hydroxy propargyl epoxides [878], and the palladium(II) catalyzed intramolecular hydroxylation of cyclohexadienes [879]. Dihydropyrans were made by the tungsten catalyzed cyclization of γ-hydroxyalkynes (Eq. (266)) [880,881].



(266)

2-H-1-benzopyrans were synthesized by palladium(0) catalyzed alkylative cyclization of ortho allylphenols [882,883], and by molybdenum(0) catalyzed cyclization of allyl phenyl ethers [884]. Other six membered oxygen heterocycles were synthesized as in Eq. (267) [885], Eq. (268) [886a], and Eq. (269) [886b].



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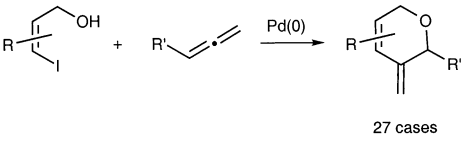

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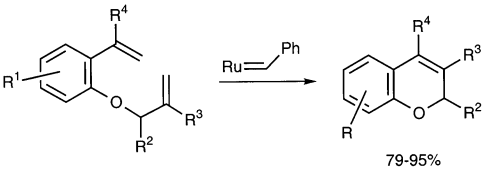
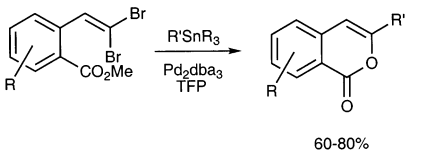
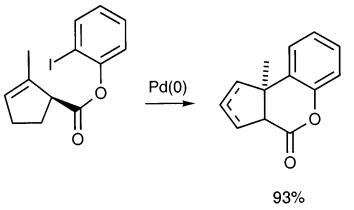


(269)

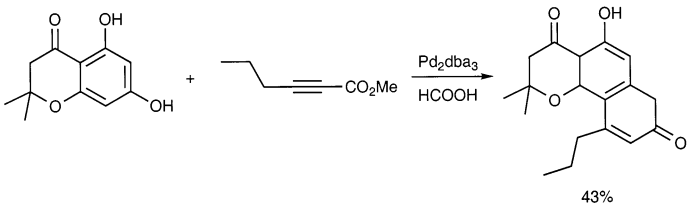
α-Methylene six membered lactones were made by platinum(0) catalyzed carbonylative cyclizations of ω-hydroxyalkynes [887,888], and by tungsten carbene chemistry [889]. Isocoumarins were formed by palladium(0) catalyzed cyclizations (Eq. (270)) [890], (Eq. (271)) [891], and by palladium(0) cyclization of propargyl esters with phenols (Eq. (272)) [892] α-Pyrones resulted from the reaction of β-iodo conjugate esters with alkynes in the presence of palladium catalysts [893], while benzodioxanes resulted from palladium catalyzed alkylative cyclization of monopropargyl ethers of o-hydroquinone [894]. Rhodium(II) catalyzed diazodecomposition produced oxygen heterocycles (Eq. (273)) [895] and (Eq. (274)) [896].



(270)



(271)



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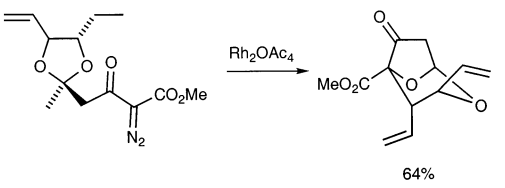

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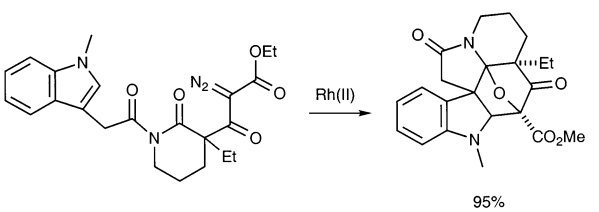
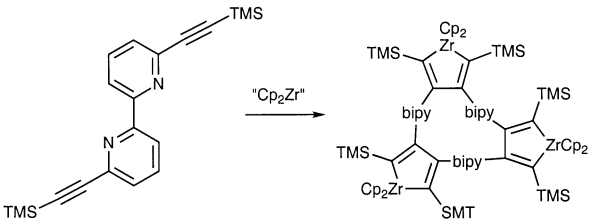


(274)

The synthesis of seven membered oxacycles has been reviewed (127 references) [897]. A variety of seven and eight membered oxacycles have been made by metal catalyzed diazo decomposition [898–900]. Azepines were made by the palladium catalyzed reaction of allenes with 2-iodo-6-benzylamino hexene [901]. Eq. (275) is a fitting way to end this section on heterocycles [902].



(275)

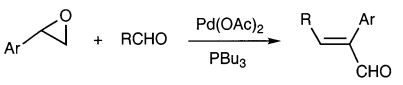
3.6. Alkenes, alkanes

Palladium(0) complexes catalyzed the elimination of allyl carbonates to dienes [903,904] and the reduction of gem vinyl dibromides to vinyl bromides [905]. Tetrasubstituted olefins were made by the $Cp_2Ti(OiPr)_2$ assisted methylenation of ketones by gem diiodoalkanes [906]. Ruthenium(II) complexes catalyzed the reaction of aldehydes with α-diazoesters to produce conjugated esters [907] and the rearrangement of β-alkoxy-α-diazo esters to α-alkoxyenones [908].

Allyl carbonates were reduced to alkenes with high ee by Pd_2dba_3, formic acid and proton sponge in the presence of chiral ligands [909,910]. Samarium iodide in the presence of palladium(0) also reduced allyl carbonates, acetates, and phosphates to olefins [911]. Phenyl silane reduced conjugated ketones 1,4 in the presence of $[Ph_3PCu]$ [912]. The complex (*R*,*R*)-EtDuPhos)Rh^+ selectively reduced α,β-γ,δ-unsaturated amino acids at the α,β-double bond with high ee [913]. Samarium iodide reduced chromium tricarbonyl complexes of styrenes to complexes of ethyl benzenes [914], while sodium cyanoborohydride reduced chromium indole complexes, to indoline complexes [915] and potassium hydride desilylated chromium complexed silyl arenes [916].

3.7. Ketones, aldehydes

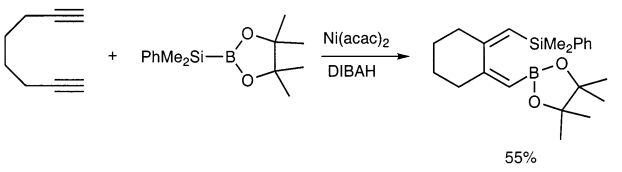
Water soluble catalysts for Wacker oxidation of alkenes to ketones have been developed [917], as have improved catalysts requiring only catalytic amounts of copper [918], zeolite supported catalysts [919] and biphasic catalysts [920]. The active oxidizing species in Wacker oxidation has been tentatively identified [921]. Palladium acetate catalyzed the oxidation of primary, secondary [922], benzylic, and allylic alcohols [923], as well as the oxidation of ketones to α,β-unsatured ketones [924]. Ruthenium(II) complexes deallylated homoallylic alcohols to give ketones [925]. Palladium acetate converted trimethylsilyl dienol ethers to γ-acetoxy unsaturated aldehydes [926]. Palladium acetate catalyzed the condensation of styrene oxides with aldehydes (Eq. (276)) [927]. Amine oxides oxidized α-OH groups to aldehydes on diene iron complexes [928].



(276)

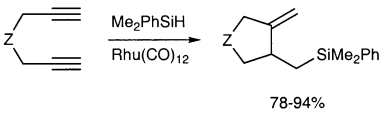
3.8. Organoboranes, silanes, germanes and stannanes

Platinum complexes catalyzed the 1,2-bis addition of $(RO)_2BB(OR)_2$ to allenes to give mixtures of regioisomers [929]. Reduced nickel catalysts dimerized alkynes with silylboronates (Eq. (277)) [930], while stannyl boranes added 1,4 to conjugated dienes in the presence of palladium(0) catalysts [931].



(277)

'Zirconocene' catalyzed the conversion of terminal alkynes into terminal alkenyl silanes, and internal alkenes into allyl silanes by triethylchlorosilane [932]. Rhodium complexes catalyzed the direct silylation of arenes by triethylsilane [933], and the Michael hydrosilylation of conjugated amides (Si α to C=O) [934]. Copper(II) triflate catalyzed the β-silylation of enones [935] while palladium(II) complexes catalyzed the intramolecular hydrosilylation of alkenes to give cyclic silanes [936]. Rhodium catalyzed silylcyclization are shown in Eq. (278) [937] and Eq. (279) [938] while a related palladium catalyzed one is shown in Eq. (280) [939].



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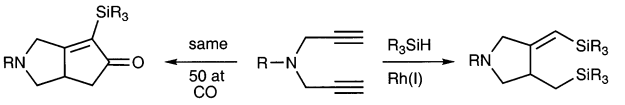

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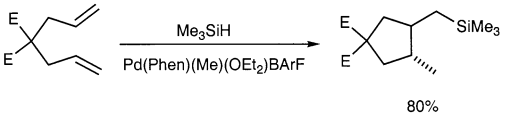
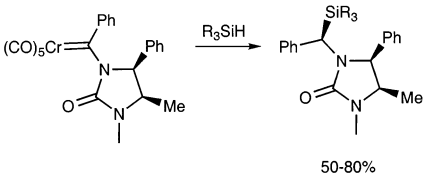
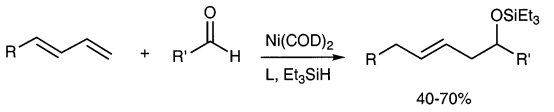


(280)

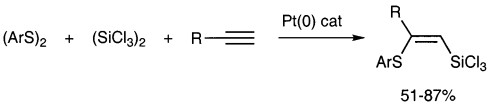
Other unusual silylation processes are shown in Eq. (281) [940], Eq. (282) [941], Eq. (283) [942], and Eq. (284) [943].



(281)



(282)



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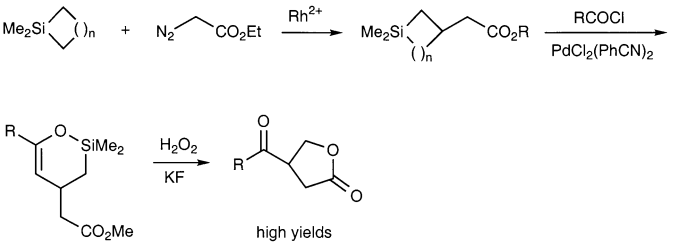

(284)

Treatment of zirconacyclopentanes, -pentenes, and -pentadienes with R_2SnCl_2 and a copper catalyst replaced zirconium with tin [944]. Palladium catalysts promoted the addition of alkynylstannanes to alkynes to give β-alkynyl vinylstannanes [945].

3.9. Organophosphorous and sulfur compounds

Palladium(0) complexes catalyzed the coupling of aryl bromides with thiophenols and was used to make polyphenylenesulfide 'molecular wires' [946]. Similar complexes catalyzed the coupling of aryl triflates and thiophenols [947], allyl carbonates [948], and allyl acetates [949] with thiols and sulfinates respectively, and the reaction of allyl alcohols with thiols and carbon monoxide to give β,γ-unsaturated thioesters [950].

Palladium acetate catalyzed the reaction of allenes with tosyl hydrazine to give allyl sulfinates and N_2 [951]. Copper iodide promoted the coupling of silyl acetylene

with aryl selenyl bromides to give arylselenyl acetylene (replace Si with Se) [952]. Tungsten(VI) chloride catalyzed the reaction between dithiols and ketones or ketals to give dithianes [953]. Ruthenium(0) [954] and palladium(0) [955,956] catalyzed reaction between $Ph_2P(O)(OH)$ and $Ph_2P(O)H$ respectively to give the corresponding addition products.

4. Reviews

The following reviews have appeared.

- Chemical biology of epothiolones (138 references) [957]
- Recent advances in the generation of non-racemic ferrocene derivatives and their application to asymmetric synthesis (122 references) [958]
- Titanocenes as electron transfer catalyst: reagent controlled catalytic radical reactions (37 references) [959]
- Recent advances in asymmetric catalytic metal carbene transformations (181 references) [960]
- Transition metals in organic synthesis: highlights for the year 1996 (1135 references) [961]
- The catalytic enantioselective construction of molecules with quaternary carbon stereocenters (61 references) [962]
- Application of transition metals to hydroformylation. Annual survey 1996 (133 references) [963]
- Alkene and alkyne complexes of zirconocene: their preparations, structure, and novel transformations (69 references) [964]
- Allylic protecting groups and their use in a complex environment part II. Allylic protecting groups and their removal through catalytic palladium π-allyl methodology (231 references) [965]
- Catalytic asymmetric epoxidations and aziridination mediated by sulfur ylides, evolutions of a project (48 references) [966]
- Symmetric chiral bis(oxazoline)-metal complexes in catalytic asymmetric synthesis (>89 references) [967]
- Catalytic applications of transition metals in organic synthesis (143 references) [968]
- Transition metals in organic synthesis 1997 (912 references) [969]
- Transition metal catalyzed reactions using glass bead technology (59 references) [970]
- Tricarbonyl iron complexes: an approach to acyclic stereocontrol (30 references) [971]
- Catalytic asymmetric processes (314 references) [972]
- Stereoselective intramolecular C–H insertion reactions of metal carbenes (56 references) [973]
- Recent developments in solid phase synthesis (214 references) [974]

- Lectures from 9th IUPAC OMCOS [975].
- A 'Tetrahedron Symposium in Print' dealing with nickel-catalyzed processes contained the following articles:
- Carbon halogen bond activation by nickel catalyst: synthesis of alkenes, from 1,2-dihalides [976].
- β- And γ-lactams by nickel powder mediated 4-*exo* and 5-*endo* radical cyclisations. A concise construction of the mesembrine skeleton [977].
- Nickel-catalyzed regio- and stereoselective homo 1,4-dialkenylation of conjugated dienes [978].
- Nickel-catalyzed coupling reaction of sterically congested cis bromides and lithium alkenylborates [979].
- Nickel-catalyzed tandem coupling of allyl electrophiles, alkynes, and alkynyltins [980].
- Synthetic and kinetic aspects of nickel-catalysed amination of allylic alcohol derivatives [981].
- A straightforward route to polyenylsilanes by palladlium- or nickel-catalyzed cross-coupling reactions [982].
- Catalytic generation and trapping of acylmetals containing Ni and Cu with enolates [983].
- Scope of the nickel catalyzed asymmetric reductive ring opening reaction. Synthesis of enantiomerically enriched cyclohexenols [984].
- Phosphine-directed stereo- and regioselective Ni-catalyzed reactions of Grignard reagents with allylic ethers [985].
- Synthetic studies and mechanistic observations in nickel-catalyzed polycyclizations [986].
- *Ortho*-bis(amino)arylnickel(II) complexes containing perfluoroalkyl chains as model catalyst precursors for use in fluorous biphase system [987].
- Stereoselective synthesis of nitrogen-containing heterocycles via nickel-catalyzed cyclization of 1,3-diene and aldehyde: formal total synthesis of (−)-elaeokanine C [988].
- Carbocyclic ring expansions with alkyne and carbene sources mediated by nickel(0) complexes: structure of the critical organonickel intermediates [989].
- Ni-catalyzed nucleophilic conjugate additions of Grignard and organozincate reagents to substituted 4-vinylpyridines. General synthesis of phosphodiesterase IV inhibitors [990].
- A general synthesis of vinylic silyl hydrides using nickel catalysis. Applications to the syntheses of silylene-tethered conjugated polymers [991].
- Stereocontrol in nickel mediated syntheses of cyclobutabenzenes. The selective preparation of cis and trans derivatives of 7,8-dibromocyclobutabenzene [992].
- Diastereocontrolled synthesis of pyrrolidines by nickel promoted tandem cyclization-quenching of aminobromodienes [993].
- Nanostructured nickel-clusters as catlaysts in [3 + 2]cycloaddition readtions [994].

- An expeditious route to CoQ_n, vitamins K_1 and K_2, and related allylated *para*-quinones utilizing Ni(0) catalysis [995].
- Transition metal-catalyzed intramolecular [4 + 2] cycloadditions: mechanistic and synthetic investigations [996].
- Pyridine-based mono(ligand)nickel(0) complexes of 1,6-heptadiene, 1-phenyl-2-trimethylsilylacetylene and 1,4-bis(trimethylsilyl)-1,3-butadiyne [997].
- Electrochemical cross-coupling between 2-halopyridines and aryl or heteroaryl halides catalysed by nickel-2,2′-bipyridine complexes [998].
- A nickel-catalyzed carbozincation of aryl-substituted alkynes [999].

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