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Coordination Chemistry Reviews 247 (2003) 79–145
COORDINATION CHEMISTRY REVIEWS
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Transition metals in organic synthesis: highlights for the year 2001

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Accepted 27 June 2003

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doi:10.1016/S0010-8545(03)00123-1

Abstract

A review with 1663 references to transition-metal catalyzed or mediated reactions and functional group preparations.

Keywords: Transition metals; Organic synthesis; Catalysis

1. General comments

This survey highlights the use of transition metals in organic synthesis for the year 2001. A fairly comprehensive literature coverage with extensive citations is presented. The field of organometallic chemistry continued to significantly expand in 2001. Considering the large number of papers published, a complete coverage of all reported reactions employing transition metals is outside the scope of this review. A number of transition-metal catalyzed transformations are now standard reactions in most organic chemists' toolbox. Thus, some of the more common applications have not been included. Reactions of unusual substrates, new reaction conditions, and new catalyst systems have been included. Applications in total synthesis of more complex organic molecules have also been reviewed. Only the more unusual or significant reactions were presented in equation form. Some emphases were put on reactions used in total synthesis of biologically active compounds. Alkylations and most Michael-type reactions of pre- or intermediately formed copper reagents have not been reviewed. The chemistry of metal carbon multiple bonds, e.g. reactions of Fischer and Schrock carbenes, are covered in a separate review, although decompositions of diazo compounds and metathesis reactions were included herein. Papers describing new ligands for palladium-catalyzed asymmetric allylic alkylation of, primarily, 1,3-diphenyl-allylacetate and most hydroformylations have not been included.

2. Carbon–carbon bond-forming reactions

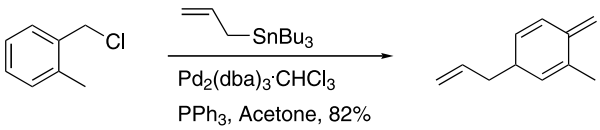
2.1. Alkylations

2.1.1. Alkylations of organic halides, tosylates, triflates, nonaflates and hypervalent species

Palladium-catalyzed coupling reactions of organotin reagents with a large variety of electrophiles continue to be developed. This reaction is generally referred to as the Stille reaction, and this name will be used herein. Reactions in ionic liquids [1] and supercritical carbon monoxide [2] were reported. Microwave-assisted Stille reactions on potassium fluoride–alumina in the absence of solvent was reported [3]. Stille-type coupling catalytic in tin was described [4,5]. The use of the air-stable ligand precursor $[(t\text{-Bu})_3\text{PH}]\text{BF}_4$ was reported [6]. Addition of diethylamine increased the yield of coupling products and reduced the amount of β-hydride elimination using tetralkylstannanes [7].

Bromo-1,4-benzoquinone, protected bromo-hydroquinones [8], and *N*-alkoxyimidoly bromides [9] were used as electrophiles in Stille couplings. Carboxylic acid chlorides were used in place of halides forming unsymmetrical ketones [10]. 3,4-Dibromo-2(5*H*)-furanone underwent regioselective coupling with arylstannanes to give 4-aryl-3-bromo-2(5*H*)-furanones [11].

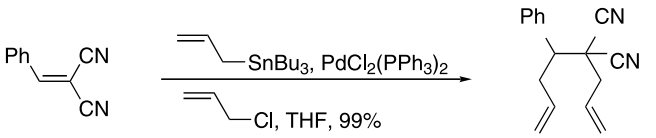
Stille-type couplings of allylic acetates having a removable dimethylpyridylsilyl group were described [12]. Allylic chlorides were coupled with allyl tributyltin [13]. A facile dearomatization was catalyzed by palladium (Eq. (1)) [14]. Vinyl epoxides were reacted with vinyltin reagents [15]. A (2-pyridyldimethylsilyl)methyl group was transferred using (2-pyridyldimethylsilyl)methyl tributyltin and aryl halides [16]. Intermolecular coupling of 2-bromopyridines with aryl bromides in the presence of hexamethylditin and a palladium catalyst was reported [17].



(1)

Tributylstannylcyclooctatetraene [18,19], an α-tributylstannyl enamide [20], a 2,2-difluoro-1-tributylstannyl MEM protected vinyl ether [21], (*E*)-tributylstannylpropanoyltriphenylsilane [22], a vinylstannane-substituted quartenary amino acid [23], an organostannatrane [24], and a fluorinated acylstannane [25] were used in cross-coupling reactions. Couplings of vinyl and aryl halides and triflates with hypervalent organostannanes in the presence of a palladium-catalyst were reported [26,27]. 1,2-Bis(tributylstannyl)ethene and 1,2-bis(tributylstannyl)ethyne were used to prepare brassinolide mimetics [28].

A double allylation of activated alkenes using allyl chlorides and allyltin reagents was described (Eq. (2)) [29]. This reaction was utilized in an interesting [*n*+2]cycloaddition using tin reagents. A cascade reaction using 1,2-ditin-substituted ethane forming polycyclic compounds was described (Eq. (3)) [30].



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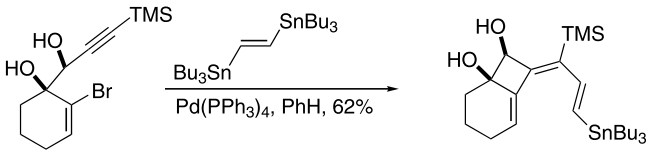

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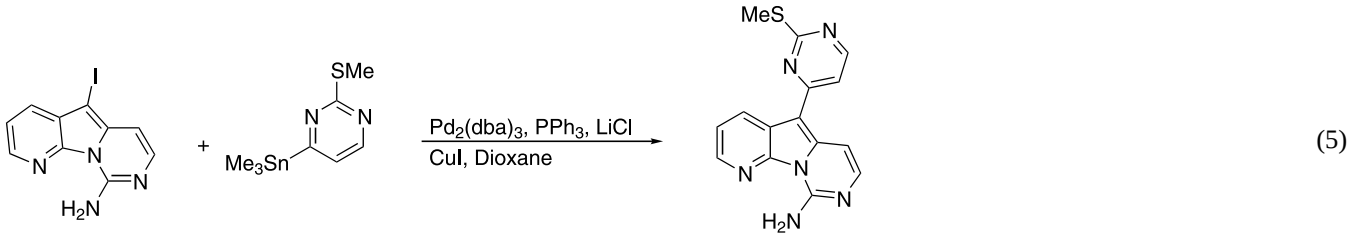
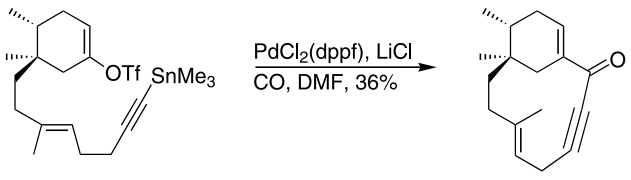


(5)

Nickel catalyzed the selective coupling in the 2-position of pentafluoropyridine with vinyltributyltin [31]. Copper mediated the coupling of silyloxyvinylstannanes with 1-iodo-1-hexene [32]. A copper-mediated coupling of a vinyl iodide with a vinyltin reagent was used in synthesis of himbacine derivatives [33]. A carbonylative coupling of a polymerbound aryltin compound with aryl halides was used to prepare diaryl ketones [34]. Carbonylative couplings were used in the synthesis of the core of phomactin (Eq. (4)) [35], of acetophenones [36], and of γ- and δ-fluorine-substituted ketones [37].

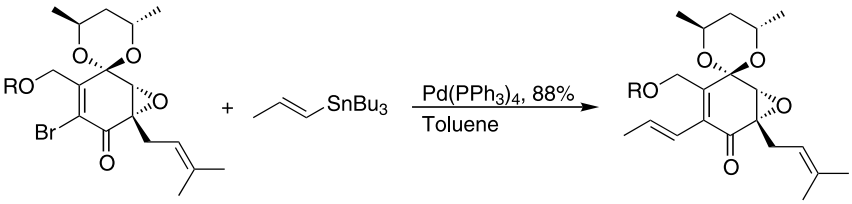


(4)

A large number of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and vinyltin reagents. For example, reactions of derivatives of 4-bromo- and 4-bromomethyl-oxazole [38], 4- and 5-iodoisoxazole [39,40], 4-iodoimidazoles [41], 5-trifloxyoxazole [42], 5-iodo-2(2*H*)-pyranones [43], halopyrimidines [44,45], 7-iodoisatins [46,47], halopyridines [48,49], bromo-, chloro-, and trifloxyquinolines and isoquinolines [50], 4-iodoindole [51], 5-bromo-5-azaoxindole [52], 1-trifloxy-β-carboline in the synthesis of pyridindolols [53], 8-bromo-1-ribofuranosidylpurin [54], polymer-bound 8-bromopurine [55], bromopyrazines [56,57], 3- and 5-chloro-2-pyrazinone [58,59], and 2-bromobenzo-thiophen and -furan [60] were reported. Coupling of 2,3-dibromothiophene [61] and 2,5-dibromopyridine [62] selectively occurred in the 2-position. Stille reaction of a 3-iodo-7-azaindole derivative was used in the synthesis of deoxyvariolin B (Eq. (5)) [63].

A variety of heterocyclic stannanes such as 2-pyridinyl [64–66], 4-pyridinyl [67], 3-2′2′-bipyridinyl [61], 2-thienyl [68–72], 2-furyl [73], 4-oxazolyl [38], 3-cephalosporin [74], 3-benzofuryl [75], 2-indolyl [60,76], tetrathiafulvalenyl [77], 4-imidazolyl [78], 4-pyrazolyl and 4-isoxazolyl [79] were used in Stille-type couplings.

The Stille reaction continued to be extensively used in organic total synthesis [80–84]. Synthetic targets include, concanamycin F [85], fostriecin [86], ratjadone [87], (−)-reveromycin B [88], rutamycin B and oligomycin C [89], analogues of CC-1065 and duocarmycins [90], lankacidins [91], (−)-physostigmine, (−)-physovenine, and (−)-aphanorphine [92], (*S*)-ipsenol [93], (−)-ichthyothereol [94], HIV-1 reverse transcriptase inhibitors [95], 9-*cis*-renioic acid analogs [96], oxadecaline core of phomactin A [97], (−)-jesterone (Eq. (6)) [98], toward FR182877 [99], taxifolial A and *iso*-caulerpenyne [100], (−)-salicylhalamide [101], reserpine [102], (+)-crocacin C [103], rugulovasines [104], (+)-amphidinolide K (Eq. (7)) [105], phorboxazole A [106], epothilones [107], lapidilecitin B [108], asperazine (Eq. (8)) [109], bafilomycin A_1 [110], (+)-phorboxazole A [111], (−)-cycloepoxydon [112], dioncophyllin B [113], (−)-hennoxazole [114], herbertendiol [115], dermostatin A [116], toward viridenomycin [117,118], lancifolol (Eq. (9)) [119], toward bufadienolides [120] FGHI ring system of azaspiracid [121], sanglifehrin A [122], fostriecin [123], a subunit of oximidines [124], epibatidine [125], benzo[*b*]phenanthridines [126], an analog of medermycin [127], astaxanthin analogs [128], and ellipticine [129].



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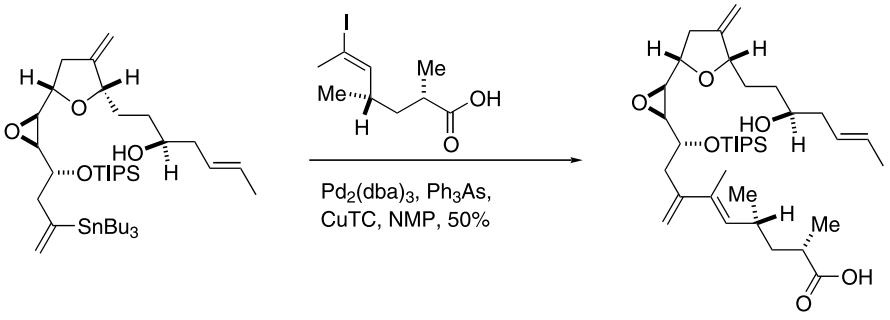

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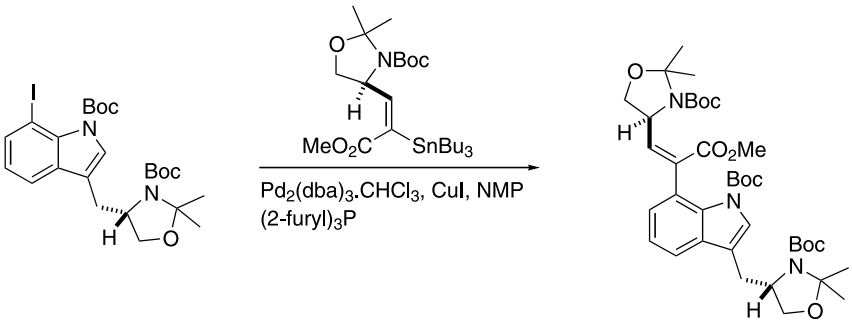

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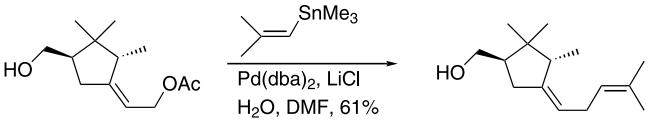


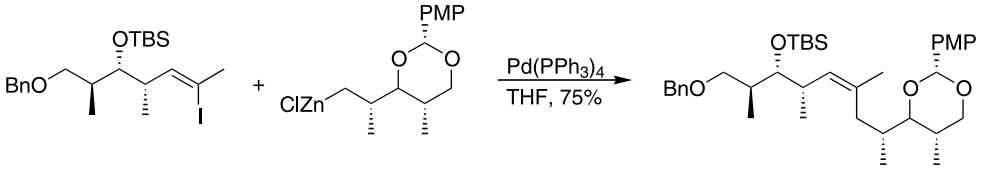
(9)

Intramolecular Stille-type macrocyclization was used in synthesis of concanamycin F [85] and toward apoptolidin [130]. Intermolecular formation of a stannane using trimethylsilyl tributyltin followed by an intramolecular coupling with a tethered electrophile was used in synthesis of nodulisporic acid A subunit [131].

Nickel and palladium complex-catalyzed cross-couplings of Grignard reagents with halides and triflates [132] were used in synthesis of (+)-ambruticin [133] and (−)-hennoxazole [114]. Coupling of a vinyl triflate with trimethylsilylmagnesium bromide was used in the synthesis of laulimalide [134]. Coupling reactions of bromopyridines [135], vinyl triflates, in the synthesis of hydroazulene ring system of guanacastepene, [136], 3-bromofuran [137], and bromothiophens [138] with Grignard reagents were reported.

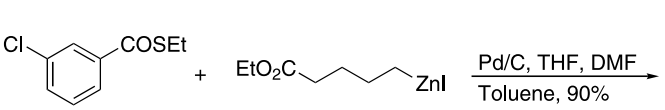
Nickel catalyzed the coupling of aryl Grignard reagents with aryl nitriles to give unsymmetrical biaryls [139]. Nickel-catalyzed asymmetric cross-coupling of a benzylic Grignard with vinyl bromide was reported [140]. Copper catalyzed the coupling of vinyl triflates with Grignard reagents [141]. Copper also catalyzed enantioselective reactions of allylic acetates with Grignard reagents [142,143]. Iron catalyzed the coupling of vinyl bromides and iodides with aryl Grignard reagents [144].

Coupling reactions of halides and triflates with organozinc reagents, usually referred to as the Negishi coupling, continued to grow as a viable alternative to tin and boron reagents [23,145–148]. Nickelocene catalyzed the coupling of aryl bromides with arylzinc reagents [149]. Palladium-catalyzed cross-coupling of organozincs with 1-trimethylsilyl-1-iodoalkenes was used in synthesis of (*Z*)-trisubstituted alkenes (Eq. (10)) [150].



(10)

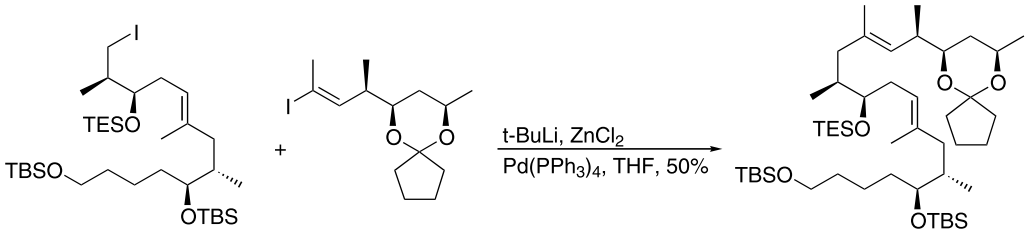
Bis(trimethylsilylmethyl)zinc was used, introducing a trimethylsilylmethyl group [151]. Aryl iodides were coupled with benzylzinc chlorides [152], alkynylzinc [153,154], and cyclopropylzinc reagents [155]. Aryl and vinyl chlorides were coupled with zinc reagents [156]. An acid halide was coupled with an amino acid derived zinc reagent [157]. Palladium catalyzed the coupling of thioesters with zinc reagents to give polyfunctionalized ketones (Eq. (11)) [158]. Alkyl zinc reagents coupled with aryl and heteroaryl halides [159–163] without β-elimination.



(11)

Heterocyclic substrates also participated in Negishi couplings. For example, 4-tosylcoumarins [164], 4-diethylphosphonyloxycoumarin [165], 3-bromobenzothiophen [166], 5-iodoisoxazole [39], halopyridines and phenanthrolines [167,168], terpyridyltriflate [169], bromo-, chloro-, and trifloxyquinolines and isoquinolines [50], and iodopyrazoloisoquinolines [170]. Imidazolylzinc on a solid support was also used in coupling reactions [171]. Heterocyclic zinc reagents were used, including 3-pyrazolyl-1-oxide [172], 2-4-pyridinyl [173,174], 5-(1,3-dioxin-4-one)yl [175], 5-pyranyl-2-ones [176], 3-indolyl [177], and 2- and 5-thiazolyl [178] derivatives.

Negishi-type cross-coupling reactions were used in the synthesis of (−)-deoxypukalide [179], carotenoids [180], neodysherbaine A [181], the core of phomactin [35], the core of mycolactones (Eq. (12)) [182], toward gambierol [183], and (*Z*)- and (*E*)-γ-bisabolene [184]. A sequence of coupling reactions was used in the synthesis of cystothiazole E (Eq. (13)) [185]. Hydrozirconations of terminal alkynes followed by transmetallation to zinc and coupling with vinyl iodides were used in the synthesis of euncenone [186] and FR901464 analogs (Eq. (14)) [187]. Transmetallation from aluminum to zinc of an intermediately formed vinylaluminum species followed by palladium-catalyzed cross-coupling was used in the synthesis of carotenoids [180].



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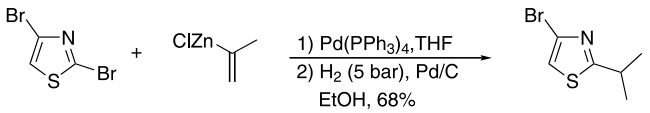

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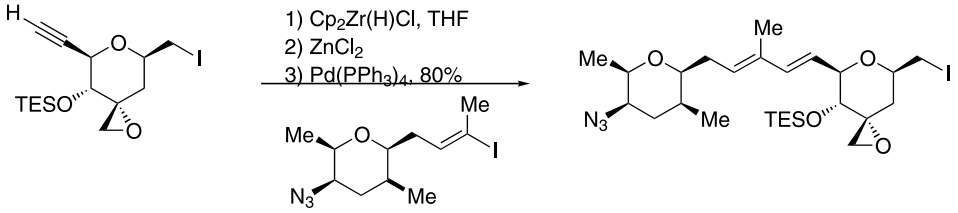
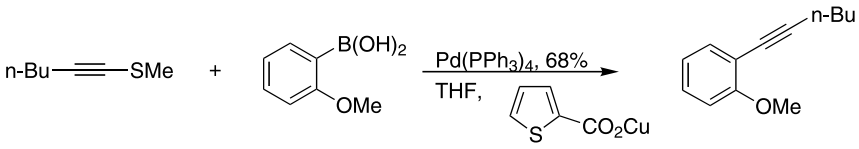


(14)

The Suzuki (or Suzuki–Miyaura) cross-coupling, i.e. palladium-catalyzed coupling of organoboronic acids and esters with organic halides and triflates, continued to see substantial use in organic chemistry [159,188,189]. A number of new ligands and catalyst systems were developed [190–199]. Reactions catalyzed by a palladium–diazabutadiene system [200], palladium on carbon in the absence of ligands [201,202], and palladium mediated by copper thiophene-2-carboxylate at room temperature [203] were reported. Heterocyclic carbene ligands [204,205] and the use of the air-stable ligand $[(t\text{-Bu})_3PH]BF_4$ were reported [6].

Coupling of aryl halides with organoboronic acids in aqueous solution catalyzed by palladium complexes [206–208] and microwave assisted reactions on potassium fluoride–alumina in the absence of solvent were reported [3]. Polymer supported Suzuki reactions attracted a fair amount of attention. Polymer supported palladium catalysts [209,210] and a polymer-supported ligand were used [211]. Polymer bound boronic esters [212], polymer bound aryl halides [213–215], 2-iodoindole [216], and a vinyl bromide [217] were used in coupling reactions. Molecular imprinting was used to prepare heterogenous catalysts [218].

Dicoupling of a vinyl-1,1-bis(pinacolyl)diboron [219], coupling of quinone boronic ester derivatives [220], cyclopropylboronic acid esters [146,221], α-triflouromethyl ethenyl boronic acid [222], and hydroboration of 1-alkynylphosphonates followed by Suzuki coupling were described [223]. Thioalkynes were coupled with boronic acids in the presence of a palladium catalyst and a copper carboxylate (Eq. (15)) [224]. Trifloxyazulene was coupled with an alkylboronic ester in the synthesis of KT1-32 [225]. A regio and stereoselective coupling of arylboronic acids with peracetylated glycals was described (Eq. (16)) [226]. β-Chloroacroleins were coupled with arylboronic acids in aqueous media [227]. β-Chlorovinamidium salts [228], 1,1-difluoro-4-bromo-3-triisopropylsilyl-1,2-butadiene (Eq. (17)) [229], and bromoacetic acid derivatives [230] were coupled with boronic acid derivatives. A carbonylative coupling of halopyridines with arylboronic acids was reported (Eq. (18)) [231]. Phenyltrifluoroacetate was coupled with organoboron compounds to give trifluoromethylketones [232]. Aryl tosylates were coupled with arylboronic acids using a nickel catalyst [233]. Intermediately formed anhydrides were coupled with arylboronic acids, using a palladium catalyst, to give ketones [234].



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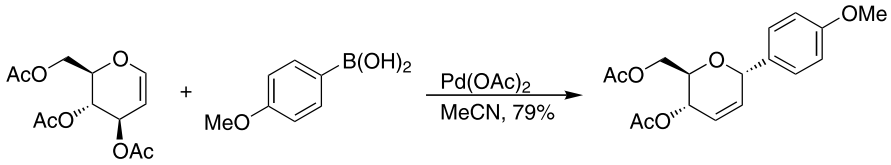

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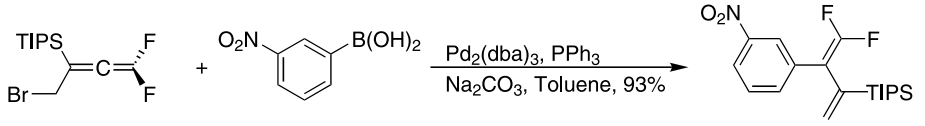

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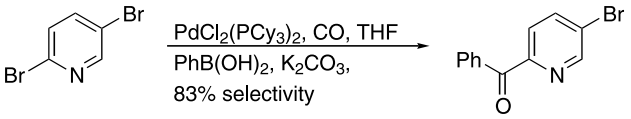
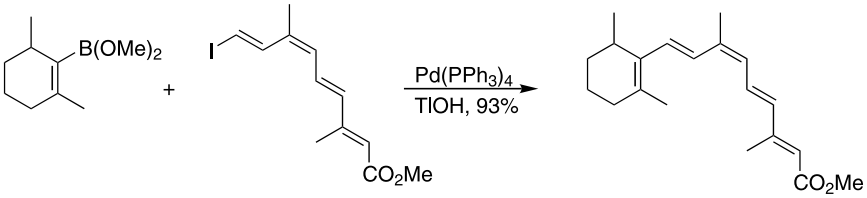


(18)

Tetraphenylborate was used in microwave assisted reactions with heteroaromatic compounds [235]. Coupling of tetraalkylammoniumtrifluoroborates was reported [236]. Palladium catalyzed the coupling of indolylborates with prop-2-ynyl carbonates [237,238]. Ethoxy dialkylvinylborates were used in Suzuki couplings [239]. Palladium catalyzed the coupling of aryl- and alkenyltriflates with potassium alkyltrifluoroborates [240] and with lithium alkylborates [241]. Also, diethylpyridylborane was used

in coupling reactions with calixarenes in the presence of cesium fluoride [242].

A plethora of total syntheses wherein boronic acids or esters were coupled with an aryl-, heteroaryl-, vinyl-halides or -triflate [243,244] were reported. Examples include, dendrobine [245], (−)-lepadins [246], tagretin and 7- and 9-*cis*-retinoic acids and derivatives (Eq. (19)) [96,247,248], khafrefungin [249], combrestatins [250], callipeltoside A side chain [251], the 15-membered macrocycle of RP 66453 [252], diazonamide A [253,254], herbertendiol [115], (−)-equisetin [255], mesembranol [256], methyl (14*E*)-dehydrocrepenynates [257], toward nakadomarin [258], toward bufadienolides [123], macrocyclic core of TMC-95A [259], apoptolidin [260], epothilone A [261], and (−)-salicylhalamide [262]. An intramolecular coupling [263] was used in the synthesis of rutamycin B (Eq. (20)) [264].



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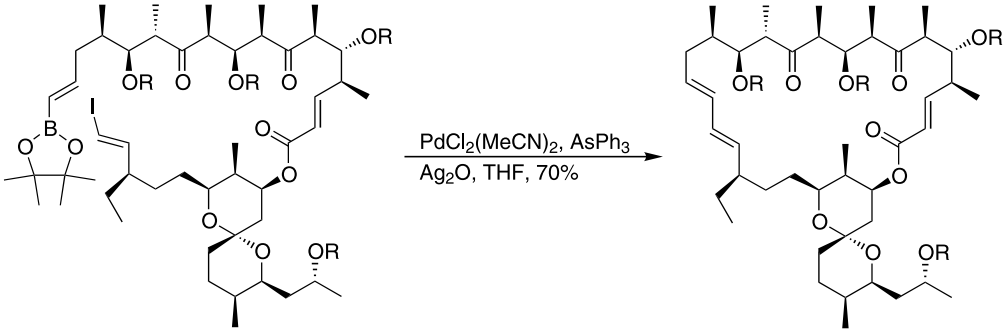

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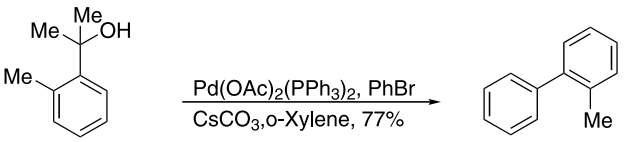
A large variety of functionalized heterocyclic halides and triflates were employed in the Suzuki reaction. For example, six- and seven-membered lactam derived vinyltriflates [265], 5-bromoquinoline [266], halo- and trifloxypyridines [49,168,173,267,268], haloindoles [269,270], 2-iodo-6- and 7-azaindoles [271], 5-iodoisoxazole [39], 7-iodoisatin, in the synthesis of TMC-95, [272], halonucleosides [273], chloropyrimidine and triazine [274], halopyridazinons [275–277], 6-haloimidazol[1,2-*a*]pyridines [278], 6-chloroimidazolo[1,2-*b*]pyridazine [279], 5-bromo-[1,2,4]-triazolo-[1,5-*a*]-pyridine [280], polymer-bound 8-bromopurine [55], 2-, 4-, and 6-halopurines [281,282], 4-chloropyridazines [283], 4-iodoisoxazole [40], 1-chloroisoquinoline [284], 4-chloroquinazoline [285], 2-bromothiophen [286], 2-piperidone derived vinylphosphates [287], 1-trifloxy-β-carboline [78], chlorophthalazines [288], 3,5-dibromopyrazine [289], and halopyrazolo-quinolines and isoquinolines [170] derivatives were all used in Suzuki-type couplings. 3,4-Dibromofuran-2(5*H*)-ones were selectively coupled in the 4-position using boronic acids [11,290]. A palladium-tetraphosphine catalyst efficiently coupled a variety of aromatic and heteroaromatic bromides with arylboronic acids [291,292]. In addition, a variety of heteroaryl boronic acids were used such as, thiophenyl [293–295], 3-furyl, toward eudesmanolides, [296], 4-pyridinyl [67], and 3-indolyl [78] derivatives.

One major advantage of the Suzuki reaction over the Stille reaction is the ability to use alkyl-substituted boronic acids (or esters) without competitive β-hydride elimination. Silver salts were shown to promote this reaction [297]. Hydroboration of alkenes using 9-BBN followed by cross-coupling with vinyl and aryl halides [298–301] was reported. Bromopyridines, iodothiophene, 4-iodopyrazole, 5-bromopyrimidine, 2,5-dibromopyridine, and nucleoside derivatives also participated in this reaction [302,303]. Coupling reactions of 2,5-dibromopyridine [304] and 2,3-dibromothiophene were shown to occur in the 2-position [61,305]. A room temperature alkylbromide–alkylboron Suzuki coupling was described [306].

Cross-coupling of vinyl and heteroaryl iodide or bromides with alkyl boronic acids or esters was used in synthesis of 15-azaepothilones [307], (−)-indolizidine 209D [308],

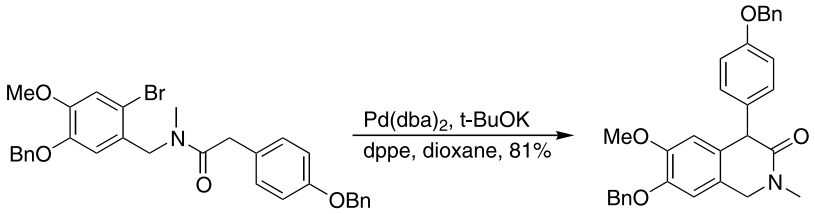
3-hetero-13,14-dihydro prostaglandin F_{1a} analogs [309], 5,6-dihydrocineromycin B [310], sphingofungins [311,312], and desoxyepothilone F [313]. The polyether core of gambierol was prepared using vinyl phosphates [314,315].

Carbon nucleophiles were also employed in alkylation reactions of halides and triflates. α,α-Disubstituted arylmethanols were coupled with aryl bromides (Eq. (21)) [316]. Palladium catalyzed the allylation of zinc enolates [317]. Multiple arylations were observed upon reaction of enolizable aryl alkyl ketones with aryl halides in the presence of a palladium catalyst [318]. Intermolecular arylation of enolizable ketones followed by annulation produced 1-vinyl-1*H*-isochromene derivatives (Eq. (22)) [319]. Both arylation and nucleophilic addition to the carbonyl group was observed in some cases (Eq. (23)) [320]. Intermolecular palladium-catalyzed vinylation of ketone enolates was reported [321].



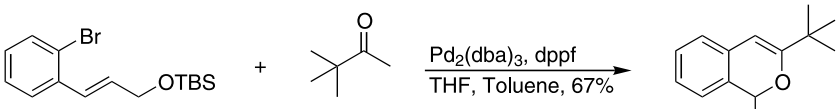
(21)

Intramolecular reaction of an amide-enolate was used in the synthesis of cherylline and latifine (Eq. (24)) [322]. A palladium-catalyzed intramolecular arylation was used to prepare oxindoles. Substituted oxindoles were obtained by a combined intra- and intermolecular arylation of 2-bromoanilides [323]. Decomposition of isolated aryl-palladium enolates derived from ketones, esters, and amides gave the expected coupling products [324]. Palladium catalyzed α-arylation of esters and protected amino acids [325,326] and a triethylborane-mediated α-allylation of 2-hydroxyacetophenone were reported [327]. Arylation of diethylmalonate was followed by decarboxylation [328].



(24)

A number of silicon reagents were developed as alternative transmetallation reagents. Palladium catalyzed the arylation of allylic benzoates using hypervalent siloxane derivatives [329]. Vinyl- and arylpolysiloxanes [330,331], dimethylpyridylsilylalkenes, [332], aryl alkyl halosilanes [333], trialkylsilylalkynes [334], triethoxyvinyl- and arylsilanes [335,336], alkynylsilanols [337], and (α-fluorovinyl)diphenylmethylsilane [338] were used in coupling reactions. A fluoride free cross-coupling was reported [339]. Alkylidenesilacyclopentanes (Eq. (25)) [340] and silyloxacyclohexenes [341] were efficiently coupled with aryl and vinyl halides. A one-pot sequential platinum-catalyzed hydrosilylation followed by a palladium-catalyzed coupling was developed (Eq. (26)) [342]. Copper mediated and palladium/silver-catalyzed couplings of vinyl iodides and triflates with TMS-alkynes were reported [343,344].



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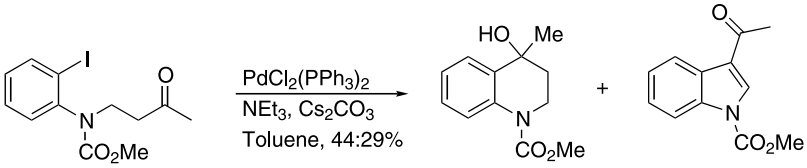

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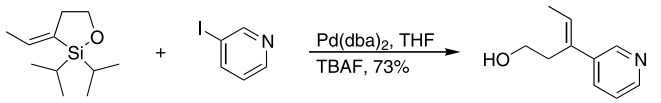

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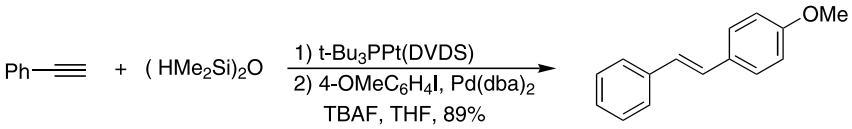
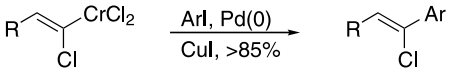


(26)

Some more uncommon cross-coupling reactions employing organometallic reagents were described. Palladium-catalyzed coupling reactions of vinyl tellurides [345,346], organotelluriums with iodonium salts [347], organoindiums [50,348], in aqueous media [349], in situ formed allylindium reagents [350], chromium vinylidene carbenoids (Eq. (27)) [351], organobismuths [352,353], triarylbismuth compounds and iodonium salts [354], triarylbismuth compounds with organostannanes in the presence of carbon monoxide [355], trimethylaluminum [356], a vinylcopper [357], and alkynylsilanes were coupled with triarylantimony compounds [358]. Nickel and palladium catalyzed the coupling of aryl halides with intramolecularly stabilized dialkylaluminum reagents [359]. Palladium catalyzed the cross-coupling of silver acetylides with vinyl triflates [360]. Oxovanadium-induced oxidative coupling of arytrimethylzincates was reported [361]. A lead/nickel/aluminum catalyst system was developed for the arylation and vinylation of 3-trifloxy-, 3-bromo-, and 3-chloro-Δ^3-cephems [362].



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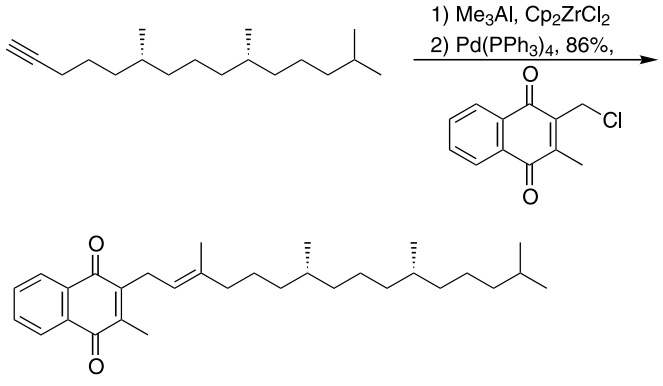
A zirconocene-catalyzed methylalumination of a terminal alkyne followed by palladium catalyzed cross-coupling with a vinyl bromide was used in the synthesis of (−)-fumagillol [363]. A related methylalumination cross-coupling was used in the synthesis of vitamin K (Eq. (28)) [364]. Vinyl zirconocenes prepared in situ by hydrozirconation of alkynes were used in cross-coupling reactions with vinyl halides [146]. Cross-coupling of a vinylzirconocene with a 4-bromoxazole derivative was used in the synthesis of diazonamide A (Eq. (29)) [365].



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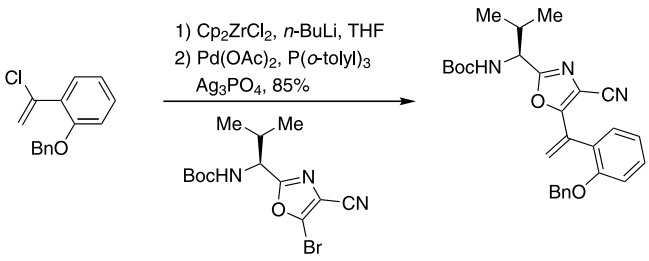


(29)

2.1.2. Alkylations of alkenes and allenes

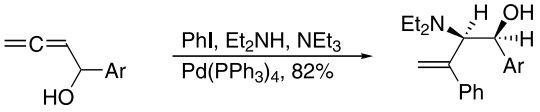
The Heck reaction continued to be one of the most versatile methods for the alkylation of alkenes. A kinetic study of the Heck reaction identified the oxidative addition adduct as the resting state in the catalytic cycle [366]. The mechanism using phosphapalladacycles as the catalyst was investigated [367]. Microwave assisted Heck reactions on potassium fluoride–alumina in the absence of solvent [3], and using palladium nanoparticles in ionic liquids [368] were reported.

A number of papers dealing with different aspects of the catalyst system and the reaction conditions appeared. Several highly efficient catalyst systems were reported [196,369,370] for example, an air-stable palladium tetraphosphine complex [371], palladium modified zeolites [372], a polymer supported ligand [373], catalysts for room temperature reactions were identified [374], catalysts for aryl chlorides [375,376], palladium–carbene complexes [377–379], and a phosphine free catalyst [380]. The formation of homogenous catalysts in heterogenous catalyst systems were demonstrated [209]. The use of the air-stable ligand [(*t*-Bu)$_3$PH]BF$_4$ was reported [6]. A semi-continuous nanofiltration-coupled Heck reaction was described [381].

The use of non-standard organic solvents for the Heck reaction was described. Aqueous DMF–potassium carbonate was used as a substitute for thallium and silver salt in Heck reactions [382]. A palladium-catalyzed Heck reaction under pressure in hydrothermal, sub-critical water was described [383]. Reactions in water and aqueous biphasic media [384,385]. Double bond isomerization in intramolcular reactions was suppressed using supercritical carbon dioxide as the solvent [386]. Heck reactions in molten tetrabutylammonium bromide were described [387]. Polymer supported acrylate [388] and dendrimer-encapsulated palladium nanoparticles [389] were used in supercritical carbon diox-

ide. Regioselective arylation of electron-rich alkenes in ionic liquids was reported [390]. Palladium carbene complexes [391,392] and palladium on carbon [393] were used as catalysts in ionic liquids.

Heck reaction of aryl halides with 2,3-allenols in the presence of an amine produced aminoalkenols (Eq. (30)) [394]. Coupling of α-trimethylsilylstyrenes with aryldiazonium salts gave 1,2-diaryl alkenes (Eq. (31)) [395]. Aryldiazonium salts [396,397] and nonaflates [398,399] were used in place of aryl halides. A highly stereoselective Heck reaction of vinyl sulfoxides was reported [400]. Regioselective arylation of the internal position of *N*,*N*-disubstituted allylamines was observed [401]. Sequential triple coupling of (2-pyridyl)dimethylsilylethene gave trisubstituted ethenes (Eq. (32)) [402]. A polymer supported alkene was used in Heck reactions [403]. Rhodium catalyzed a Heck-type coupling of arylboronic acids with alkenes in aqueous media [404]. Palladium catalyzed a related reaction of organoboron reagents using copper diacetate as the oxidant (Eq. (33)) [405].



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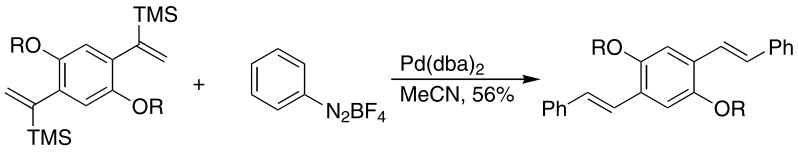

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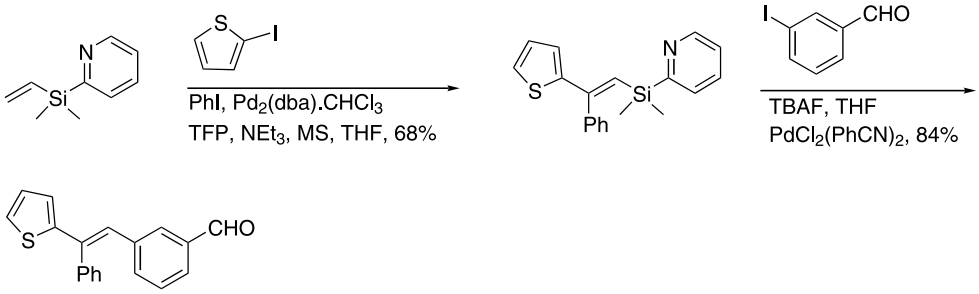

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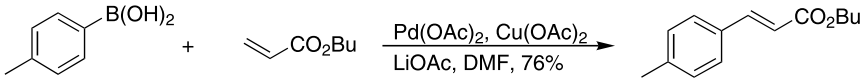
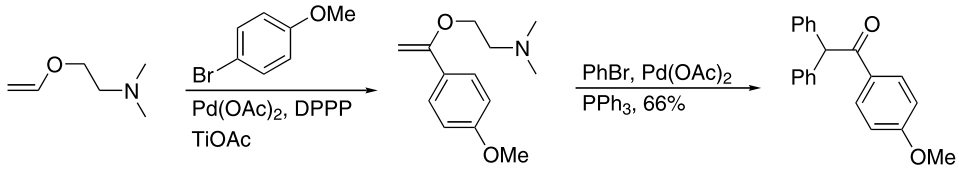


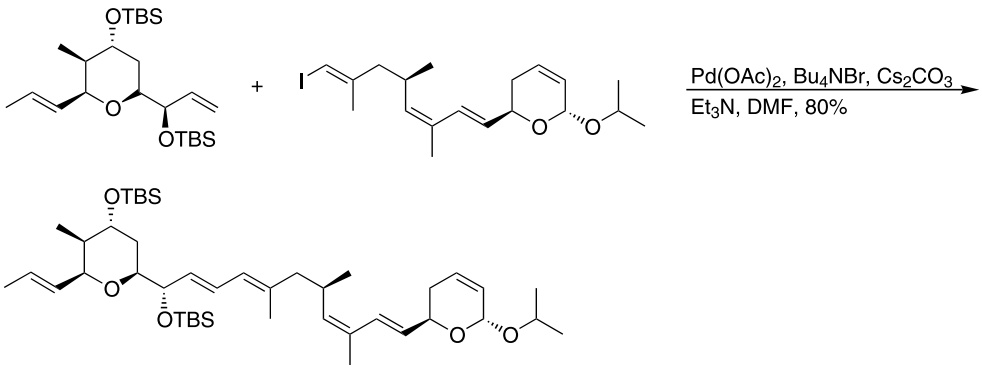
(33)

Heck reactions of α-acetamidoacrylate [406], 3,3-dimethoxy-1-propene [407], butyl vinyl ether [408], and 2-vinylpyrrole [409], were described. Phthalimide or *N*-BOC protected allylic amines were selectively arylated at the internal position [410]. Heck-type reaction of propargylic alcohols produced β-substituted vinylketones [411]. An interesting multiple arylation of vinyl ethers having tethered coordinating functionalities was described (Eq. (34)) [412]. Heck reaction followed by nucleophilic substitution using bicyclopropylidene, phenyl iodide, and various nucleophiles was described [413]. Halides and triflates of a variety of heterocyclic ring systems were used, such as haloindoles [51,414], 2-iodothiophen [415], 4-iodoisoxazole [40], 2- and 3-halopyridines [173,416], bromo-, chloro-, and trifloxyquinolines and isoquinolines [50], and 5-iododeoxyuridine [417]. Pyrrolidine C-nucleosides were prepared using 5-iodouracil [418].



(34)

The intermolecular Heck reaction was used extensively in organic synthesis [47,136,419–421]. Examples include, ratjadone (Eq. (35)) [90], (+)-ambruticin [133], cryptophycin-24 [422], subunit of TMC-95A [423], callystatin A [424], ergoline ring system [425], toward rubromycins (Eq. (36)) [426], 9-methoxystrobilurin K [427], (+)-5-epiindolizidine 167B and indolizine 223AB [428], and pinnaic acid [429]. Multiple alkene insertions to form polycyclic products were reported [430]. The competing β-hydride elimination was suppressed using DABCO. A tandem Heck reaction was used to prepare polycyclic heterocycles (Eq. (37)) [431].



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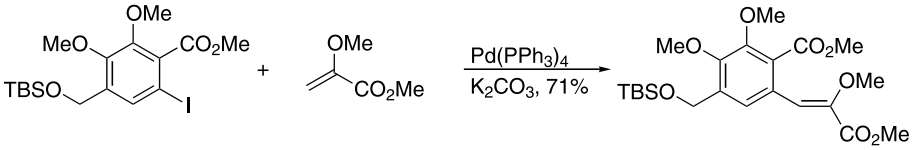

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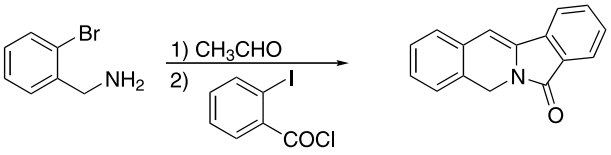
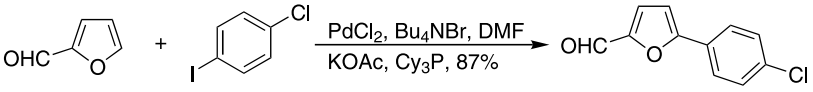


(37)

Aromatic systems were shown to undergo, at least formally, Heck-type arylations [432]. 2-Furaldehyde was arylated exclusively in the 5-position (Eq. (38)) [433]. This type of reaction was used in the synthesis of chiral biscarbazole alkaloids (Eq. (39)) [434], and trisphaeridine and norchelerythrine [435].



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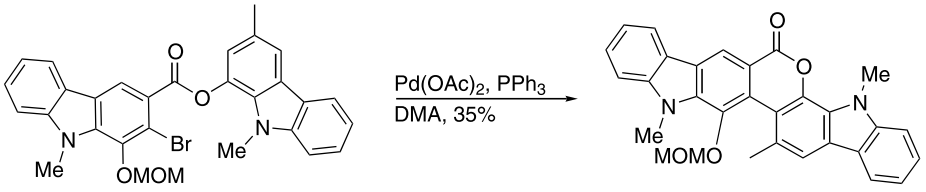
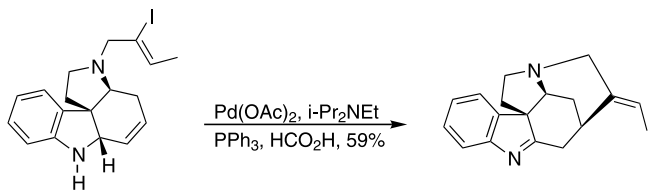


(39)

The intramolecular variation of the Heck reaction [436,437] was used to prepare fused indoles [414,438], and as the key step toward an array of natural products, for example γ-lycorane [439], an A-ring synthon for 1α-hydroxyvitamin D_3 [440], (*S*)-camptothecin [441], mastigophorenes [442], (−)-dehydrotubifoline and (−)-tubifoline (Eq. (40)) [443], herbertenes [444], asperazine (Eq. (41)) [109], helioxanthin (Eq. (42)) [445], the 2-carboxybenzo[*b*]azepine ring system [446], toward zoanthenol [447], macrocyclic core of TMC-95A [259], galanthamine [448], diazonamide A (Eq. (43)) [365], fragments of stemodane and stemarane diterpenes [449], and 19-nor-steroids [450]. Intramolecular macrocyclization of a polymer-bound substrate was described [451]. An interesting silicon-tethered Heck reaction forming dienols was reported (Eq. (44)) [452]. A cascade sequence was used to prepare (−)-nitraraine [453]. A Heck reaction followed by addition of a nucleophile to an intermediately formed η^3-allyl complex was used in the synthesis of mycophenolic acid (Eq. (45)) [454].



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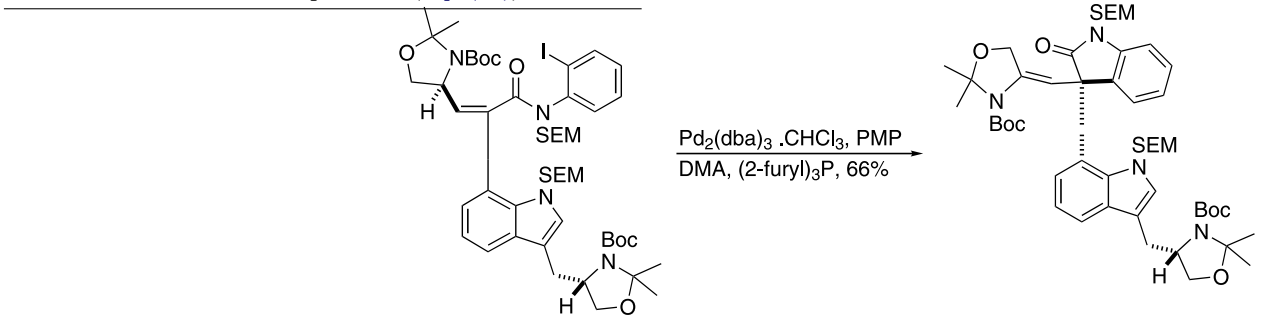

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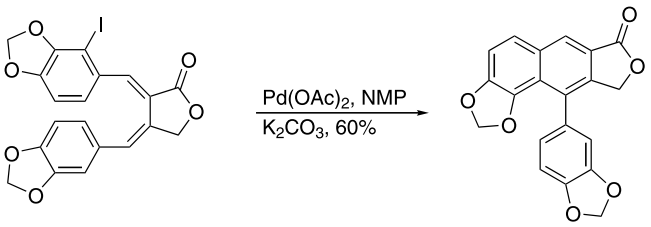
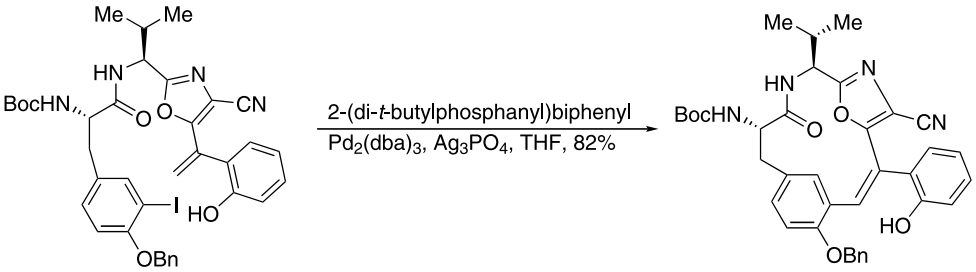


(42)

New chiral ligands for asymmetric Heck reactions were developed [455–460]. An asymmetric intramolecular Heck reaction was used to prepare heterocycles containing a quaternary center [47]. Chiral α,β-unsaturated sulfoxides were used in asymmetric Heck reactions [461].

Reductive Heck reactions were employed in a synthesis of (+)-phorbol (Eq. (46)) [462] and epibatidine analogs [463]. An interesting spirocyclic dihydrofuran was used (Eq. (47)) [81]. A related enantioselective Heck reaction was used in the synthesis of an NK-1 receptor agonist [464]. An intermolecular asymmetric reductive Heck reaction of norbornene derivatives was described [465]. Standard and reductive Heck reactions were used in the synthesis of dihydropyrido(2,1-*a*)isoindolones [466].



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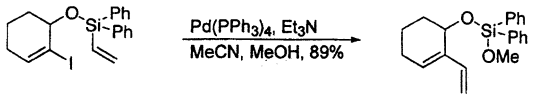
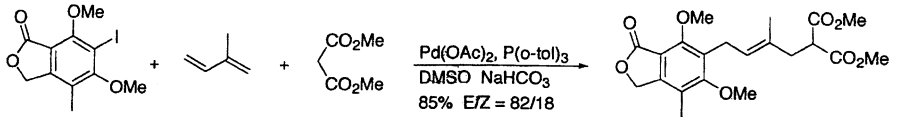


(43y2)



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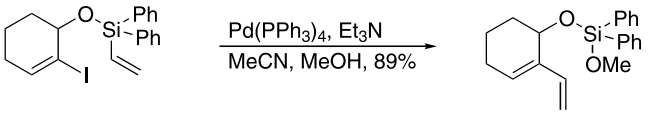

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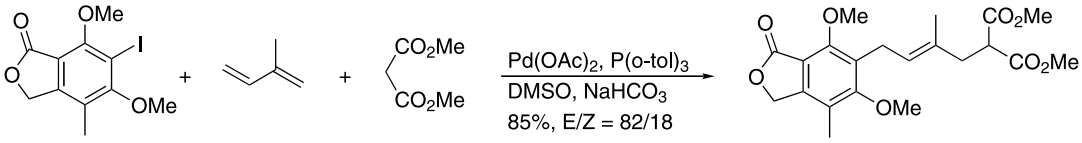

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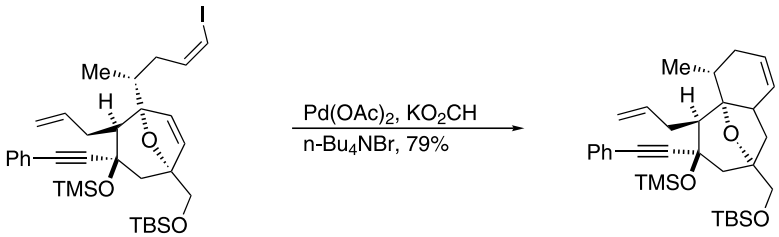

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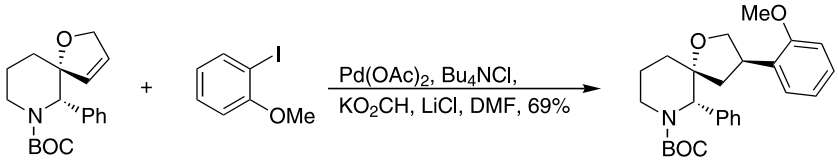
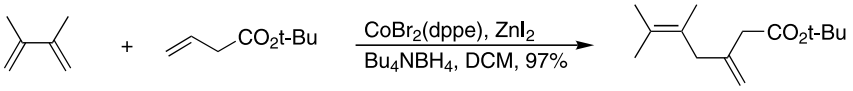


(47)

Cobalt catalyzed a 1,4-hydrovinylation of 1,3-dienes (Eq. (48)) [467]. Ruthenium-catalyzed hydrovinylation of styrenes (Eq. (49)) [468]. Palladium catalyzed the reaction of active methylene pronucleophiles with allenes forming dienes via multiple insertions [469].



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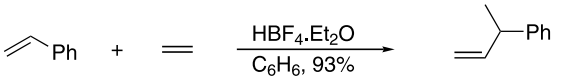


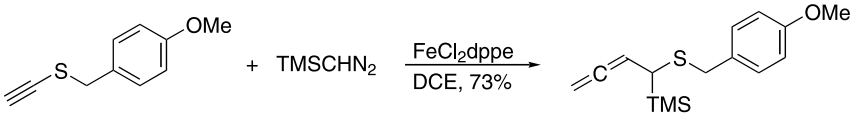
(49)

2.1.3. Metal-catalyzed diazo decomposition (including other cyclopropanations)

Either [2,3]- or [3,3]-rearrangements of α-diazoketone derived propargyloxy enols was observed depending on the catalyst [470]. Electronic effects of the migrating group on the ratio of [2,3]sigmatropic-, [1,2]- and [1,4]-rearrangements of bicyclic oxonium ylides were examined [471]. Enantioselective [2,3]-sigmatropic and [1,2]-Stevens rearrangement of allylic oxonium or sulfonium ylides formed from decomposition of diazo compounds were reported [472–474]. A rhodium carbene complex catalyzed a number of reactions of diazo compounds [475].

A rhodium-catalyzed Wolff rearrangement was used to prepare α-phosphono-γ-lactones [476].

An iron-catalyzed addition-rearrangement was observed upon reaction of propargylic sulfides with trimethylsilyldiazomethane (Eq. (50)) [477]. A rhodium-catalyst initiated [3.3]rearrangement was used in the synthesis of (+)-latifolic acid and (+)-latifoline [478] and of K252a analogs [479]. Both 1,2-aryl- and 1,2-hydride migration was reported upon decomposition of diazo compounds in the presence of rhodium [480]. A stereoselective oxonium ylide formation [2,3]-shift was used as an approach to polycyclic ethers [481]. Related reactions leading to cyclic and bicyclic amines were reported [482, 483].



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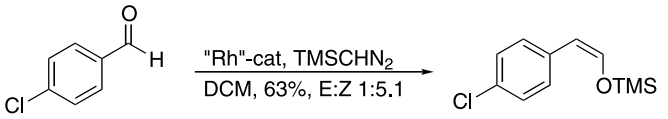
Reaction of trimethylsilyldiazomethane with aromatic aldehydes in the presence of a rhodium catalyst furnished *cis*-vinyl ethers (Eq. (51)) [484]. Copper catalyzed an asymmetric ring-expansion of oxetanes to tetrahydrofurans via carbon–oxygen bond insertion [485]. Rhodium-catalyzed intermolecular diastereoselective insertions of α-diazo carbonyl derived carbenoids into oxygen–hydrogen bonds were reported [486].



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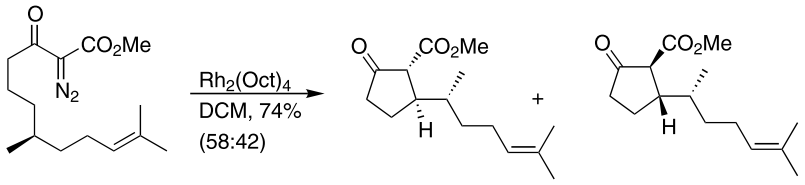
Intramolecular rhodium catalyzed carbon–hydrogen bond insertions via decomposition of diazo compounds were used in a number of synthetic applications forming five- and six-membered rings [487]. Examples include, (−)-astrogoriadiol (Eq. (52)) [488], (−)-thiocyanatoneopupukeanane (Eq. (53)) [489], epimagnolin A [490], asarinin, epimagnolin A, and fargesin [491], (1*R*,7*R*,8*R*)-(−)-turneforcidine [492], (−)-eburnamonine [493], dioxabicyclo[3.2.1] core of zaragozic acids (Eq. (54)) [494], (−)-pupukeanone [495], and grandisol [496].



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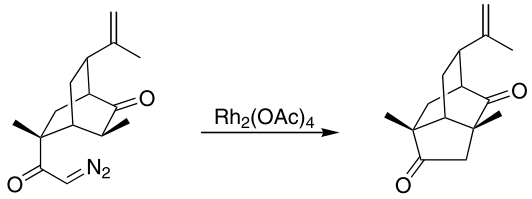

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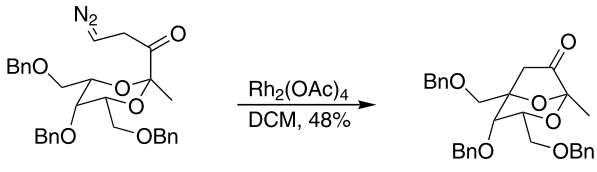

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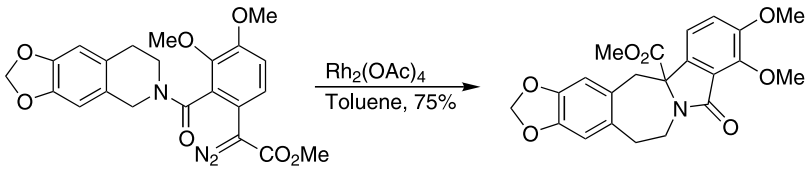
Carbon–hydrogen bond insertion with high regio- and stereoselectivity was reported [497]. Asymmetric rhodium catalyzed carbon–hydrogen bond insertions were described



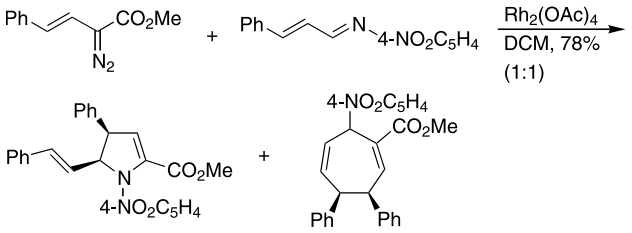
(56)

[498–500]. A double insertion was used to prepare a spirocyclic compound [501]. Monosaccharide derived diazo-esters produced both six- and seven-membered insertion products [502]. Depending on the ligand on rhodium, stereoidal diazoacetates insert into carbon–hydrogen bonds to give four- or five-membered lactones [503,504]. Indole-substituted α-diazo-β-ketoesters were decomposed in the presence of rhodium to give a variety of products depending on the starting indole (Eq. (55)) [505]. Intermolecular carbon–hydrogen bond insertions initiated by diazo decomposition were also reported [506].

(55)

The carbenoid derived from rhodium-catalyzed decomposition of dimethyldiazomalonate inserts into nitrogen–hydrogen bonds of secondary amines to form tertiary amines [507]. Polymer-bound α-diazo-β-ketoesters were used in insertion reactions with primary amides [508]. Asymmetric intramolecular insertions of aryldiazoacetates were reported [509]. Rhodium-catalyzed oxygen–hydrogen bond insertion was used to prepare cyclic ethers [510,511]. A palladium-catalyzed Kirmse reaction of allyl sulfides with trimethylsilyldiazomethane was reported [512].

Rhodium-carbenoids participated in intermolecular [3+2] reactions [513]. An ammonium ylide-Stevens rearrangement sequence was used as an approach toward isoindolobenzazepines (Eq. (56)) [514]. Rhodium catalyzed the formation of vinylcarbonyl ylides followed by ring closure to give oxiranes and dihydrofuranes [515]. Ylide formation followed by cycloaddition was used in a synthetic route toward pseudolaric acid [516], decahydrobenzocarbazoles [517], substituted furans [518], and in reactions with quinones [519] and isatins [520]. Furans participated in cycloadditions to give 8-oxabicyclo[3.2.1]octa-2,6-dienes [521]. Sulfur ylides were generated as intermediates in functionalization of thioether-substituted indoles [522]. Rhodium catalyzed the generation of an ylide from styryldiazoacetates followed by cycloaddition to form five- and seven-membered heterocycles (Eq. (57)) [523]. An asymmetric [3+2]cycloaddition of vinyldiazocarbonyl derivatives with enol ethers to give cyclopentenes was catalyzed by a chiral rhodium complex (Eq. (58)) [524].



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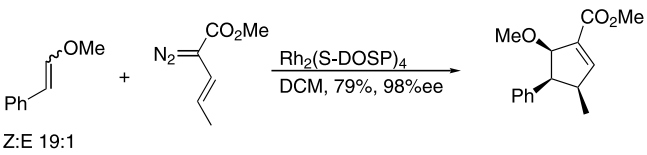

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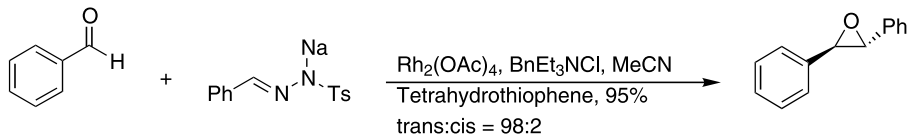
A novel ligand for rhodium-catalyzed enantioselective epoxidation of aldehydes using phenyldiazomethane was described [525]. Rhodium catalyzed an asymmetric epoxidation of aldehydes using sulfur ylides and in situ generation of the diazo-compound (Eq. (59)) [526]. Epoxides and aziridines were prepared from imines and aldehydes, respectively, via ylide intermediates [527,528]. Synthesis of 3-hydroxy-2-sulfonyltetrahydrofurans from β-triethylsilyoxy-substituted aldehydes and *p*-toluenesulfonyldiazomethane was described (Eq. (60)) [529]. A related synthesis of tetrahydrofurans from protected β-hydroxyketones with benzyl diazoacetate in the presence of zirconium tetrachloride was reported [530]. Copper catalyzed the formation of 1,3-oxazolidines from acetone, ethyl diazoacetate, and an imine [531]. Dipolar cycloadditions of rhodium generated carbonyl ylides with vinylethers to form furane derivatives [532,533], with *para*-quinonimides to give bicyclo-[3.2.1] and -[2.2.1] systems [534], and with *N*-phenylmaleimide [535] were developed. Medium-ring carbocycles were prepared by rearrangement of oxonium ylides derived from diazo decomposition (Eq. (61)) [536]. A related ring expansion of ketals was described [537].



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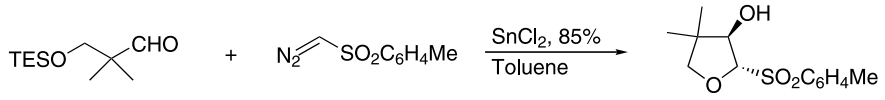

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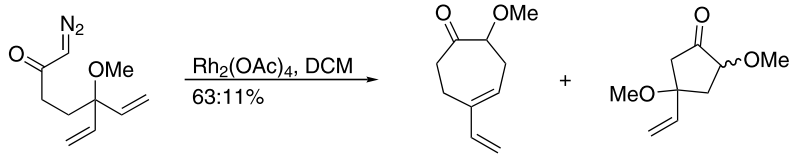

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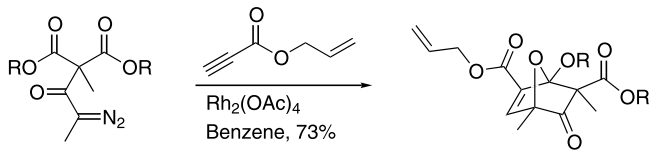
A rhodium-catalyzed diazo decomposition–cycloaddition was used in the preparation of epoxysorbicillinol (Eq. (62)) [538]. Decomposition of 3-diazobenzopyran-2,4(3*H*)-dione in the presence of terminal alkynes furnished furocoumarins [539]. Rhodium catalyzed the decomposition of aryldiazoketones or ethyl diazoacetate adding the resulting carbenoid to 2-alkoxyfurans to give *trans,trans*-2,4-hexadien-1,6-carbonyl compounds [540].



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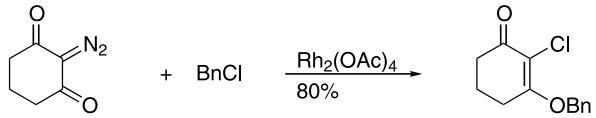
Rhodium catalyzed the synthesis of β-substituted α-haloenones by decomposition of 2-diazo-1,3-dicarbonyls in the presence of benzyl halides (Eq. (63)) [541].



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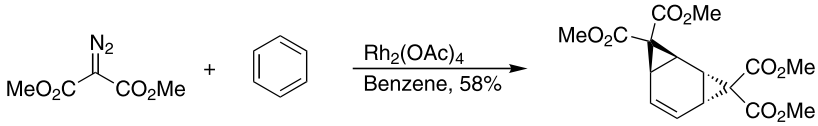


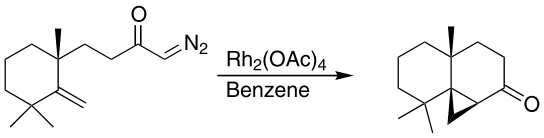
(64)

Cyclopropanation of alkenes via transition-metal catalyzed decomposition of α-diazo-carbonyl or -cyano compounds continued to be developed [542–546]. A copper homoscorpionate catalyst was used in a highly *cis*-diastereoselective cyclopropanation [547]. Rhodium-catalyzed asymmetric cyclopropanation and aziridination using chiral sulfides and in situ generated diazo compounds [548]. An asymmetric solid-phase cyclopropanation was described [549] as well as reactions in ionic liquids [550], and protic media [551]. Enantioselective cyclopropanations using copper salts together with polymer supported chiral ligands [552,553], ruthenium [554–557], rhodium [558–560], and cobalt catalysts [561], and reactions in aqueous or alcoholic solvents [562] were reported. An enantioselective copper-catalyzed cyclopropanation was used in the synthesis of (−)-roccellaric acid [563].

Palladium-catalyzed cyclopropanation of alkenes using diazomethane [564,565] was used in the synthesis of (*S*)-α-cyclopropyl-4-phosphonophenylglycine [566]. A vinylborane was cyclopropanated in a similar fashion [146]. Rhodium-catalyzed double cyclopropanation of benzene (Eq. (64)) [567] and a related cyclopropanation of the central ring of phenantherene were reported [568]. A number of transition-metals catalyzed cyclopropanations of electron-rich alkenes by decomposition of in situ derived diazo compounds prepared from tosylhydrazone salts [569]. Osmium also catalyzed related cyclopropanation reactions [570]. A rhodium–porphyrin complex-catalyzed cyclopropanation of alkenes using glycine methyl ester hydrochloride and sodium nitrite was developed [571]. Copper [572] and rhodium [573] catalyzed cyclopropanation using phenyliodonium ylides.

Rhodium catalyzed the intramolecular cyclopropanation of glycals [574]. The influence of substrate and catalyst on the intramolecular cyclopropanation vs carbon–hydrogen bond insertion was examined [575]. Rhodium catalyzed intramolecular cyclopropanations with high enantiocontrol [576]. Metal-catalyzed intramolecular cyclopropanation of α-diazoketones having a pendant unsaturation gave bicyclo[3.1.0]hexanes [577], tricyclo[4.3.0.0]nonane [578], and a bicyclic lactone [579], and was used in formal syntheses of mayurone and thujopsenes (Eq. (65)) [580], asitane-type diterpenoids [581], and (+)-ambruticin S [582]. Intramolec-

ular, rhodium-catalyzed, cyclopropanation of benzene rings followed by rearrangement produced azulenones and tetralones [583]. Azulenes were prepared using a similar strategy [225]. The influence of a chiral tether on the reaction rate of intramolecular benzene cyclopropanation-expansion to form fused cycloheptatrienes was examined [584].



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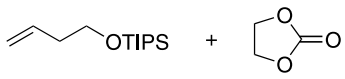
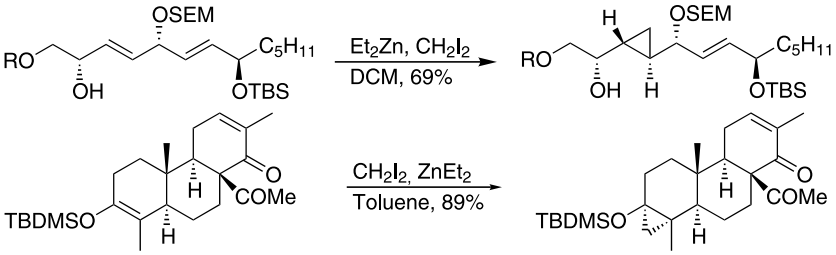


Cyclopropanation of alkenes using Et_2Zn and CH_2I_2 or CH_2ClI was employed in organic synthesis by a number of groups [244]. Examples of synthetic applications include, halicolactone (Eq. (66)) [585], callipeltoside A side chain [251], callipeltoside aglycon [586], (+)-ferruginol [587], *cis*-sabinene hydrate [588], tagretin and 9-*cis*-retinoic acid derivatives [247], herbertendiol [115], asitane-type diterpenoids (Eq. (67)) [582], deoxymannojirimycin analogs [589], and toward kempanes [590]. A hydroxy-group directed diastereoselective cyclopropanation was described [591].



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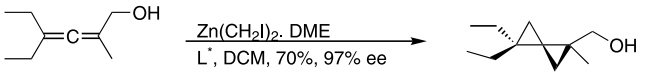
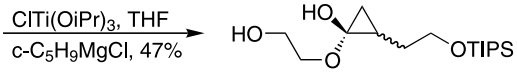
(67)

Titanium mediated the cyclopropanation using $CFBr_3$ or $CFCl_3$ introducing a fluorobromo- or fluorochlorocyclopropane ring [592]. Halomethylzinc alkoxides were used in cyclopropanations of allylic alcohols and ethers [593]. Cyclopropanation of alkenes using ethylzincmethyl perfluoropentanoate was described [594]. Asymmetric cyclopropanations of allylic alcohols using $Zn(CH_2I)_2$ and a chiral dioxaborolane ligand were used in the synthesis of (−)-doliculide [595], (+)-ambruticin [133], epothilone analogs [596], solanoeclepin A subunit [597] and spiropentanes (Eq. (68)) [598]. A related asymmetric cyclopropanation using a chiral titanium catalyst was reported [599].



(68)

A number of titanium alkoxides and aryloxides mediated cyclopropanation of alkenes with esters [600,601]. A titanium-mediated cyclopropanation of ethylenecarbonate with terminal alkenes gave cyclopropanone hemiacetals (Eq. (69)) [602]. Related titanium-mediated cyclopropane formations from esters and terminal alkenes [603], from ethyl 3-chloropropanoate and Grignard reagents [604], and intramolecular reactions [605] were reported. Titanium mediated an aminocyclopropane formation using nitriles and Grignard reagents (Eq. (70)) [606]. Intramolecular cyclopropanation of *gem*-dihalide tethered alkenes using titanocene was reported (Eq. (71)) [607]. A three-component aldimine-addition cyclopropanation was developed (Eq. (72)) [608].



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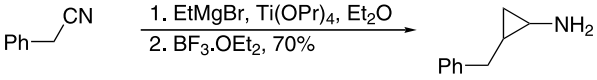

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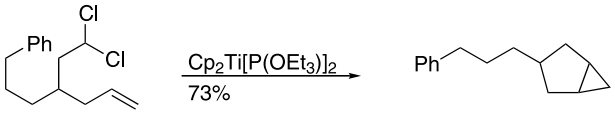

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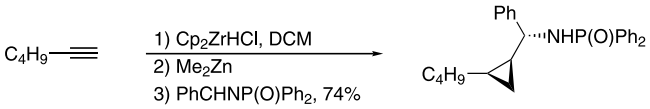
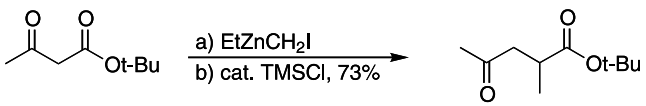


(72)

An interesting reaction of β-ketoesters and β-ketoamides with ethyliodomethylzinc to form α-methylated-γ-ketoesters via cyclopropanation of intermediately formed enolates was reported (Eq. (73)) [609]. A zinc-carbenoid mediated the chain extension of β-keto amides [610]. Platinum catalyzed an intramolecular cyclopropylcarbene formation rearrangement of alkyne tethered furans (Eq. (74)) [611].



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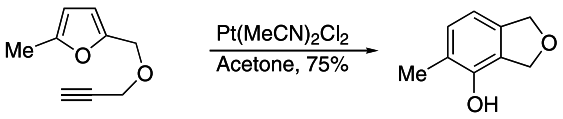


(74)

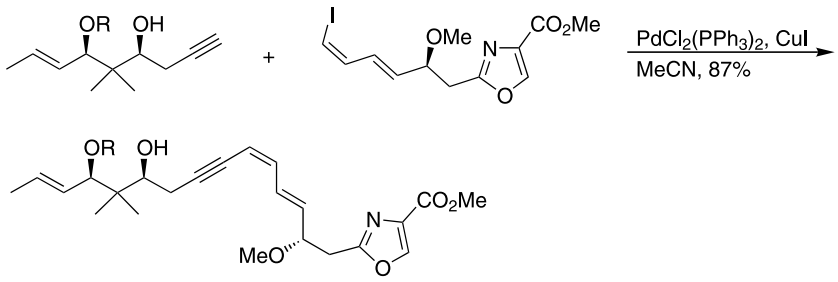
2.1.4. Alkylation of alkynes and arynes

The palladium-catalyzed coupling between terminal alkynes and aryl and vinyl halides or triflates, usually called the Sonogashira reaction, was one of the more frequently utilized carbon–carbon reactions. Coupling reactions in water and aqueous media were reported [612,613]. Solventless microwave assisted Sonogashira couplings on potassium fluoride doped alumina in the presence of palladium powder [614], using palladium–copper doped KF/Al_2O_3 under microwave irradiation [615], using the air-stable ligand $[(t\text{-Bu})_3PH]BF_4$ [6], and on solid support [616–619] were described. Palladium catalyzed reactions promoted by tetrabutylammonium fluoride [620] and microwave irradiation [368,621].

Bromoazulenes [622], diaryl iodonium salts [623], sulfur and selenium-substituted vinylic tosylates [624], and alkenyliodonium salts [625] were used in Sonogashira-type couplings. Diaryl sulfides were obtained from reaction of arylpropargylsulfides with aromatic iodides [626]. Coupling of 4-bromo-1,1-difluoro-3-triisopropylsilyl-1,2-butadiene with terminal alkynes furnished 1,1-difluoro-2-alkynylsubstituted-3-triisopropylsilyl-1,3-butadienes [229]. Sugar derived alkynes [627,628] were used as substrates. Sonogashira coupling of 1,1-dibromoalkenes gave 1,3-diynes or 1,1-disubstituted alkenes [629].

Heterocyclic ring systems were shown to readily participate in Sonogashira-type couplings. Reactions of 4-tosylcoumarins [164], 5-iodo-2-chloropyridine [630], halopyridines [168,597,631–634], 2- and 3-halothiophens [138,635], 5-iodoisoxazole [39], 4-iodoisoxazole [40], 5-iodo-1-ribofuranosidylpyrimidine and 8-bromo-1-ribofuranosidylpurin [34], 5-iodo-2′-deoxyuridine [636,637], halonucleotides [638], 2-iodopurine [639], 2-and 5-halopyrimidines [640], 2-iodothiazoles [641], 2-chloroquinoline [642], trifloxy- and chloro-3(2*H*)-pyridazinones [643], 4-bromo-4,4′-bipyridine [644], 4-iodopyrazole [645], 3-iodoindole [646], iodopyrazoloisoquinoline [170], and 2-haloquinoxalines [647,648] derivatives were described. Regioselective coupling in the 2-position of 2,4-dibromo-5-methylthiophen [295] and 2,3-dibromothiophen [61] were observed. A related coupling of 2,3,5-tribromobenzofuran was used as a key step in the synthesis of eupomatenoid 15 [649].

The Sonogashira reaction continued to be extensively used in organic syntheses [650–654]. Examples of synthetic applications include tonkinecin 1 [650], disorazole C_1 (Eq. (75)) [655], himandravine [656], AB-ring system of macquarimicins [657], (−)-dysidiolide [658], toward spirolucidine [659], imerubrine, isoimerubrine, and grandirubrine [660], frondosin B [75], N1999-A2 [661], callipeltoside aglycon (Eq. (76)) [587], N1999-A2 (Eq. (77)) [662], pyrinodemins [663], toward rubromycins [427], methyl (14*E*)-dehydrocrepenynates [257], C-1027 chromophore [664], 15-*epi*-lipoxin A_4 [665], (+)-20_R-dihydrocleavamine [666], tashiromine [667], himbacine derivatives [33], and cyclamenol A [668].



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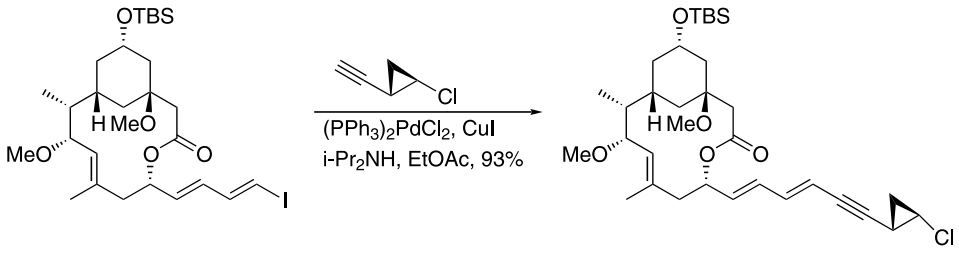

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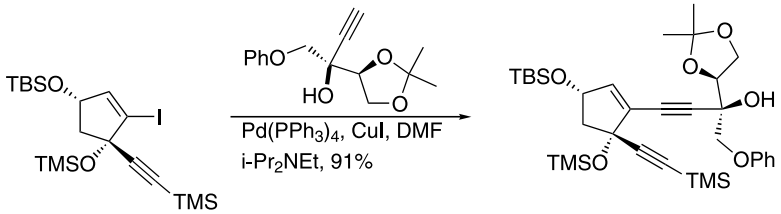
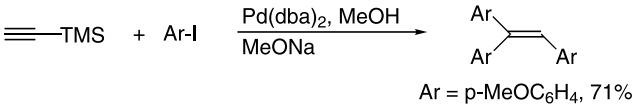


(77)

Sonogashira coupling of trimethylsilylacetylene with an excess if aryl iodide in the presence of sodium methoxide gave diarylethynes or triarylethenes depending on the substrate (Eq. (78)) [669]. Reaction of 3-iodopropenoic acid with terminal alkyne resulted in cross-coupling and intramolecular lactonization [670]. An intramolecular Sonogashira coupling was used to prepare cyclodeca-1,3-diynes [671]. An interesting benzannulation to give aminonaphthalenes was observed upon reaction of alkynes with 2-bromoacetophenone (Eq. (79)) [672]



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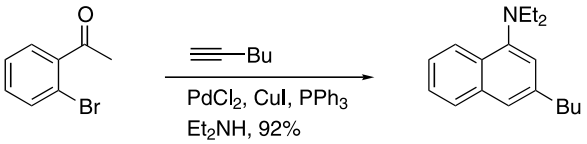


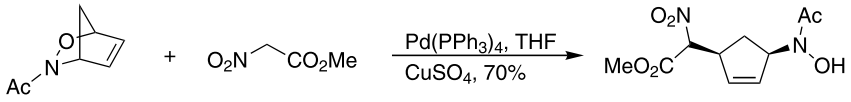
(79)

Copper catalyzed the coupling of terminal alkynes with aryl- and alkenyl-iodonium salts [673] and with aryl iodides [674]. Palladium catalyzed the coupling of a heterocyclic iodinium salt with terminal alkynes in the presence of carbon monoxide to give aryl–alkynyl ketones [675]. Palladium catalyzed the coupling of 2-(butyltelluro)thiophen [676], 2-(butyltelluro)furan [677], β-telluro vinylphosphonates [678], and organolead compounds [679] with terminal alkynes.

2.1.5. Alkylation of allyl, propargyl, and allenyl systems

Palladium continues to dominate as the catalyst of choice for allylic alkylation reactions. Microwave assisted alkylations on potassium fluoride–alumina, in the absence of solvent, were reported [3]. A polymer supported palladium catalyst was used [210]. Cellulose was used as solid support for allylic alkylations in aqueous solvents [680]. A tetraphosphine was used as a ligand for palladium [194].

A nitroalkane [681], tributylstannyl-diethylaluminum, in a synthesis toward laulimalide [682], and methylnitroacetate, in the synthesis of N^{ω}-acetyl-N^{ω}-hydroxyornithine and -lysine (Eq. (80)) [683] were used as nucleophiles in allylic alkylations. Diethylmalonate was used as the nucloephile in the synthesis of nafuredin [684]. Palladium catalyzed the α-allylation of aldehydes with allylic alcohols (Eq. (81)) [685].



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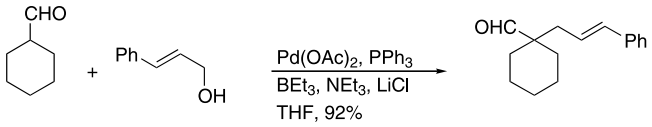
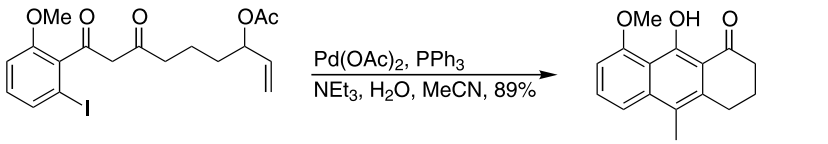


(81)

Regioselective alkylations directed by a removable dimethylpyridylsilyl group were described [12]. An interesting domino allylic alkylation–Heck reaction was reported (Eq. (82)) [686]. A sequential allylic alkylation–Michael addition was used in the synthesis of dihydroerythramine (Eq. (83)) [687].



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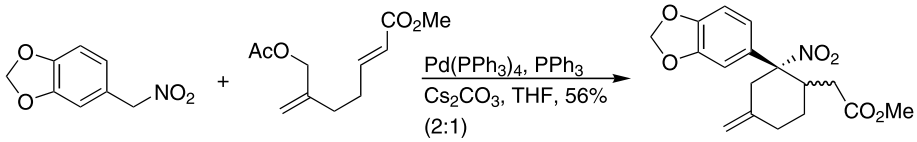
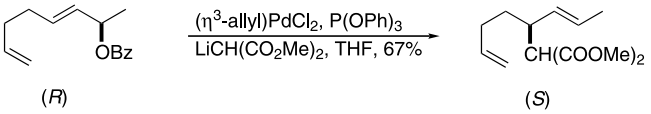


(83)

Palladium-catalyzed asymmetric allylic alkylation continued to receive substantial attention [688]. A chiral phase transfer catalyst was used [689]. Reactions in water were reported [690]. A tethered alkene directed the regioselectivity of alkylation (Eq. (84)) [691]. Regio- and enantioselective reactions of a vinylepoxide with carbon nucleophiles were reported [692]. Palladium catalyzed asymmetric allylic alkylations were used in the synthesis of hygromycin subunits [693,694] and of (+)- and (−)-cyclophellitol [695]. Palladium catalyzed asymmetric alkylations of chiral (*E*)-γ-acetoxy-α,β-unsaturated *p*-tolylsulfoxides [696]. An

iron-mediated allylic asymmetric alkylation was used in the synthesis of (−)-(*S*)-myoporon (Eq. (85)) [697].



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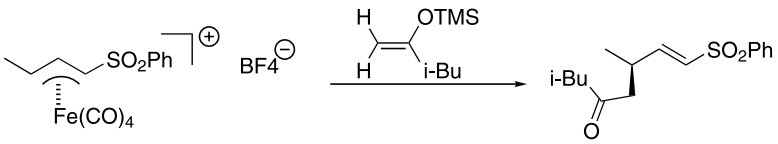

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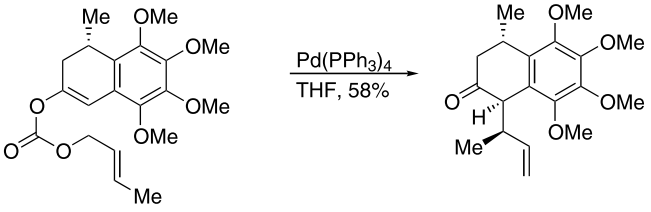
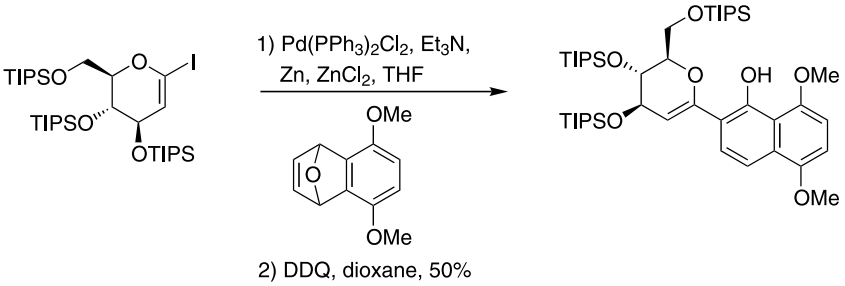
Asymmetric alkylation of geminal allylic dicarboxylates was described. High regioselectivity for substitution at the carboxylate-substituted carbon was achieved [698,699]. Some of the substrates were prepared by a palladium catalyzed hydro-acetoxylation of propargylic acetates. An amide TMS–enolate [700], and ketone enolates [701] were used in palladium catalyzed asymmetric alkylations. An intramolecular allylation of a vinyl allyl carbonate was used in the synthesis of colombiastatin A (Eq. (86)) [702,703].



(86)

The mechanism of the palladium-catalyzed ring-opening of oxabicyclic alkenes using dialkyl zinc reagents was studied. An η^3-allyl mechanism was ruled out and a carbapalladation as a key step was indicated [704]. This type of ring-opening was used in the synthesis of C-aryl glycosides (Eq. (87)) [705]. Rhodium catalyzed the asymmetric ring opening of oxabenzonorbornadiene with carboxylates [706] and alcohols [707].



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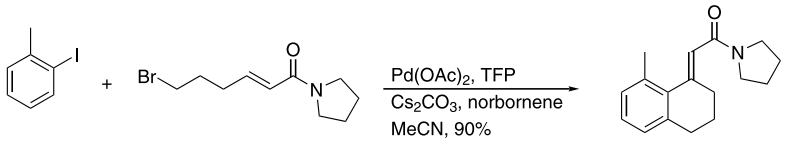
Palladium catalyzed a sequential alkylation–alkenylation of aryl iodides, in the presence of norbornene, forming substituted tetrahydronaphthalenes (Eq. (88)) [708]. Related reactions were reported forming phenanthrenes from 2-substituted aryliodides and diphenyl or alkylphenylalkynes [709], and aceanthrylenes from 5-bromoanthracene and terminal alkynes [710]. Palladium catalyzed an intramolecular allylic alkylation to form 4-vinyl-2-pyrrolidones [711]. A related tandem intramolecular alkylation Heck reaction was also described [712].



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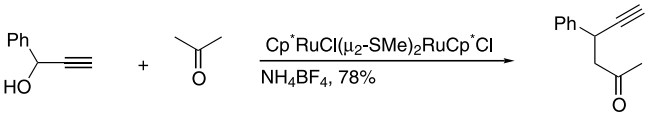
In addition to palladium, a number of other transition-metals catalyzed allylic alkylation reactions. Rhodium-catalyzed allylic alkylation [713] was used to construct *anti*-1,3-carbon stereogenic centers and C_2-symmetric compounds [714]. Highly enantio- and regioselective alkylations using molybdenum complexes were reported [715]. Molybdenum and tungsten catalysts, both showing preferences for alkylation at the more substituted carbon were described [716]. Direct alkylation of propagylic alcohols with ketones to afford γ-ketoalkynes was catalyzed by ruthenium (Eq. (89)) [717].

A three component coupling of benzylidenemalonitrile, allylstannanes, and allylic chlorides was reported [718]. Palladium catalyzed a metallo-ene reaction of allylic phosphates with acrylonitrile [719]. Palladium catalyzed allylation of 2-chloro-1,3-butadiene affording terminal allenes was reported (Eq. (90)) [720]. Palladium catalyzed acylation of allylic trifluoroacetates with acylsilanes (Eq. (91)) [721].



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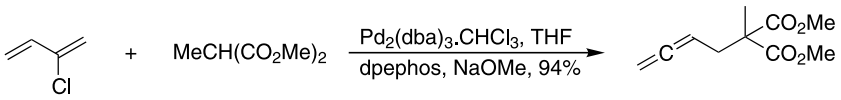

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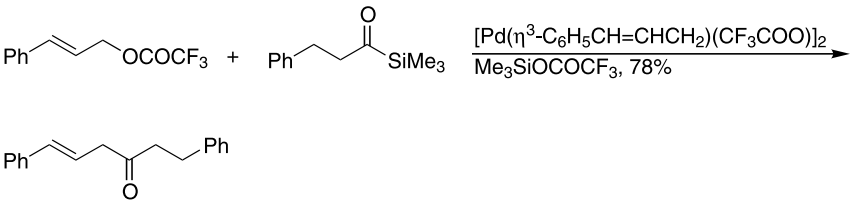

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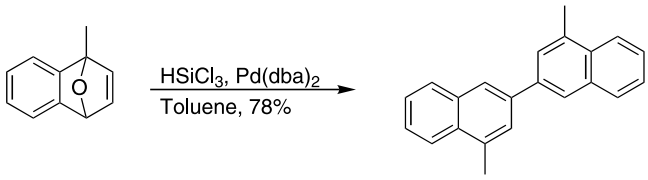
2.1.6. Coupling reactions

Nickel catalyzed the intramolecular coupling of bis-chloroarenes to form macrocycles [722]. A nickel-catalyzed homo-coupling was used in the synthesis of a bisbenzopyran [723]. Nickel-mediated homo-coupling of an allylic bromide was used in the synthesis of testudinariol A [724]. Palladium catalyzed the dimerization or trimerization of 2-halobornene derivatives [725,726]. Palladium catalyzed the formation of unsymmetrical biaryls via coupling of aryl halides [727]. Nickel catalyzed the homo-coupling of a variety of aryl, heteroaryl, and vinyl halides [728,729] for example, of 3-bromo-9,10-phenanthroline [730]. Cobalt catalyzed an electrochemically driven cross-coupling of aryl halides with 4-chloroquinolines [731].

A number of organometallic reagents undergo carbon–carbon coupling reactions. A palladium-catalyzed homo-coupling of a quinone boronic ester was used in the synthesis of a bismurrayaquinone A analog [220] and of an indolestin compound in the synthesis of himastatin [732]. Related palladium-catalyzed homo-couplings of bicyclo[1.1.1]pentyl Grignards [733], aryl boronic acids [734], arylzinc reagents [735], and of aryl- and heteroaryltin reagents [736] were reported.

Copper- and cobalt-catalyzed the homo-coupling of alkynes and silyl-substituted alkynes [737–739]. Copper catalyzed intramolecular couplings of alkynes [740]. Palladium catalyzed the head-to-head coupling of terminal arylalkynes. The regioselectivity was attributed to an agostic interaction [741]. Palladium catalyzed an unusual deoxygenative dimerization of 1,4-epoxy-1,4-dihydroarenes in the presence of zinc and trichlorosilane (Eq. (92)) [742]. Ruthenium catalyzed the dimerization of propargyl alcohols (Eq. (93)) [743]. Palladium catalyzed a reductive dimerization of allenes to give conjugated dienes [744].



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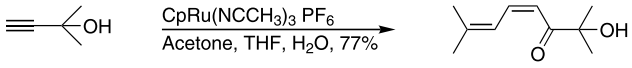

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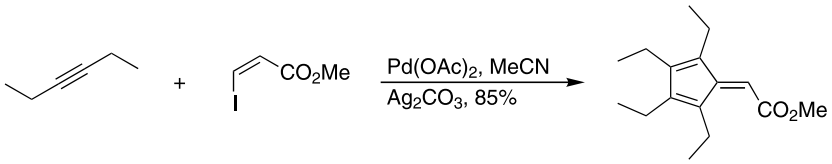
A palladium-catalyzed intramolecular 2,2′-indole oxidative coupling was used in the synthesis of rebeccamycin aglycon [745]. Related 2-indole-2′-benzo-thiophen or -furan couplings were also described [60]. A palladium-catalyzed coupling-cyclization was used in the synthesis of 2α-substituted-1α,25-dihydroxyvitamin D_3 [746]. Palladium catalyzed the reaction of aryl and heteroaryl iodides with diphenylethyne to give 9-alkylidene-9*H*-fluorenes [747]. Palladium catalyzed the coupling of two alkynes and an alkenyl iodide to give penta-substituted fulvenes (Eq. (94)) [748]. Palladium catalyzed the dimerization of benzene to give biphenyl [749]. A palladium catalyzed alkyne–alkyne coupling to give a conjugated enyne was used in a synthesis toward streptogramin (Eq. (95)) [750].



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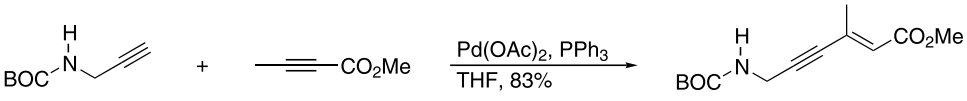

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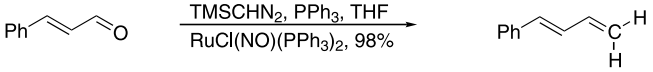
2.1.7. Alkylations of carbonyl compounds

Alkenylation of carbonyl compounds using CH_2Br_2–Zn–$TiCl_4$ [751] was used as a key step in the synthesis of (−)-β-necrodol [752], 8-*O*-methylpopolohuanone E [753], and kelsoene [754]. Methylenation of an ester carbonyl employing $TiCl_4$–CH_2Br_2–Zn–$PbCl_2$ was used in synthetic applications toward the A–D ring system of gambierol [755] and of disaccharides [756]. Reaction of CH_3CHBr_2–Zn–$TiCl_4$–$PbCl_2$ with a lactone carbonyl was used to introduce an ethylidene group in the synthesis of (+)-phorboxazole A [114]. Tebbe's reagent, $Cp_2TiCH_2ClAlMe_3$, was used to methylenate ketones [245], a lactone [757], and was employed in total synthesis of, for example, sclerophytins [758], ratjadone [90], (−)-aphanorphine [759], frondosin B [75], N1999-A2 [659], and epothilone B and D [760]. Tebbe alkenylation of polymer-supported esters was reported [213]. Petasis reagent, Cp_2TiMe_2, was employed in the synthesis toward CP-263,114 [761], (+)-phorboxazole A [114], (+)-zampanolide [762], (+)-altohyrtin A and spongistatin 1 [763], a velbanamine fragment [764], diprionyl acetate [765], and cyclobut A [766].

Selective methyleneation of ketones having an α-oxygen atom, using $CH_2(ZnI)_2$, was reported [767]. Aldehydes were methylenated using TMS-diazomethane and a rhodium catalyst (Eq. (96)) [768]. An intramolecular alkenylation of esters using alkyl bis(phenylthio)alkanoates was mediated by titanocene (Eq. (97)) [769].



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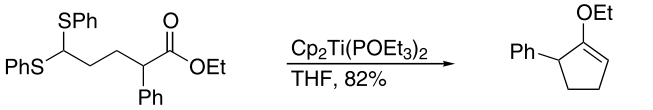

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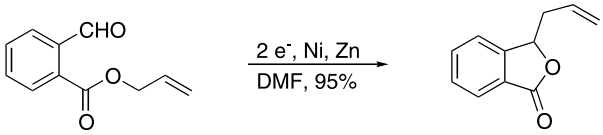
Palladium-catalyzed diasteroselective alkylation of aldehydes by chiral propargylic mesylates in the presence of Et_2Zn was used in the synthesis of the C1–C21 segment of tautomycin [770], sugar derived carbocycles [771], and analogs of the C14–C22 segment of callystatin A [772]. In related reactions, palladium catalyzed allylations of aldehydes with allylic substrates in the presence of indium iodide [343,773]. The η^3-allylic intermediate can be prepared from a vinyl epoxide and reacted with ketones and aldehydes to afford 2-pentene-1,5-diol derivatives [774]. A related reaction of vinyl aziridines with palladium and indium iodide in the presence of an aldehyde gave 1,3-amino alcohols [775]. A cascade reaction of 3-bromo-1-iodopropene leading to homoallylic alcohols was developed [776].

Palladium catalyzed allylation of polymer-supported aldehydes with allylic alcohols in the presence of $SnCl_2$ was reported [777]. Chiral 2-ethynylaziridines were stereoselectively transformed into 1,3-aminoalcohols bearing three chiral centers by a palladium catalyzed, indium-mediated, reaction [778]. A nickel-catalyzed allyl-transfer was also reported (Eq. (98)) [779]. Palladium catalyzed the alkylation of an aldehyde using 1-ethenylcyclopropyl mesylate in the presence of Et_2Zn [780].



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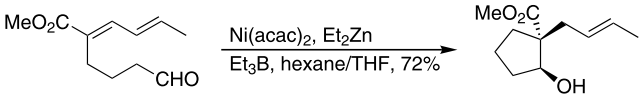
Nickel catalyzed an intramolecular homoallylation of 6,8-dienylaldehydes (Eq. (99)) [781]. Titanium complexes mediated the intramolecular cyclization of 1,3-diene-tethered esters to give *trans*-2-alkenyl cycloalkanols and/or cycloalkanones [782]. Titanium mediated a reductive cyclization of 5-ene-1-ones to give cyclopentanols (Eq. (100)) [783]. Rhodium catalyzed the addition of boronic acids to anhydrides affording unsymmetrically substituted ketones [784]. A platinum catalyzed asymmetric ene reaction was described [785].



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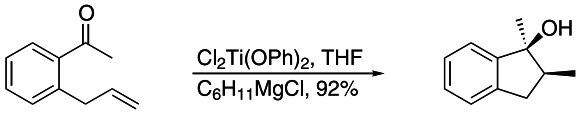

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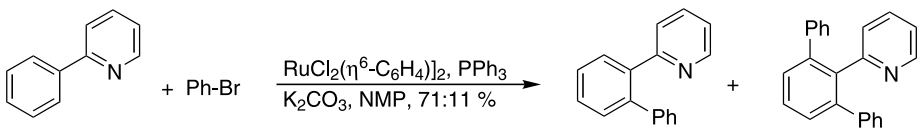
2.1.8. Carbon–hydrogen bond insertions

Ruthenium catalyzed *ortho*-arylation and -alkenylation of arylpyridines (Eq. (101)) [786]. Ruthenium catalyzed the carbon–hydrogen bond activation of *N*-(2-pyridyl)amines (Eq. (102)) [787], and aromatic hydrazones [788]. An interesting imine-directed, rhodium-catalyzed activation was described (Eq. (103)) [789,790]. Rhodium catalyzed a related *ortho*-alkenylation of 2-phenylpyridines with alkynes [791]. An iridium catalyzed carbon–hydrogen bond insertion of alkynes into the *peri*-position of naphthalene derivatives was developed (Eq. (104)) [792]. Carbon–hydrogen bond insertion using vinylsilanes and a ruthenium catalyst was reported (Eq. (105)) [793].



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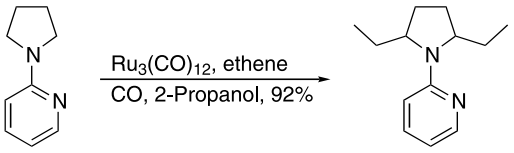

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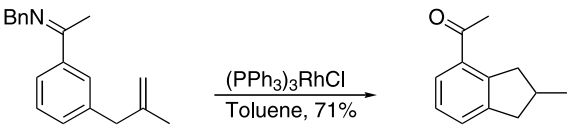

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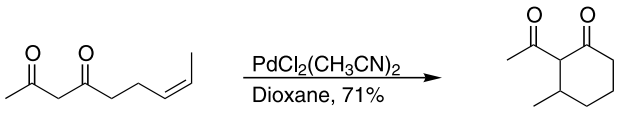

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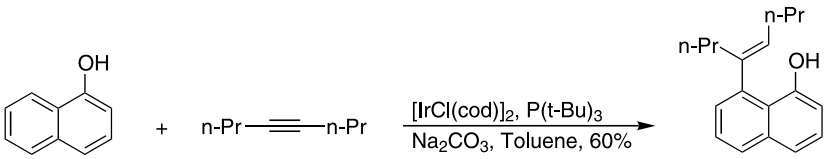
A Wilkinson's catalyst-2-amino-3-picoline system was used in a dehydrogenation–transimination sequence leading to unsymmetrical ketones (Eq. (109)) [799]. Carbon–carbon bond activation and skeletal rearrangement of cycloalkanone imines using a rhodium catalyst was described (Eq. (110))



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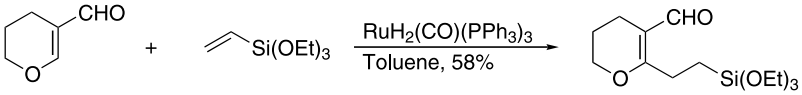

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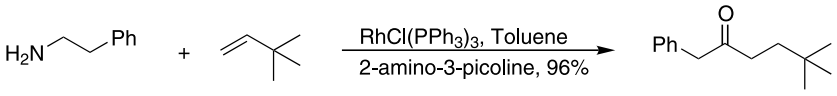
Ruthenium catalyzed an oxidative coupling of arenes with alkenes, in the presence of oxygen, affording styrenes [794]. A carbonylative carbon–hydrogen bond activation was developed using a mass spectrometric labeling strategy [795]. A rhodium-catalyzed annulation of alkenyl-substituted heterocycles was described (Eq. (106)) [796]. Rhodium catalyzed the allylation of styrenes with allyl tosylate (Eq. (107)) [797]. An interesting intramolecular addition of 1,3-diones to alkenes forming cyclohexanes was described (Eq. (108)) [798].

[800]. A rhodium-catalyzed asymmetric cyclization of 4-pentenals to pentanones was reported [801,802]. Rhodium catalyzed an oxidative carbon–hydrogen bond insertion forming oxazolidinones (Eq. (111)) [803] and the cyclization of 4-pentynals to give cyclopentenones (Eq. (112)) [804]. Silicon-tethered ynals were cyclized using a nickel catalyst [805]. An enantioselective carbon–hydrogen bond amination using a manganese catalyst was developed [806].



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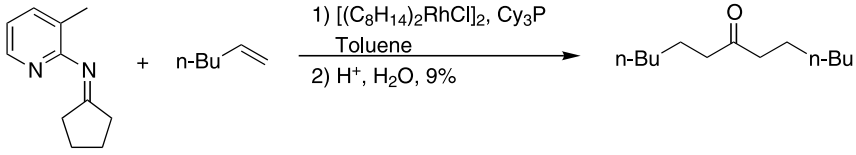

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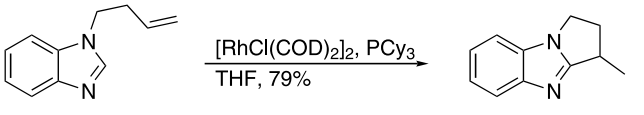

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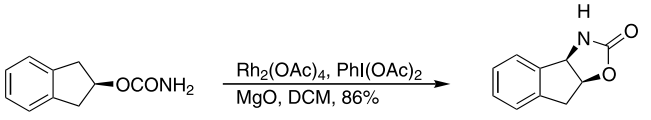

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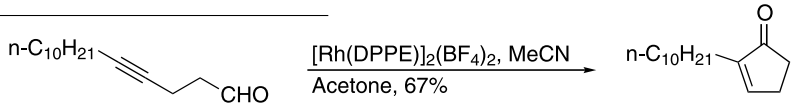

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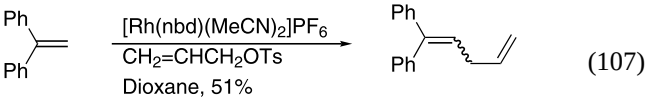
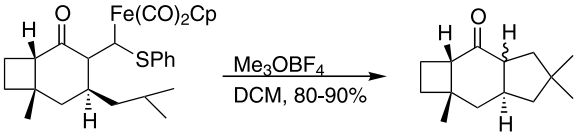


(107)

Intramolecular carbon–hydrogen bond insertion reactions of cyclopentadienyldicarbonyliron carbene complexes were used in the synthesis of sterpurene and pentalene (Eq. (113)) [807]. Novel polycyclic compounds were obtained from a tandem [3+2]cycloaddition carbon–hydrogen

bond insertion (Eq. (114)) [808]. Zirconocene mediated the formation of homoallylic alcohols from acid chlorides via allylic carbon–hydrogen bond activation (Eq. (115)) [809].



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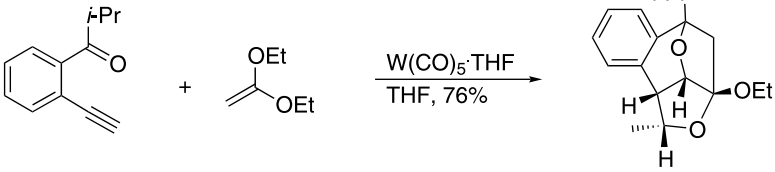

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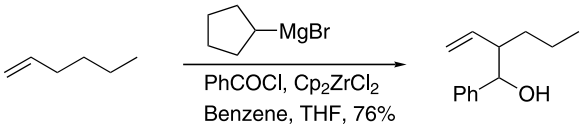

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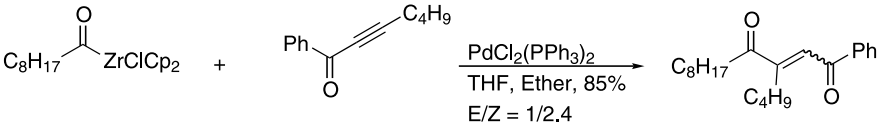
2.2. Conjugate addition

Rhodium complexes catalyzed Michael addition of tin and silane reagents in water under air [810–812] and of arylboronic acids [813]. Rhodium-catalyzed Michael additions of dichloromethylsilane to methyl acrylate esters produced a silylketene acetal [814]. Ruthenium-catalyzed addition of terminal alkynes to enones was reported [815]. Asymmetric conjugate addition of arylboronic esters to α,β-unsaturated carbonyl compounds catalyzed by rhodium(I)-(*S*)-BINOL-based catalysts was described [816]. Palladium catalyzed Michael-type amidation of α,β-unsaturated ketones [817]. Palladium catalyzed the Michael-type addition of acylzirconocenes to conjuated ynones to produce 1,4-diketones (Eq. (116)) [818]. A related reaction of acylstannanes to enones followed by addition of an aldehyde furnished 2-hydroxymethyl-1,4-diketones [819].

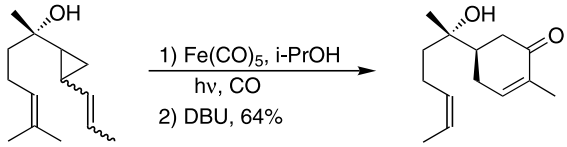


(116)

2.3. Acylation reactions excluding most hydroformylations

2.3.1. Carbonylations of alkenes and arenes

An iron-mediated carbonylation of a vinyl cyclopropane was used in the synthesis of (−)-delobanone (Eq. (117)) [820]. A dicarboxylation and an arylative carboxylation of 1,3-dienes were developed (Eq. (118)) [821]. A [2+2+1]cycloaddition between an alkene, a ketone and carbon monoxide, reminiscent of a Pauson-Khand reaction was reported (Eq. (119)) [822].



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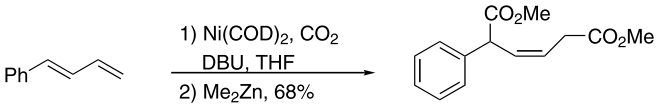

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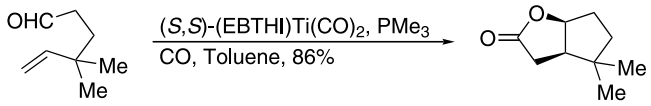

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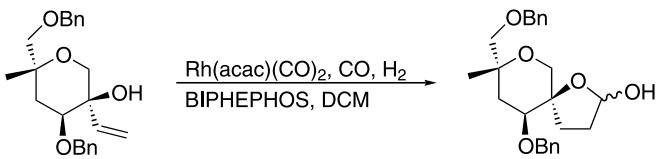
Rhodium catalyzed a stereoselective hydroformylation of alkenes [823]. Hydroformylation was employed as an approach to spirocyclic γ-butyrolactones (Eq. (120)) [824], (+)-ambruticin [133], fluspirilenpenfluridol [825], and lepadiformine [826]. Palladium catalyzed an enantioselective thiocarbonylation of 1,3-dienes (Eq. (121)) [827]. Reaction of vinyl sulfones with carbon monoxide, hydrogen, and an amine in the presence of a rhodium catalyst affords enamines as the major product [828]. Rhodium catalyzed a highly regio- and diastereoselective hydroformylation of allylic ethers [829].



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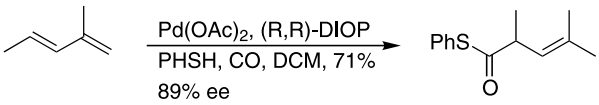


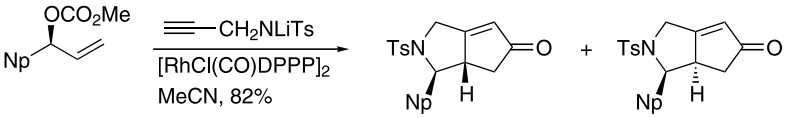
(121)

Palladium catalyzed a regioselective carbonylation-lactonization of dihydromyrcenol [830]. Seven- and eight-memebered lactones were prepared by carbonylation-lactonization of allen-ols [831]. Carbonylation of (*Z*)-3-iodo-3-trifluoromethyl-2-propenols and 3-trifluoromethyl-2-propyn-1-ols produced γ-lactones [832,833].

2.3.2. Carbonylations of alkynes (including the Pauson-Khand reaction)

Metal-mediated [2+2+1]cycloadditions forming cyclopentenones continued to be extensively studied, in particular the cobalt-mediated, Pauson-Khand reaction [751,834–840]. A density functional study of the Pauson-Khand reaction was reported [841]. In some cases, the regiochemistry was found to be dependent on the electronic nature of substituents on the alkyne [842]. Alkene insertion was found to be an irreversible step determining the stereo- and regiochemistry of the reaction. Moderate remote substituent effect on the regioselectivity of Pauson-Khand reactions of 2-substituted norbornenes were observed [843].

Catalyst systems [844,845] employed in the Pauson-Khand reaction, included $Co_2(CO)_8$, $Co_4(CO)_{12}$, and enyne-$Co_2(CO)_6$ complexes together with cyclohexylamine under one atmosphere of CO [846], cobalt on carbon [847], colloidal cobalt nanoparticles [848], and a rhodium complex [849]. A molecular sieve-promoted Pauson-Khand reaction was used to prepare tricyclic aromatic compounds [850]. A tandem rhodium-catalyzed allylic alkylation Pauson-Khand sequence was developed (Eq. (122)) [851]. Asymmetric Pauson-Khand reactions using camphor derived chelating thioalkynes [852] chiral sulfinamides [853], and chiral 1-sulfinylenynes [854], chiral borane-2,10-sultam [855] were reported.



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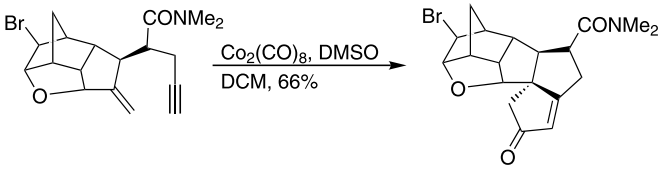
The Pauson-Khand reaction was used in synthesis of asteriscanolide [856], (+)-arnicenone (Eq. (123)) [857], the A-ring of nitiol [858], cedrone [859], tagretin and 9-*cis*-retinoic acid derivatives [247], medium-sized rings fused to a cylopentenone [860], chiral cyclopenta[*c*]pyranes [861], tetracyclic indolidines [862], (−)-dendrobine [863], (+)-taylorine [864], tritium labeled methyl jasmonate [865], and magellanine [866].

Cyclization of triynes to tetracyclic compounds [867], a double Pauson-Khand reactions of acyclic and cyclic diynes [868], an *endo*-selective intramolecular Pauson-Khand reaction of γ-oxygenated α,β-unsaturated phenylsulones [869], and reactions of 7-azanorbornenes [870] were described. An intermolecular Pauson-Khand reaction using cyclopropene was reported [871]. In situ irradiation of the reaction mixture furnished 2-substituted phenols (Eq. (124)) [872]. The regioselectivity and the β-effect of silicon substitutents in the intermolecular allenic Pauson-Khand reactions were examined [873,874].



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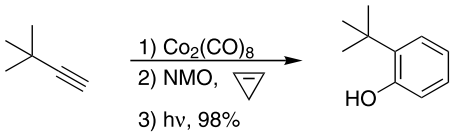

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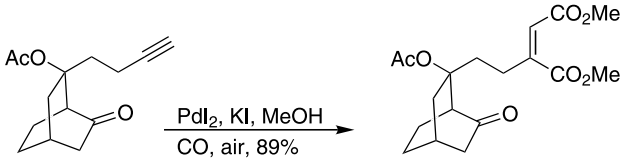
Palladium-catalyzed carbonylation of terminal alkynes to give acetylenecarboxylates was described [875]. Palladium-catalyzed double carbonylation of terminal alkynes were reported [876,877] and used in a synthesis toward CP-263,114 (Eq. (125)) [761]. An asymmetric 1,2-dicarbonylation of terminal alkenes using a palladium–copper catalyst system was reported [878]. Palladium catalyzed the thioesterification of alkynes using *O*-methyl-*S*-phenylthiocarbamate (Eq. (126)) [879]. Carbonylation of TMS-terminated propargylic alcohols using a rhodium catalyst gave 3-silyl-2(5*H*)-furanones (Eq. (127)) [880].



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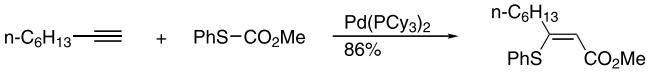

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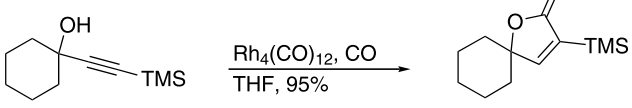

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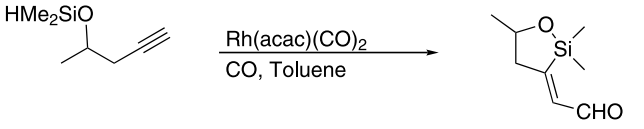
Iridium catalyzed the cyclization of 1,6-diynes in the presence of carbon monoxide to form bicyclic cyclopen-

tadienones [881]. Rhodium-catalyzed intermolecular silylformylations of alkynes were reported [882,883]. Rhodium catalyzed the silylformylation of alkynes in an ionic liquid [884]. Rhodium-catalyzed intramolecular silylhydroformylation (Eq. (128)) [885], and an interesting tandem silylformylation–allylsilylation was described (Eq. (129)) [886] was reported.



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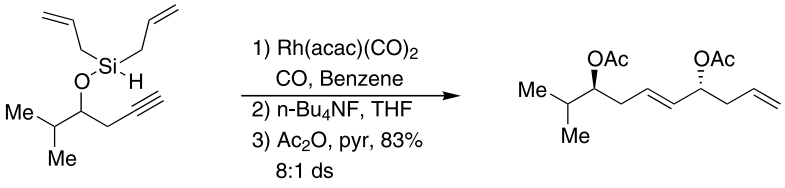

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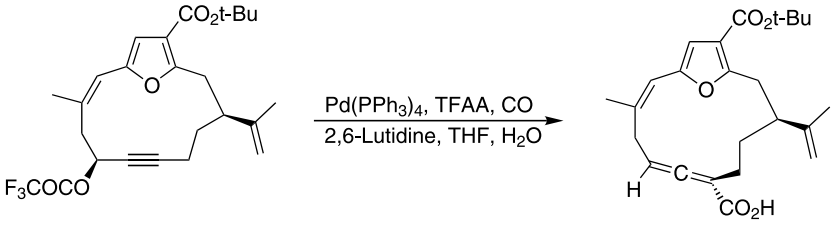
2.3.3. Carbonylation of halides, triflates, epoxides, and phosphonates

Palladium complexes catalyzed the carbonylation of a variety of substrates to form carboxylic acids and derivatives. The formation of homogenous catalysts in heterogenous catalyst systems was demonstrated [209]. Esters were prepared by carbonylation of halides [887–891] and triflates [892–894]. Synthetic applications include, (+)-galiellalactone [895], acromelic acids [896], toward gambierol [183], analogs of martinellic acid [897], martinellic acid [898], and SB-214857-A [899]. Carbonylation of a vinyl epoxide was used in the synthesis of euncenone [186]. Carbonylation of a propargylic trifluoroacetate to give an alleneic acid was used in the synthesis of (−)-deoxypukalide (Eq. (130)) [179].



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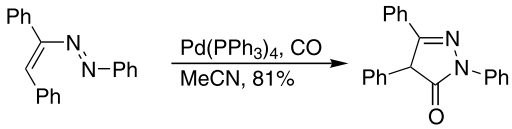
Palladium-catalyzed carbonylation of a vinyltriflate in the presence of tributyltin hydride to give the corresponding α,β-unsaturated aldehyde was used in the synthesis of a nodlisporic acid A subunit [900]. A related synthesis of aryl aldehydes was described [901]. A double carbohydroamination of aryl iodides affording α-aminoamides was developed [902]. Primary aromatic amides were prepared by palladium-catalyzed carbonylation of aryl halides using formamides as an ammonia equivalent [903]. Methoxymethylamine was used to prepare a Weinreb's amide in the synthesis of euplotin A [418]. Aryltin compounds were used in place of arylhalides and triflates [904].

Palladium-catalyzed carbonylation-lactone formation was utilized in organic synthesis of isovelleral [905], (+)- and (−)-wilforonide [906], mycophenolic acid [455]. A related carbonylation using C-11 labeled carbon monoxide and 2-bromobenzamide to give a phthalimide was described [907]. A double carbonylation of aryl iodides in the presence of primary amines to give α-ketoamides was reported [908]. Cobalt catalyzed the formation of β-lactones from epoxides in the presence of a Lewis acid [909], and the carbonylation of aziridines to give β-lactams [910].

2.3.4. Miscellaneous carbonylations

Palladium catalyzed the carbonylation of cyclic ketones to give mainly acyclic diesters [911]. Ruthenium-catalyzed carbonylation of amino-tethered allenes to afford unsaturated lactams [912]. Palladium catalyzed the carbonylation of 1,2-diaza-1,3-butadienes to give 2,3-pyrazole-1(5*H*)-ones (Eq. (131)) [913]. Palladium catalyzed the carbonylation of hypervalent iodonium salts with amidoximes to give oxadiazoles (Eq. (132)) [914] and with organolead compounds to give symmetrical ketones [915].



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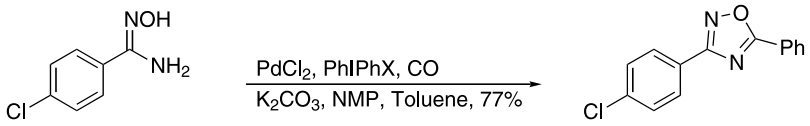

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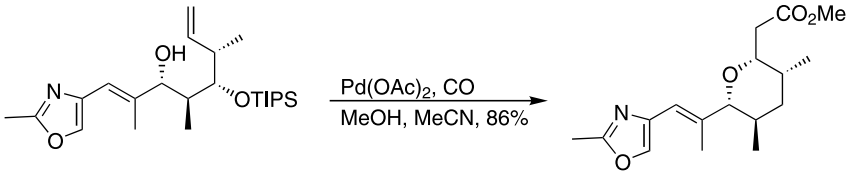
A palladium-mediated hydroxy-palladation–carbonylation sequence was used in the synthesis of a segment of phor-

boxazole A (Eq. (133)) [916]. Palladium catalyzed the carbonylation of an *N*-silylprotected indole to give an indole-3-carboxylic acid [917]. Oxidative carbonylation of 2-ethynylanilines to give (*E*)-(methoxycarbonyl)methylene-1,3-dihydroindol-2-ones was described (Eq. (134)) [918].



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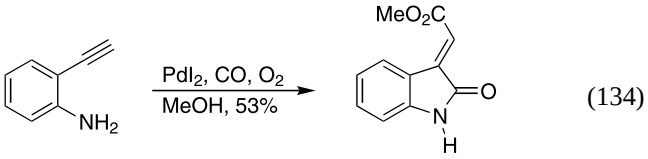

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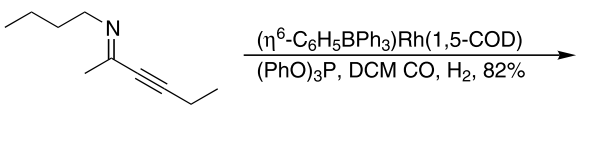
Rhodium catalyzed the preparation of 2,6-(4*H*)-thiazepin-5-ones from alkyne-substituted thiazoles [641], the formation of substituted 4-carbaldehydepyrrolin-2-ones from ynimines (Eq. (135)) [919], and the intramolecular reaction of 5- and 6-alkynoylsilanes to give α-alkylidenecycloalkanones (Eq. (136)) [920]. Oxazolidin-2-ones were prepared by an electrochemical palladium-catalyzed carbonylation of 2-amino-1-alkanols [921].



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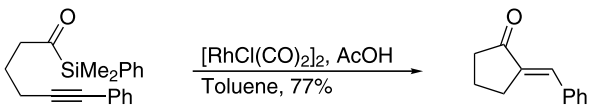

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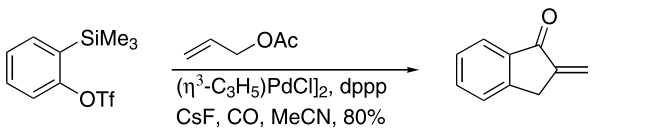
Palladium-catalyzed carbonylations of benzyne intermediates affords anthraquinones in the absence and indanones having an exocyclic double bond in the presence of an allylic acetate (Eq. (137)) [922]. Palladium catalyzed the amidocarbonylation of aldehydes to form *N*-acyl aminoacids (Eq. (138)) [923]. Gold catalyzed the carbonylation of amines to give carbamates [924]. Nickel mediated a cyclization of α,*o*-dienals followed by carbonylation to give bicyclic compounds (Eq. (139)) [925]. Formylation of an alkylmercury compound was used in a synthesis of mycoticin A (Eq. (140)) [926].



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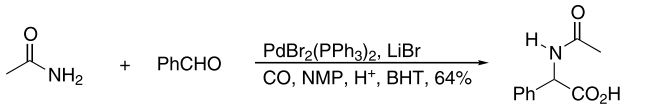

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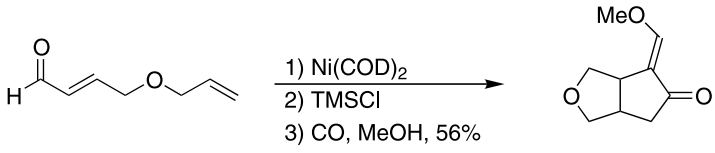

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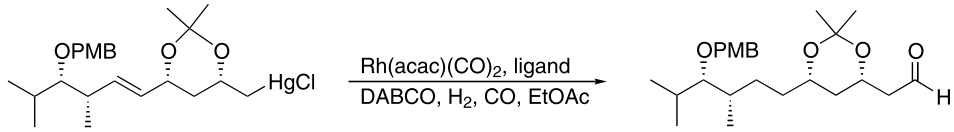

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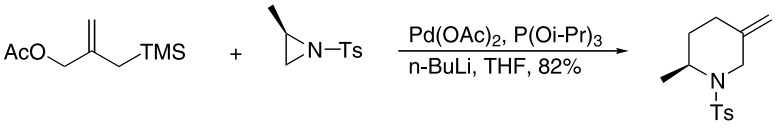
2.4. Oligomerization (including cyclotrimerization/cyclobenzannulation/cycloaddition)

Palladium catalyzed a [3+2]cycloaddition of alkylidenecyclopropanes with imines to give pyrrolidines [927]. A related reaction of alkylidenecyclopropanes and aldehydes produced 3-methylenetetrahydrofurans [928]. Cobalt and ruthenium complexes catalyzed the [2+2]cycloaddition of bicyclic alkenes with alkynes [929,930]. A cobalt-catalyzed diastereoselective intramolecular [2+2]cycloaddition of bis-enones was described [931]. The exo-selectivity and yield of reaction of ketene–alkene [2+2]cycloaddition was

improved in the presence of a palladium catalyst [932]. A [3+3]cycloaddition of aziridines with trimethylenemethane palladium complex furnished functionalized piperidines (Eq. (141)) [933]. Cobalt catalyzed Diels–Alder reactions of 1,3-dienes with internal alkynes [934]. An intramolecular [4+2]cycloaddition of a dicobalt hexacarbonyl complex with a dienolsilyl ether was used in the synthesis of a fredericamycin A analog (Eq. (142)) [935].



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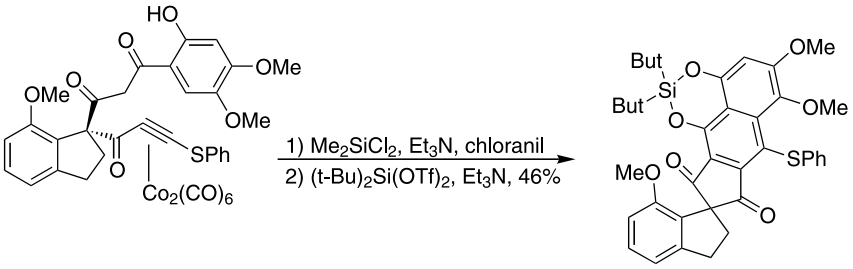

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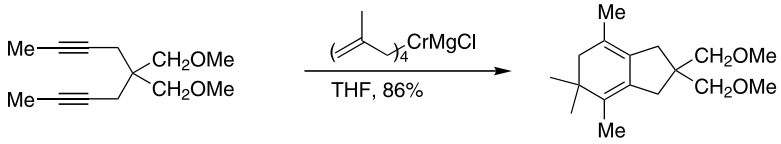
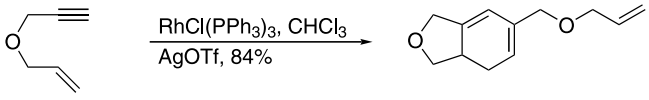
Cobalt mediated the intramolecular [2+2+2]cyclotrimerization [936], and this reaction was used in a synthesis of strychnine [648]. Cyclotrimerization in supercritical carbon monoxide was described [937]. Cyclotrimerization of 6-ethynylpurines was reported [938]. Penta-substituted benzenes were prepared via a chemo- and regioselective, titanium-mediated [2+2+2]cyclobenzannulation of three different alkynes [939]. A palladium–copper catalyst system [940], immobilized palladium complexes [941], iridium [942] nickel and zinc phenoxides [943] were used for cyclotrimerization of alkynes. Wilkinson's catalyst was used for [2+2+2]cyclobenzannulation of a polymer-bound diyne with an alkyne [944]. A titanium-mediated cyclobenzannulation leading to a metallated arene followed by electrophilic quenching was developed [945].

Ruthenium was used for regio- and stereoselective [2+2+2]cyclobenzannulation of 1,6-diynes with dicyanides [946], and of electron deficient nitriles [947]. A novel annulation of 1,6-diynes mediated by methallylchromate or methallylmagnesium chloride using a chromium catalyst was reported (Eq. (143)) [948]. Cobalt-mediated [2+2+2]diyne-nitrile cyclobenzannulation [949] was used to prepare pyridine containing cyclophanes [950]. Cobalt-mediated [2+2+2]cycloadditions of substituted enediynes [951], and of 5-hexynenitrile with 1,3-diynes [952] were reported.



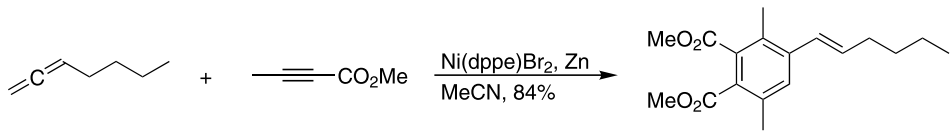
(143)

Bicyclic pyridones were prepared by a ruthenium-catalyzed [2+2+2]cycloaddition of 1,6-diynes with isocyanates [953]. An interesting ruthenium-catalyzed [2+2+2]annulation of 1,6-enynes was described (Eq. (144)) [954].



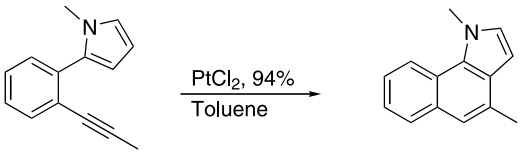
(144)

Cobalt-catalyzed a [4+2+2]cycloaddition of bicyclo[2.2.1]hepta-2,5-dienes and bicyclo[2.2.2]octa-2,5-dienes with 1,3-dienes [955]. A highly regio- and chemoselective nickel-catalyzed cyclotrimerization of propiolates with allenes was described (Eq. (145)) [956].



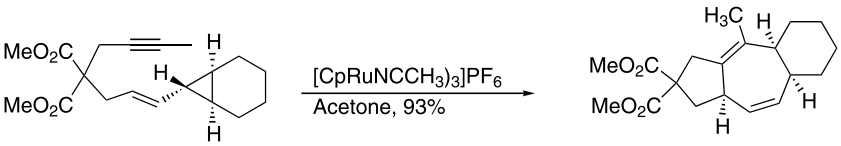
(145)

Nickel-catalyzed homo-benzannulation of perfluoroalkyl enynes was reported [957]. Palladium catalyzed the benzannulation of conjugated enynes under fluorous biphasic conditions [958] and an intermolecular enyne–diyne benzannulation forming cyclophanes [959]. A number of transition metals catalyzed the benzannulation of aromatic enynes to give functionalized naphthalenes (Eq. (146)) [960].



(146)

Rhodium catalyzed an intermolecular [5+2]cycloaddition of alkynes with unactivated vinylcyclopropanes [961]. Ruthenium catalyzed the intramolecular [5+2]cycloaddition of alkyne tethered vinylcyclopropanes to give tricyclic compounds (Eq. (147)) [962], such as the core of cyathanediterpene[963]. Tandem, rhodium-catalyzed, [5+2]- and [4+2]cycloadditions of vinylcyclopropanes with conjugated enynes in the presence of a dieneophile was described (Eq. (148)) [964].



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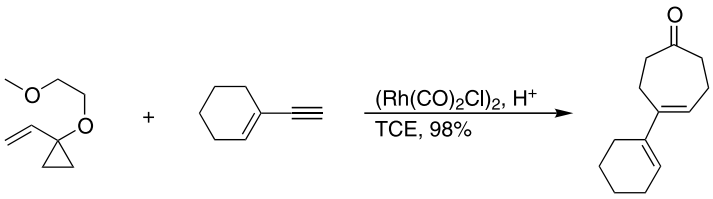
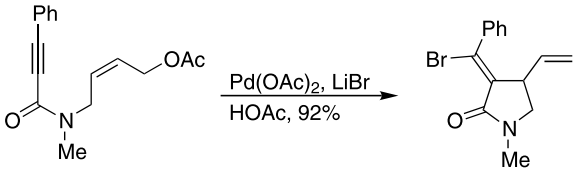


(148)

The mechanism of the cobalt-mediated cycloisomerization of enynes was examined [965]. Cobalt mediated the cycloisomerization of allyl propargyl ethers to form dihydrofurans [966]. Cyclization of 1,6-enynes, in the presence of lithium bromide was used in the synthesis of isocynodine and isocynometrine (Eq. (149)) [967]. Cycloisomerization in organoaqueous medium was used to prepare a podophyllotoxin intermediate [968]. Titanium-mediated 1,6-enyne cyclization was used in the synthesis of (+)-phorbol (Eq. (150)) [462]. A related palladium-catalyzed asymmetric cyclization affording lactones was reported [969]. Palladium catalyzed a reductive cycloisomerization of 1,6-enynes in the presence of formic acid and triethylsilane to afford cyclopentanes having an exocyclic double bond [970]. Palladium-catalyzed 1,6-enyne cycloisomerization using trimethylsilyl tributyltin afforded silicon and tin-substituted products [971]. Cyclization of 1,7-allenynes in the presence of a palladium or rhodium catalyst gave a variety of cyclic products depending on the catalyst and additives (Eq. (151)) [972]. A highly enantioselective palladium-catalyzed 1,6-enyne cycloisomerization was reported [973]. Iridium catalyzed the cycloisomerization of 1,6-enynes, having a terminal alkyne, to give vinylcyclopentenes, and substrates having internal alkynes to give 1,2-alkylidenecyclopentanes [974].



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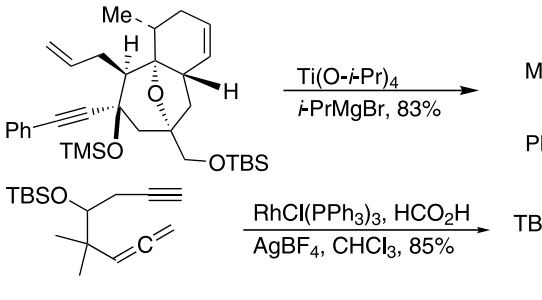
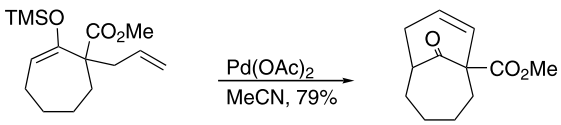


(150)

(151)

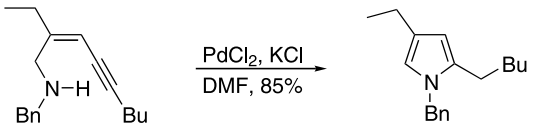
A cationic palladium complex catalyzed the cycloisomerization of 1,6-dienes [975]. *N*-Heterocycles were formed by palladium-catalyzed 1,6-diene cycloisomerization [976]. Depending on the Lewis acid additive, 1,6-dienes were cyclized to give cyclopentenes having either an endo- or exocyclic double bond [977]. Palladium mediated the cyclization of 2-silyloxy-substituted 1,5- and 1,6-dienes to form bicyclic alkenones (Eq. (152)) [978]. Titanium mediated the cyclization of 1,5- and 1,6-bis propagylic alcohol derivatives to give four- and five-membered rings having conjugated exocyclic bisallenes [979].



(152)

A ruthenium-catalyzed three-component reaction between lithium bromide, an alkene, and an alkyne was used to prepare 4-bromo-3-alkenones [10]. Ruthenium catalyzed the

reaction of alkynes with silyl protected allylic alcohol to give *E*-enol ethers [980] and of alkynes with allylic amides to afford enamides (Eq. (153)) [981]. Related reactions of terminal alkynes with alkenes produced 1,1-disubstituted alkenes [982]. The ruthenium-catalyzed Alder-ene reaction was used in the synthesis of deschlorocallipeltoside A (Eq. (154)) [983]. Ruthenium-catalyzed reaction of allenes with α,β-unsaturated carbonyl compounds furnished 1,3-dienes (Eq. (155)) [984].



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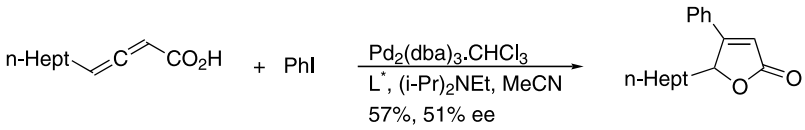

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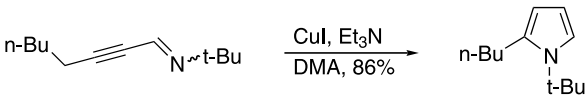

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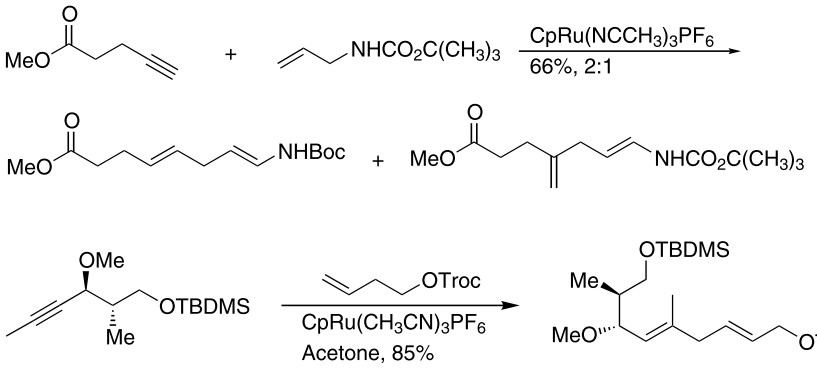

(153)

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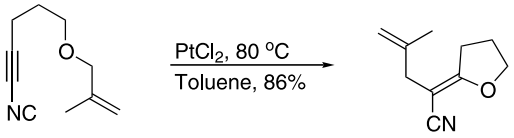
(155)

Platinum catalyzed the alkoxy and hydroxycyclization of 1,6-enynes [985]. Platinum catalyzed three different cycloisomerizations of enynes, a metathesis, a cyclopropanation, and an allylic shift (Eq. (156)) [986,987]. Palladium catalyzed the cycloisomerization of 5-amino-3-alkene-1-ynes to give substituted pyrroles (Eq. (157)) [988]. Palladium-catalyzed tandem dimerization and cycloisomerization of 1-yne-3-ones produced 3,3′-bifurans [989]. Tungsten-catalyzed cycloisomerization of a 5-hydroxy-1-alkyne to give a dihydropyrane, was used in a synthesis of digitoxin [990]. Copper catalyzed the cycloisomerization of 2,3-dienoicacids to 2(5*H*)-furanones [991]. An enantioselective palladium-catalyzed synthesis of 5-aryl-2(5*H*)-furanones from 2,3-dienoic acids and aryl halides was described (Eq. (158)) [992]. Copper mediated the cycloisomerization of alkynyl imines to give pyrroles and pyrrole containing heterocycles (Eq. (159)) [993].

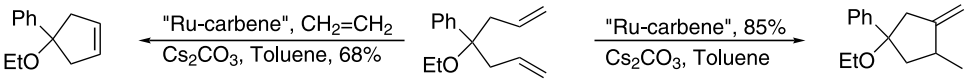


(156)

2.5. Rearrangements

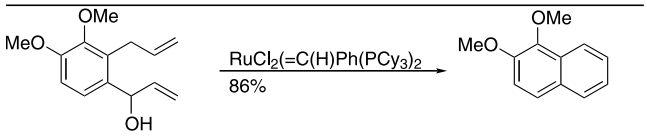
2.5.1. Metathesis

A substantial number of reports on ring-closing metathesis reactions using ruthenium catalysts were published. New ruthenium catalysts were developed [994–998]. The effects of ligand on the mechanism and activity was studied [999]. A mechanistic study indicated the intermediacy of a 14-electron catalyst intermediate [1000]. A ruthenium-catalyst was developed able to catalyze 1,6-diene cycloisomerization or, in the presence of ethane, catalyze ring-closing metathesis (Eq. (160)) [1001]. Ruthenium by-products from metathesis reactions were removed by treatment of the crude products with triphenylphosphine oxide or dimethylsulfoxide followed by silica gel filtration [1002]. Polymer supported, recyclable catalysts were reported [1003–1005]. Ring-closing metathesis was used in solid phase synthesis [1006,1007]. Ruthenium- or molybdenum-catalyzed ring-closing metatheses in ionic liquids [1008], and in supercritical carbon dioxide [1009] were reported.



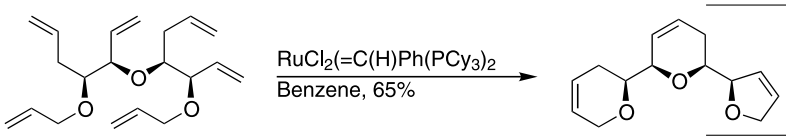
(160)

Enantioselective ruthenium- and molybdenum-catalyzed ring-closing metathesis were reported [1010–1013]. A one-pot tandem palladium-catalyzed allylic acetate isomerization and ruthenium-catalyzed ring-closing metathesis may be possible [1014]. A large variety of ring systems [317,1015,1016] for example, cyclic phosphonates [1017,1018], cyclic enol silyl ethers [1019], five- to eight-membered cyclic amines [1020–1026], fused bicyclic amines [1027], cyclic amino-substituted Fischer carbene complexes [1028], six- to nine-membered lactams [1029–1031], fused bicyclic lactams [1032–1034], macrocyclic lactams [1035], lactones [1036], macrocyclic lactones [1037–1040], fused bicyclic lactones [1041], six-membered cyclic ethers [824,1042–1044], macrocyclic silsesquioxanes [1045], polyoxygenated cyclooctenes [1046], macrocyclic peptides [1047,1048], cyclic pseudo-peptides [1049,1050], five- to seven-membered cycloalkenes [335,691,711,1051–1054], macrocyclic alkenes [1055], spirocycles [1056–1060], a spirocyclic α,β-unsaturated cyclohexenone [1061], fused bicyclic amino acids [1062], cyclic α-aminoacid esters [1063], dihydroquinolines [1064], bicyclic oxazolidines [1065,1066], cyclic α-thiophosphonates [1067], five- to six-membered cyclic enamides [1068], 1,3,2-diazaphosphepine 2-oxides [1069], phosphorous containing mono- and bicyclic rings [1070,1071], silyloxacycloalkenes [341,1072], 1-silyl-2,8-dioxacyclooctenes [1073,1074], silacycloalkenes [1075,1076], bicyclic nucleotides [1077], concave 1,10-phenanthrolines [1078], and optically active bridged oxabicycles [1079] were all prepared using ring-closing metathesis methodology. A triple ring-closing metathesis was reported (Eq. (161)) [1080]. Ruthenium complexes catalyzed sequential metathesis hydrogenation and hydride transfer reactions [1081]. Both ring-closing metathesis and cross-metathesis were observed using polymer supported alkenes [1082]. Ring-closing metathesis of dicobalt hexacarbonyl complexed dienynes was reported [1083]. Ring-closing metathesis of 1,2-diallylaromatic compounds produced substituted naphthalenes (Eq. (162)) [1084]. Schrock-type carbenes were used to prepare a six-membered ether in the synthesis of hemebrevetoxin B [1085] and to prepare a cyclopentene [1086].



(162)

Ring-closing metathesis saw substantial use in organic total synthesis [1087]. Synthetic applications toward halicholactone (Eq. (163)) [1088], tarchonathuslactone [1089], conduritols [1090], indolizidines [1091], zizaene [1092], pyrrolidine and piperidine alkaloids [1093], carbocyclic nucleoside [1094], (+)-calystegine B_2 [1095], disaccharides [756], methyl 4a-carba-D-arabinofuranosides [1096], laulimalide [134,682,1097,1098], umuravumbolide [1099], (+)-3-deoxy-D-glycero-D-galacto-2-nonulosonic acid [1100], the spirocyclic core of pinnaic acid [1101], 1-desoxyhypnophilin [1102], (−)-pyrenophorin [1103], hydroazulene ring system of guanacastepene [136], an inosine derivative [1104], securinine [1105], the carbocyclic skeleton of ingenol [1106], salicylhalamides (Eq. (164)) [101,1107], teubrevin G and H [1108], asteriscanolide (Eq. (165)) [857], C1–C12 fragment of fostriecin [1109], cryptocarya diacetate [1110], toward the A–D ring system of gambierol [755], cylindrophanes [1111], (+)-ambruticin S [580], (−)-isolaurallene [1112], roseophilin [1113], ent-(−)-roseophilin [1114], (−)-fumagillol [391], 4-hydroxypipecolic acids and (−)-SS20846A [1115], halichlorensin [1116,1117], the ergoline ring system (Eq. (166)) [425], salicylhalamides [262,1118,1119], toward FR901483 [1120], toward zoapatanol analogs [1121], toward havellockate [1122], a sarcodictyin analog [1123], a fragment of ciguatoxin CTX3C [1124,1125], the FGHI ring system of azaspiracid [121], laulimalide [1126], apoptolidine [1127], 1,6-germacradiene-5-ol [1128], the HIJKLM rings of ciguatoxi CTX3C [1129], (−)-A26771B [1130], (−)-coiceine [1131], (−)-4a,5-dihydrostreptazolin [1132], (*Z*)- and (*E*)-γ-bisabolene [184], epothilones (Eq. (167)), (+)-acoradiene [1133,1134], and the ADE-ring of nakadomarin A (Eq. (168)) [1135]. A double enantioselective ring-closing metathesis was used in the synthesis of an NK-1 receptor antagonist (Eq. (169)) [464]. An asymmetric molybdenum-catalyzed metathesis was developed and employed in the synthesis of tipranavir (Eq. (170)) [1136].



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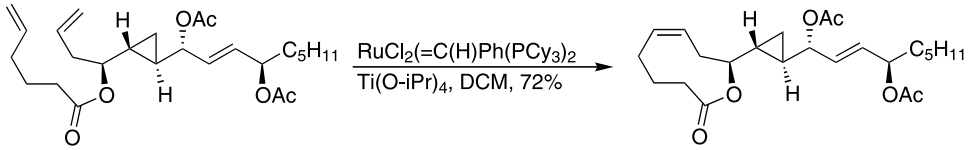

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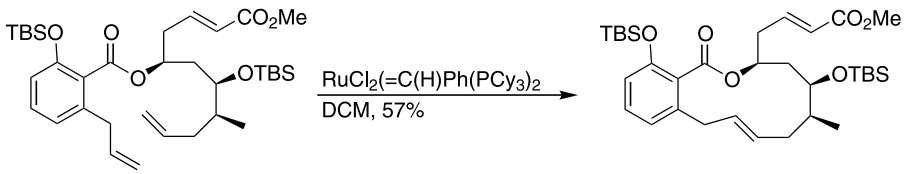

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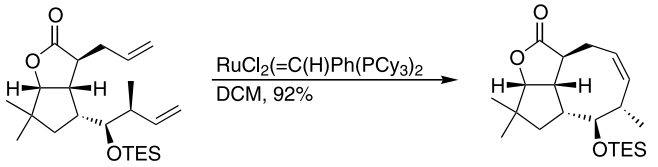

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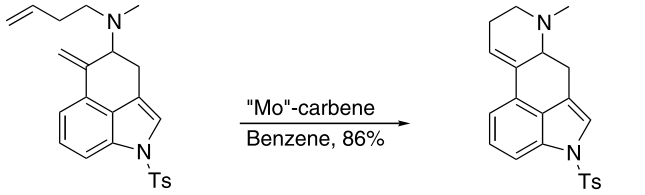

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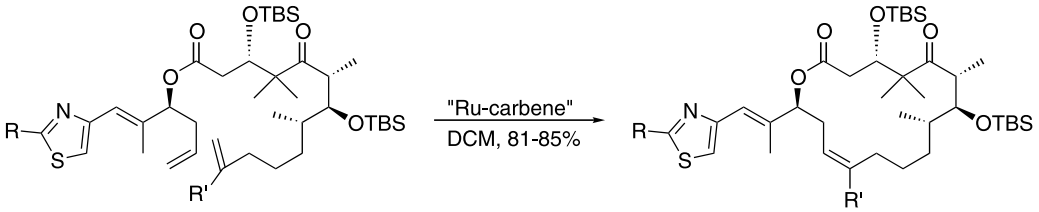

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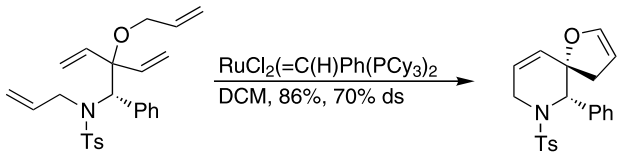

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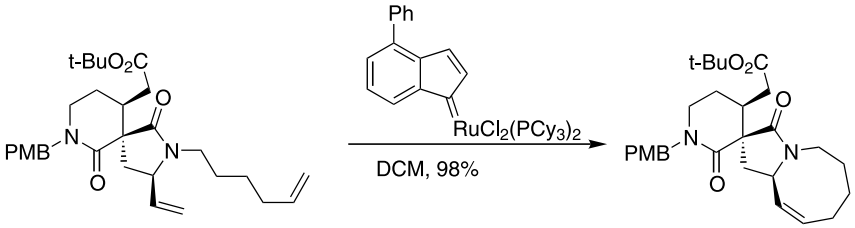

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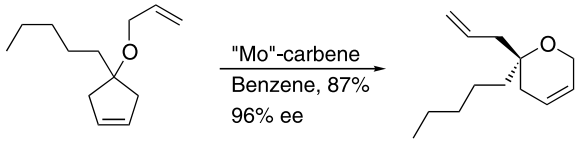
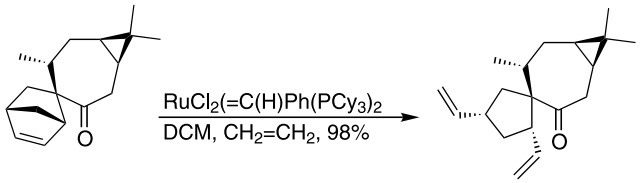


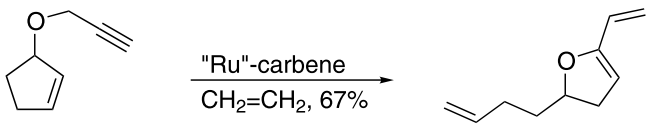
(170)

Ring-opening cross-coupling metathesis [1137] was used to prepare 4-pyrone derivatives [1138], polyethers, and related compounds [1139,1140]. Asymmetric ring-opening cross-metathesis reactions leading to optically active cyclopentanes were described [1141]. A ring-opening metathesis was used in the synthesis of the carbocyclic core of ingenol (Eq. (171)) [1106] and sporochnol A [1142].



(171)

Alkyne–alkyne cross-metathesis, using molybdenum catalysts, was used in the synthesis of prostaglandin E_2 [1143]. An alkyne–alkene ring-closing metathesis was used in a synthesis toward manzamine alkaloids [1144]. Alkyne–alkyne ring-closing metathesis was used to prepare epothilone A and C using $Mo[N(t\text{-}Bu)(Ar)_3]_3$ as the catalyst [1145,1146]. Sequential enyne metathesis, ring-closing metathesis, ring-opening metathesis, and cross-metathesis were reported (Eq. (172)) [1147]. Ruthenium-catalyzed ring-opening ring-closing metathesis of enynes (Eq. (173)) [1148], and a tandem dienyne cross-metathesis ring-closing metathesis [1149,1150] were reported. A tandem dienyne metathesis was used to prepare bicyclic ring systems (Eq. (174)) [1151]. 4-Vinyl-substituted 1-silyl-2-oxa-4-cyclohexenes were prepared by metathesis of silyloxa-tethered enynes [1152].



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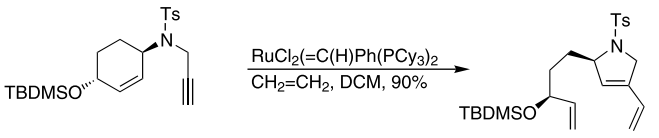

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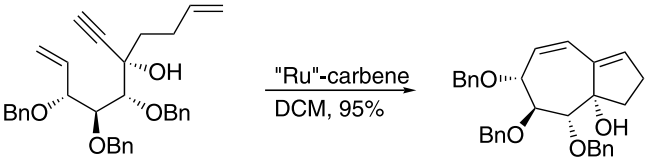
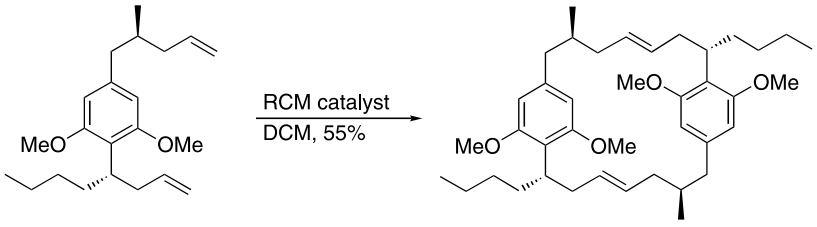


(174)

Intermolecular alkene–alkene cross-coupling metathesis reactions are becoming more frequently used [1072,1153–1157]. Homoallylic alcohols with anti-allylic substituents displayed *E*-alkene selectivity in cross-metathesis reactions with allyltrimethylsilane [1158]. Cross-metathesis reactions of phenyl vinyl sulfone with terminal alkenes [1159], allyl-substituted nucleosides [1160], 2-allylphenols with styrenes [1161], fluorinated alkenes [1162], acrylonitrile with alkenes [1163], diethyl vinylphosphonate with alkenes [1164], vinyl ferrocene [1165], and vinylsilanes with alkenes [1166–1168] were reported. Cross-metathesis was used to prepare vinylphosphonate-linked nucleic acids [1169].

Intermolecular cross-metathesis was used in the synthesis of (−)-roccellaric acid [563], a fragment of amphidinolide [1170], and vancomycins [1171,1172]. A cross-metathesis ring-closing metathesis dimerization was used in the synthesis of (−)-cylidrocyclophanes (Eq. (175)) [901].



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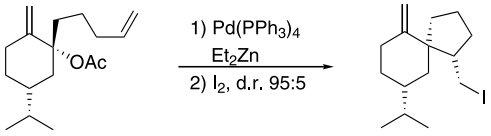
A ruthenium-catalyzed intermolecular enyne cross-coupling metathesis was reported [1173]. A number of intramolecular enyne metathesis reactions were described [1174–1181]. Both five- and six-membered rings were obtained using a ruthenium carbene complex [1182].

2.5.2. Cycloisomerizations

Transition-metal catalyzed carbocyclization of 1,5- and 1,6-dienes to give substituted cyclopentanes or cyclohexanes continued to be extensively examined. Cyclization reactions of 8-acetoxy-1,6-dienes in organoaqueous media to give vinyl-methylene substituted five-membered rings were reported [1183] A ruthenium-catalyzed cycloisomerization of 1,6-dienes to *exo*-methylenecyclopentanes was reported [1184]. Palladium-phenanthroline complexes catalyzed cycloisomerization of 1,5- and 1,6-dienes to give cyclopentenes [1185]. An interesting palladium-catalyzed spirocyclization of a 8-acetoxy-1,6-diene in the presence of diethyl zinc was reported (Eq. (176)) [1186]. Cyclizations in the presence of silanes were reported [1187]. Palladium catalyzed an enantioselective 1,6-diene cyclization–silylation [1188,1189]. Related platinum-catalyzed reactions of 1,6-diynes were also described [1190]. Reaction of 1,2-allene-7-ynes with silicon–tin or tin–tin bonded compounds in the presence of a palladium catalyst produced difunctionalized cyclopentanes (Eq. (177)) [1191].



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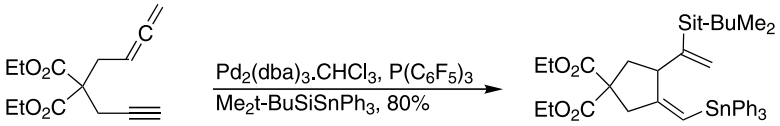

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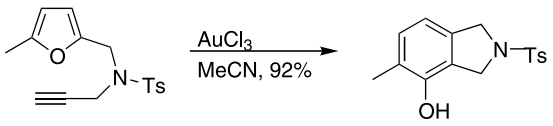
Gold-catalyzed cycloisomerizations of 1,2-diene-3-ols to form 2,5-dihydrofurans was reported [1192]. Gold also catalyzed the formation of phenols from alkynyl tethered furans (Eq. (178)) [1193]. Ruthenium catalyzed the cycloisomerization of yneoates to give enol-lactones [1194].



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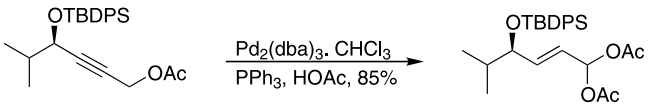
2.5.3. Alkene isomerizations

Palladium catalyzed the formation of vinyl acetals from propargylic acetates (Eq. (179)) [698,1195]. Palladium-catalyzed 1,3-allylic transposition [699] was used in the synthesis of stemodinone (Eq. (180)) [1196], ambrox like compounds [1197,1198], prostaglandin E_1 methyl ester [1199], sphingofungins E and F [311], and 4-demethoxyadriamycinone [1200]. Wilkinson's catalyst was used to prepare vinyl ethers from allylic ethers [1201,1202], and this reaction was used in the synthesis

of hemebrevetoxin B [1087]. Palladium catalyzed a rearrangement of an allylic epoxide (Eq. (181)) [1203], and of chiral 2-alkynyl sulfinates to allenylsulfones [1204]. Ruthenium catalyzed the isomerization of allylic amines to enamines [1205]. A rhodium-catalyzed asymmetric isomerization of allylic amines to enamines was used in the synthesis of citronellal [1206]. Double-bond isomerization of 1-aryl-2-propenyl to 1-aryl-1-propenyl was achieved using a palladium catalyst [1207]. A rhodium-catalyzed double-bond isomerization to the more stable alkene was used in the synthesis of nakijiquinone C [1208].



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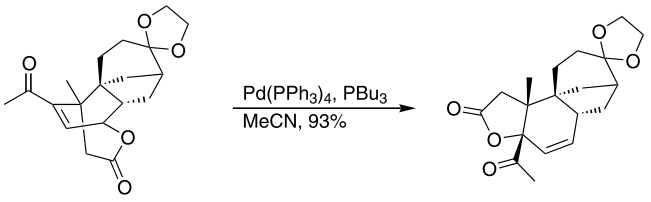

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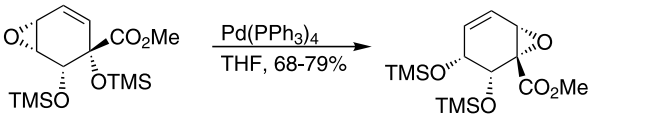
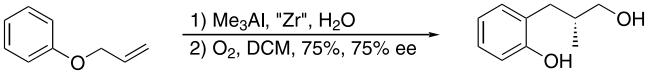


(181)

Iron, rhodium, and ruthenium catalyzed the isomerization of allylic alcohols to saturated carbonyl compounds [1209,1210]. Related reactions using Grubb's catalyst resulted in isomerization to ethyl ketones at elevated temperatures and methyl ketones (a loss of one carbon) at ambient temperature [1211]. Ruthenium catalyzed the isomerization of a propargylic alcohol to an α,β-substituted aldehyde [311].

2.5.4. Skeletal and miscellaneous rearrangements

Copper and ytterbium catalyzed Claisen rearrangements [1212]. A copper catalyzed asymmetric Claisen rearrangement was described [1213]. Water-accelerated a tandem Claisen rearrangement–zirconium catalyzed asymmetric carboalumination (Eq. (182)) [1214]. Palladium-mediated Ferrier-II carbocyclizations [1215], and asymmetric, Wagner-Meerwein rearrangements were described (Eq. (183)) [1216].



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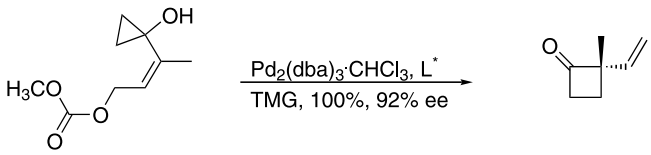


(183)

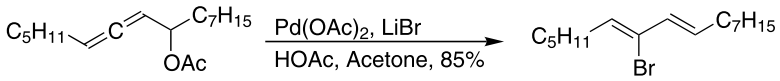
3. Functional group preparation

3.1. Halides, nitriles, azides, ureas, imidates, thioimidates

Vinyl iodides were prepared by the chromium(II) mediated Takai olefination [1217]. This reaction was used in synthesis toward (−)-lepadins [246], mosin B [1218], laulimalide [1096], himandravine [654], (+)-crocacin C [103], (+)-roxaticin [1219], (−)-amphidinolide P [1220], rutamycin B [264], and (−)-equisetin [255].

Palladium catalyzed the transformation of α-allenic acetates to (*Z*,*E*)-2-bromo-1,3-dienes in the presence of lithium bromide (Eq. (184)) [1221]. Hydrozirconation of silyl-substituted alkynes followed by addition of iodine produced silyl-substituted vinyl iodides [150]. A related reaction of tin-substituted alkynes followed by addition of iodine or bromine gave gem dihalo-substituted alkenes [1222].

Palladium catalyzed the cyanation of aromatic halides using zinc dicyanide [1223–1225], dialkyl cyanoboronates [1226], or potassium or sodium cyanide [1227,1228]. Nickel catalyzed an asymmetric hydrocyanation of ethenylarenes [1229]. A three-component palladium-catalyzed route to allylcyanamides employing allyl carbonate, isocyanides, and trimethysilyl cyanide was reported [1230]. A one pot synthesis of β-cyanohydrins catalyzed by zirconium was described [1231].



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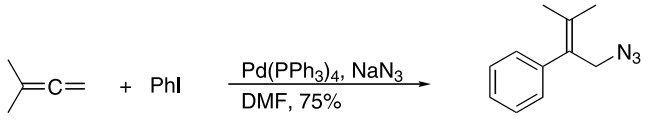
Palladium-catalyzed reaction of isocyanides with an aromatic halide and alkoxides or thioalkoxides produced imidates and thioimidates, respectively [1232]. A palladium-catalyzed azidation of allylic cyanohydrinecarbonates was developed [1233]. Palladium supported on sulfate modified zirconia catalyzed the oxidative carbonylation of amines to form ureas [1234]. Reaction of aryl halides with allenes in the presence of sodium azide afforded allylic azides (Eq. (185)) [1235]. Palladium catalyzed the reaction of sodium azide with allylic acetates forming allylic azides [1236].



(185)

3.2. Amines, alcohols

A large variety of aromatic chlorides, bromides, and triflates were aminated using palladium-based catalyst systems [1237–1246]. A polymer-supported ligand was used for palladium-catalyzed aminations [211]. The mechanism of amination of aryl chlorides [1247] and the kinetics of amination of aryl bromides and triflates were examined [1248]. Bis(trimethylsilyl)amide, triphenylsilylamide, and lithium amide were used as an ammonia equivalents in aminations of aryl halides [1249,1250].

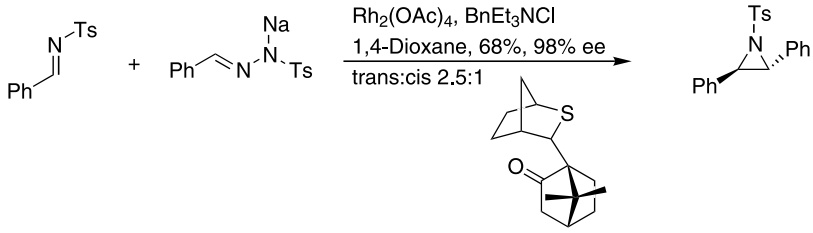
Palladium catalyzed the *N*-arylation of 2-oxazolidinones [1251,1252], benzophenone imine [1253] in the synthesis of a muscarine receptor agonist [1254], hydrazine derivatives using pyridine halides and triflates [1255], chiral sulfoximines [1256,1257], chiral pyrrolidinones [1258], aminocyclopropanes [1259], 7-azabicyclo[2.2.1]heptane [1260], aza-crown ethers [1261], carbazole [1262], urea [1263], polyamines [1264,1265], benzophenone hydrazone [1266], tetrahydro-isoquinoline and -quinoline [1267] and benzotriazole using diaryl iodonium salts [1268]. Palladium-catalyzed amination was used in the synthesis of isocynodine and isocynometrine [967], a staurosporine aglycone analog [1269], and of enantiopure 1,2,4,5-tetrahydro-1,4-benzodiazepin-3-(3*H*)-ones [1270]. Secondary and tertiary aminothiophens were prepared via palladium-catalyzed amination of 3-bromothiophen [1271]. Sequential diamination of 1,2-dibromobenzene was used in a synthesis of benzimidazolium salts [1272].

Palladium-catalyzed amination of 8-bromo-2′-deoxyguanosine [1273], halonucleosides [273], 8-bromoadenosin [1274], 2-bromopyridine [1275], 2-chloropurine [281], 2-bromothiophen [1276], and chlorophthalazines [288] derivatives were reported. Intramolecular amination of *N*-aryl-*N*′-(2-bromobenzyl)hydrazines produced 1-aryl-1*H*-indazoles [1277]. Selective amination in the 2-position of dichloropyridines was observed [1278].

Transition metals other than palladium also catalyzed amination reactions. Nickel catalyzed the amination of dichloroarenes [1279]. Copper catalyzed reactions were described [674,1280–1282]. Copper catalyzed the coupling of aryl halides with amides, nitrogen heterocycles, *N*-BOC-hydrazide, benzophenone hydrazone [1283], and ammonia [1284]. The regioselectivity, terminal vs internal, of the amination using hydrazides depended on the hydrazide [1285]. Copper and nickel but not palladium catalyzed the amination of 5-iodouracil derivatives [1286]. Ruthenium catalyzed the *N*-monoalkylation of anilines using tetralkylammonium halides [1287].

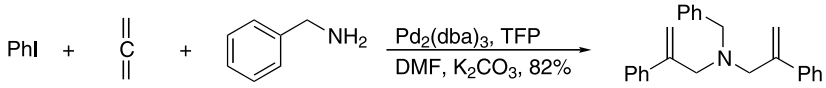
Copper complexes promoted the amination of aryl and vinyl boronic acids with a variety of amines [1288,1289], heteroaromatic amides [1290], purine [281], and imidazoles [1291]. Reactions using aqueous solvent were described [1292]. Copper catalyzed the transformation of allylic stannanes to allylic amines using (*N*-(*p*-toluenesulfonyl)imino)phenyliodinane [1293].

Rhodium catalyzed an asymmetric aziridination of imines using sulfur ylides and in situ generation of the diazocompound (Eq. (186)) [548]. Nickel catalyzed the sequential amination of aryl- and heteroaryl di- and trichlorides [1294].



(186)

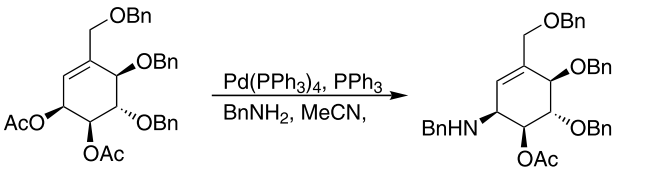
Palladium catalyzed a variety of aminations of allylic substrates via η^3-allyl intermediates. Allylic amination reactions in water were reported [1295]. The rate of amination was accelerated using a tetraphosphine ligand [1296]. 6-Aminopurine [1094], 6-chloropurine [1297], 8-methylthioimidazo[4,5-*g*]quinazoline [1298], and N^6-benzoyladenine [1299] were used as *N*-nucleophiles. Palladium catalyzed the amination of 2-chloro-1,3-butadiene affording 4-amino-substituted 1,2-butadienes [720]. Palladium-catalyzed intramolecular allylic aminations was used in the synthesis of (2*R*,3*R*)-3-hydroxy-3-methylproline [1300], and pancracine [1301]. An intramolecular amidation of an allylic chloride was used to prepare the bicyclic spiro core of halichlorine and pinnaic acid [1302]. Palladium catalyzed a three component reaction between an aryl iodide, allene, and a primary amine forming bis(2-arylallyl)amines (Eq. (187)) [1303]. Palladium and platinum catalyzed the direct reaction between anilines and allylic alcohols [1304,1305].



(187)

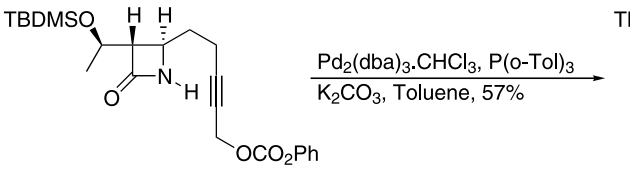
A number of palladium-catalyzed asymmetric allylic aminations were published using optically active ligands. Asymmetric allylic amination using a chiral phase-transfer catalyst was reported [1306]. A regio- and enantioselective amination using sodium diformylamide as the nucleophile was described [1307]. Reaction of racemic 5-vinyloxazolidinones with phthalimide in the presence of a chiral catalyst gave optically enriched regioisomeric

products [1308]. Regioselective amination of an optically active allylic acetate was used in the synthesis of valienamine (Eq. (188)) [1309]. Asymmetric allylic amination was used in the synthesis of (−)-dehydrotubifoline and (−)-tubifoline [443]. Rhodium catalyzed a regioselective and enantiospecific asymmetric allylic amination using *N*-(arysulfonyl)anilines [1310]. Palladium-catalyzed asymmetric [1311], and room temperature [1312], hydroamination of 1,3-dienes were reported.



(188)

Iridium catalyzed allylic aminations of allylic esters [1313]. Palladium catalyzed the amination of phenoxyallene [1068]. A palladium-catalyzed regio- and stereoselective aminohalogenation of styrenes was reported [1314]. An intramolecular amination of propargylic carbamate or phosphonate tethered β-lactams was reported (Eq. (189)) [1315]. Rhodium- and titanium-catalyzed hydroamination of terminal alkynes were described [1316]. The latter catalyst was used under microwave irradiation [1317]. Imidotitanium complexes were used to form imines from allenes and amines (Eq. (190)) [1318]. A silica supported palladium catalyst was used in cyclizations of amino-substituted alkynes to form nitrogen heterocycles [1319].



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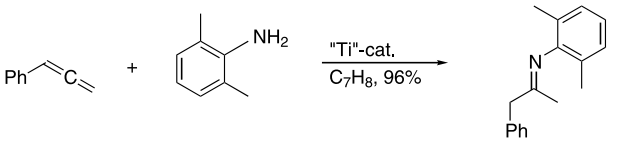
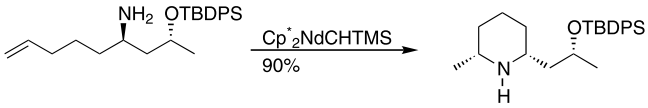


(190)

A ruthenium-catalyzed intramolecular hydroamination of tethered alkene to give five- to seven-membered rings was described [1320]. A neodymium-catalyzed intramolecular hydroamination was used in the synthesis of pinidinol (Eq. (191)) [1321]. Tandem bis-allylation of isocyanates using allyl chlorides and allyl stannanes catalyzed by palladium was reported (Eq. (192)) [1322]. A palladium-catalyzed allylic oxidation-amination was used in the synthesis of (−)-dehydrotubifoline and (−)-tubifoline [443]. Homoallylic amines were prepared by a tandem zirconocene homologation-aldimine allylation (Eq. (193)) [1323].



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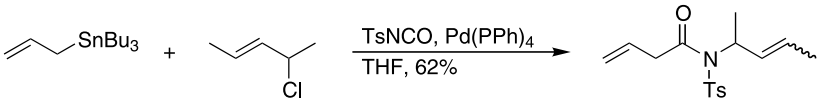

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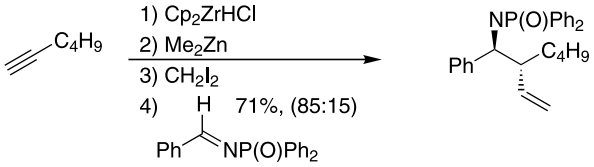
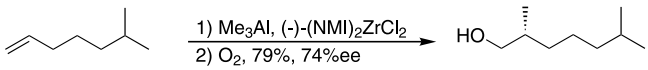


(193)

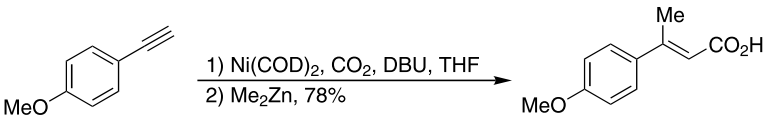
Palladium catalyzed the hydroalumination of 1-alkenes followed by oxidation affording primary alcohols [1324]. Zirconocene-catalyzed enantioselective methylalumination of terminal alkenes followed by oxidation was used in total synthesis (Eq. (194)) [364].



(194)

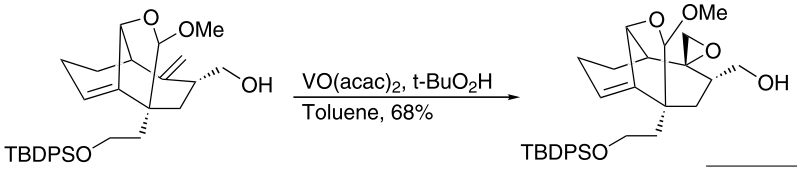
3.3. Ethers, esters, lactones, acids

Ortho esters were prepared by the rearrangement of epoxyesters in the presence of Cp_2ZrCl_2 and silver perchlorate [1325]. A nickel-promoted alkyl or aryl carboxylation of alkynes using carbon dioxide was developed (Eq. (195)) [1326]. A copper-catalyzed asymmetric allylic oxidation of cycloalkenes, using *t*-butylperbenzoate followed by addition of an organozinc reagent, forming allylic esters was reported [1327].



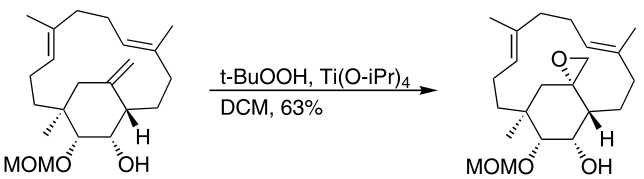
(195)

Vanadyl acetylacetonate-catalyzed epoxidations of allylic alcohols using peroxides continued to be a valuable tool in organic total synthesis. For example, sclerophytins and cladiellin diterpenes [1328,1329], phomactin analogs [1330], the *celastracea* sesquiterpene core [1331], (+)-arteanniun M [1332], functionalized 1,2,3,4-tetrahydroquinolines [404], fredericamycin [1333], FR901464 analogs [187], (−)-bulgecinine [1334], (+)-cheimonophyllon E [1335], the CD ring of paclitaxel [1336], stolonidiol [1337], toward daphnanes and (+)-resiniferatoxin [1338], toward the tetracyclic core of CP-225,917 (Eq. (196)) [1339], and (−)-13-hydroxy-11,12-epoxyneocembrene [1340] were prepared using this methodology. Epoxidation of alkenes in the presence of a tethered alcohol resulted in the formation of tetrahydrofurans [1341,1342].



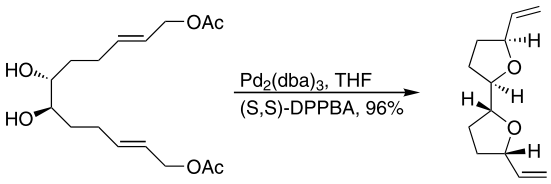
(196)

Sharpless enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continue to see extensive use in organic synthesis. For example, this reaction was used in synthetic approaches to (−)-doliculide [637], sarcodictyins [1343], (−)-laulimalide [1344], 1,N^2-doexyguanosine [1345], teubrevin G and H [1108], (+)-crocacin A [1346], ^{13}C-labeled 2-deoxyribonolactones [1347], the octacyclic core of gambierol [314,1348], posticlure [1349], azaspiracids [1350], cryptophycin A unit [1351], radicol and monocillin I [1352], (+)-prelactone C [1353], (−)-amphidinolide P [1222], pamamycin 607 [1354], laulimalide [1126], the HIJKLM rings of ciguatoxi CTX3C [1129], (+)-3,4-epoxycembrene A [1355], and a puffball pigment [1356]. A related epoxidation of an homoallylic alcohol was used in the synthesis of (−)-fumagillol [363] and of trinervitane and kempane skeletons (Eq. (197)) [1357].



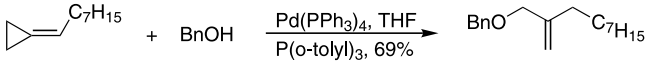
(197)

Palladium-catalyzed asymmetric allylic additions of alkoxides were used in a formal synthesis of LY 333531 [1358] and deschlorocallipeltoside A [983]. A double intramolecular alkoxylation was used in the synthesis of uvaricin (Eq. (198)) [1359]. Palladium catalyzed the reaction of phenols with sugar derived allylic carbonates [1360]. The effects of halide ligands and protic additives on the enantioselectivity in rhodium-catalyzed asymmetric ring opening of oxabicyclic alkanes with phenols was reported [1361].



(198)

Palladium catalyzed the intermolecular reaction of alcohols with aryl halides to form aryl ethers [1362]. Intramolecular reactions to give benzannulated oxygen containing compounds were also reported [1363]. Palladium-catalyzed addition of an alcohol to methoxyallene was used in a synthesis toward laulimalide [682]. Palladium catalyzed the addition of alcohols to alkylidenecyclopropanes to give allylic ethers (Eq. (199)) [1364]. Palladium catalyzed the carbon–oxygen coupling of sodium *t*-butoxide and unactivated aryl halides [1365].



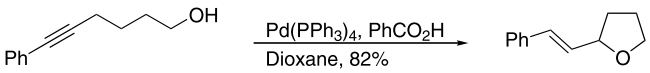
(199)

A palladium-catalyzed hydroxy-palladation–carbonylation–lactonization sequence was used in natural product synthesis forming bicyclic compounds [1366], and palladium mediated the same reaction in the preparation of benzo[*b*]furo[3,4-*d*]furane-1-ones [1367].

Copper mediated the cross-coupling of *N*-hydroxyphthalimide with phenylboronic acids forming *N*-aryloxyphthalimides [1368]. Intramolecular *O*-arylation of phenols with phenylboronic acids, catalyzed by copper, was used in the synthesis of macrocyclic metalloprotein inhibitors [1369]. Copper catalyzed the intermolecular etherification of phenols with aryl bromides [674].

A palladium-catalyzed reaction of a phenol with an arylboronic acid was used in the synthesis of teicoplanin aglycon

[1370]. Palladium-catalyzed addition of phenols to allenes furnished dienylethers [1371]. An interesting palladium catalyzed hydroalkoxylation of alkynes in the presence of benzoic acid was developed (Eq. (200)) [1372].

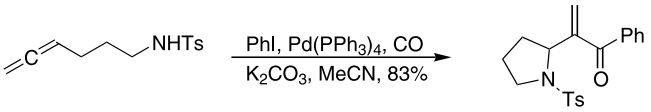


(200)

3.4. Heterocycles

A variety of complex polycyclic heterocycles were obtained by reactions usually initiated by an oxidative addition of a low valent transition metal to an aryl or a vinyl halide [1373–1385]. The product isolated depended on the sequence of events that follow, for example, alkene and alkyne insertions, carbonylations, and terminations. These cascade-type reactions are unfortunately not readily categorized within this review and are therefore only briefly discussed. A palladium-catalyzed intramolecular insertion of an alkyne was used in the synthesis of the ergoline ring system [425], and quinoxaline derivatives [1386].

Reaction of allene tethered sulfonamides with carbon monoxide and aryl iodides affords 3-aroyl-2- or 3-pyrrolines and pyrrolidine- or piperidine-substituted enones depending on the length of the tether (Eq. (201)) [1387]. Reaction of 2-halobenzamides with allenes gave *N*-heterocycles (Eq. (202) [1388]. A diastereoselective cobalt-mediated acylation of amine-tethered allenes to give pyrrolidines was reported (Eq. (203)) [1389].



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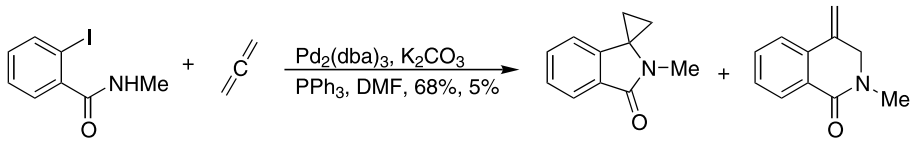

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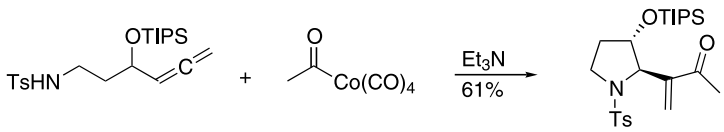
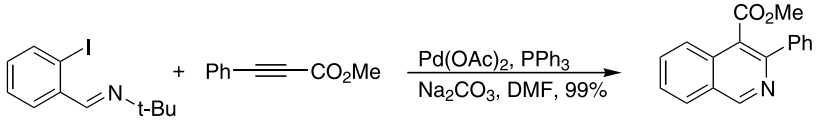


(203)

Reaction of *N*-allyl-2-iodoaniline or *O*-allyl-2-iodophenol with carbon monoxide in the presence of a palladium catalyst gave mixtures of 3-methoxycarbonylmethyl derivatives of dihydroquinoline and dihydrochromeme, respectively. The product ratio was dependent on the carbon monoxide pressure [1390]. Palladium catalyzed the annulation of 2-halobenzaldehyde imines with alkynes forming 3,4-disubstituted isoquinolines (Eq. (204)) [1391]. Pyridines [1392], isoindolo[2,1-*a*]indoles [1393], and β- and γ-carbolines [1394] were formed in a similar fashion. Palladium catalyzed the reaction of aryl halides with *N*-propargylamides to give 2,5-disubstituted oxazoles (Eq. (205)) [1395]. Pyridines, quinolines, pyrroles, and spiro amines were prepared by palladium-catalyzed cyclization of unsaturated oximes (Eq. (206)) [1396,1397]. Indole[1,2-*c*]quinazolines were prepared by a palladium-catalyzed reaction of aryl and vinyl halides and triflates with bis(2-trifluoroacetamidophenyl)ethyne [1398]. Palladium catalyzed the reaction of 2-iodophenols or 2-iodoacetaniline with terminal alkynes, in the presence of an excess of methylmagnesium chloride, an aryl- or vinyliodide, or carbon monoxide forming 2,3-substituted benzofurans and indoles [1399]. Rhodium-catalyzed coupling-cyclization of *N*-aryl trifluoroacetimidoyl chlorides with alkynes furnished quinolines (Eq. (207)) [1400].



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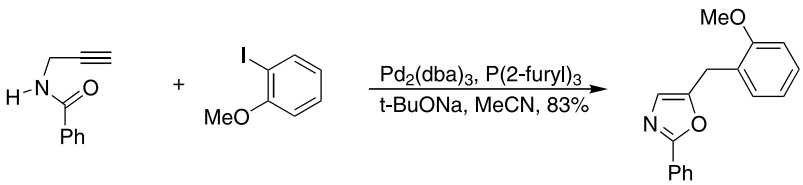

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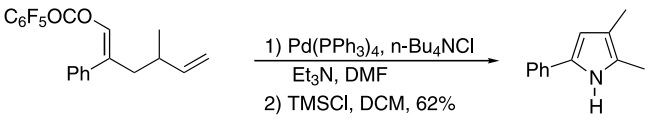
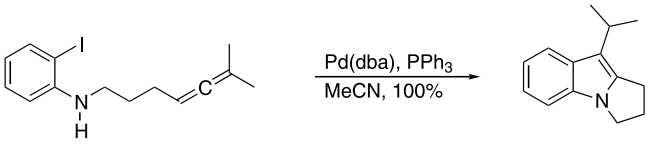


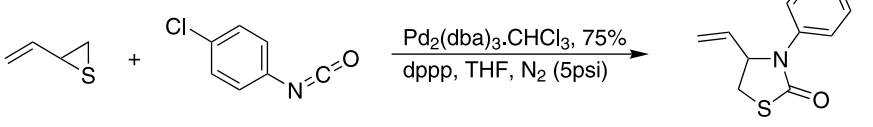
(206)

Palladium catalyzed the cyclization of 2-vinylazetidines with heterocumulenes affording tetrahydropyrimidones, tetrahydropyridinimines, and thiazinanimines [1401]. Palladium-catalyzed reaction of optically active allenes having an aminofunctionality tethered to the chain with aryl iodides produced three- to five-membered nitrogen heterocycles [1402]. An interesting synthesis of fused indoles by palladium-catalyzed annulation of *N*-allene tethered 2-iodoaminobenzenes was developed (Eq. (208)) [1403].



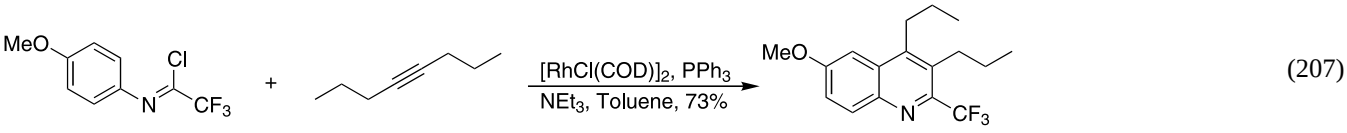
(208)

Palladium-catalyzed cyclization of 2-vinylthiiranes with heterocumulenes furnished thiazolidine, oxathiolane, and dithiolane derivatives (Eq. (209)) [1404]. A palladium–copper catalyzed regio- and stereoselective synthesis of (*E*)-2-(2-arylviny)-3,1-benzoxathiin-4-ones was reported [1405].



(209)

Vinylogous 2-sulfonylethenylindolines were prepared by reaction of 2-iodoanilines with dienyl-sulfones in the presence of a palladium catalyst [1406]. A three component, palladium-catalyzed synthesis of imidazolines using carbon monoxide, an acid chloride, and imines was described (Eq. (210)) [1407]. γ-Lactams were prepared from α-bromoacrylamides and 1,3-dienes [1408].



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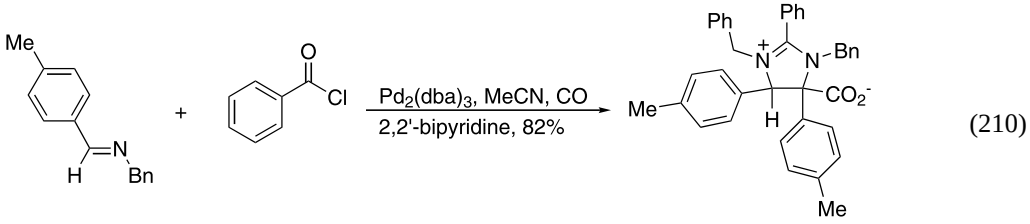
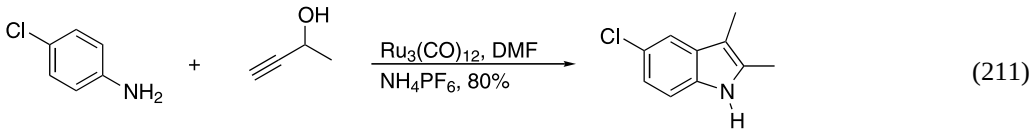


(210)

Sequential palladium-catalyzed Sonogashira coupling followed by intramolecular amino-palladation of polymer-bound 2-iodoanilines gave indoles [269]. A related reaction was used in synthesis [270]. A palladium-catalyzed tandem amino-palladation of allenyl *N*-tosylcarbamates followed by a reductive Heck reaction was reported to give 2-oxazolidinones [1409]. A 2,3-disubstituted indole was prepared from a 2-iodoaniline and an internal alkyne [271]. Heck reaction of 2-haloanilines gave indoles [1410]. An interesting ruthenium-catalyzed synthesis of 2,3-disubstituted indoles by reaction of anilines with propargylic alcohols was developed (Eq. (211)) [1411]. Tryptophan analogues were prepared by reaction of internal alkynes with 2-iodoarylamines [1412]. Reaction of alkanolammonium chlorides with aryl amines in the presence of a ruthenium catalyst and a Lewis acid produced indoles [1413]. A copper-catalyzed cyclization was used to prepare (*E*)-2-(2-arylvinyl)quinazolinones (Eq. (212)) [1414,1415]. A ruthenium-catalyzed oxidative cyclization of 2-aminobenzyl alcohols and ketones to give quinolines was developed [1416].



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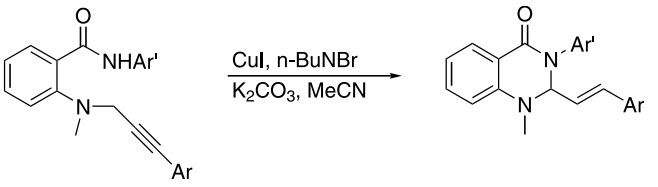
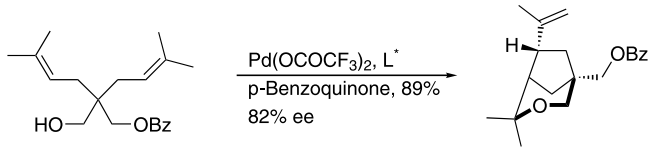


(212)

Poly(pyrazolyl)borate–copper complexes catalyzed the aziridination of alkenes [1417]. Copper-catalyzed aziridination of an alkene was used toward the synthesis of kalihinane diterpenoids [1418]. Copper-catalyzed aziridination mediated by PhI=O was reported [1419]. Diethylzinc, chloroiodomethane, and tetrahydrothiophen were used to form aziridines from imines [1420].

Intramolecular hydroxy-palladations forming five-membered rings were reported [447,1421]. Palladium-catalyzed intramolecular hydroxy-palladation of intermediately formed cumulenes was used to prepare tetra-substituted furans (Eq. (213)) [1422]. A three component reaction of an aryl or vinyl halide and triflate with a propargylic alcohol, and an alkylidene malonate, using a palladium catalyst, produced 3-arylidene or 3-alkylidene tetrahydrofurans (Eq. (214)) [1423]. This reaction was used in a synthesis of burseran [1424]. Asymmetric reaction of activated alkenes with hydroxy-group tethered allylic carbonates, in the presence of a palladium catalyst, produced cyclic ethers [1425].



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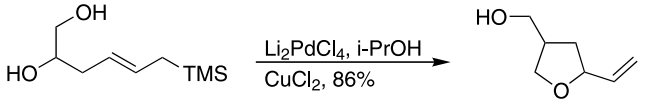
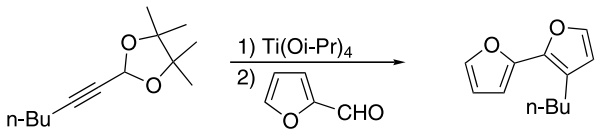


(216)

1,3-Oxazolidines were prepared by palladium catalyzed reaction of 5-vinyl-1,3-oxazolidin-2-ones [299]. Titanium mediated the formation of 2- and 2,3-substituted furans from 2-alkynal acetals and aldehydes (Eq. (217)) [1431]. A nickel-catalyzed cyclization of oxanorbornenes and alkyl propiolates to give benzocoumarin derivatives was developed (Eq. (218)) [1432].



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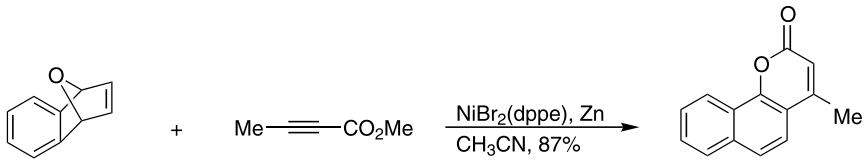
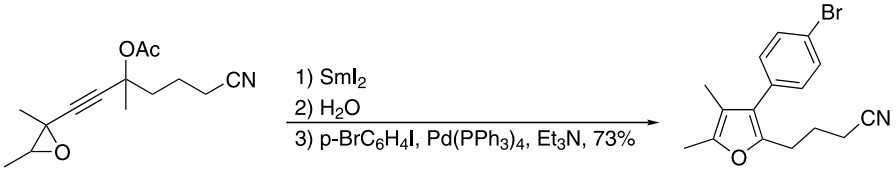


(218)

An asymmetric hydroxy-palladition alkene cyclization was described (Eq. (215)) [1426]. Palladium-catalyzed hydroxypalladation of allylic silanes forming tetrahydrofurans was reported (Eq. (216)) [1427]. Palladium catalyzed the reaction of benzene-1,2-diols with propargylic carbonates to give 2-alkylidene-1,4-benzodioxanes [1428], and with the biscarbonate of 2-(hydroxymethyl)allyl alcohol to give 3-methylene-3,4-dihydro-2*H*-1,5-benzodioxepines [1429]. Intramolecular hydroxy-palladation methoxycarbonylation of 4-hydroxyalkynes was used to prepare (*E*)-cyclic β-alkoxyacrylates [1430].

3.5. Alkenes, allenes, alkanes, arenes

A variety of functional groups were replaced by a hydrogen using palladium-catalyzed methodologies. Palladium-complex catalyzed reduction, together with a hydride source, such as tributyltin hydride, silanes, or ammonium formates, were used in synthesis of (−)-dehydrotubifoline and (−)-tubifoline [483], D-erythro-sphingosine and L-lyxo-phytospingosine [1433], the ABCD ring system of azaspiracid [1434], and (−)-a-eudesmol [1435]. Palladium catalyzed the reductive cleavage of polymer-bound aryl



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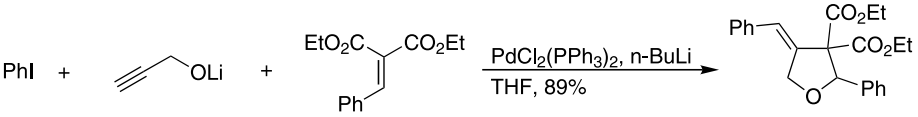


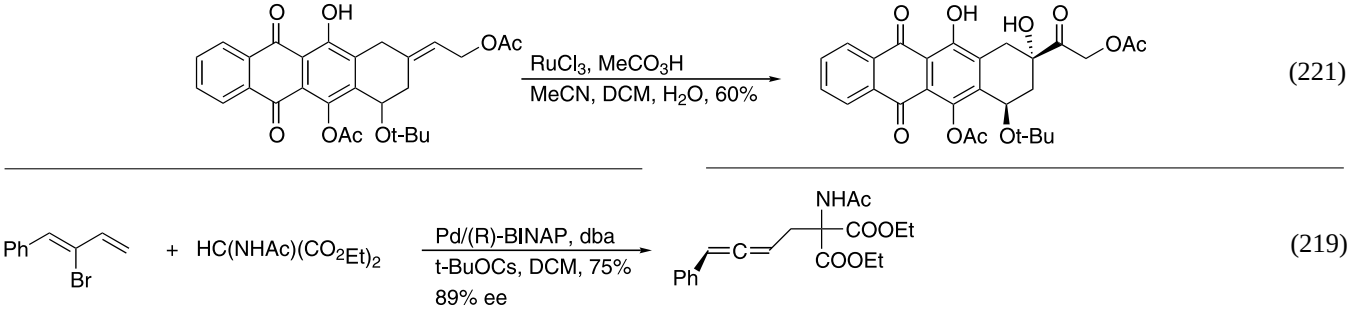
(214)

perfluoroalkylsulfonates [1436]. The trifloxy groups of a 3-trifloxyfuran [1437] was removed using similar methodologies.

Monodebromination of 1,1-dibromoalkenes was achieved using tributyltin hydride, in the presence of a palladium catalyst, forming *cis*-substituted bromoalkenes [147,437]. This reaction was used in the synthesis of combrestatins [250]. Palladium catalyzed an interesting intramolecular redox reaction resulting in debromination-enone formation [1438]. Nickel catalyzed the reduction of aromatic halides to arenes in the presence of boron–dimethyl amine complex [1439,1440]. A palladium-imidazolium catalyst was used to dehalogenate aryl halides [1441].

Palladium-catalyzed regioselective ammonium formate reductions of allylic carbonates and carboxylates were reported [1442,1443], and this reaction was used in synthesis of 5,7-dideoxypaclitaxel derivatives [1444]. A related regioselective palladium-catalyzed reduction of a vinyl epoxide to a homoallylic alcohol was used in synthesis of a precursor of 9-nor-1α,25-dihydroxyvitamin D_3 [1445]. Palladium catalyzed the reductive ring opening of a vinyl-substituted lactone [446]. A palladium-catalyzed samarium mediated reduction of propargylic phosphonates to give allenes was described [1446].

Palladium-catalyzed transformation of allylic acetates to dienes were used in a synthesis of mesembranol [256] and a homoheptalene derivative [1447]. A terminal triple bond was reduced to a double bond in the presence of internal double or triple bonds using sodium borohydride and a palladium catalyst [1448]. A palladium-catalyzed synthesis of chiral allenes from 2-bromo-1,3-dienes was reported (Eq. (219)) [1449]. Wilkinson's catalyst was used to remove an aldehyde functionality [1450].



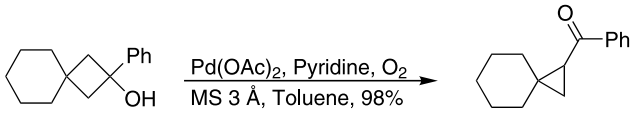
(219)

3.6. Ketones and aldehydes

A number of applications of the Saegusa reaction, i.e. the transformation of silyl enol ethers to α,β-unsaturated ketones, were published [1451–1453]. For example, synthesis of variecolin [1454], cedrone [862], stemonamide and isostemonamide [1455], FR901464 analogs [187], kelsoene [1456], and naewedine and galanthamine [1457] were described. A related palladium-catalyzed reaction of a methyl enol ether was used in the synthesis of stelletta-dine A [1458]. The oxidation was also accomplished using tributyltin methoxide and allyl methyl carbonate in the presence of palladium diacetate [1459].

Wacker-type oxidation, i.e. reaction of mono-substituted terminal alkenes with palladium(II) and water to afford methyl ketones, was utilized in a number of syntheses [1043,1454,1460,1461]. Frontaline analogs [1462], (−)-fumagillol [363], sclerophytins [758], β-elemene [1463], toward eudesmanolides [296], (+)-roxaticin [1219], 2-thiocyanatoneopupukeanane [1464], herbertenediol and mastigophorenes [1465], and diterpenoids [1466] were prepared using a Wacker reaction in one of the steps. A correlation of relative rates of oxidation of alkenes vs alkene ionization potentials, HOMOs and LUMOs was reported [1467]. Reversal of regioselectivity of the addition of water was observed comparing styrene and 1-phenylpropene [1468].

Palladium catalyzed a ring-expansion of 1-allenyl-1-cyclobutanols in the presence of a Michael acceptor to give substituted cyclopentanones [1469]. Palladium also catalyzed a ring-opening of tertiary cyclobutanols to give unsaturated ketones, ring-expanded ketones, or ring-contracted ketones in good yields (Eq. (220)) [1470].



(220)

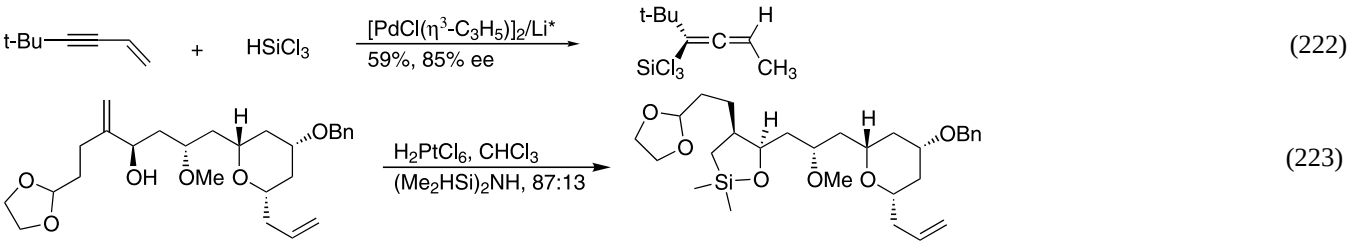
A ruthenium-catalyzed oxidation of a trisubstituted allylic alcohol to a hydroxy ketone was used as a key step in the synthesis of demethoxyadriamycinone (Eq. (221)) [1200]. Ruthenium-catalyzed anti-Markovikov additions of water to terminal alkynes affording aldehydes [1471].

(221)

3.7. Organoboranes, silanes, germanes and stannanes

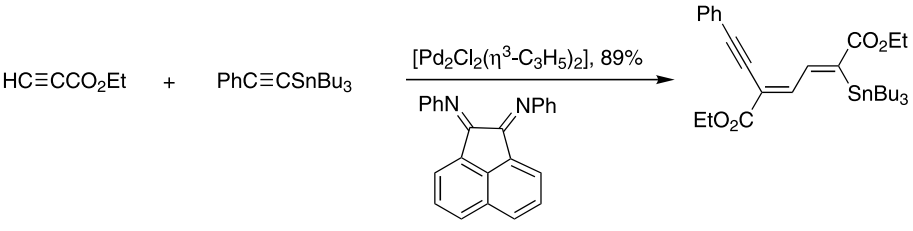
A thioether-directed platinum-catalyzed hydrosilylation of alkenes was reported [1472]. Palladium-catalyzed asymmetric hydrosilylations [1473–1475] for example, of 4-substituted 1-buten-3-ynes to give chiral allenylsilanes (Eq. (222)) [1476]. Wilkinson's catalyst was used in hydrosilylations of ynals [1477], alkenes [1478], and was used in tandem hydrosilylation–aldol reactions [1479]. Cobalt catalyzed the hydrosilylation of sugar derived al-

lenes [1480]. A platinum-catalyzed intramolecular hydrosilylation was used in the synthesis of a leucascandrolide A fragment (Eq. (223)) [1481]. Reaction of aromatic bromides and iodides with triethoxysilane in the presence of a palladium catalyst gave triethoxysilane-substituted arenes [1482]. A rhodium-catalyzed desymetrization of 4-dimethylsilyloxy-1,6-diynes via sequential silyl-formylation was reported [1483].

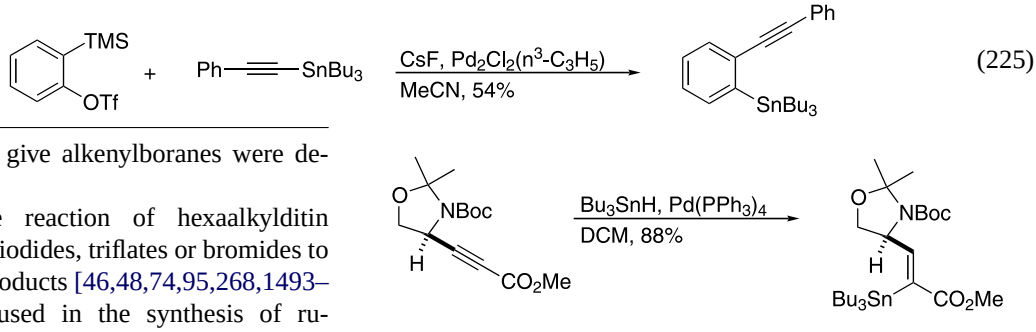


Palladium catalyzed the borylation of benzylic carbon–hydrogen bonds using bis(pinacolato)diboron or pinacoleborane [1484], and rhodium catalyzed regioselective borylation of aromatic carbon–hydrogen bonds using pinacolborane [1485]. Palladium catalyzed the cross-coupling of aromatic halides, triflates with dipinacolylborane or pinacoleborane producing aromatic pinacolyl boronic esters [120,258,259,1371,1486–1489]. Cedranediolborane was used in related reactions [1490]. A palladium-catalyzed 1,2-diboration of allenes was reported [1491]. Related borasilylations of allenes to give alkenylboranes were described [1492].

Palladium catalyzed the reaction of hexaalkylditin reagents with aryl, and vinyl iodides, triflates or bromides to give trialkyltin-substituted products [46,48,74,95,268,1493–1497]. This reaction was used in the synthesis of rutamycin B and oligomycin C [1498], cladiellin diterpenes [1328], the core of phomactin [35], toward FR182877 [99], lapidilecitin B [1499], and himastatin [732]. Palladium-catalyzed the dimerization–carbostannylation of alkynes to give conjugated dienylstannanes (Eq. (224)) [1500].



A palladium–iminophosphine catalyzed carbostannylation of arynes was developed (Eq. (225)) [1501]. Palladium-catalyzed hydrostannation of terminal alkynes using tributyltin hydride, forming vinylstannanes, [47] was used in total synthesis of ratjadone [87], (−)-reveromycin B [88], (−)-ichthyothereol [94], bafilomycin A_1 [110] phorboxazoles [106,1502], a segment of gambieric acid [1503], apoptolidin [130,260], himbacine derivatives [33], and astaxanthin analogs [128]. Regioselective hydrostannation of a 1,4-dihydroxy-2-alkyne [81], and a regiospecific hydrostannation on the carbon adjacent to the aryl group of an 1-aryl-1-alkyne were reported [95]. The regiochemical outcome was influenced by oxo-substitution of terminal alkynes [1504]. Regioselective palladium-catalyzed hydrostannations of internal alkynes [47,1505] were used in the synthesis of asperazine (Eq. (226)) [109], and (+)-phorboxazole A [111]. Hydrostannation of a bromoalkyne affording a vinylstannane was used in the synthesis of sanglifehrin A [122]. Hydro-, methyl-, and butyl-stannations of *N*-benzyl-*N*-tosylynamine were reported [1506].

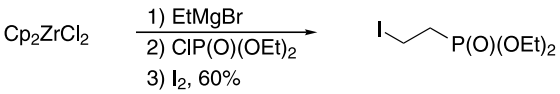


Nickel-catalyzed the acylstannation of 1,2-dienes [1507] and of ynoates [819]. A palladium-catalyzed allylic stannylation of allyl chloride, using $Bu_3SnAlEt_2$ as the nucleophile was employed in a synthesis of a spongistatin 1 fragment [1508]. A rate acceleration of the palladium-catalyzed hydrogermylation of terminal alkynes and dienes was observed in water [1509].

3.8. Organophosphorous, arsenic, selenium, and sulfur compounds

Aryl diethylphosphonates were prepared from aromatic halides using diethylphosphite [1510], aryl dialkylphosphonates were prepared from dialkyl phosphates and diaryl iodonium salts [1511], and vinyl phosphonates were prepared from vinyl iodides and dibenzylphosphite [1512] all using palladium catalysts. Palladium catalyzed the reaction of bromopyridines with dinucleosidephosphonates [1513] and with isopropyl methylphosphinate [1514]. Vinyl phosphonate linked oligonucleic acids were prepared in an analogous fashion [1515]. Palladium catalyzed the phosphorous–carbon coupling of aryl triflates with diarylphosphine oxides to give a triarylphosphine oxides [892,1189,1516,1517]. Reaction of arylphosphinic esters with aryl iodides gave diarylphosphinic esters [1518]. Palladium catalyzed the formation of triaryl phosphines from aryl phosphines and aryl iodides, bromides, and triflates [1519–1521]. Palladium catalyzed the coupling of vinyl triflates with diphenylphosphine and diethylphosphine [455]. Nickel catalyzed the coupling of aryl halides and triflates with diarylphosphines [1522,1523], and of iododihydropyridones to give phosphonates and phosphine oxides [1524]. Mono-substituted phosphinic acids were prepared by palladium-catalyzed coupling of anilinium hypophosphites with aryl halides [1525].

Palladium and nickel catalyzed the hydrophosphination of alkynes. Complete reversal of regiochemistry was obtained depending on the catalyst and solvent [1526]. A palladium-catalyzed hydrophosphorylation of 1,3-dienes forming allyl phosphonates was developed [1527]. Rhodium catalyzed a regioselective addition of diphenylposphine oxide to terminal alkynes affording *E*-alkenyl diphenylphosphine oxides [1528]. Rhodium and palladium catalyzed the hydrophosphorylation of terminal alkenes [1529–1531]. Reaction of zirconocene–ethene complex with diethylchlorophosphate, followed by an electrophile, gave functionalized phosphonates (Eq. (227)) [1532]. Palladium catalyzed a solvent free arsination of aryl triflates [1533].



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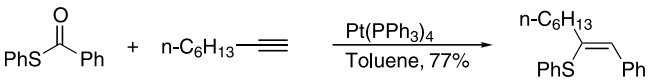
Palladium catalyzed a regioselective carbothiolation of alkynes (Eq. (228)) [1534]. Palladium-catalyzed coupling of aryl and heteroaryl iodides and chlorides with thiols to give thioethers was described [166,1240,1535]. This reaction was used in the synthesis of chuangxinmycins (Eq. (229)) [1536]. Reaction of 3-trifloxymorphine with tributyltin *t*-butylsulfide under similar conditions was described [1537]. Rhodium catalyzed the addition of dialkyldisulfides to alkenes forming (*Z*)-bis(1,2-alkylthio)alkenes [1538]. Palladium catalyzed the formation of 1,2-bis-triisopropylsilanylsulfanyl-alkenes from alkenes and bis-triisopropylsilyldisulfide [1539]. A palladium-catalyzed sulfinylzincation of alkynyl sulfoxides was reported [1540].



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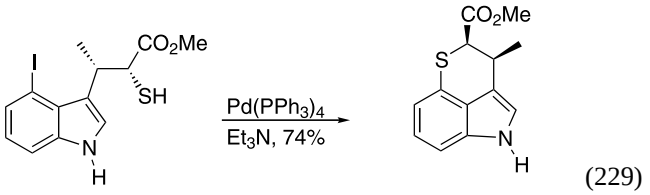

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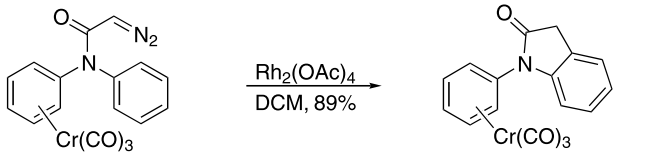
4. Preparations and reactions of isolated metal complexes and metal intermediates

A number of transition-metal catalyzed reactions were published wherein metal complexes were allowed to react on one or more of the ligands without demetallation of the complex. Iodoferrocene was carbonylated to give α-ketoamides [1541]. Intramolecular Heck reactions were reported using arenechromium tricarbonyl complexes [1542]. Heck reaction of vinylferrocene was reported [1543]. Sonogashira-type coupling of ferrocenylethyne [1544,1545], 1,2-diethynylcyclobutadiene cobalt complexes [740], and Sonogashira, Stille, Suzuki, and Negishi couplings of iodo-substituted ferrocene [1546–1549] were reported. Palladium catalyzed the coupling of 1,1′-dizincferrocene with 8-bromoquinoline [1550]. Bis(ferrocene)mercury was coupled with aryl halides under palladium catalysis [1551]. A Suzuki coupling of tricarbonyl chromium aryl bromide was used in a synthesis of the A and B ring system of vancomycin [1552]. Stille, Sonogashira, and Suzuki couplings of arene tricarbonyl chromium complexes were reported [1553,1554], using iodo-substituted ferrocene [1555], and ferrocenylstannane [1556]. An asymmetric synthesis of a protected ent-actinoidinic acid using a Suzuki coupling of a chiral arene chromiumtricarbonyl was described [1557]. Reaction of chloroarene tricarbonyl chromium with hexalkylditins in the presence of palladium resulted in the transfer of one of the alkyl groups [1558]. Ferrocenophanes were prepared via a ruthenium catalyzed ring-closing metathesis [1559].

Arene tricarbonyl chromium complexes continued to be extensively used as templates for organic reactions. Reactions on functional groups attached to complexes of this type include, radical reaction of pendant carbonyl groups [1560], Michael additions [1561], ring-closing metathesis [1562], Mannich reactions [1563], and stereoselective Diels–Alder reactions [1564]. An arene tricarbonyl chromium complex was used in the synthesis of 11-epi-helioporin B [1565]. Silylketene acetals reacted with arene tricarbonyl chromium complexes to give lactones [1566].

Reactions on the aromatic ring of arene tricarbonyl chromium complexes were also described, for example, enantioselective palladium-catalyzed methoxycarbonylation of 1,2-dichlorobenezene chromium tricarbonyl complex [1567], aromatic nucleophilic substitution using an alkoxide [1568], amines [1569], and phosphoryl-stabilized carbanions [1570]. Clean *ortho*-functionalization of 4-*t*-butyl-benzylalcohols complexed to chromium tricarbonyl was reported [1571]. Sequential addition of a nucleophile and an electrophile to enantiomerically pure arene chromium tricarbonyl complexes to give chiral-substituted cyclohexadienes was described [1572].

Arene chromium carbonyl complexes were used as polymer-bound linkers [1573,1574]. The arene chromium carbonyl moiety does not undergo intra- or intermolecular cyclopropanations using carbenoids derived from diazacompounds (Eq. (230)) [1575]. Propargylic cations adjacent to a chromium tricarbonyl arene group underwent electrophilic substitution reactions with nucleophiles affording products with retention of configuration [1576]. Allenic thioethers were obtained from related reactions with thiols [1577].



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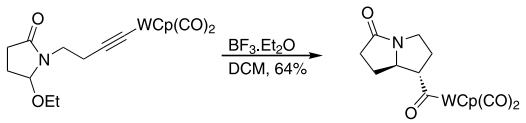
Tungsten-σ-alkyne complexes were used to prepare indolizidine and quinolizidine derivatives via intramolecular cyclization with *N*-acyliminium ions (Eq. (231)) [1578]. Formal [3+3] and [3+2]cycloadditions of epoxide-substituted tungsten-σ-alkyne complexes leading to bicyclic pyranes were reported (Eq. (232)) [1579,1580]. Enantioselective synthesis of naturally occurring butanolides was accived starting from tungsten-σ-alkyne complexes [1581].



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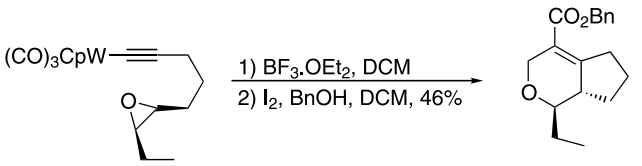

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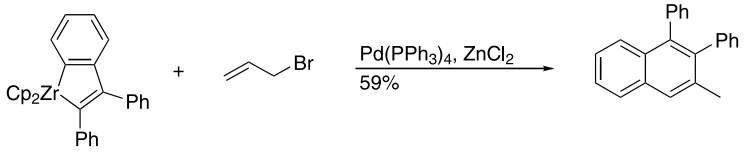
Intermolecular nucleophilic aromatic substitution of ruthenium chloroarene complexes forming 2-tetralones was reported [1582]. Aza crown ethers were used as nucleophiles in related reactions [1583]. An intramolecular reaction was used toward the BCDF ring system of ristocetin A [1584]. Palladium catalyzed the alkoxycarbonylation of a η^5-1-chlorocyclohexadienyl manganese complex [1585]. Intramolecular [2+2+1]cycloaddition of oxygen-thetered diynes using iron pentacarbonyl gave bicyclic iron tricarbonyl complexes [1586]. Palladium catalyzed the reaction of zirconaindenes with allyl bromide to form naphthalenes (Eq. (233)) [1587]. Carbonylation of titanacyclobutanes complexes prepared by free radical alkylations produced cyclobutanones [1588].



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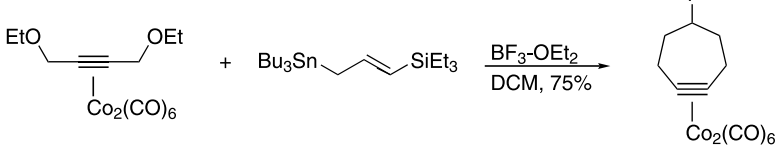
Alkynes complexed to dicobalt hexacarbonyl were used as templates for organic synthesis. An interesting cycloaddition of butyne-1,4-diethers complexed to cobalt with allyl silanes forming seven-membered rings was described (Eq. (234)) [1589]. An epoxide to pyrane conversion was used in the synthesis of (−)-ichthyothereol [94]. Cobalt coordinated propargylic acetals exhibited enhanced diastereoselectivity in crotylation reactions compared to uncomplexed substrates [1590]. Diastereoselective reactions of chiral propargylic acetals were reported [1591]. Nicholas-type reactions [1592] of cobalt alkyne complex stabilized cations were used in synthesis toward ciguatoxins [1593,1594], and pseudopteropsin G–J aglycon [1595]. Cations derived from bispropagylic alcohols complexed to dicobalt hexacarbonyl reacted to form diethers [1596]. An intramolecular oxonium-ene type reaction was described (Eq. (235)) [1597]. Ruthenium-catalyzed ring-closing metathesis of a molecule containing a hexacarbonyl dicobalt alkyne complex was reported [1598].



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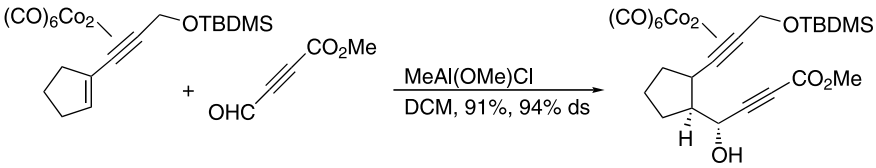

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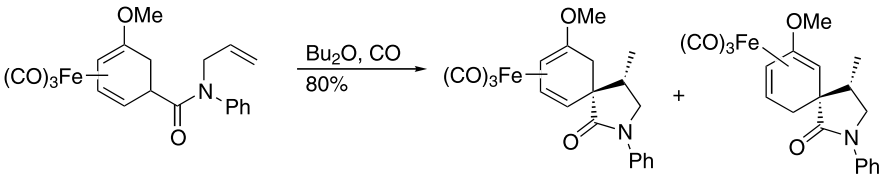
Iron tricarbonyl diene templates were used to prepare 11*Z*-9-demethyl-9-((3-indolyl)methyl)retinal [1599], and in an asymmetric synthesis of halicholactone [1600]. Intramolecular coupling between iron tricarbonyl-1,3-cyclohexadiene complexes and a pendant alkene produced spirolactams stereospecifically (Eq. (236)) [1601]. Related reactions of pendant alkenes and arenes were reported [1602]. An efficient synthesis of optically pure tricarbonyl(methyl 6-oxo-2,4-hexadienoate)iron was described [1603]. Regioselective deprotonation followed by 1,3-migration of phosphorous was observed for iron coordinated 1,3-cyclohexadienes (Eq. (237)) [1604]. Stereospecific 1,3-migration of the irontricarbonyl moiety on acyclic conjugated polyenes were applied to remote and iterative asymmetric induction [1605]. Removal of the iron tricarbonyl moiety and reduction of the diene to and alkane using Raney nickel was used in the synthesis of (+)-[6]-gingerdiol [1606]. Hydrogenation of tricarbonyl iron cycloheptatriene complexes gave the corresponding diene complex [1607]. The 1-ethoxycarbonyl-2-methylpentadienyl iron tricarbonyl cation was shown to react predominately at the C-5 carbon [1608].



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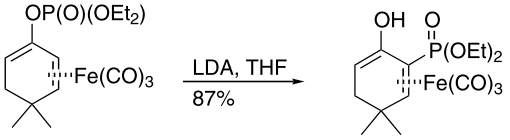
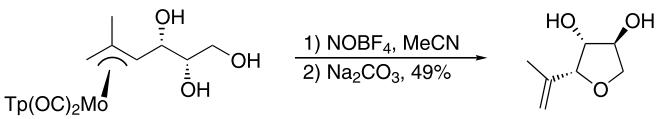


Diastereoselective synthesis of γ-butyrolactones using η^3-allyl molybdenum complex was reported (Eq. (238)) [1609]. η^3-Pyranylmolybdenum complexes were used as chiral scaffolds for regio- and sterocontrolled [4+2]cycloadditions [1610], and in synthesis of 2,3,6-trisubstituted piperidines [1611]. Iron and molybdenum η^3-allyl molybdenum complexes were used in synthesis [1595]. Acyclic 1,3-diols were prepared from vinyl η^3-allyl tungsten complexes [1612].



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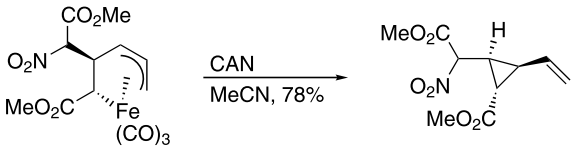
The long-range regio- and steroinduction of nucleophilic addition to vinyl-substituted cationic cyclohexadienyl iron complexes was examined [1613]. An η^5-iron pentadienyl complex was used as a template for cyclopropane synthesis (Eq. (239)) [1614]. η^5-Iron pentadienyl cations reacted with poor nucleophiles such as furan and allyl trimethylsilane [1615].



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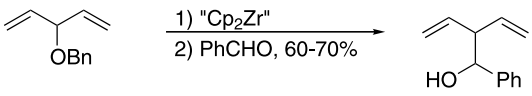
Hydrozirconation of alkynes followed by addition of iodine to produce vinyl iodides was used in the synthesis of the macrocyclic core of ratjadone [87], callystatin A [424], FR901464 analogs [187], apoptolidin [130,1127], and apoptolidinone [1616]. Related palladium-catalyzed hydrostannation-iodination of a terminal alkyne was use in the synthesis of cyclamenol A [668]. Hydrozirconation of a terminal alkyne followed by transmetallation to zinc and reaction with an aldehyde was used in the synthesis of (−)-ratjadone [1617]. Methylzirconation using trimethyl aluminum and $ZrCp_2Cl_2$, followed by treatment with acetaldehyde, gave an allylic alcohol [1618]. Reaction of phosphonate-substituted zironacyclopropene with alkynes furnished 1,3-butadienylphosphonates [1619].

Reaction of terminal alkynes with trimethylaluminum and a catalytic amount of zirconocene dichloride, followed by iodine to give trisubstituted vinyl iodides [1087], was used in syntheses of concanamycin F [85], bafilomycin A_1 [110], the core of phomactin [35], ratjadone [87], and bis-deoxylophotoxin [1620]. Methyl and ethylaluminations followed by water quench was used in the synthesis of (10*R*,11*S*)-(+)-juvenilehormones [1621].

Insertion of aldehydes into zirconacycle phosphonates affords alkenylphosphonates [1622]. Intermediately formed pentadienylzirconium compounds reacts with carbonyl compounds to give 3-alkylated 1,4-dienes (Eq. (240)) [1623].



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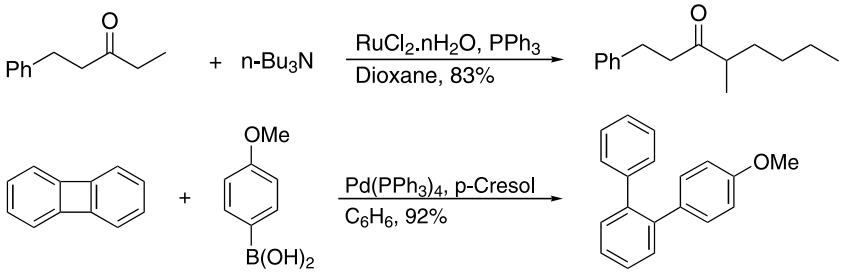
5. Miscellaneous

Grubbs' catalyst was shown to catalytically remove allyl groups form amines [1624]. Aromatization of enamines derived from cyclohexanone, 1,3-cyclohexandione, and tetralones employing a stoichiometric amount of palladium(II) salts was reported [1625]. A chiral cobalt catalyst was developed for enantioselective carbonyl-ene reactions [1626]. Tungsten mediated the annulation of γ-acetylenic silyl enol ethers (Eq. (241)) [1627].



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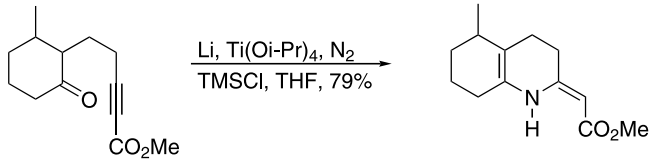

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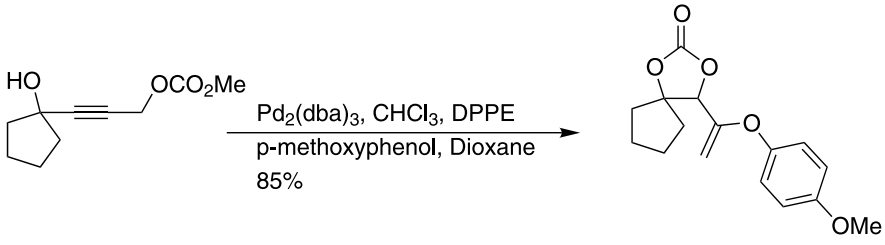

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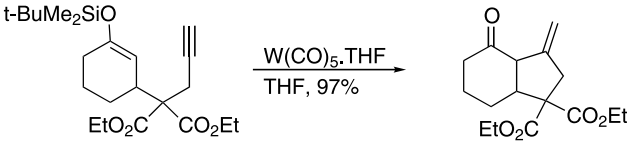

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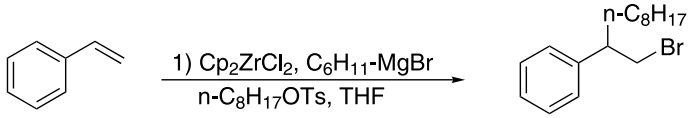
Ruthenium catalyzed an interesting regioselective alkylation of ketones using trialkylamines (Eq. (242)) [1628]. Palladium-catalyzed coupling of alkenes, boronic acids, or ketones with biphenylene afforded products derived from C–C-bond cleavage (Eq. (243)) [1629]. A synthesis of pumilotoxine C using molecular nitrogen as the nitrogen source was reported (Eq. (244)) [1630]. Cycloproparenes were reacted with a ruthenium carbene to give ruthenacyclopentenes that decompose to form *o*-xylylenes [1631]. Cyclic carbonates were derived from a domino reaction of 4-methoxycarbonyloxy-2-butyn-1-ols with phenols (Eq. (245)) [1632].

Zirconium-catalyzed addition of two electrophiles to alkenes was described (Eq. (246)) [1633]. Vanadium-catalyzed additions of propargyl alcohols (Eq. (247)) [1634] and allenic alcohols [1635] to aldehydes were developed. Iron catalyzed the formation of aldol products from reaction of allylic alcohols with aldehydes under irradiation (Eq. (248)) [1636], and related reactions using rhodium or ruthenium were developed [1637].



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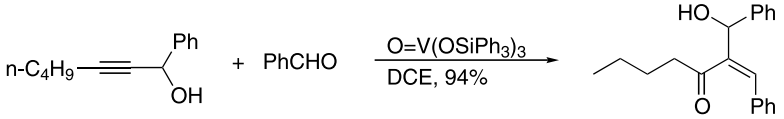

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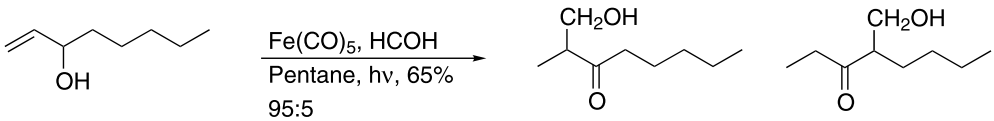

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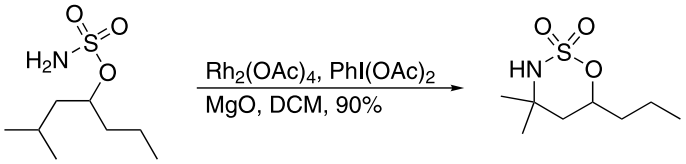

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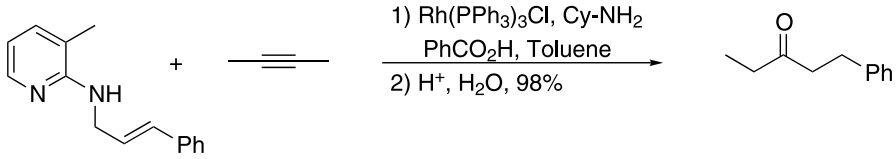

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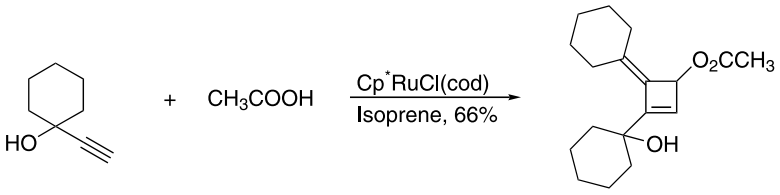
A titanium-mediated coupling of 1,4-diynes with allylic electrophiles to give substituted cyclopentanes was described [1638]. An oxidative insertion of an amine into a carbon–hydrogen bond using rhodium catalysts and $PhI(OAc)_2$ was developed (Eq. (249)) [1639]. Cleavage of an alkyne via hydroiminoacylation in the presence of rhodium was described (Eq. (250)) [1640].

Ruthenium catalyzed a dimerization of propagylic alcohols to cyclobutenes (Eq. (251)) [1641]. Reaction of alkynes with propagylic carbonates mediated by titanium gave, after electrophilic quenching, cyclobutene derivatives (Eq. (252)) [1642]. Nickel catalyzed a three component coupling of diphenylzinc with 1,3-dienes and an aldehyde to give unsaturated alcohols (Eq. (253)) [1643]. Iridium catalyzed a three component coupling of an aldehyde, an amine, and an alkyne to give an allylimine (Eq. (254)) [1644]. An interesting palladium-catalyzed carboacetoxylation of allene-substituted dienes was developed (Eq. (255)) [1645]. Rhodium (Eq. (256)) [1646], and nickel [1647] catalyzed hydroarylations of alkynes using arylboronic acids.



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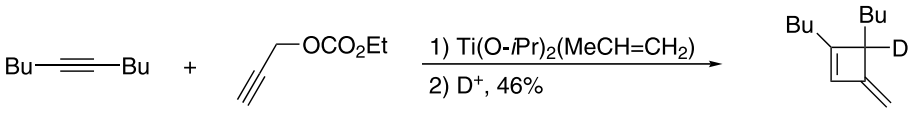

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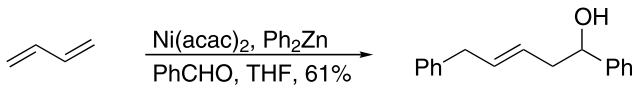

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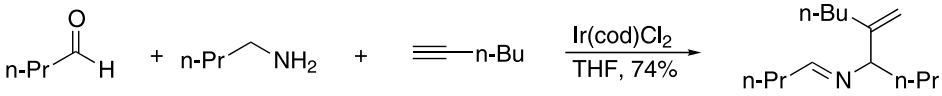

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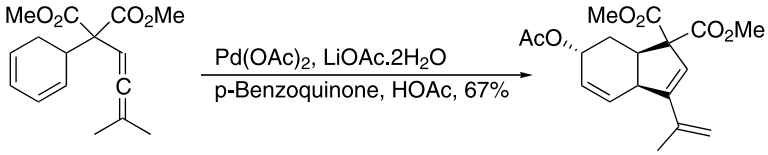

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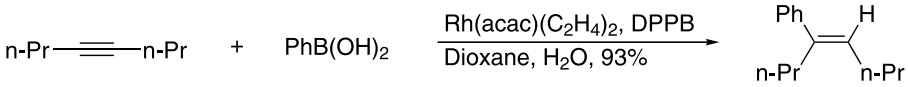

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6. Reviews

The following reviews appeared in 2001:

- Metal mediated carbometallation of alkynes and alkenes containing adjacent heteroatoms (52 references) [1648].
- Advances in the Heck chemistry of aryl bromides and chlorides (114 references) [1649].
- Synthetic applications of the dearomatization agent pentaamineosmium(II) (66 references) [1650].
- Synthesis of cyclopropane containing natural products (160 references) [1651].
- Polymer-supported catalysis in synthetic organic chemistry (81 references) [1652].
- 2-Furylphosphines as ligands for transition-metal-mediated organic synthesis (177 references) [1653].
- Non-metathesis ruthenium-catalyzed C–C bond formation (171 references) [1654].
- The *B*-alkyl Suzuki–Miyaura cross-coupling reaction: Development, mechanistic study, and applications in natural product synthesis (101 references) [1655].
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- Macrocycle formation from catalytic metal carbene transfer (28 references) [1659].
- Applications of stoichiometric transition metal complexes in organic synthesis (93 references) [1660].
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