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Transition metals in organic synthesis: highlights for the year 2002

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Abstract

A review with 1717 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 2002. Comprehensive literature coverage with extensive citations is presented. The field of transition metal chemistry continued to expand in 2002 and have a significant role in functional group transformations and organic total synthesis. 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 standard applications have not been included in this review. Reactions of unusual substrates, new reaction conditions, and new catalyst systems have been included. Also, applications in total synthesis of more complex organic molecules have been reviewed. Only the more unusual or significant reactions were presented in equation form. Some emphasis was 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, is covered in a separate review although decompositions of diazo compounds and metathesis reactions were included herein. Papers describing new chiral ligands for palladium-catalyzed allylic alkylations and allylic aminations of, primarily, 1,3-diphenyl–allylacetate and ligands for metal-catalyzed asymmetric cyclopropanations were not included. Most hydroformylations were not included apart from some reactions used in total synthesis.

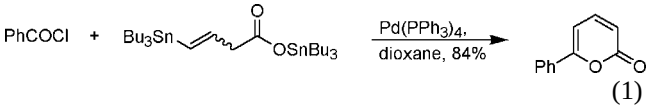
2. Carbon–carbon bond-forming reactions

2.1. Alkylations

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

Palladium-catalyzed cross-coupling reactions of organotin reagents (Stille couplings) with a large variety of electrophiles continued to be developed and extensively used in organic synthesis. Palladium nanoparticles [1], palladium encapsulated in polyurea [2], layered double hydroxide-supported palladium [3], iminophosphine–palladium complexes [4], and catalysts for couplings of unactivated aryl chlorides were developed [5,6].

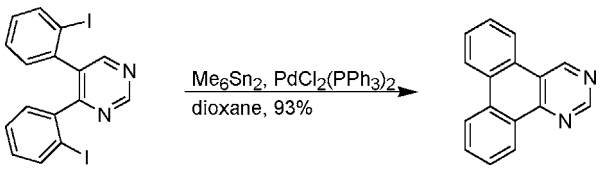
Enol nonaflates [7], 3-iodo-2-alkenoic acids [8], and 1-bromo-1-fluoro-2-arylethenes [9] were used as electrophiles in Stille couplings. Carboxylic acid chlorides were coupled with tributylstannyl-4-(tributylstannyl)but-3-enoate to give α-pyrones (Eq. (1)) [10]. 2,3-Difluoro-1,3-dienes were obtained from reaction of 1-bromo-1-fluoroalkenes with hexabutyl ditin in the presence of a palladium catalyst [11]. (*E*)-(1,2-Difluoro-1,2-ethenediyl bis[bibtributylstannane) [12], 1-stannyl-2-silyl-1-aminoalkenes [13], and 6-stannylazulene [14] were used in cross-coupling reactions.



(1)

A large variety of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and vinyltin reagents. For example, reactions of 2-bromothiazole [15], 3-iodofuro[3,2-*b*]pyridine [16], 2-chloro- and 4-bromooxazoles [17], 5-bromoisatin [18], 5-iodo- and 4-bromo-2(2*H*)-pyranone [19–21], 5-bromo-6-phenyl-3(2*H*)-pyrazinone [22], bromo-pyridinium cations [23], 2-trifloxybenzo[*b*]furans [24], 3- and 5-halopyrazoles [25], 4-iodoindole [26], 6-chloropurine [27], and 5-chloro and 5-tosyl-1,2,3-triazolo[1,5-*a*]quinazolines [28], or derivatives thereof, were reported. An iodopyridine derivative was coupled with a 2-stannylpyridine in a synthesis of orelline [29].

Regioselective couplings in the 2-position of 2,4-dibromothiazole [30], 3-position of 3,5-dibromo-2-pyrone [31], the 3-position of 3,5-dichloropryridazin-2(1*H*)-one [32], and 4-position of 3,4-dibromo-2(5*H*)furanone [33] were observed. The latter substrate was used in a synthesis of rubrolide M and analogs [34]. Intermolecular formation of an aromatic stannane followed by intramolecular coupling with an aryl halides was used to prepare phenanthro[9,10-*d*]heterocyclic compounds (Eq. (2)) [35]. Benzo[4,5]furopyridines were prepared by related intramolecular coupling of diodides in the presence of hexamethylditin (Eq. (3)) [36].



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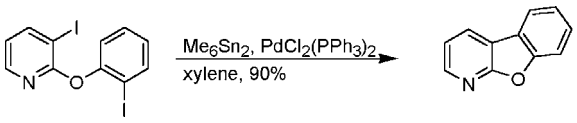
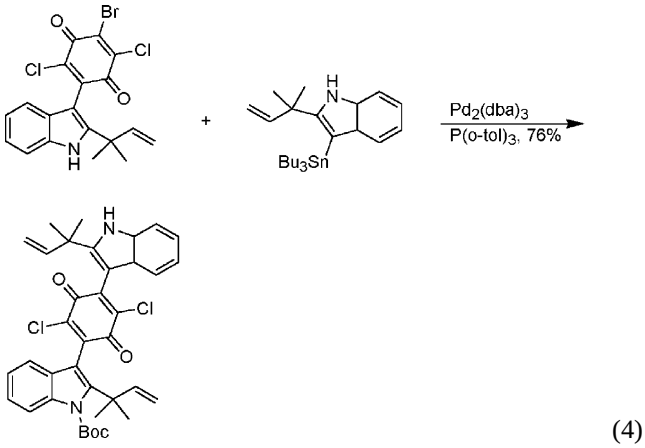


(3)

A variety of heterocyclic stannanes such as 5-stannylthiazol [37,38], 2- and 4-stannylpyridines [39–43], 2-stannyl-5-bromopyridine [44], 2-stannylfurans [45–47], 2,5-bisstannylthiophene [48], 3,4-bisstannyl-2(5*H*)-furanones [49], stannylthiophenes [50], 4-stannyl-1-benzyloxypyrazole [51], 5-stannylpyrrolo[1,2-*c*]pyrimidin-1-(2*H*)-one [52], 2-stannylpurine [53], and 3-stannyl-5-bromo-2-pyrone [54], or derivatives thereof, were used in Stille-type couplings with a variety of aromatic and heteroaromatic halides and triflates. Palladium-catalyzed coupling of 3-tributylstannylpyridine with an acid chloride was used in a synthesis of nicotine analogs [55].

The intermolecular Stille reaction continued to be extensively used in organic total synthesis. Synthetic targets include bafilomycin V_1 [56], 3′-substituted stavudine analogs [57], β-carotene and (3*R*,3*R*′)-zeaxanthin [58], momilactone A [59], 9-*cis*-retinoid derivatives [60], epothilones and derivatives [61–63], rhizoxin D [64], demethylasterriquinones A1 and B1 (Eq. (4)) [65], eupomatilones (Eq. (5)) [66], a subunit of ET 743 [67], (+)-crocacin D [68], the core of apicularen A [69], the 18-epi-tricyclic core of garsubellin A (Eq. (6)) [70], AB-carbon backbone of halichomycin (Eq. (7)) [71], himbacin derivatives [72], 4,4a-didehydrohimbacine and 4,4a-didehydrohimandravine (Eq. (8)) [73], ring-system of guanacastepenes (Eq. (9)) [74], BMS-337197 [75], toward SNF4435 C and SNF4435 D [76], madindoline A and B [77], gambierol [78,79], 7-substituted farnesyl diphosphate analogs [80], luminacin D [81], lobatamide analogs [82], polycyclic ring-systems [83], cytostatin [84], rubiginones A_2 and C_2 [85], polycephalin C [86], fostriecin [87,88], histrionicotoxins [89], (+)-puraquinonic acid [90], 9,12-anhydroerythronolide aglycone [91], analogs of calcitrol [92], cystothiazole A and C [93], (−)-macrolactin A [94], (−)-sanglifehrin A (Eq. (10)) [95], lobatamide C [96], manzamine A [97], quadrigemine C and psycholeine [98], (+)- and (−)-furocaulerpin [99], azido analogs of medermycin [100], oxazolomycin antibiotics [101], WS 75624 A (Eq. (11)) [102], callipeltoside A [103], (−)-strychnine [104], α- and β-zearalenol [105], AK-toxins [106], (−)-pregaliellalactone [107], (20*R*)-homocamptothecin [108], dermostatin A [109], variolin B [110], 1,2-dihydro-4(3*H*)-carbazolones [111], garsubellin A [112], monocillin I and radicicol [113], clusiparalicoline [114], apicularen A [115], and oxazolomycin [116]. A diastereomer of amphidinolide A was prepared using a combination of inter- and intramolecular coupling (Eq. (12)) [117]. Cross-coupling of a 1,1-dibromo-1-alkene with concurrent elimination to give a conjugated enyne was used in the synthesis of callipeltoside A (Eq. (13)) [118]. Intramolecular Stille-type macrocyclization was used in synthesis of rhizoxin D (Eq. (14)) [119] and (−)-lasonolide A (Eq. (15)) [120].



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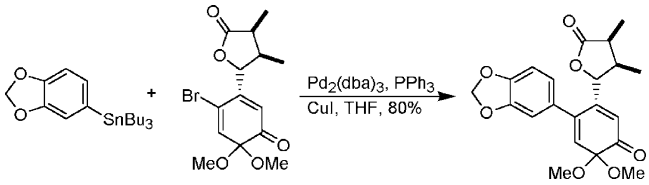

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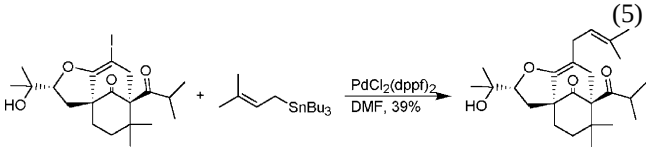

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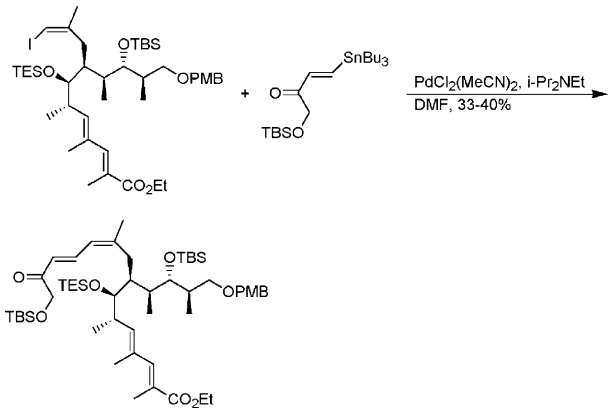

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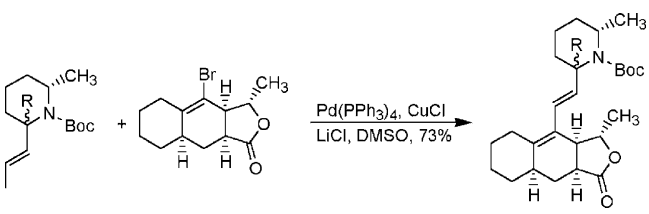

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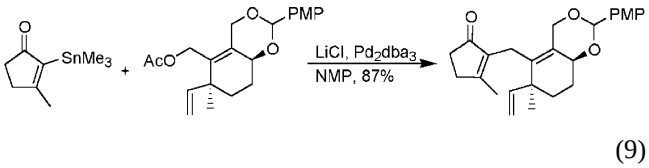

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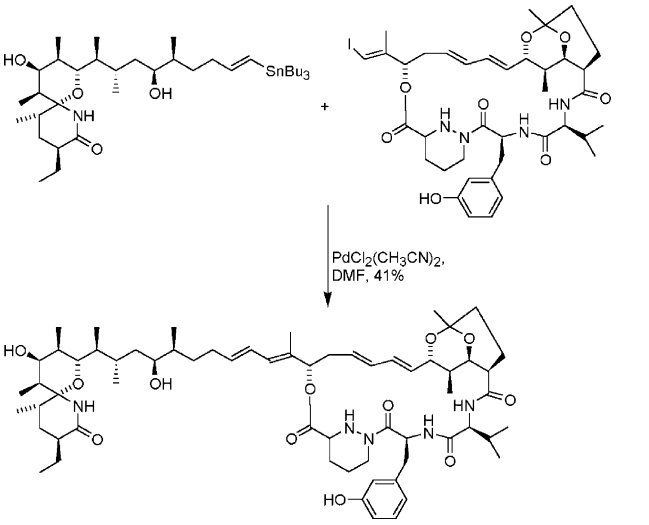

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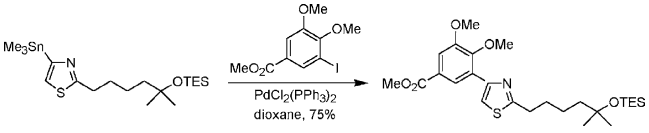

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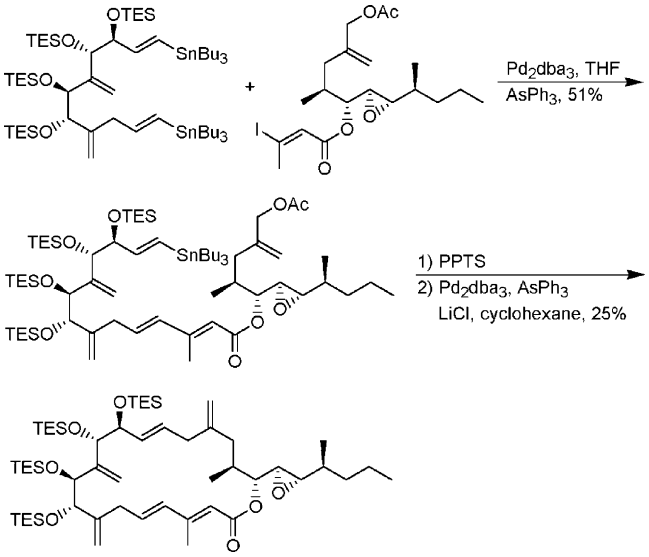

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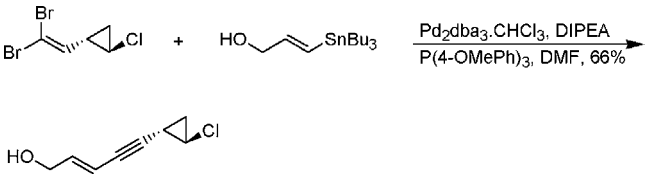

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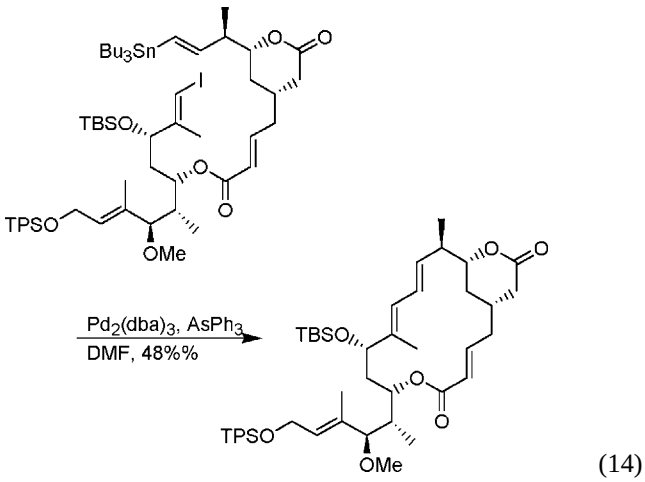

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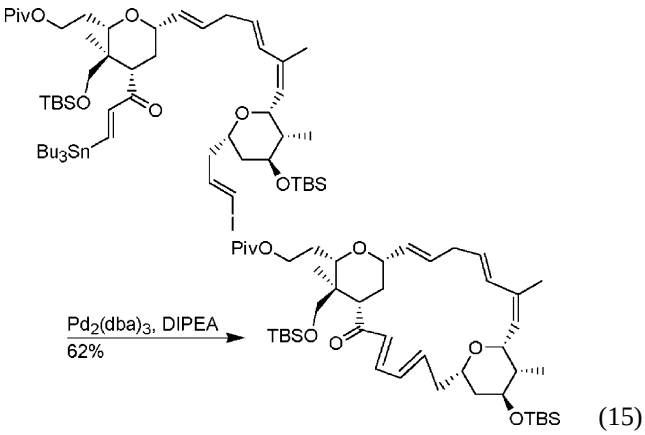
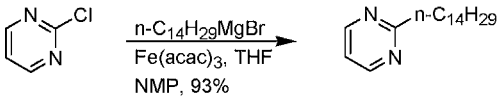


(15)

Nickel and palladium complex-catalyzed cross-couplings of Grignard reagents with halides and triflates were used in organic synthesis [39,121] of cycloaraneosene and ophiobolin [122], (−)-laulimalide [123], and strobilurin analogs [124].

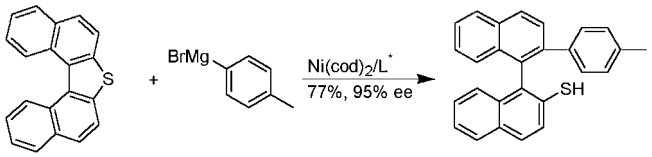
Alkyl chlorides [125], arylchlorides [126], 2-bromobenzenamines [127], and halo-pyridines, -quinolines, -pyrimidines, and -pyrazine [128] were coupled with Grignard reagents in the presence of a palladium catalyst. Nickel-catalyzed reactions of aryl chlorides [129], of aromatic and heteroaromatic fluorides [130], alkyl halides, and tosylates with Grignard reagents were described. In the latter case, best results were obtained in the presence of butadienes [131]. Iron-catalyzed couplings of alkyl and aryl Grignards with a large variety of aromatic and heteroaromatic triflates, tosylates, and chlorides were reported (Eq. (16)) [132–134]. Cobalt catalyzed the coupling of alkyl halides with allylic Grignard reagents [135].



(16)

New ligands were described for asymmetric coupling of benzylic Grignard reagents with vinyl bromides using nickel as the catalyst [136]. Nickel catalyzed an asymmetric cross-coupling of dinaphthothiophene with Grignard reagents to give axially chiral 1,1′-binaphthyls (Eq. (17))

[137]. A nickel-catalyzed ring opening coupling of methyl magnesium bromide with a 2,3-dihydrofurane was used in a synthesis of (−)-1(10),5-germacradien-4-ol (Eq. (18)) [138] and of 3-methylalkan-2-ols [139].



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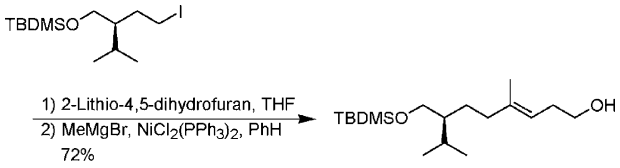
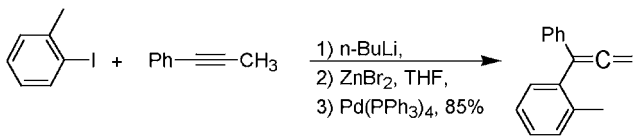


(18)

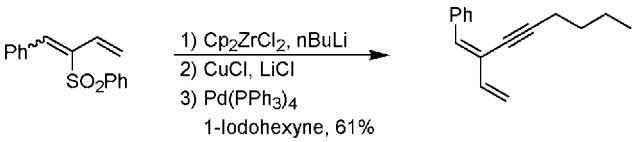
Palladium- or nickel-catalyzed couplings of halides and triflates with organozinc reagents (Negishi couplings) continued to grow [140–145]. Catalysts for the coupling of aryl chlorides with arylzinc reagents were described [146]. Primary and secondary alkylzinc chlorides were coupled, using a nickel catalyst, with primary alkyl halides [147]. Alkylzinc reagents were coupled with 2-bromopyridines [148]. Reactions of intermediately formed allenic/propargylic zinc reagents with organic halides in the presence of a palladium catalyst can be tuned to give allene or alkyne products (Eq. (19)) [149].



(19)

3-Iodo-2-alkenoic acids [8], aromatic nonaflates [20], acetyl chloride [150], and vinyl phophonates [151] were used in Negishi-type reactions. Methyl 2,3-dibromopropenoate was selectively coupled in the 3-position using an alkynyl zinc reagent [20]. Cyclic ketene acetal triflates were coupled with alkylzinc reagents and used in synthesis of polycyclic ethers [152].

α-Thioether substituted vinyl zinc reagents were coupled with α-bromovinyl ethers [153]. An interesting synthesis of zirconated conjugated dienes from nonconjugated dienylethers followed by palladium-catalyzed coupling was developed (Eq. (20)) [154]. Zinc reagents derived from ethyl 2-bromo-propenoate [155] and in situ prepared 1,2,2-trifluoroethenylzinc [156] were utilized in cross-coupling reactions. An interesting palladium-catalyzed coupling of thioethers with alkylzinc reagents was described (Eq. (21)) [157]. Nickel catalyzed the cross-coupling of alkyl zinc reagents with thiolesters to give unsymmetrical ketones [158].



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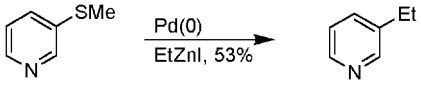
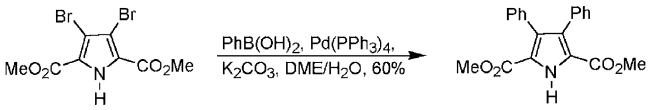


(21)

Heterocyclic substrates also participated in Negishi couplings [159,160]. For example, 2-bromothiazole [15], 2-chloro- and 4-bromooxazoles [17], 2-chlorinated pyridines [161,162], 2- and 5-bromopyrimidine and 3- and 5-halopyrazoles [25,163,164], iodopyrazines [165], 3,4-dibromopyrrole (Eq. (21)) [166], 5-chloro-1,2,3-triazolo[1,5-*a*]quinazolines [28], or derivatives thereof, were used. An iodopyridine derivative was coupled with a pyridylzinc reagent in the synthesis of orelline (Eq. (22)) [167]. 3-Bromobenzo[*b*]furan was coupled with methylzinc chloride in a synthesis of eupomatenoids [168]. Exclusive coupling in the 2-position of 2,4-dibromothiazole [30], the 6-position of 2,6-dichloropyrimidine, the 2-position of 2,3-dichloropyridine, and the 4-position of 4,7-dichloroquinoline [169] was observed.



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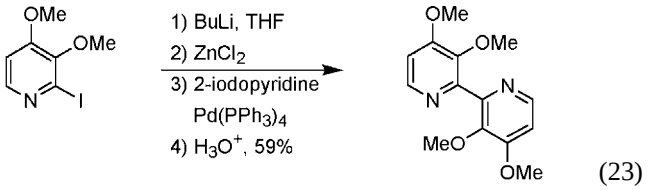
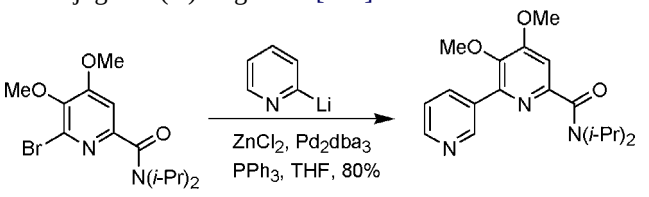


(23)

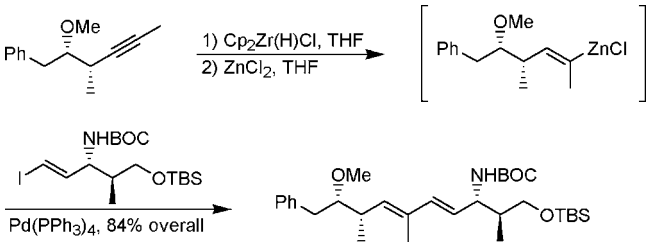
Heterocyclic zinc reagents were used including, pyrazolyl [170,171], 4-indolyl [172], 3-pyridyl [173], 2-pyridyl in the synthesis of caerulomycin C (Eq. (24)) [174], 7-indolyl with 4-iodoindole in the synthesis of diazonamide fragment [175], and 2(2*H*)-pyranone-5-yl [19,20].

1,1-Difluoro-1-alkenyl-2-zinc derivatives derived from transmetallation from zirconium were used in coupling reactions using palladium [176]. Triorganozincates were used in palladium-catalyzed cross-coupling reactions with chloropyridines and aryl bromides [177]. Hydrozirconation of a terminal alkyne followed by transmetallation to zinc and coupling with vinyl iodides was used in the synthesis of conjugated (*E*)-oligoenes [178].



(24)

Synthetic targets using the Negishi coupling include caerulomycin A and B [179], pumilotoxins A and B (Eq. (25)) [180,181], coenzyme Q_{10} (Eq. (26)) [182], C1–C18 segment of lophotoxin and pukalide [183], sphingofungin F [184], amphidinolide T4 (Eq. (27)) [185], strobilurin analogs [124], ecteinascidin 743 [186], gambierol (Eq. (28)) [187], (+)-α-onocerin [188], (−)-longithorone A [189], *trans*-epothilone A [190], oleandolide [191], amino acid derivatives [192], muriocin [193], (−)-pregaliellalactone [107], and superstolide [194]. Regioselective hydrozirconation of an internal alkyne followed by transmetallation to zinc and palladium-catalyzed coupling with a vinyl iodide was used in the synthesis of (−)-motuporin (Eq. (29)) [195].



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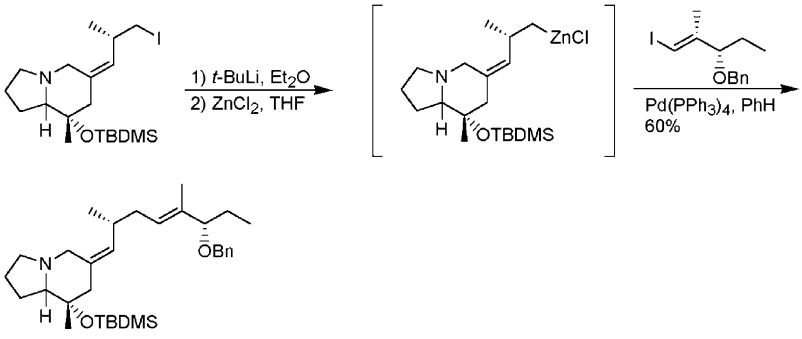

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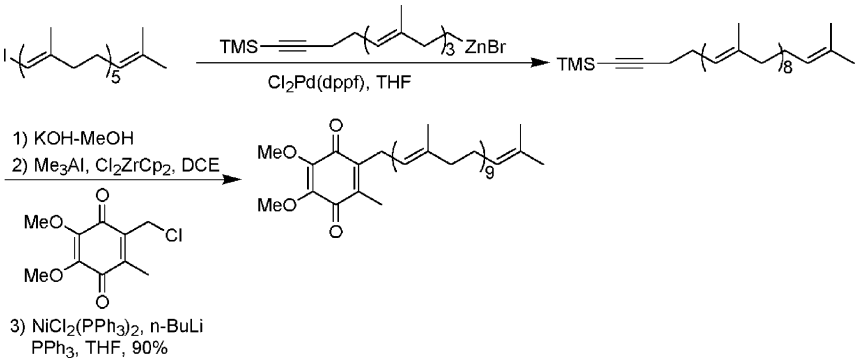

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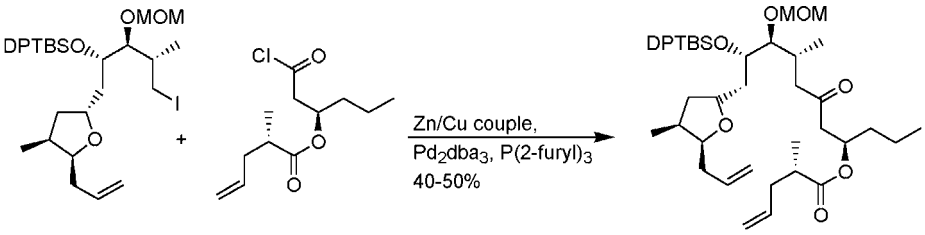

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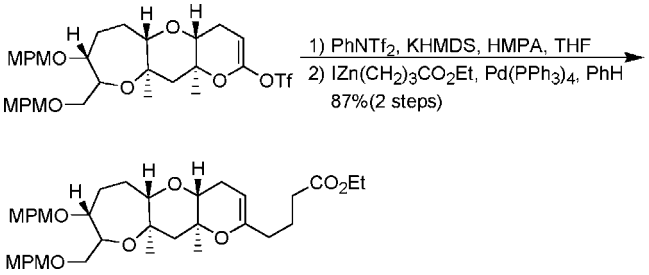


(28)

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. A number of new ligands and highly active catalyst systems were developed [196]. Oxime-derived palladacycles [197,198], palladium-

2-aryl-2-oxazoline complexes [199], recyclable hollow palladium spheres [200], arylpalladium halide phosphine complex [201], palladium on carbon [202], palladium on hydroxyapatite [203], palladium nanoparticles [1], macrocyclic triene palladium complex [204], layered double hydroxide supported palladium [3], palladium–carbene complex [205], fiber-bound palladium catalyst [206], sepiolite clay-supported palladium catalyst [207], palladium encapsulated in polyurea [2,208], and recyclable hollow palladium spheres [209] were reported.

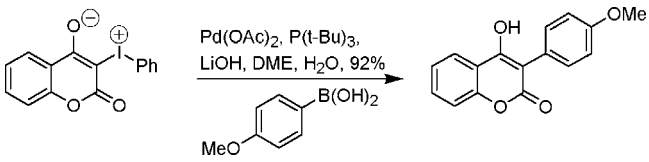
Ligands including a phosphine–pyrazole [210], *N*-alkyl-*N*′-arylimidazolium [211], iminophosphine [212], phosphorous functionalized aminopyridines [213], perfluoroalkylphenylphosphines [214], pro-azaphosphatranes [215], ruthenium-containing phosphine [216], tetraphosphine ligand [217], and air-stable ferrocenyl dialkylphosphines [218], were described. The effectiveness of tris(*t*-butyl)phosphine and related ligands was attributed to their propensity to form monophosphine–palladium complexes [219].

Couplings of haloaromatic compounds with organoboronic acids in aqueous solutions catalyzed by palladium were reported [220–222]. Catalysts for the coupling of vinyl and aryl chlorides were described [146,223–225]. Microwave assisted reactions using palladium catalysts in water [226,227] and ionic liquids were reported [228].

Polymer-bound Suzuki reactions have attracted a fair amount of attention. A rate acceleration was observed for a polymer-bound aryl iodide in an ionic liquid [229]. Polymer-bound boronic esters were used in coupling reactions [230]. Polymer-bound 4-bromotryptamine, aryl and heteroaryl halides and triflates were used together with aromatic and heteroaromatic boronic acids [53,231–235]. Polymer-bound catalysts [236] were employed using water [237] or supercritical carbon dioxide [238] as the solvent.

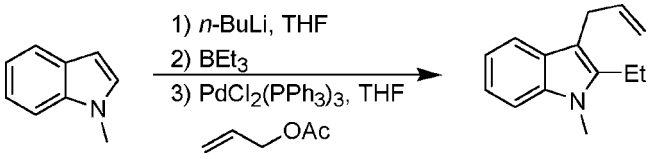
Anhydrides were coupled with aromatic boronic acids using a palladium catalyst to give ketones [239]. A related coupling of carboxylic acids in the presence of dimethyl carbonate or pivalic anhydride was described [240–242]. An anhydride is probably formed in situ. Aroyl chlorides were coupled with sodium tetraphenylborate using palladium-doped KF/Al_2O_3 [243].

Lactam-derived vinyl triflates were coupled with α-alkoxyboronates [244]. Suzuki coupling of 2-bromo-1,*n*-dienes produced mainly cyclic products derived from an oxidative-addition alkene insertion-transmetallation sequence [245]. (*Z*)-2-Bromovinyl carboxylates [246], α-bromoalkenyl phosphonates [247], 2-iodocarborane [248], haloaroylmethylidenetriphenylphosphoranes [249], phenyliodonium zwitterions (Eq. (30)) [250], and 2-fluoro-1-iodo-1-alkenes [251] were coupled with boronic esters and acids. Halomethylene-substituted sugars were coupled with aromatic boronic acids [252]. Sequential allylic nucleophilic substitution followed by Suzuki coupling using 2,3-dibromo-1-propene was reported [253].



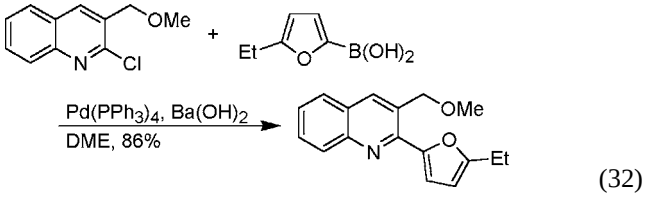
(30)

Suzuki couplings of 2,4,6-trivinylcyclotriboroxane [254], cyclopropyl boronic acid [255], 1,1-difluoro-1-alkenyl-2-boronic acid derivatives [176], 1-alkoxy-1,3-dienyl- and 1-alkoxy-2-phenylalkenylboronic esters [256], and a fluorous tagged boronic ester [257] were described. The boron-bearing carbon of geminal dimethylphenylsilylborylated alkenes was coupled with aryl-, vinyl-, alkynyl-, and allyl-halides [258]. Palladium catalyzed the coupling of air and moisture stable potassium alkynyl- [259,260], alkenyl- [261], and aryl-trifluoroborates [262] with aryl and heteroaryl halides and triflates. Intramolecular alkyl migration and allylation of indolylborates was used to prepare indole and carbazole derivatives (Eq. (31)) [263].



(31)

A number of total syntheses wherein aryl or vinyl boronic acids or esters were coupled with an aryl-, heteroaryl-, vinyl-halides or -triflate were reported. Synthetic targets included, nothapodytine B (Eq. (32)) [264], (−)-spinosyn A [265], orevactaene [266], amaryllidaceae alkaloids (Eq. (33)) [267], dragmacidin D (Eq. (34)) [268,269], (+)-fostriecin (Eq. (35)) [270], F–N ring fragment of gymnocin A [271], 4,4a-didehydrohimbacine and 4,4a-didehydrohimandravine [73], (−)-ebelactone A [272], pyridomacrolidin [273], ciguatoxins [274], the spinosyn A tricyclic nucleus [275], (−)-sibirine [276], the protesome inhibitors TMC-95A and TMC-95B [277,278], (−)-FR182877 (Eq. (36)) [279], (+)-epoxyquinols A and B [280], C2-aryl pyrrolobenzodiazepines [281], ficuseptine [282], (−)-bafilomycin A [283], E2040 [284], khafrefungin [285], vicenistatin [286], halitulin [287], (+)-curacin A [288], amino acid derivatives [289–292], COX-2 inhibitor [293], monocillin I and radicicol [113], C292 and hypothemycin [294,295], and clusiparalicoline [114]. Asymmetric Suzuki couplings were used to prepare axially chiral biaryls [296]. Intramolecular couplings were used in synthesis toward xestocyclamine A (Eq. (37)) [297], salicylhalamide A core (Eq. (38)) [298], epothilone analogs [299], and strychnine (Eq. (39)) [300].



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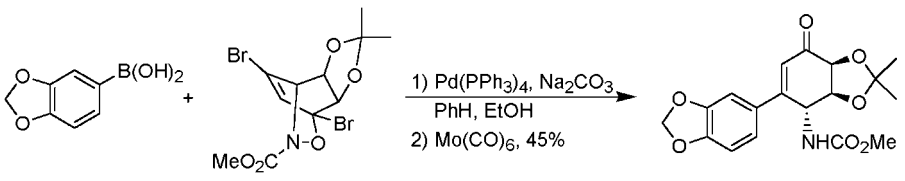

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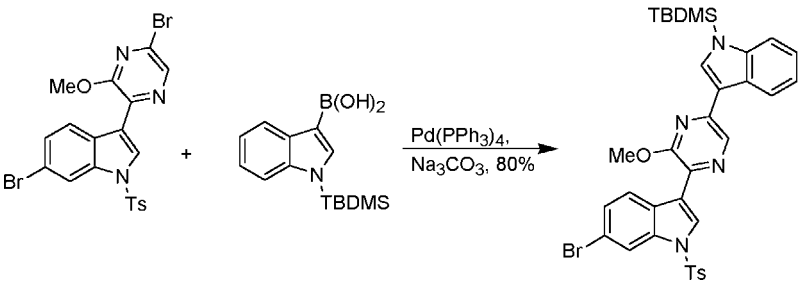

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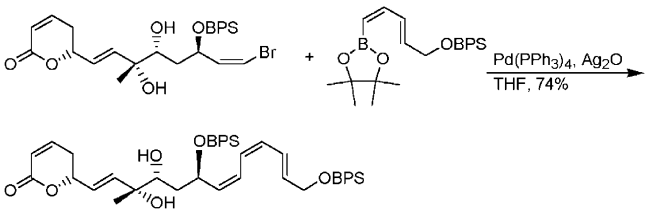

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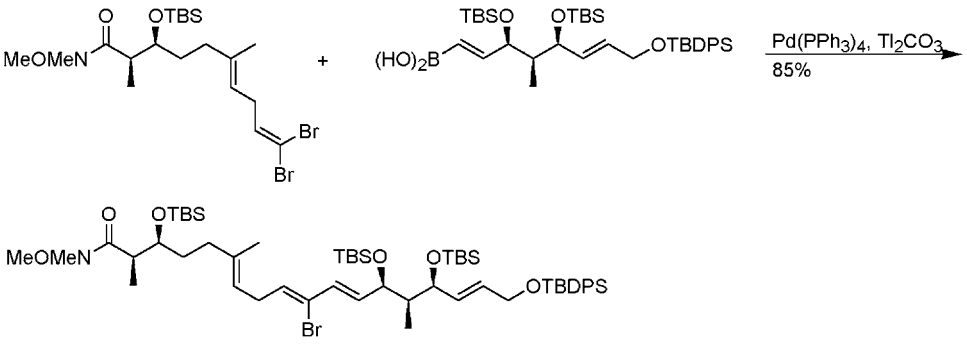

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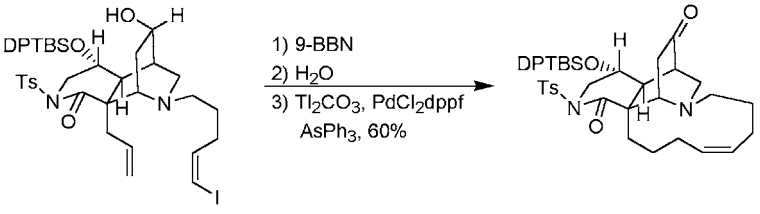

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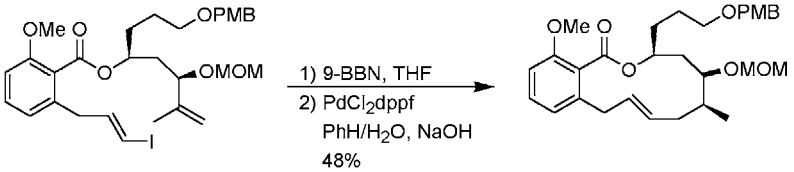

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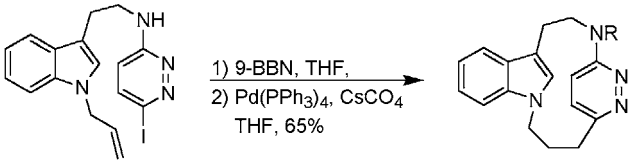
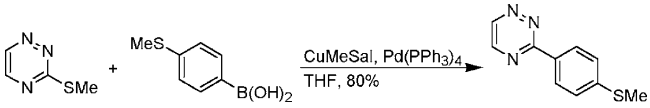


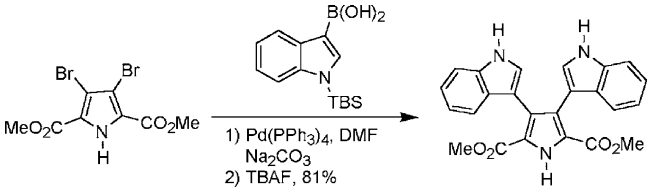
(39)

A large variety of functionalized heterocyclic halides and triflates were employed in the Suzuki reaction. For example, 5-bromo-2-iodopyrimidine [301,302], 2-bromo-6-iodopyridine [303], 2-chloro-3-nitropyridine [304], 2-bromothiazole [15], 3-bromofuran-2(5*H*)-one [305], 2′-deoxyguanosine-O^6-sulfonates [306], iodofuropyridines [16,307], 2-chloro- and 4-bromooxazoles [17], 3,4-dibromo- or 3,4-dichloro-5-hydroxy-5*H*-furan-2-ones [308], 4-trifloxy-2*H*-chromenes [309], 2,9-dihalo-1,10-phenanthrolines [310], 8-chloro- and 8-bromotheophylline [311], 5-iodo-2′-deoxyuridine and 8-bromo-2′-deoxyguanosine [312], 6-chloropurine [27], 3-iodopyrazole [171], 4-bromopyrazole in a synthesis of withasomnine [313], 4-iodoimidazole, 5-iodouracil, 3-chloropyridazine, and 6-chloropurine [314], 3-iodo-6-arylpyridazines [315], 5-bromo-, 4-trifloxy-, and 5-trifloxy-substituted 3(2*H*)-pyrazinones [22,316], bromobenzo[*b*]thiophenes [317,318], 4-bromo-3-chloro-3-phenylpyridazine [319], 5-chloropyridazin-3(2*H*)-one, 6-chlorouracil, and 2-chloropyrazine [320], 2-trifloxy- and 3-bromo-benzo[*b*]furans [24,321], 1-bromo- and 1-chloroisoquinoline [322], 6-chloropurine [323], 3-bromo-pyrroline nitroxides [324], iodopyrazines [165], 4-tosyloxycoumarins [325], 5-iodo 4-bromo-2(2*H*)-pyranones [21,326], and 3-chloro-1,2,4-benzotriazole [327], or derivatives thereof, were all used in Suzuki-type cross-couplings. 4-Chloro-6-iodoquinoline was successively coupled with aromatic boronic acids [328]. 3-Thiomethyltriazine was coupled with organoboron reagents using a palladium catalyst (Eq. (40)) [329]. Heteroaromatic thioethers [330] and aromatic and heteroaromatic amidines [331] were coupled with boronic acids in the presence of a palladium catalyst and a copper carboxylate. 3,4-Dibromofuran-2(5*H*)-ones were selectively coupled in the 4-position using boronic acids [332].



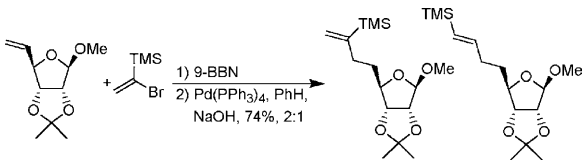
(40)

A variety of heteroaryl boronic acids were used such as benzothiophene boronic acids [333], 2-pyrrolyl [334], 2,5-dihydro-3-pyrolinyl and 3-pyrolinyl [335], 3-bromo- and 3-chloro-isoxazolyl [336], halopyridin-3- and -4-yl [337–339], and 3-indolyl (Eq. (41)) [166] boronic acids or derivatives. Pyridinylboronic acids derivatives were coupled with a large selection of heteroaromatic halides [340].



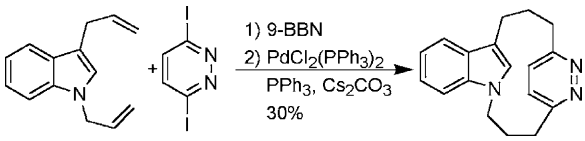
(41)

Coupling reactions of simple alkyl chlorides [341] and tosylates [342] with alkylboron reagents were developed. Aryl and vinyl halides and triflates were coupled with primary alkylboronic acids using palladium [343–345]. Room temperature couplings of alkyl bromides with aryl, alkyl, or vinyl boronic acids were described [346]. Coupling of alkyl boronic esters with 1-bromo-1-trimethylsilylethene produced two isomeric products (Eq. (42)) [347].



(42)

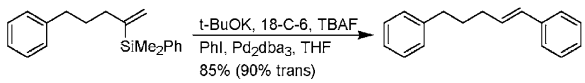
Cross-coupling of vinyl halides with alkyl boronic acids or esters was used in synthesis of callystatin A [348], prostaglandins [349], C26-(1,3-dioxolanyl)-12,13-desoxyepothilone B [350], wortmannin [351], sphingofungin E [352], the carbon skeleton of vannusal A [353], *trans*-epothilone A [190], and sphinganines [354]. Hydroboration of *N*,3-diallylindole followed by coupling with 3,6-diiodopyrazine was reported (Eq. (43)) [355]. Cross-coupling of alkylboronic acids with enolphosphates were used in syntheses of (−)-gambierol [79] and the ABCD ring fragment of ciguatoxins [356].



(43)

Copper(I) oxide co-catalyzed palladium-catalyzed couplings of aromatic boronic acids with ethyl bromacetate [357]. Copper nanoclusters catalyzed the cross-coupling of aryl boronic acids with aryl halides [358]. Platinum catalyzed the coupling of aromatic bromides and iodides with aromatic and vinylic boronic acids [359,360]. Nickel catalyzed the cross-coupling of aromatic boronic acids with aromatic halides [361]. Nickel-catalyzed Suzuki-type coupling of 1-acetoxy-4-hydroxy-cyclo-2-pentenone [362], and of organic borates with aryl mesylates [363].

A number of silicon reagents were developed as alternatives to organotin and boron compounds in palladium-catalyzed cross-coupling reactions with organic halides and triflates. Palladium catalyzed cross-couplings of alkenylsilanols [364,365], of arylhalosilanes [366], alkenyldimethyl(2-thienyl)silanes [367], and alkylidenesilacyclopentanes [368] with vinyl and aryltriflates, iodides, and nonaflates. Exclusive cine substitution was observed for dimethylphenylsilyl-substituted alkenes (Eq. (44)) [369]. Palladium-catalyzed sequential coupling of tributyl (2-pyridyl)silylmethylstannane to give diarylmethanes (Eq. (45)) [370]. Intramolecular couplings of silyloxycyclo-hexenes and -heptenes with a pendant vinyl iodide were reported (Eq. (46)) [371]. This type of reaction was used in a synthesis of (+)-brasilenyne [372].



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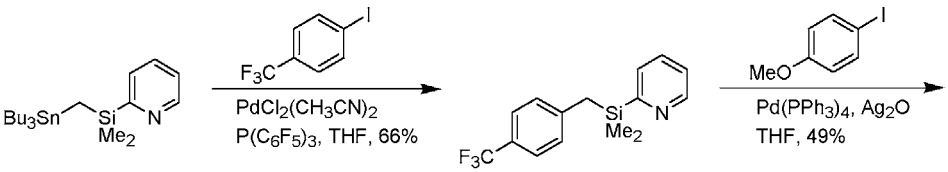
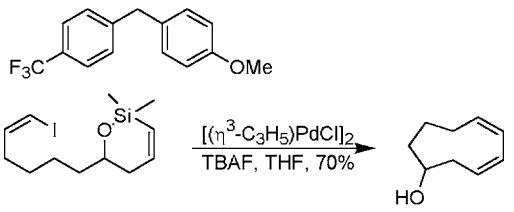
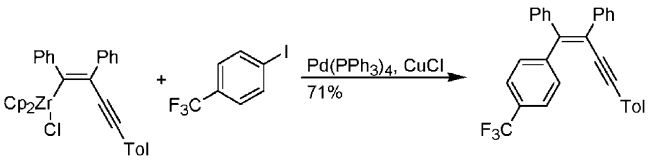




(46)

Some more unusual cross-coupling reactions employing organometallic reagents were described. Palladium–copper catalyzed the coupling of vinylzirconium compounds derived from alkynylzirconation of alkynes (Eq. (47)) [373]. Acylzirconocenes were coupled with diaryliodonium salts to give ketones [374]. A zirconium-catalyzed diastereoselective intramolecular reductive alkylation of alkenes having a pendant tosylate was developed (Eq. (48)) [375]. Palladium-catalyzed coupling reactions of allyl indium [376], aryl indium [377], vinylindium [378], and methyl indium [379] reagents with a variety of aryl and vinyl halides and triflates. Palladium catalyzed the coupling of propargylic indium species with a variety of halides and triflates to give allenes (Eq. (49)) [380]. A methylindium reagent was used in a palladium-catalyzed coupling of chromium tricarbonyl complexes with aryl chlorides [381].



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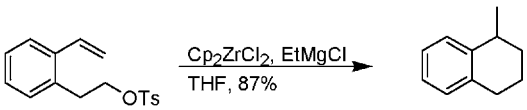

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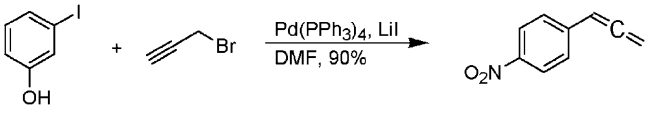


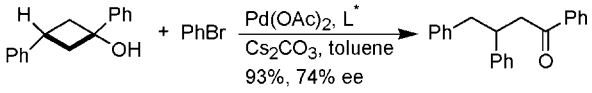
(49)

Palladium also-catalyzed cross-coupling reactions of vinylcopper reagents derived from copper promoted 1,4-carbon to oxygen silyl migration [382], a vinylcopper [383], of a variety of organo-germane reagents [384,385], vinylgallium reagents [386], and dialkoxybismuth reagents [387]. Palladium-catalyzed coupling of chloromethyl substituted quinones with vinylaluminum reagents was used in the synthesis of coenzyme Q [182]. Related reactions of benzylic halides with vinylaluminum reagents derived from terminal alkynes and trimethylaluminum in the presence of a zirconium catalyst were described [388].

(45)

Vinylaluminum intermediates formed from regioselective hydroalumination of propynols were coupled with aryl and vinyl iodides to give 3,3-disubstituted allylic alcohols [389]. Palladium catalyzed the coupling of trialkylaluminum reagents with 5-chloro-7-nitro-8-trifloxyquinoline [390] and sodium of tetraalkynylaluminates with aryl bromides [391]. Palladium and nickel catalyzed the coupling of organotitanium reagents with aryl triflates and halides [392].

Carbon nucleophiles were also employed in alkylation reactions of halides and triflates. Air-stable palladium catalysts for the alkylation of aromatic chlorides with ketone enolates were developed [224,393]. Palladium catalyzed the coupling of nitroalkanes [394], malonates, and cyanoesters [395], 1,3-cyclopentadione [396], a TBS-ketene acetal derived from *t*-butyl acetate [333], ester enolates [397], ketones [398,399], aldehydes [400], alkylnitriles [401,402], and activated methylenes [403,404] with aryl and vinyl bromide and chlorides. Palladium catalyzed an asymmetric reaction between aryl halides and triflates with tertiary cyclobutanols affording acyclic arylketones (Eq. (50)) [405,406]. Intramolecular α-arylation of 2-bromophenyl-tethered α-aminoacids was described [407]. Intramolecular palladium-catalyzed alkylations were used in the synthesis of a number of indole alkaloids (Eq. (51)) [408–410], oxindoles [411], hetisine (Eq. (52)) [412], and of physovenine [413]. Palladium- and nickel-catalyzed asymmetric enolate alkylations using chiral ligands were described [414–418]. Copper catalyzed the arylation of diethylmalonate with aryl- and heteroaryliodides [419]. Ruthenium catalyzed a regioselective α-alkylation of ketones with primary alcohols (Eq. (53)) [420]. Palladium catalyzed the reaction of 3-methoxy- and 3-cyanobenzo[*b*]thiophene with aromatic and heteroaromatic halides forming 2-substituted products [421].



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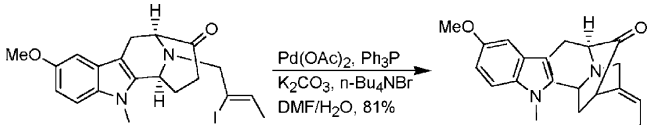

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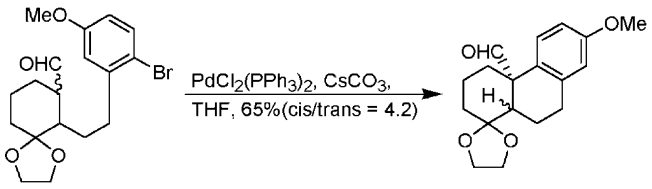

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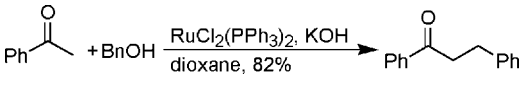


(53)

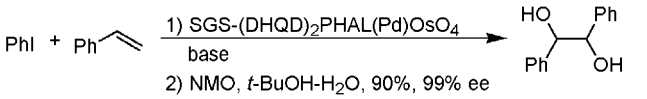
2.1.2. Alkylations of alkenes and allenes

Alkylations of alkenes and allenes refer to reactions wherein, usually, the double bond(s) of the alkene/allene remain(s) intact in the product.

The Heck reaction continued to be one of the most versatile methods for the alkylation of alkenes. Microwave-assisted Heck reactions using organic [422,423], ionic liquids [424], or water [425] as the solvent were reported. Reactions in water [426] using a polymer-bound palladium catalyst [427], and reactions in ionic liquids using a silica-supported palladium catalysts [428] were developed. A palladium–carbene catalyst was used for reactions of aromatic chlorides in ionic liquid [429]. Reactions in molten salt mixture were described [430].

A number of papers dealing with different aspects of the catalyst system and the reaction conditions appeared [223]. Several highly efficient catalyst systems were reported, a thermomorphic fluorous palladacycle [431], a palladium–imidazolium carbene for reactions of aryl diazonium salts [432], a palladium-metallated cyclophosphazene-containing polymer [433], palladium on hydroxyapatite [203], a palladium–carbene [434], bidentate pyridine ligands [435], a palladium–tetraphosphine complex [436,437], an aryl-palladium halide phosphine [201], palladium nanoparticles [1,438], a polymer-bound catalyst used in supercritical carbon dioxide [238], palladium-mordenite catalysts [439], palladium on carbon [202], layered double hydroxide-supported palladium catalyst [3], palladium complexed to a macrocyclic triene [204], and palladium encapsulated in polyurea [2] were used in Heck reactions. Palladium on carbon catalyst loading as low as 0.01 ppm gave quantitative conversion within two hours [440]. Polyethylene glycol was used as a reusable solvent [441]. Reactions of both polymer-bound aryl iodides [232,442] and polymer-bound alkenes [443] were described. A number of ligands for asymmetric Heck reactions were reported [444–447].

A new bifunctional palladium–osmium catalyst was developed for a tandem Heck reaction-asymmetric dihydroxylation of alkenes (Eq. (54)) [448]. Allylic amidines and guanidines were used as the alkene in Heck reactions [449]. Intramolecular palladium-migration after oxidative-addition to 2-halo-biaryls was observed (Eq. (55)) [450,451]. Palladium catalyzed a tandem cyclization Heck reaction of 2-(1-alkynyl)benzaldimines to give substituted isoquinolines (Eq. (56)) [452]. Heck reaction under oxygen gave a mixture of the expected Heck-type and dealkylation-addition product obtained from triethylamine [453]. A one-pot Heck-type reaction–cyclization of 2-trifloxybenzaldehydes with 2-hydroxyethyl ethenyl ether forming protected indanones was described (Eq. (57)) [454].



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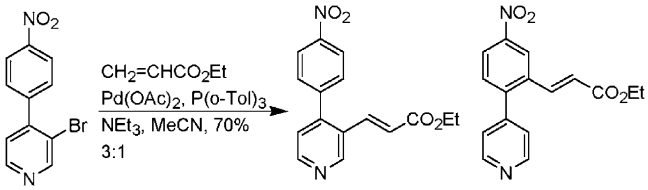

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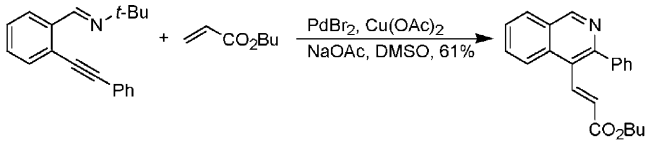

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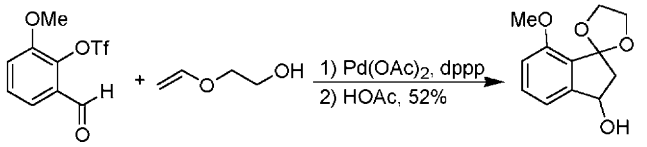
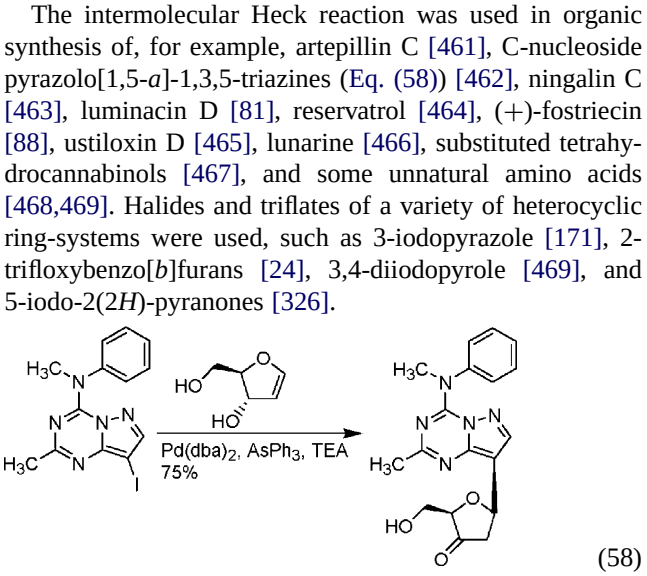


(57)

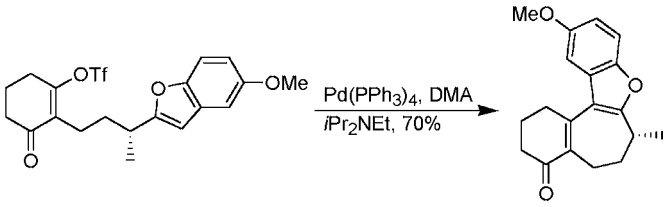
Substrates other that organic halides and triflates participated in the Heck reaction. Heck reactions of the enol acetate derived from ethyl 3-bromopyruvate [455] and aryldiazonium salts [456,457] were described. Aromatic carboxylic acids and esters were used in place of halides and triflates via an initial decarboxylation [458,459]. Heck reactions of the vinyl tosylate formed from 1,3-cyclohexadione were described [460].

The intermolecular Heck reaction was used in organic synthesis of, for example, artepillin C [461], C-nucleoside pyrazolo[1,5-*a*]-1,3,5-triazines (Eq. (58)) [462], ningalin C [463], luminacin D [81], reservatrol [464], (+)-fostriecin [88], ustiloxin D [465], lunarine [466], substituted tetrahydrocannabinols [467], and some unnatural amino acids [468,469]. Halides and triflates of a variety of heterocyclic ring-systems were used, such as 3-iodopyrazole [171], 2-trifloxybenzo[*b*]furans [24], 3,4-diiodopyrole [469], and 5-iodo-2(2*H*)-pyranones [326].



(58)

Heck-type reactions of aromatic substrates [470,471] in place of alkenes were used in a synthesis of a vancomycin fragment [472], benzo[*c*]phenanthridine alkaloids [473], and pyrimido[4,5-*b*]indoles and benzo[4,5]furo[2,3-*d*]pyrimidines [474]. Multiple arylation of thiophenes was reported [475]. Intramolecular addition to a furane was used in a synthesis of (−)-frodosin (Eq. (59)) [476]. A tandem Suzuki–Heck reaction leading to a tricyclic ring-system was described (Eq. (60)) [477].



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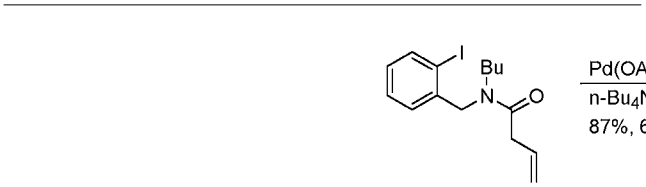
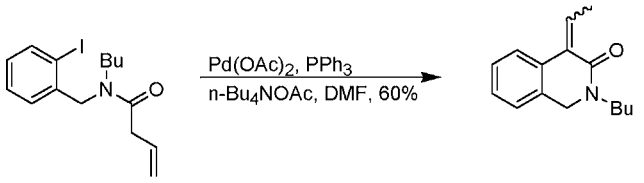


(60)

The regioselectivity for intramolecular Heck reaction could be dramatically altered by the addition of water (Eqs. (60) and (61)) [478]. A tandem intramolecular Heck reaction of 2-bromo-1,6-dienes was used to prepare bitricyclic skeletons [479]. Multiple intramolecular Heck reactions were used to prepare polycyclic compounds [480,481].

The intramolecular variation of the Heck reaction [482–488] was used as the key step toward an array of natural products, for example, (−)-strychnine [489], (−)-galanthamine [490], ecteinascidin 743 (Eq. (63)) [186], quadrigemine C and psycholeine (Eq. (64)) [98], gelsemine [491], (−)-codein and (−)-morphine [492], balanol [493], β- and γ-carbolinones [494], ajmaline and sarpagine alkaloid skeleton [495], cyathin diterpenoids [496], podophyllotoxin skeleton [497], and (−)-cephalotaxine [498]. An intramolecular Heck reaction of polymer-bound 2-haloenamines gave indoles [499].



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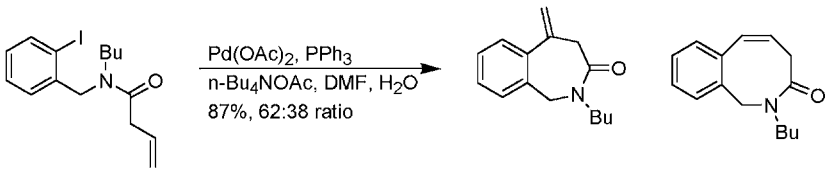

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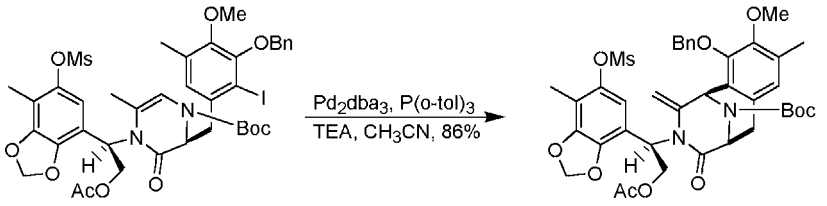

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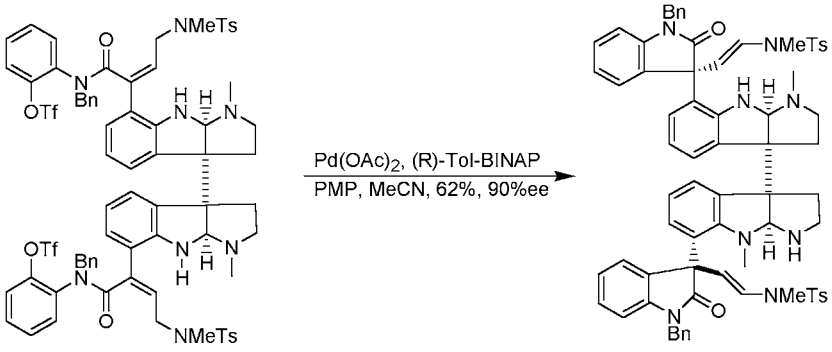

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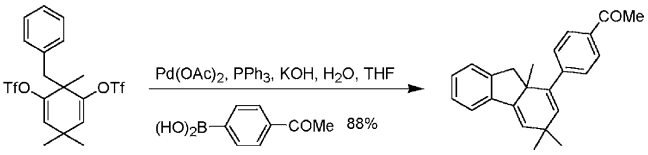
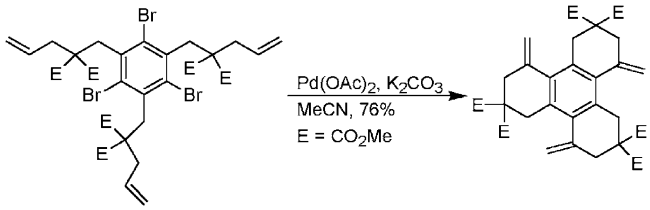


A triple Heck reaction was used to prepare triannelated benzenes (Eq. (65)) [500]. A Heck-type reaction of *N*-chloroamine having a pendant alkene was described leading to 3-chloropiperidines (Eq. (66)) [501,502]. Related reactions of pentafluorobenzoyloxime-tethered cycloheptatrienes and alkenes to give 1-azaazulenes [503] and pyrrole derivatives [504], respectively, were described. A domino Heck reaction Diels–Alder reaction was reported [505].



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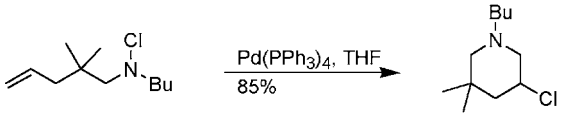
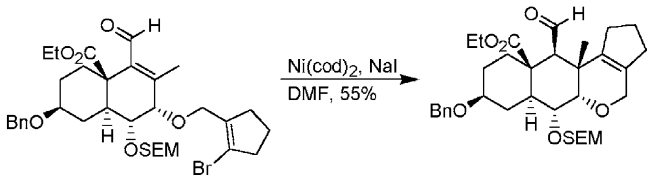


(66)

Norbornene derivatives participated in reductive Heck (or Mizoroki–Heck) reactions affording substituted norbornanes [506]. Asymmetric reductive Heck reactions of norbornene were reported [507]. A reductive Heck reaction was used in a synthesis toward zoanthenol [508]. A nickel-catalyzed electrochemical reductive Heck-type reaction was reported [509].

A nickel-catalyzed Heck reaction was used in a synthesis toward azadirachtin (Eq. (67)) [510]. A cobalt-catalyzed reaction of alkyl halides with alkenes in the presence of trimethylsilylmethylmagnesium chloride was developed (Eq. (68)) [511,512]. Rhodium catalyzed both Heck-type reactions and reductive Heck-type reactions (conjugate additions) of aryl- and alkenylstannanes [513], boranes [514], and potassium organotrifluoroborates [515].



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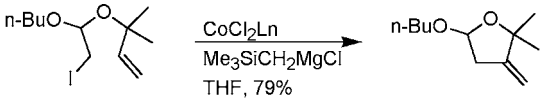
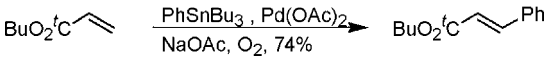


(68)

Palladium catalyzed the coupling of organotin reagents with alkenes using an oxygen or copper to reoxidize the palladium system (Eq. (69)) [516]. Carbon nucleophiles were also used to alkylate alkenes. Palladium catalyzed the cyclization of 5-allylsubstituted 2-silyloxy-1,3-cyclohexadienes to form bicyclic alkenones (Eq. (70)) [517]. A related reaction was used in a synthesis of magellanine (Eq. (71)) [518]. Polycyclic compounds were obtained from palladium-catalyzed annulations of alkenyl or aryl tethered silyl enol ethers [519]. This type of reaction was used in a synthesis toward phomoidride B [520]. The core of teleocidin B4 was prepared via an intramolecular alkylation (Eq. (72)) [521].



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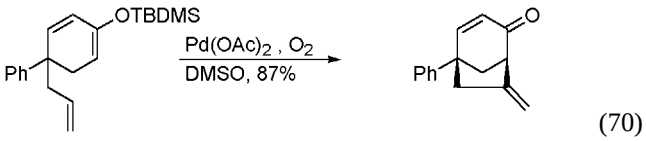

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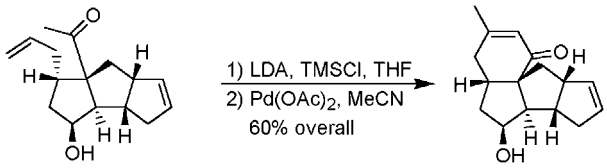

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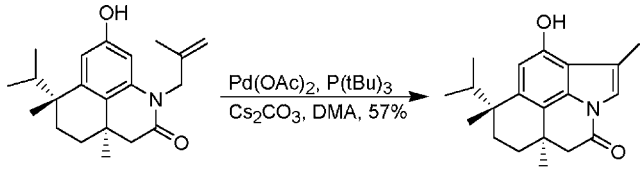
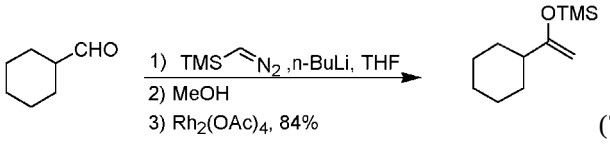


(72)

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

Reaction of trimethylsilyldiazomethane with a strong base and an aldehyde or ketone followed by protonation or methylation and diazo decomposition in the presence of a rhodium-catalyst furnished silyl enol ethers (Eq. (73)) [522]. Rhodium catalyzed the formation of enamines via decomposition of α-diazo-β-amido esters [523]. Halo-allylation or formation of furane derivatives was observed upon decomposition of cyclic diazadicarbonyl compounds using rhodium (Eqs. (74) and (75)) [524]. Ruthenium catalyzed the formation of (*Z*)-4-alkoxycarbonylmethylidene-1,3-dioxolanes from 4-alkoxy-2-diazo-3-oxoesters via an intramolecular hydride abstraction–cyclization sequence (Eq. (76)) [525,526]. Rhodium- and copper-catalyzed intramolecular amido alkoxylations of alkenes in the presence of $PhI(OAc)_2$(Eq. (77)) [527]. Rhodium catalyzed the asymmetric decomposition of [(4-nitrophenyl)sulfonylimino]phenyliodinane to give amidation products (Eq. (78)) [528].



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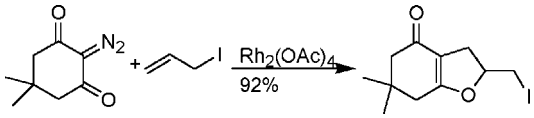

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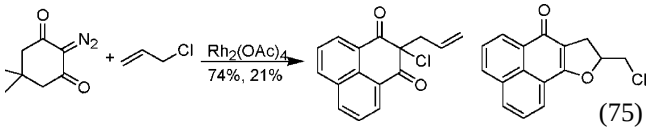

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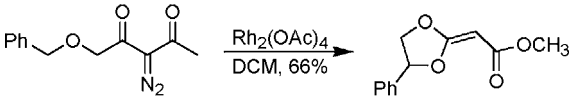

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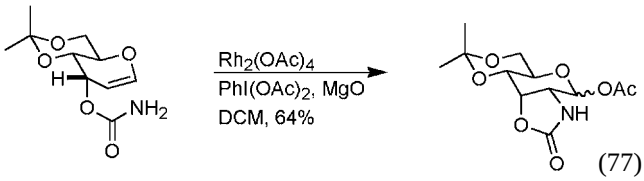

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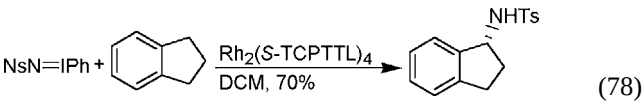
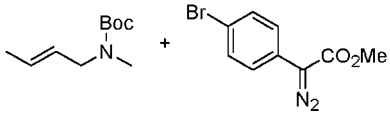
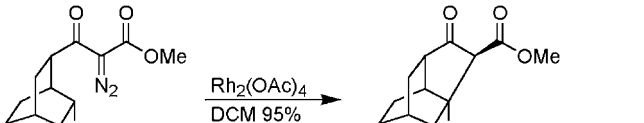


(78)

Intramolecular rhodium-catalyzed carbon–hydrogen bond insertions via decomposition of diazo compounds were used in a number of synthetic applications forming four- to six-membered rings [529,530]. For example, chiral γ-lactams [531], furofuranone derivatives [532], polycyclic ethers [533], isotwistane ring-system (Eq. (79)) [534], 7-episordinin [535], (−)-pupukeanone [536], 2-phosphonocyclopenten-2-ones [537], 3-indolyl-3′-



yloxindoles (Eq. (80)) [538], and tricyclic cycloheptatriene fused sugars (Eq. (81)) [539] were prepared. Calixarene-bound catalysts were used in cyclization reactions of diazo compounds [540]. Decomposition of α-diazophosphonates using a rhodium catalyst followed by insertion into a variety of bonds furnished indoles, benzofuranes, benzothiophenes, and isocoumarins (Eq. (82)) [541].



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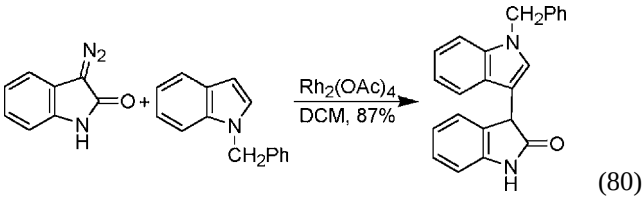

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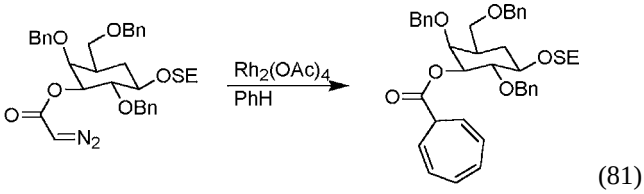

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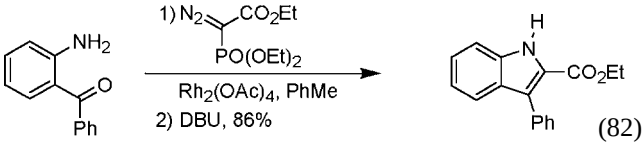
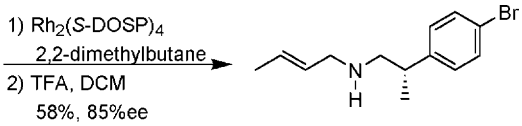


(82)

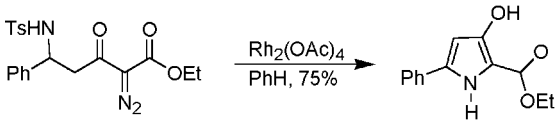
Regioselective intermolecular rhodium-catalyzed carbon–hydrogen bond insertion reactions using 3-diazo-2-oxindole with pyrroles was described [542]. (+)-Indatraline [543] and (+)-cetiedil [544] was prepared using an asymmetric intermolecular carbon–hydrogen insertion as the key step. Intermolecular copper-catalyzed carbon–hydrogen bond insertions of carbenoids in the α-position of cyclic ethers were described [545]. An intermolecular asymmetric benzylic carbon–hydrogen bond insertion via decomposition of aryldiazoactates in the presence of a chiral rhodium catalyst was reported [546]. Asymmetric rhodium-catalyzed carbon–hydrogen bond insertions affording *cis*-2-aryl-3-methoxycarbonyl-2,3-dihydrobenzofurans [547] and insertion into a primary carbon–hydrogen bond (Eq. (83)) [548] were described. Indolyl malonates were obtained from carbon–hydrogen bond insertions of dimethyl malonate carbenoids [549].



(83)

The carbenoids derived from rhodium-catalyzed decomposition of diazo compounds insert into nitrogen–hydrogen bonds of alkyl benzeneamines to form tertiary amines [550]. Intramolecular nitrogen–hydrogen bond insertions to give substituted prolines [551], pyrroles (Eq. (84)) [552], and 2-substituted 3-alkylamino-5-phenylthiophenes [553] were reported. Polymer-bound diazo compounds were used in nitrogen–carbon bond insertion reactions [499,554]. Copper catalyzed the insertion of carbenoids into amine and amide nitrogen–hydrogen bonds [555].

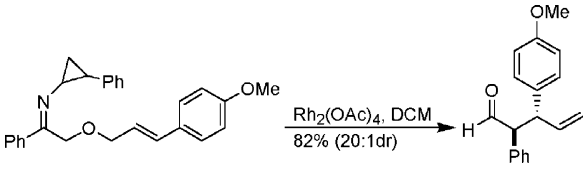
Oxygen–hydrogen bond insertions of carbenoids derived from decomposition of a diazo compound using a rhodium catalyst [556] were used in a synthesis of an E-selectin antagonist [557], and of conformationally locked phosphocholines [558]. Diastereoselective oxygen–hydrogen bond insertions using a chiral rhodium catalyst were described [559]. Rhodium also catalyzed the insertion of carbenoids into silicon–hydrogen bonds [560].



(84)

Asymmetric [2,3]-sigmatropic rearrangement of sulfur ylides formed from decomposition of diazocompounds in the presence of a copper catalyst was reported [561]. A rhodium-mediated Wolff rearrangement was used to prepare 5,6-dihydro-4*H*-thieno[3,2-*b*]pyrrol-5-ones [562]. Rhodium carbenoids derived from *N*-aziridinylimines were shown to participate in a tandem Bamford–Stevens/Claisen rearrangement (Eq. (85)) [563]. A stereoselective, copper-catalyzed, ammonium ylide-Stevens rearrangement sequence was used as an approach to hydroxylated quino-

lizidines (Eq. (86)) [564]. Sulfur ylids, generated from the reaction of 1,3-oxathiolanes and a silylated diazoacetate in the presence of a copper catalyst, gave 1,4-oxathianes via a 1,2-rearrangement (Eq. (87)) [565]. Rearrangement of ammonium ylides generated from metal carbenoids to give novel amino acids was reported (Eq. (88)) [566].



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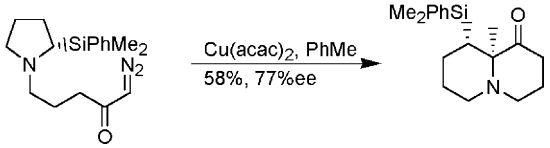

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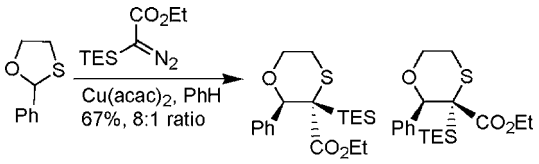

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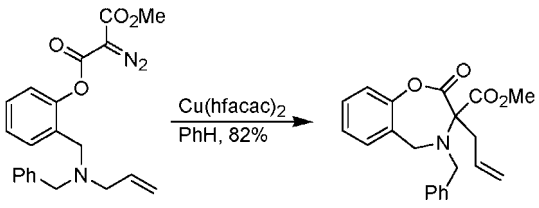
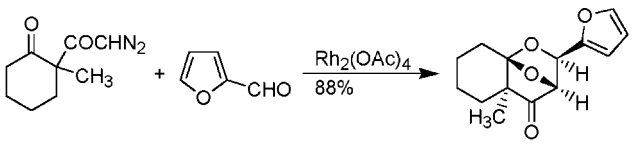


(88)

A variety of cycloaddition reactions of carbonyl ylids formed by decomposition of α-diazocarbonyl compounds were described [567]. Rhodium–carbenoids participated in intermolecular [3 + 4]cycloaddition reactions with heterocyclic dienes [568]. A tandem cyclization–cycloaddition to give oxygen-bridged tetrahydropyranones was described (Eq. (89)) [569]. Reaction of benzyl 2-trialkylsilyl-2-diazoacetates with ketones in the presence of a rhodium catalyst furnished novel dioxolones [570]. Spiro-oxabicycles were prepared from 1,2-diones (Eq. (90)) [571]. The colchicine skeleton was prepared via an intramolecular cycloaddition (Eq. (91)) [572]. Enantioselective [3 + 2]cycloadditions of benzopyrylium-4-olate derived via a rhodium-catalyzed diazo decomposition were described [573]. A [3 + 2]cycloaddition of a diazo-derived ylide with a nitrile was used in a synthesis of leuscandrolide A (Eq. (92)) [574].



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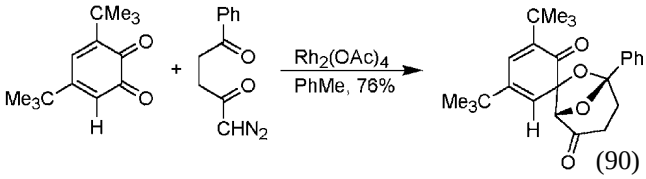

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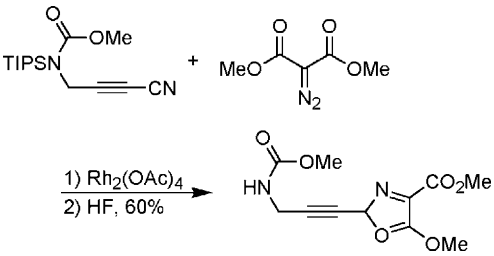
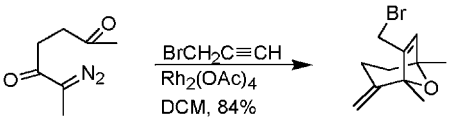


(92)

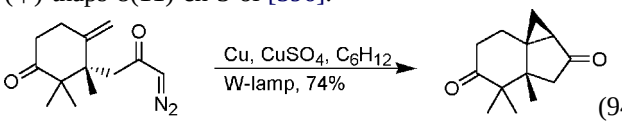
A polymer-bound ruthenium–porphyrin catalyst was used in carbonyl ylide formation–cycloadditions of α-diazoketones [575]. Dipolar cycloadditions of rhodium generated isomünchnones with vinyl ethers [576,577], of a carbonyl ylide with arylalkynes [578], of a carbonyl ylide with propargyl bromide in the synthesis of *cis*-nemorensic acid and 4-hydroxy-*cis*-nemorensic acid (Eq. (93)) [579], and of a carbonyl ylide with 2,2′-bisindole in the synthesis of K252a dimers [580]. An intermolecular diastereoselective carbonyl ylide formation cycloaddition to give 1,3-dioxolanes was reported [581].



(93)

Cyclopropanations of alkenes via transition metal-catalyzed decomposition of diazo compounds continued to be developed. A metal-catalyzed cyclopropanation in water was reported [582]. α-Nitro-α-diazocarbonyl derivatives were used in cyclopropanations of alkenes in the presence of a rhodium catalyst [583]. Chiral rhodium complexes were used for enantioselective intramolecular cyclopropanation using diazoketones [584]. Rhodium-catalyzed intramolecular cyclopropanations were used in syntheses to 2,3-disubstituted cyclopentanones [585], bicyclic phosphonates [586], chiral bicyclo[3.1.0]hexan-2-ones [587,588], 7-oxatricyclo[4.3.0.0^{2,9}]nonan-8-one [589], 12-F_2-isoprostanes [590], a prostaglandin E_2 metabolite [591], and (+)-10,11-epoxythapsan-10-ol [592]. A diastereoselective intramolecular cyclopropanation leading to [3.1.0]bicyclic phosphonates was described [593]. A rhodium-catalyzed intermolecular cyclopropanation with high enantio-control and significant *cis*-selectivity was used to prepare a HIV-1 transcriptase inhibitor [594]. A copper

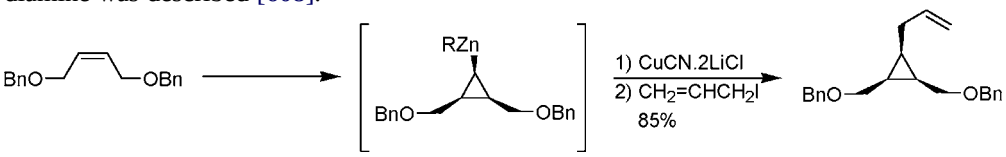
mediated intramolecular cyclopropanation was used in a synthesis of 3-methoxythaps-8-ene (Eq. (94)) [595] and (+)-thaps-8(11)-en-3-ol [596].



(94)

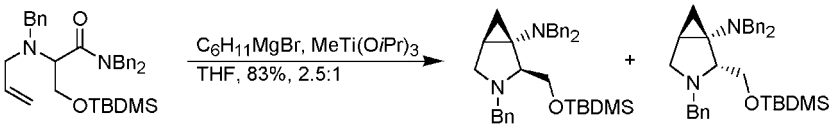
Palladium-catalyzed cyclopropanation of alkenes using diazomethane [597,598] was used in a synthesis of epothilone 490 [63]. Selective diene cyclopropanation using palladium and diazomethane of the alkene adjacent to an electron with drawing group or a boronic ester was reported [599].

Cyclopropanation of alkenes using diethylzinc and diiodomethane or chloroiodomethane was used in synthesis of bafilomycin A_1 C15–C25subunit [600], *trans*-dihydroconfertifolin [601], hirsutene [602], 9,12-anhydroerythronolide aglycone [91], (+)-epifricanol [603], tricyclo[5.3.1.0] undecanoles [604], and mispyric acid [605]. 2,2-Diiodopropane was used in a related zinc-mediated reaction of allylic alcohols [606]. Evidence for a geminal dizinc carbenoid from diethyl zinc and iodoform was reported, and a distereoselective synthesis of 1,2,3-trisubstituted cyclopropanes (Eq. (95)) [607]. Asymmetric cyclopropanation of cinnamyl alcohols using diiodomethane, diethylzinc, and a chiral diamine was described [608].



(95)

Titanium mediated an intramolecular aminocyclopropane formation using alkene tethered carboxamides or nitriles nitriles and a Grignard reagent (Eq. (96)) [609]. Related intramolecular titanium-mediated aminocyclopropanations of alkenes with amides were described [610,611]. Titanium mediated the formation of alkynylcyclopropanes from reaction of 1,1-bis(phenylthio)-2-alkynes with alkenes [612] and the formation of vinyl- and alkynyl–cyclopropanols from vinyl and alkynyl esters [613]. A zirconium-mediated cyclopropanation of allylic ethers was developed (Eq. (97)) [614]. Copper catalyzed an indirect electro-reductive cyclopropanation of activated alkenes with α,α,α-trichloro or α,α-dichloro compounds (Eq. (98)) [615].



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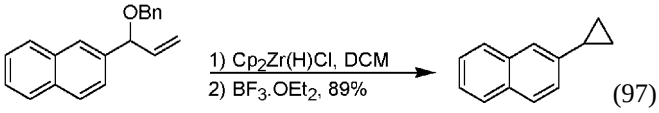

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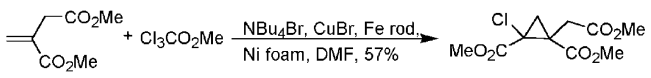

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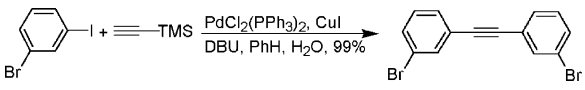
2.1.4. Alkylation of alkynes and arynes

The palladium-catalyzed coupling between terminal alkynes and aryl and vinyl halides or triflates, usually called the Sonogashira coupling, was one of the more frequently utilized carbon–carbon coupling reactions. A number of catalyst systems were described including, palladium carbene complexes [616], a complex for coupling of aryl chlorides in the presence of zinc dichloride [617], palladium on carbon [202,618], a layered double hydroxide supported palladium [3], and using perfluoroalkylphenylphosphine ligands [214,619].

Catalyst systems without copper [620,621] and without copper in ionic liquid were reported [622]. Coupling reactions in aqueous ammonia were reported [623].

Sonogashira couplings of polymer-bound aryl and heteroaryl iodides [53,232,234,624] or polymer-bound alkynes [625] were reported. Microwave-assisted couplings were described [626]. Enol nonaflates [7] and enol tosylates [627] were used in Sonogashira-type couplings. A method for in situ generation of propyne followed by coupling was described [628]. Acid chlorides were coupled with terminal alkynes affording alkynones [629]. Coupling of 2,2′-diiodobiphenyls with terminal alkynes resulted in coupling of one of the iodides and reduction of the other [630].

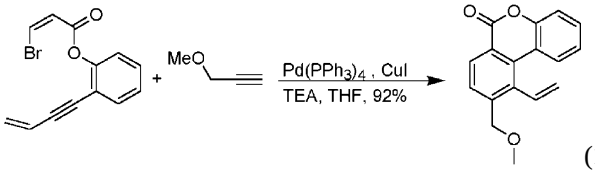
Direct formation of biarylethynes from vinyl triflates, aryl halides and triflates and trimethylsilylalkynes [631,632] using an amidine base and a stoichiometric amount of water was reported (Eq. (99)) [633].



(99)

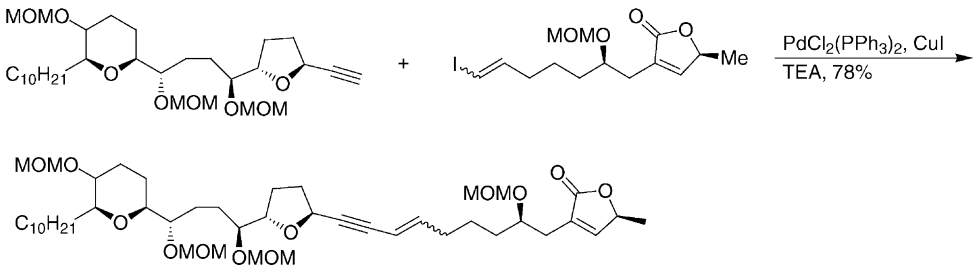
Heterocyclic ring-systems were shown to readily participate in Sonogashira-type couplings. Reactions of 2-bromothiazole [634], 5-iodouridines [635–639], 8-bromoadenosine [638], 5-iodouracils [640], 3-iodofuro[3,2-*b*]pyridine [16], 2,- 4-, and 5-trifloxy-oxazoles and -thiazoles [641], 2-chloro- and 4-bromooxazoles [17], 2,7-dichloro-1,8-naphthyridine [642], 2-bromobenzimidazole and 2-iodoimidazole [643], 2,4-dibromopyrimidine [644], 8-iodoadenosine [645], 3- and 5-iodo-2-chloropyridine [39,646], 3-iodo-1-azaazulene [647], 5-bromo-6-phenyl-3(2*H*)-pyrazinone [22], 6-iodopyrrolo[2,3-*d*]pyrimidine

[648], 2-trifloxybenzo[*b*]furanes [24], haloindoles [649–651], 3-iodoindazoles [652], 5-chloro- and 5-tosyl-1,2,3-triazolo[1,5-*a*]quinazolines [28], and 5-iodo- and 4-bromo-2(2*H*)-pyranones [21,326], or derivatives thereof, were reported. Regioselective coupling in the 3-position of 3,5-dibromo-2-pyrone was reported [653]. A domino Sonogashira coupling-benzannulation to give 6*H*-dibenzo[*b*,*d*]pyrane-6-ones was described (Eq. (100)) [654].



(100)

The Sonogashira reaction continued to be extensively used in organic synthesis [655,656]. Examples of synthetic applications include a cadenine derivative [657], mucocin (Eq. (101)) [658], the side chain of luecascandrolide [659], bicyclic core of cochleamycin [660], 1a,25-dihydroxyvitamin D_3 analogs [661], *cis*-solamin [662], disorazoles [663], a callystatin A subunit [664], analogs of Kornfeld's ketone [665], the carotenoid peridinin [666], (−)-frodosin [476], (+)-FR900482 [667], mucocin [668,669], histrionicotoxins [89], analogs of calcitrol [92], muricatetrocin C [670], cystothiazole E [93], ficuseptine [282], prelaureatin [671], callipeltoside A [103], (−)-histrionicotoxins [672], analogs of benzolactam V8s [673], pseudorubrenoic acid A analog [674], hachijodine G [675], and combrestains A-1 and B-1 [676]. Both inter- and intermolecular couplings were used to prepare kedarcidin chromophore aglycon (Eq. (102)) [677]. Copper-catalyzed couplings of terminal alkynes with alkynyl halides were used in the synthesis of montiporyne A and B [678] and (+)-virol C (Eq. (103)) [679].



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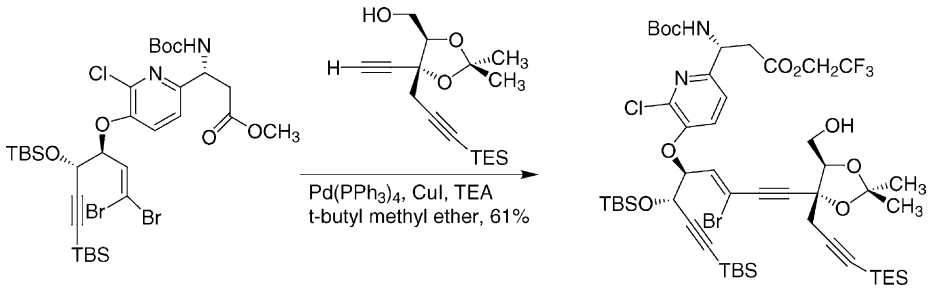

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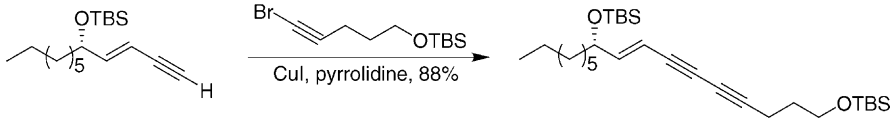


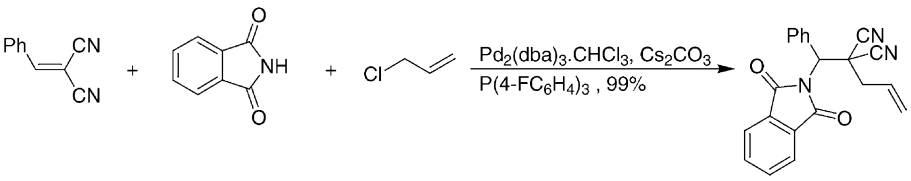
(103)

2.1.5. Alkylation of allyl, propargyl, and allenyl systems

Palladium continued to dominate as the catalyst of choice for allylic alkylation reactions. The mechanism of the formation of the η^3-intermediate, oxidative addition versus dissociation in an alkene–palladium complex was examined [680]. The regioselectivity of the addition of malonate anions to cycloalkenyl esters was predicted by analysis of the sterics of the intermediately formed η^3-allyl intermediate [681]. An intermediate in molybdenum and tungsten-catalyzed allylic alkylations was isolated. Stereospecific substitution of a chloro-group on the metal with a carbon nucleophile followed by *cis-trans* isomerization and reductive elimination was observed [682]. A number of ligands were designed to probe the binding mode in molybdenum-catalyzed reactions [683]. A polymer-bound phosphine was used in allylic alkylation reactions [684].

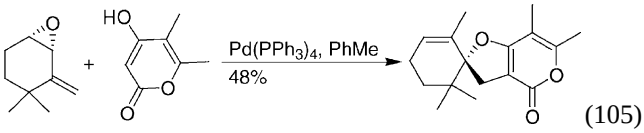
Palladium catalyzed the C-alkylation of phenols via an η^3-allyl intermediate derived from 1-arylpropynes [685].

A palladium-catalyst for the direct alkylation of allylic alcohols was developed [686]. Nucleophiles derived from Michael addition of phthalimide to alkylidenemalonitriles (Eq. (104)) [687] and anions derived from Fischer carbenes were used in palladium-catalyzed allylic alkylations [688]. Zinc enolates derived from glycine were used as nucleophiles for the preparation of optically active amino acid derivatives [689].



(104)

An intermolecular allylic alkylation followed by an intramolecular alkoxylation was used to prepare CDE ring-system of breviones (Eq. (105)) [690]. Intermolecular allylic alkylation of a dienyl carbonate was used in a synthesis toward tigliane/daphnane diterpenes [691] and of dihydromultifidene [692]. A domino intramolecular allylic alkylation–Heck reaction was used to prepare epiisopicropodophyllin aza analogs (Eq. (106)) [693]. Palladium-catalyzed intramolecular allylic alkylation was used in the synthesis of δ-araneosene and humulene (Eq. (107)) [694], toward phomoidride B (Eq. (108)) [520], (+)-brefeldin A [695], and (+)-FR182877 (Eq. (109) [696]. Allenes acts as nucleophiles in intramolecular allylic alkylations (Eq. (110)) [697].



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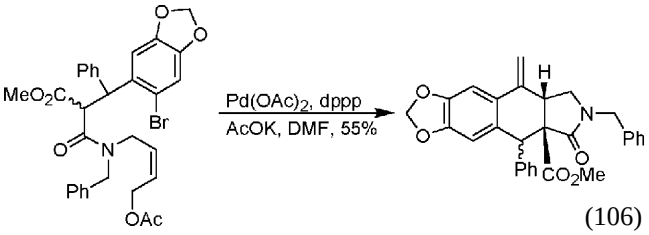

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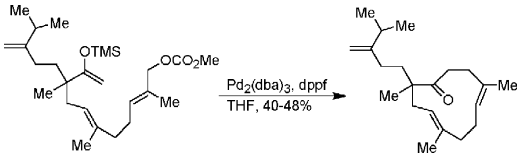

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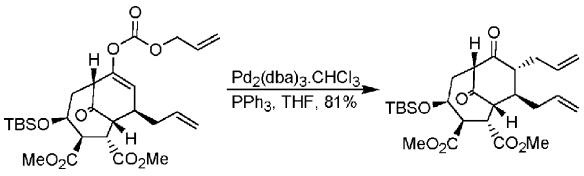

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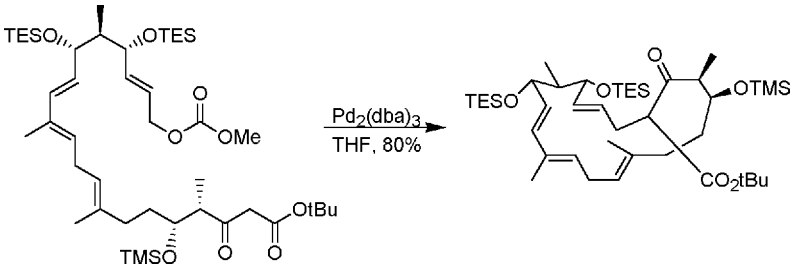

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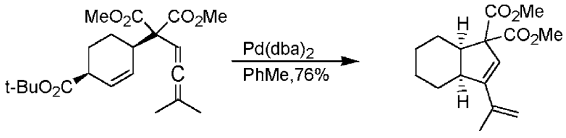

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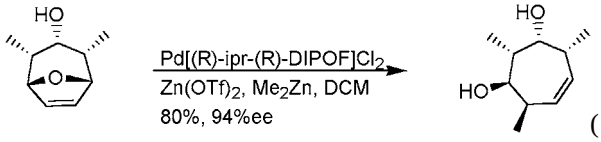
A palladium-catalyzed enantio- and diastereoselective intramolecular allylic alkylation leading to [2.2.2]bicyclic compounds was described [698]. Regioselective alkylation, using stabilized nucleophiles, at the less-hindered terminus of allylic substrates having adjacent chiral tertiary and quaternary centers was observed. Alkylation involving organotin reagents gave opposite regio- and stereoselectivity [699]. Glycine amino esters were used as nucleophiles in asymmetric allylic alkylations [700]. Palladium catalyzed the asymmetric alkylation of 2,3-dienyl phosphates to give optically active 2-(2,3-dienyl)malonates [701]. Palladium-catalyzed asymmetric allylic alkylations were used in the synthesis of jasmonoids [702], C-2-epi-hygromycin A [703], strigolactones [704], and the proposed structure of amphidinolide A [705].

Microwave-assisted molybdenum-catalyzed allylic alkylations were reported [706]. Molybdenum catalyzed asymmetric allylic alkylation at the more substituted position [585]. The solvent dependence in molybdenum-catalyzed asymmetric alkylations was studied [707]. Excellent ee's were obtained using toluene or 1,2-dichloroethane. A molybdenum-catalyzed asymmetric allylic alkylation was used to prepare quaternary amino acids [708]. A molybdenum-catalyzed a dynamic kinetic asymmetric allylic alkylation was used in a synthesis of tipranavir [709]. Stereospecific ruthenium-catalyzed allylic alkylations of chiral allylic carbonates were reported [710].

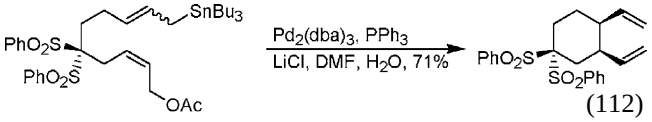
7-Oxabenzonorbornadienes having remote strong π-donating substituents gave good to excellent regioselectivity upon rhodium-catalyzed nucleophilic ring opening [711]. Related asymmetric and racemic ring-opening reactions using organoboronic acids were described [712,713]. Copper also catalyzed the enantioselective ring-opening of

oxa- and azo-bicyclic alkenes using dialkylzincs [714] and acetylides as nucleophiles [715]. Asymmetric, palladium- and nickel-catalyzed nucleophilic ring-openings were used in the synthesis of ionomycin (Eq. (111)) [716].



(111)

Palladium-catalyzed acylation of allylic trifluoroacetates with acyltin reagents [717]. Palladium catalyzed the intermolecular allylation of allyl carboxylates with allyl stannanes or allyl silanes (Eq. (112)) [718] and of allylic acetates with aryl boronic acids [719]. Intermolecular palladium-catalyzed coupling of a chiral cyclic carbonate with vinyltin reagents was used in the synthesis of styryllactones [720]. An intermolecular alkylation using silyl sulfinates and allyl acetate was reported (Eq. (113)) [721]. Allylic germanes were prepared via palladium-catalyzed coupling of allylic acetates with tri(2-furyl)germane [722]. Allyl indium reagents were used as the nucleophile in allylic alkylations [723]. Palladium catalyzed the coupling of 1,3-diarylpropagylic acetates with phenyl zinc chloride to give triaryl-1,2-propadienes [724]. Iridium catalyzed a propargylic substitution using silyl enol ethers (Eq. (114)) [725].



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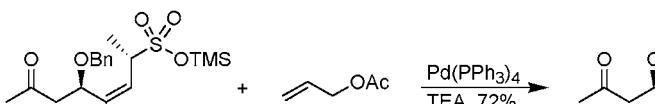

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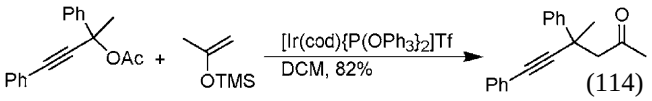
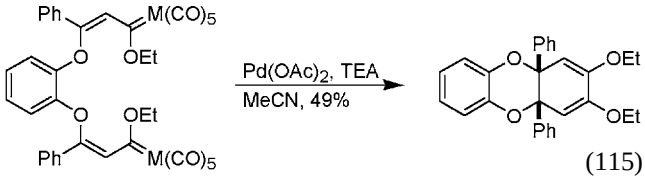


(114)

2.1.6. Coupling reactions

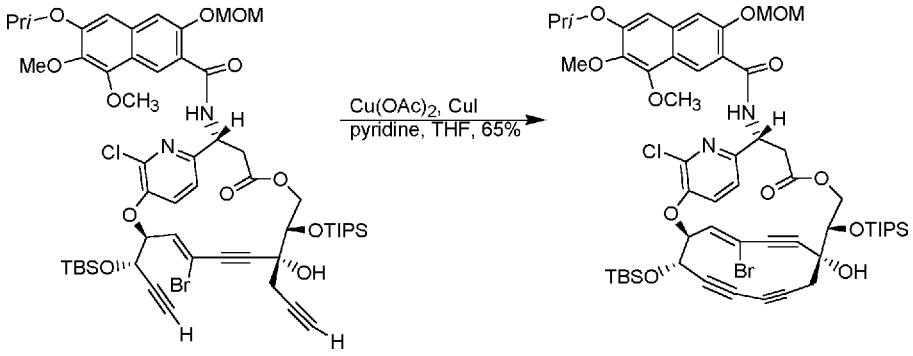
Palladium catalyzed the homocoupling of benzylic zinc and boron reagents in the presence of an α-halocarbonyl compound [726,727]. Palladium catalyzed the dimerization of organo boron compounds [728] in aqueous media [729]. Nickel catalyzed the coupling of divinyl selenides with aromatic Grignard reagents [730]. Palladium catalyzed the coupling of bis chromium carbene complexes to form polycyclic compounds (Eq. (115)) [731]. Palladium catalyzed the coupling of ketene butyltelluroacetals and terminal alkynes to give cross-conjugated triynes [732].



(115)

Nickel catalyzed the intramolecular coupling of 1,2-bis(dibromomethyl)benzene to give *trans*-5,6-dibromobenzocyclobutene [733]. Palladium and nickel catalyzed the dimerization of haloimidazoles [734]. Palladium-catalyzed the coupling of aniliniumhypophosphites with vinyl bromides [735]. Copper catalyzed the coupling of exhaustively flurorinated alkyl iodides with iodophenols [736]. An electro-reductive cobalt-catalyzed cross-coupling of aryl halides to give unsymmetrical biphenyls was reported [737].

A palladium-catalyzed intramolecular oxidative aryl–aryl coupling of diarylamines was used in the synthesis of mukonine [738] and thienocarbazoles [739]. Nickel catalyzed the addition of organoboron esters to conjugated dienes forming alkylated alkenes [740]. A copper-mediated intermolecular coupling was used to prepare kedarcidin chromophore aglycon (Eq. (116)) [677].

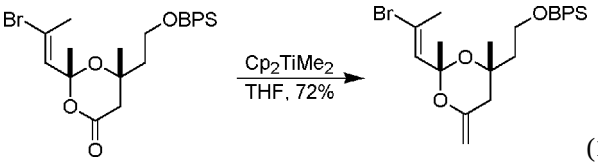


(116)

2.1.7. Alkylations of carbonyl compounds

A number of carbonyl-to-alkene transformations were reported. Reaction of CH_2Br_2-Zn-$TiCl_4$ was used in the synthesis of the taxane AB fragment having a spirocyclopropyl group [741], of orostanal [742], and (+)-arenarol [743]. Methylenation of an aldehyde carbonyl

employing Cp_2ZrCl_2, Zn-dust, and CH_2I_2 was used in a synthetic application toward the pectenotoxins-4 and -8 [744]. Tebbe's reagent, $Cp_2TiCH_2ClAlMe_2$, was used to methylenate a lactone carbonyl in a synthesis toward ciguatoxin [745], an ester in the synthesis of Vitamin D_3 side-chain [746], and an aldehyde in a synthesis toward migrastatin [747]. Petasis reagent, Cp_2TiMe_2 [748], was used in synthetic applications toward histrionicotoxins [89] and (+)-zampanolide and (+)-dactylolide (Eq. (117)) [749].



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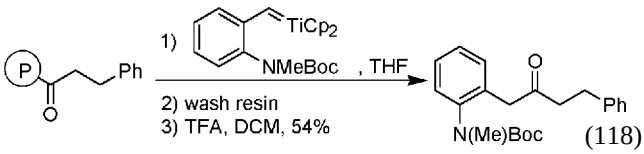
An titanium benzylidene was used in a solid-phase synthesis of indoles (Eq. (118)) [750]. A titanocene–alkyl halide system was employed to alkenylate aldehydes, ketones and esters (Eq. (119)) [751]. A palladium-catalyzed alkenylation of a thiolactone, using an alkylzinc reagent, was used in a synthesis of (+)-biotin (Eq. (120)) [752]. Rhodium catalyzed the methylenation of fluorine substituted ketones using trimethylsilyl diazomethane [753]. An iron porphyrin complex catalyzed the alkenylation of aldehydes with ethyl diazoacetate [754].



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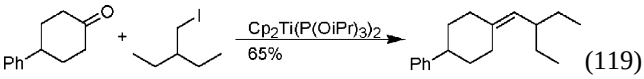

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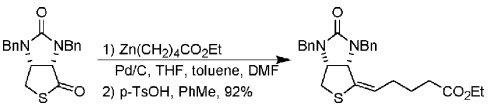
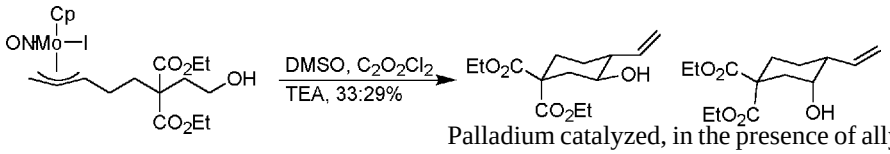


(120)

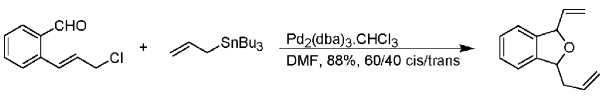
Umpolung of η^3-allyl palladium complexes by transmetallation to indium was used in a number of cases. Palladium-catalyzed diasteroselective alkylation of aldehydes by chiral propargylic mesylates in the presence of diethylzinc was used in the synthesis of bafilomycin V_1 [56] and a callystatin A subunit [664]. η^3-Allylpalladium intermediate derived from vinyl aziridines was transmetallated to indium and gave in the presence of an aldehyde 1,3-amino alcohols [755].

Cyclic homoallylic alcohols were obtained from cyclization of η^3-allyl molybdenum complexes having a pendant aldehyde (Eq. (121) [756]. Rhodium-catalyzed Reformatsky-type reactions of α-bromo esters with aldimines, in the presence of diethylzinc, forming β-amino esters or β-lactams [757].



(121)

Palladium catalyzed, in the presence of allyltributyltin, the formation of five- and six-membered heterocycles from allylic halides having a pendant aldehyde or imine (Eq. (122)) [758]. Nickel catalyzed intermolecular homoallylation of aldehydes with 1,3-dienes via transmetallation to aluminum [759]. An intramolecular variation of aldehyde-tethered conjugated dienes in the presence of $Bu_3SnSiMe_3$ (Eq. (123)) [760] and an electrochemically driven allyl transfer from a allylic ether to a pendant carbonyl group [761] were described. A related reaction in the presence of diethyl zinc was also reported [762]. An asymmetric nickel–chromium-catalyzed coupling of vinyl iodides with aldehydes to give allylic alcohols was described. This type of reaction was used in a synthesis of the C14–C26 fragment of halichondrins (Eq. (124)) [763]. Hydrozirconation of a terminal alkyne followed by transmetallation to zinc and reaction with an aldehyde was used in the synthesis of leucascandrolide A [764].



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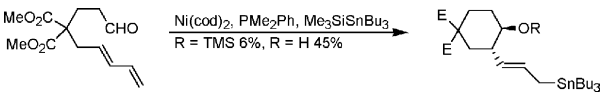

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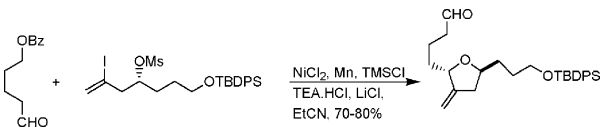
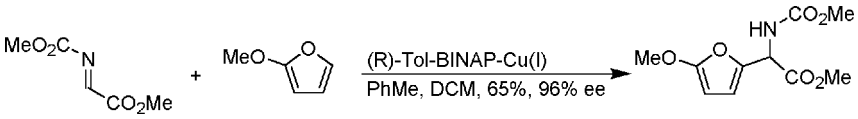


(124)

2.1.8. Carbon–hydrogen bond insertions

A chiral copper catalyst was used in the synthesis of aromatic and heteroaromatic α-aminoesters via enantioselective direct alkylation of aromatic and heteroaromatic compounds with α-imino esters (Eq. (125)) [765]. Ruthenium catalyzed the carbon–hydrogen bond insertion, carbonylation, and alkylation sequence of *N*-(2-pyridyl)indolines (Eq. (126)) [766]. Ruthenium catalyzed the formation of 3-aryl-1-alkynes from aromatic compounds and propargyl alcohols (Eq. (127)) [767]. Palladium catalyzed the addition of pyrroles to alkynoates (Eq. (128)) [768]. Rhodium catalyzed hydroarylations of alkenes using heterocycles (Eq. (129)) [769].



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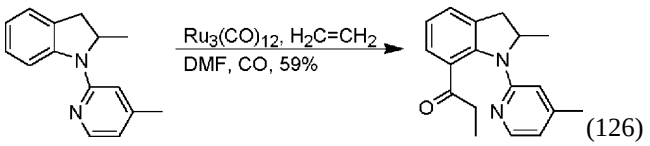

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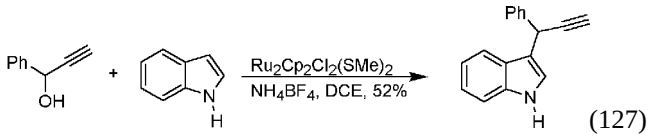

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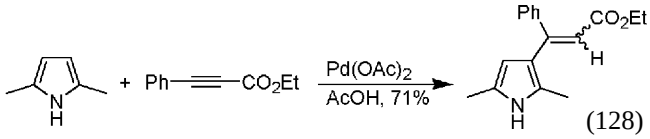

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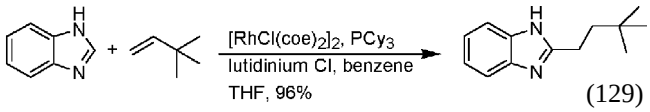
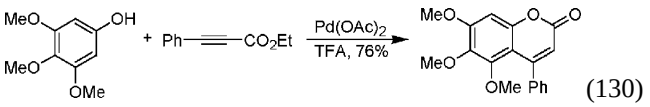


(129)

A palladium-catalyzed intermolecular hydroarylation of alkoxyphenols with alkynoates affording coumarins was described (Eq. (130)) [770]. Nickel catalyzed an asymmetric hydrovinylations of styrenes using ethene [771,772]. A cobalt-catalyzed hydrovinylation of 1,3-dienes with terminal alkenes was described (Eq. (131)) [773].



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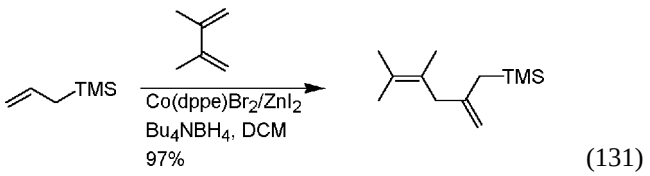
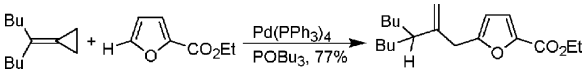


(131)

A nickel-catalyzed carbon–hydrogen bond insertion reaction of aryl iodides with aryl aldehydes affording unsymmetrical ketones was developed [774]. Alkylation of alkylidenecyclopropanes with heteroaromatic compounds (Eq. (132)) [775] and ketones [776], in the presence of a palladium catalyst, were reported. Palladium catalyzed the addition of malonitriles to methyleneaziridines [777]. An *N*-heterocyclic carbene complex was reported as an intermediate in rhodium-catalyzed annulation of *N*-alkenyl-substituted benzimidazole [778]. Rhodium catalyzed the enantioselective cyclization of 4-pentynals to give cyclopentenones (Eq. (133)) [779].



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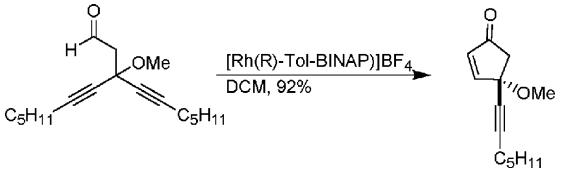
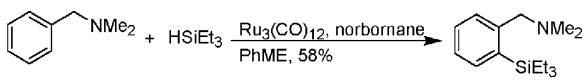


(133)

Chelation-assisted ruthenium-catalyzed silylations of aromatic carbon–hydrogen bonds were reported (Eq. (134)) [780]. Chelation-assisted ruthenium-catalyzed hydroesterifications of alkenes (Eq. (135)) [781], *ortho*-arylation of aromatic imines with aromatic halides [782], and Heck-type reaction of anilides [783] via carbon–hydrogen bond activation were described. Rhodium catalyzed a related coupling of alkenes with α,β-unsaturated ketones in the presence of a secondary amine (Eq. (136)) [784]. Acylation of alkenes using intermediately formed imines employing Wilkinson's catalyst and 2-amino-3-picoline was reported (Eq. (137)) [785]. The reaction was also performed using microwaves in the absence of solvent [786]. Rhodium catalyzed a novel formation of cycloalkanones from dienes and an allylic amine as the source of the carbonyl carbon (Eq. (138)) [787].



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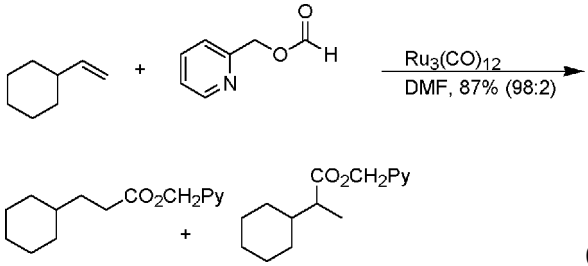

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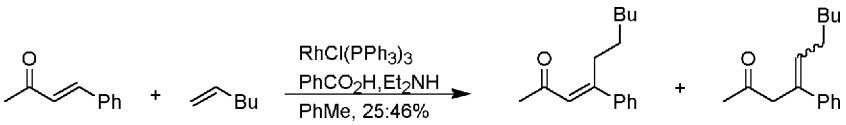

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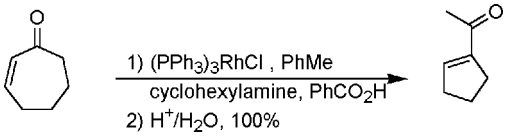

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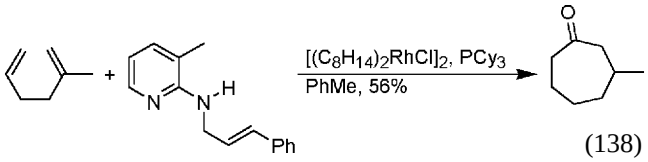
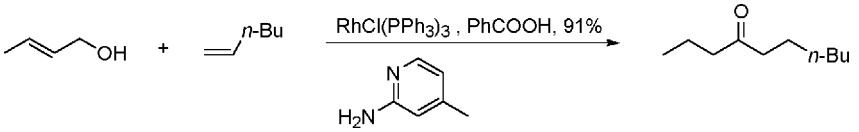


(138)

Wilkinson's catalyst was used to prepare ketones via double bond isomerization of allylic alcohols followed by hydroacylation of an alkene (Eq. (139)) [788]. Related alkylations of aromatic imines using alkenes and a rhodium-catalyst were described [789]. Selective hydroacylation of terminal alkynes with aldehydes to give α,β-unsaturated ketones was described using Wilkinson's catalyst [790]. Carbon–hydrogen bond activation using palladium as the catalyst was also developed (Eq. (140)) [791].



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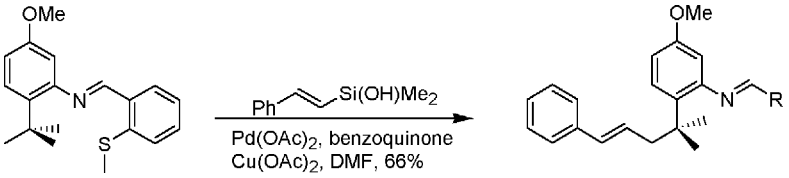
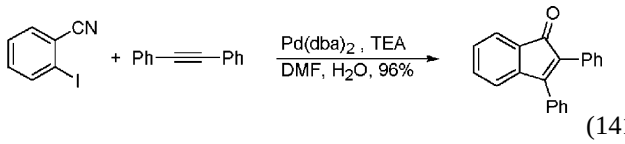


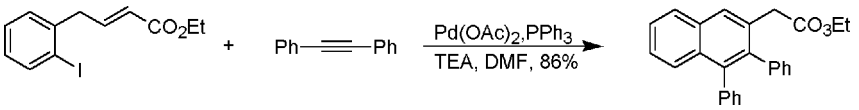
(140)

2.1.9. Cascade reactions

Reactions wherein intermediately formed σ-palladium complexes undergo sequential insertion(s) of an unsaturation followed by a termination step, usually a nucleophilic addition, are discussed in this section. Most of the reactions are catalyzed by palladium. A number of reactions initiated by oxidative addition of palladium(0) to aryl iodides and bromides followed by insertion of alkynes to give a new carbon–carbon bond were reported. Palladium-catalyzed reaction of 2-iodoarylnitriles with alkynes and alkenes furnished 2,3-disubstituted indenones and polycyclic aromatic compounds (Eq. (141)) [792]. Oxidative addition followed by insertion of 1-(1-alkynyl)cyclobutanols and ring expansion to give cyclopentanones was reported [793]. A reaction consisting of sequential oxidative addition, intermolecular alkyne insertion, and intramolecular alkene insertion leading to substituted naphthalenes was described (Eq. (142)) [794]. Cascade annulations to give bicyclic heterocycles were reported (Eq. (143)) [795,796]. Nickel catalyzed an oxidative addition—alkyne insertion—intramolecular ketone alkylation to give indenes (Eq. (144)) [797] and related reaction of 2-iodobenzylalcohols terminated by nucleophilic carboxylation to give seven-membered lactones [798].



(141)



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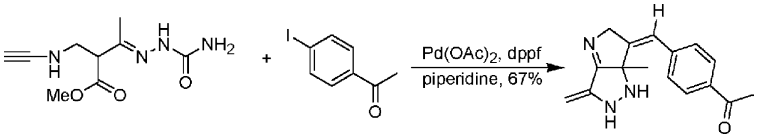

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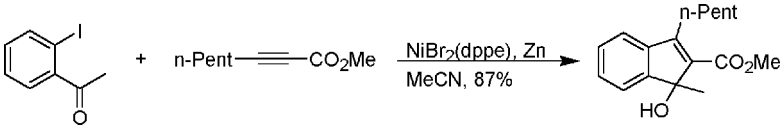
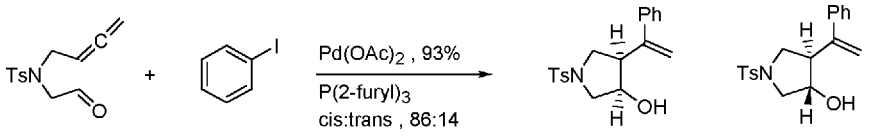


(144)

Copper catalyzed the reaction of 2-iodophenols with aryl ethynes to give 2-arylbenzo[*b*]furans [799]. A related palladium/copper-catalyzed reaction of ferrocenylethyne and terminal alkynes with 2-iodophenol was reported [321,800]. Palladium-catalyzed oxidative addition to a polymer-bound 2-iodoaniline followed by alkyne insertion and intramolecular amination was used to prepare polymer-bound indoles [801]. A similar reaction was used in the synthesis of a number of indole alkaloids [802]. Oxidative addition-carbon monoxide insertion of aryl iodides followed by alkyne insertion and cyclization using 2-alkynyl phenols to give 2-substituted-3-aroylbenzo[*b*]furans was described [803].

Oxidative addition—allene insertion followed by intramolecular alkylation of a tethered, stabilized carbanion can be tuned to give either cyclopropanes or cyclopentenes [804,805]. A similar oxidative addition—allene insertion followed by intramolecular alkylation of a tethered aldehyde, in the presence of indium, was described (Eq. (145)) [806]. A related oxidative addition, allene insertion, imine alkylation sequence, in the presence of indium, producing homoallylic amines was developed [807]. Oxidative addition—allene insertion followed by intramolecular amide carbonyl cyclization furnished iminolactones [808], and aminations furnished 2,3-dihydropyrolles [809], and 2,2-dimethyl-3-pyrrolines [810]. A related asymmetric reaction was described (Eq. (146)) [811]. A fragmentation or formation of 2,3-dihydrofurans was observed upon reaction of 5-hydroxy-1,2-dienes with aryl halides (Eq. (147)) [812,813]. Oxidative addition, allene insertion, intermolecular imine alkylation followed by intramolecular amination was used to prepare nitrogen containing compounds (Eq. (148)) [814]. An asymmetric oxidative addition, 2,3-allenoic acid insertion, cyclization was used to prepare optically active 3-aryl-substituted butenolides (Eq. (149)) [815]. Oxidative addition—intermolecular allene insertion—intermolecular amination of methyl 2-iodobenzoate followed by intramolecular amide formation furnished 4-methylene-3,4-dihydro-1(2*H*)-isoquinolin-1-ones [816]. Oxidative addition, allene insertion, alkoxylation followed by cycloaddition gave fused isoxazolidines (Eq. (150)) [817]. A palladium-catalyzed oxidative-addition–allene insertion ring-expansion to form isoquinoline diones was reported (Eq. (151)) [818]. Palladium catalyzed of aryl halides with 1-(1,2-dienyl)cyclobutanols to give α-disubstituted cyclopentanones was described [819]. Palladium catalyzed the reaction of allenes with organic halides in the presence of an arylboronic acid furnishing functionalized alkenes (Eq. (152)) [820].



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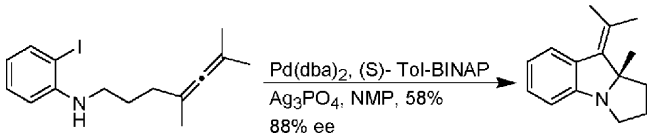

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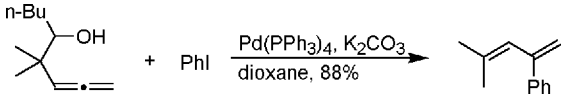

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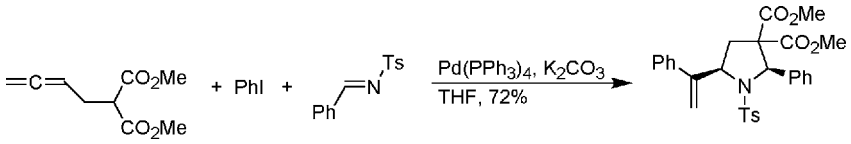

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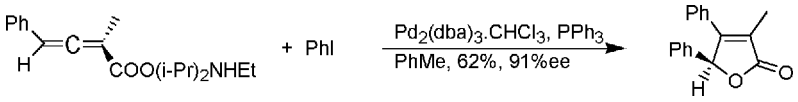

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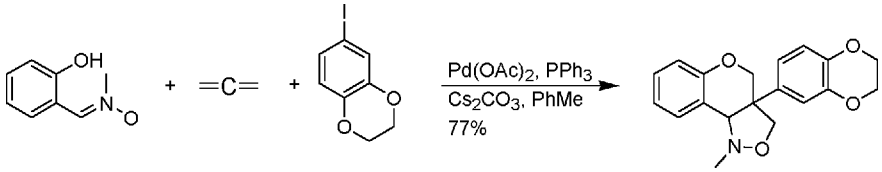

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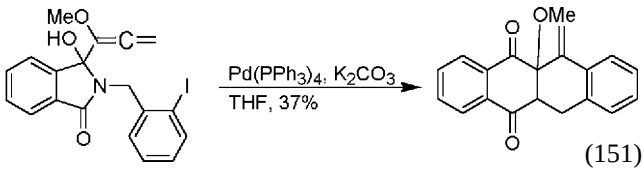

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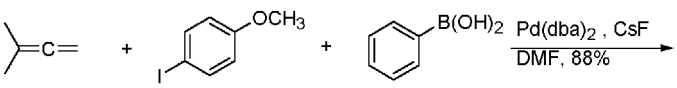
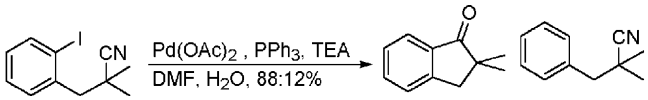


Indanones, tetralones, and cyclopentenones were obtained from palladium-catalyzed reaction of aryl and vinylhalides having a tethered nitrile (Eq. (153)) [821]. A palladium-catalyzed cascade reaction of isonitriles with 6-iodo-*N*-propargylpyridones was used in the synthesis 11*H*-indolizino-[1,2-*b*]quinolin-9-ones (Eq. (154)) [822]. Oxidative addition using 3-iodopyridine followed by insertion of nonconjugated dienes, palladium migration, and nucleophilic attack using nitrogen nucleophiles was used to prepare a number of pyridine alkaloids (Eq. (155)) [823]. Oxidative addition to propargylic carbonates having a pendant alkene furnished azabicyclo[3.1.0]hexanes (Eq. (156)) [824].



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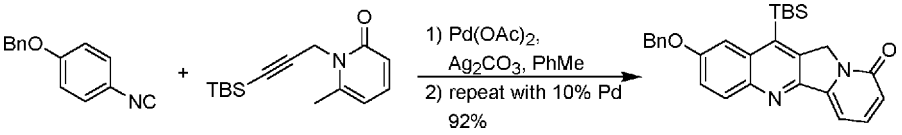

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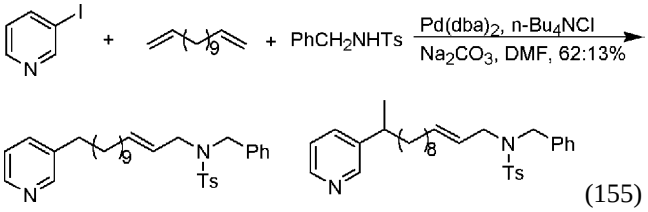

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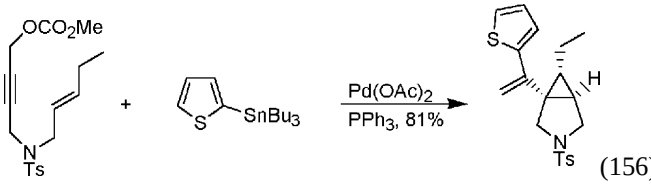
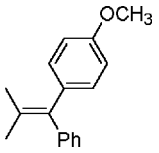


(156)

2.2. Conjugate addition

Conjugate addition of alkylzirconocenes was reported [825]. Rhodium-catalyzed Michael additions of organosiloxanes [826]. Palladium and copper-catalyzed Michael-type addition of acylzirconocenes to conjugated ene- and ynones produced 1,4-diketones [827,828]. Palladium-catalyzed hydroarylations of nitroalkenes using aryltin compounds in the presence of a catalytic amount of bismuth [829]. A stereoselective synthesis of indanes involving transmetallation from boron to rhodium–alkene insertion and Michael-type addition to a pendant acceptor was reported (Eq. (157)) [830].



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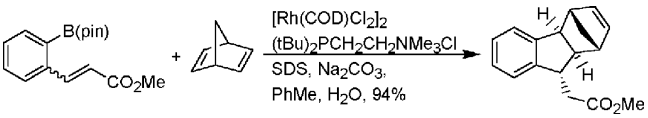
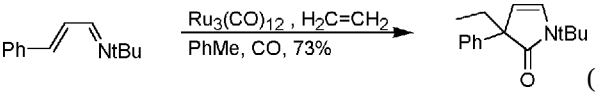


(157)

2.3. Carbonylation reactions excluding most hydroformylations

2.3.1. Carbonylations of alkenes and arenes

A palladium-catalyzed dicarboxylation of alkenes using a palladium–molybdovanadophosphate catalyst system under carbon monoxide and air was developed [831]. Ruthenium-catalyzed carbonylation of α,β-unsaturated imines in the presence of alkenes furnished β,γ-unsaturated γ-butyrolactams (Eq. (158)) [832]. Palladium catalyzed the formation of lactones from 2-allylphenols (Eq. (159)) [833].



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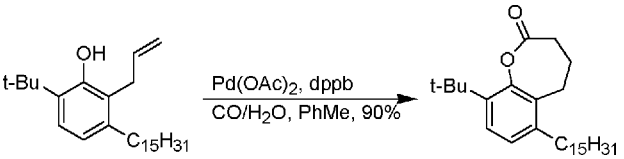

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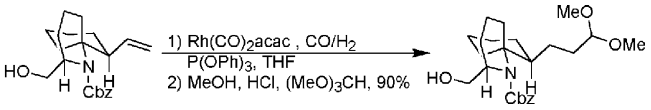
Rhodium-catalyzed hydroformylation was employed in synthesis of lepadiformine Eq. (160)) [834] and carba-D-fructofuranose [835]. A rhodium-catalyzed formylation-annulation sequence to give azabicyclo[4.4.0]alkane amino acids was developed (Eq. (161)) [836]. *N*-Phenyl alkyl amides were prepared from alkenes and anilines using

cobalt on charcoal in the presence of carbon monoxide [837]. Rhodium-catalyzed intramolecular silylformylations of pendant alkenes or alkynes followed by allylation was reported (Eq. (162)) [838]. Silylcarbocyclization formylation of 1,6-enynes using rhodium-catalysts were also described (Eq. (163)) [839].



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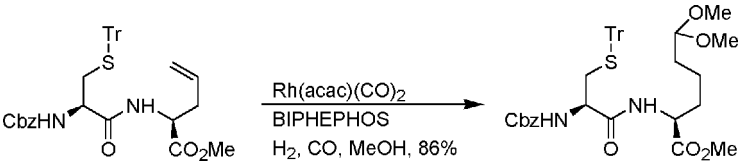

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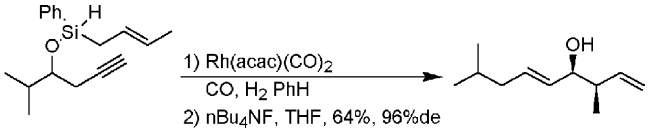

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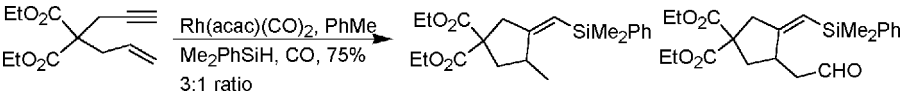
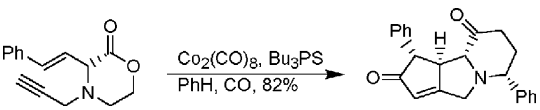


(163)

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 [840,841]. Catalyst systems for the Pauson–Khand reaction, included a polymer-bound catalyst [842], heptacarbonyl(triphenylphosphine)dicobalt [843], and dicobalt octacarbonyl and molecular sieves [844].

An intramolecular Pauson–Khand reaction in water using colloidal cobalt nanoparticles was described [845]. Supercritical ethane [846] and ionic liquids [847] were used as solvents. Both Pauson–Khand and reductive Pauson–Khand reactions were reported using cobalt nanoparticles on charcoal as the catalyst [848]. Diastereoselective intramolecular Pauson–Khand reactions using a catalytic amount of cobalt together with Bu_3PS were reported (Eq. (164)) [849]. Palladium and cobalt nanoparticles immobilized on silica catalyzed a tandem allylic alkylation Pauson–Khand reaction (Eq. (165)) [850,851]. Pauson–Khand reactions promoted by microwave irradiation were reported [852,853]. Tungsten–cobalt alkyne complexes undergo intermolecular Pauson–Khand reactions [854].



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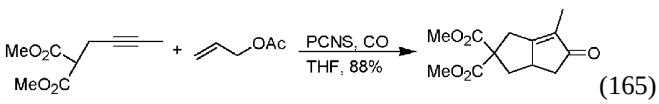

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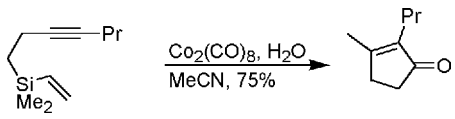
Pauson–Khand reactions of *S*-alkyl substituted alkynes gave cyclopentenones having the *S*-alkyl group α to the ketone [855]. Reductive Pauson–Khand reactions of alkynes having a pendant vinyl silane were described (Eq. (166)) [856]. Reductive and standard Pauson–Khand type reactions were observed depending on additives and reaction conditions upon thermolysis of 1,6- and 1,7-enynes (Eq. (167)) [857]. Trimethylgermyl-substituted enynes participated in Pauson–Khand reactions [858]. Pauson–Khand reactions of silicon-tethered enynes [859] and 2-aryl-1,6-enynes [860] were reported. Molybdenum–cobalt complexed alkynes were shown to undergo endo-selective and intermolecular Pauson–Khand reactions [861]. A rhodium-catalyzed Pauson–Khand type reaction using aldehydes as the carbon monoxide source was described (Eq. (168)) [862–864]. Vinyldimethyl-2-pyridylsilanes smoothly undergo intermolecular Pauson–Khand type reactions in the presence of a ruthenium catalyst and carbon monoxide (Eq. (169)) [865].



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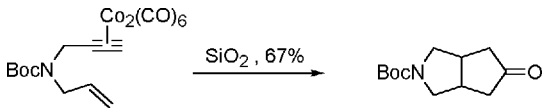

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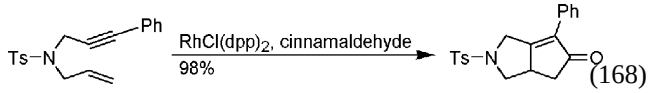

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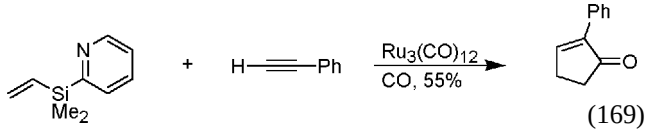
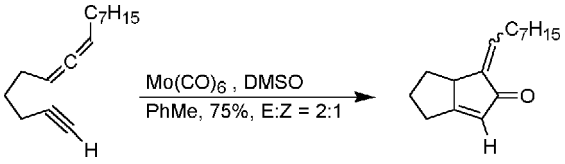


(169)

Asymmetric Pauson–Khand reactions using cobalt and chiral arylphosphites [866] or chiral N-oxides [867] were reported. Asymmetric intramolecular Pauson–Khand reactions using optically active 1,6-enynes were described [868], and diasteroselective Pauson–Khand reactions using propynal derived allylic acetals were described [869]. Molybdenum mediated Pauson–Khand-type reactions using optically active 1,2-diene-7-ynes to give α-alkylidene cyclopentenones (Eq. (170)) [870].



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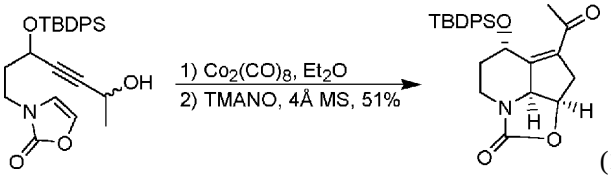
The Pauson–Khand reaction was used in synthesis of 8α-hydroxystreptazolone (Eq. (171)) [871], indole and quinoline fused cyclopenenones [872], [5.n.1.$0^{1,5}$]tricyclic ring-systems [873], ceratopicanol [874], and the ABC rings of nakadomarin and manzamine [875]. Cyclization of molecules containing a nonconjugated diyne and a conjugated diene gave [5.5.5.6]fenestrane system (Eq. (172)) [876].



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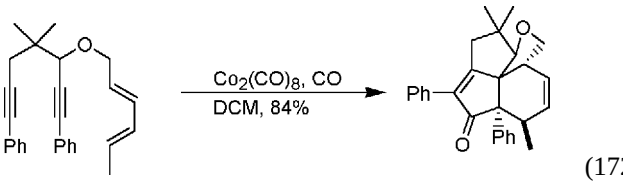
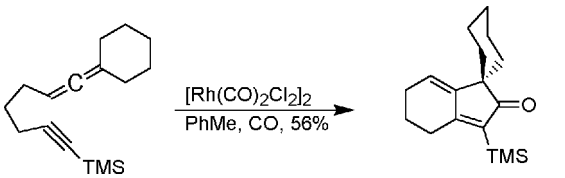


(172)

Intramolecular, molybdenum- and rhodium-catalyzed allenic Pauson–Khand reactions were described [877]. A silicon tethered variation of this reaction was also reported [878]. The π-bond selectivity was found to be catalyst dependent (Eq. (173)) [879]. Allene–yne substituted β-lactams was employed to give tricyclic compounds [880].



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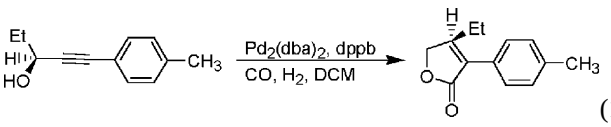
Palladium catalyzed the formation of oxazolidinones from propargylic amines and carbon dioxide [881]. A palladium-catalyzed carbonylation–lactonization of a propargylic alcohol was used in a synthesis of (+)-incrustoporin (Eq. (174)) [882].



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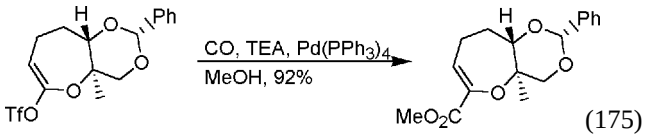
2.3.3. Carbonylation of halides, triflates, epoxides, and phosphates

Palladium complexes catalyzed the carbonylation of a variety of substrates to form carboxylic acids and derivatives. Palladium catalysts were developed for the alkoxycarbonylation of aromatic halides [883,884] and halomethylketones [885]. Carbonylations using a dendrimer-palladium catalyst on silica [886] and palladium encapsulated in polyurea [2] were described. A palladium–carbene complex was used to carbonylate aromatic halides in ionic liquids [887]. Carbonylation of 3-iodofuropyridines was described [16]. Iridium-catalyzed alkoxycarbonylations of allylic phosphates [888]. Nickel catalyzed an electrochemically driven carboxylation of vinyl triflates [889].

The palladium-catalyzed alkoxy-carbonylation of halides and triflates was used in synthetic applications toward 8,10-di-*O*-methylbergenine [890], analogs of Kornfeld's ketone [665], the carotenoid peridinin [666], gambierol (Eq. (175)) [187], endothelin A [891], (−)-acromelobic acid and (−)-acromelobinic acid [892], pyrrolo[3,2-c]quinolone alkaloids [893], griseoviridin (Eq. (176)) [894], and alkaloid 223A [895].



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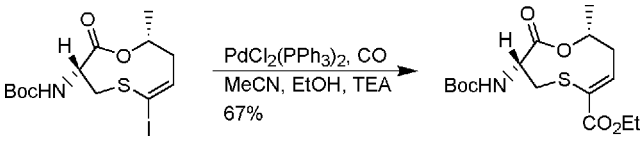

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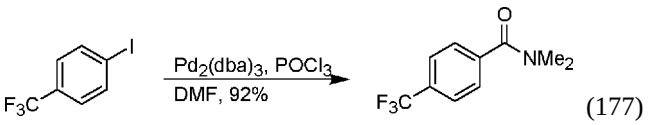
Palladium catalyzed the aminocarbonylation of 1-bromo-1,2-dienes to give 2,3-unsaturated amides [808]. Direct formation of amides from aryl or alkenyl halides using a palladium-catalyst, $POCl_3$, and DMF as the amide source was reported (Eq. (177)) [896]. Dimethylformamide was also used as the source of carbon monoxide in palladium-catalyzed formation of aromatic amides from aryl bromides under microwave irradiation (Eq. (178)) [897]. Direct formation of *N*, *N*-dimethylamides via palladium-catalyzed reaction of aromatic halides with *N*, *N*-dimethylcarbamoyltrimethylsilane was also described [898]. Carbonylation of aromatic bromides in the presence of amides produced unsymmetrical imides [899]. A trifloxyisoquinoline derivative was transformed into an amide using ^{11}C carbon monoxide [900]. This type of reaction was also used toward the triptoquinone-triptinin A ring-system [901]. Palladium catalyzed the formation of quinolin-4-ones via carbonylation and intermolecular acylation [902]. Palladium catalyzed the formation of 3-methyleneisoquinolin-3-ones from 2-bromoacetophenone, carbon monoxide, and an amine [903].



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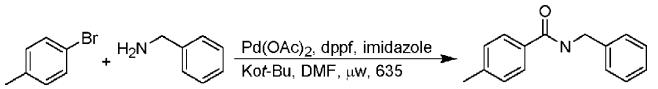

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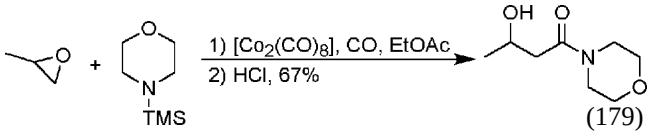
Palladium-catalyzed cyclocarbonylation of 2-halobiphenyls to give fluoren-9-ones was developed [904]. Palladium catalyzed the carbonylation of diaryliodonium salts in the presence of indium to give symmetrical ketones [905] and a carbonylative coupling of aryl diazonium salts with aryl boronic acids to give diarylketones [906]. Coupling of a trimethylsilyl pyridylethyne with aryl iodides in the presence of carbon monoxide gave aryl alkynyl ketones [907]. Palladium catalyzed a carbonylative coupling of 1-tributylstannyl-1-fluoroethene with aromatic iodides and triflates to give aryl α-fluoroethenyl ketones [908], and of a methyl indium reagent with chromium tricarbonyl complexed aromatic chlorides to give methyl ketones [909]. Carbonylative coupling of phenyl tributylstannyl selenide with aromatic iodides to give the corresponding selenol esters was reported [910].

Enantiopure β-hydroxy amides were prepared using a cobalt-catalyzed carbonylation of optically active terminal epoxides (Eq. (179)) [911]. Cobalt catalyzed the carbonylation of aziridines and epoxides to give β-lactams and β-lactones, respectively [912]. A silica-bound palladium catalyst was used in formylations of aromatic halides using sodium formate [913].



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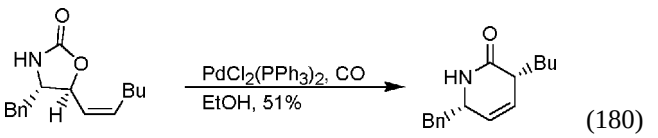
2.3.4. Miscellaneous carbonylations

Intramolecular Heck reaction followed by carbonylation of the formed σ-palladium intermediate was reported [914]. A diastereospecific decarboxylative-carboxylation of 5-vinyloxazolidin-2-ones to give 3,6-disubstituted 3,6-dihydro-1*H*-pyridin-2-ones using a palladium catalyst was described (Eq. (180)) [915]. Palladium catalyzed a carbonylation–cyclization of aryl iodide with *N*-*t*-butyl-2-(1-alkynyl)benzaldimines to give 4-aroylisoquinolines (Eq. (181)) [916,917]. Palladium catalyzed the carbonylation of diaryliodonium salts in the presence of amidoximes to give 3,5-disubstituted-1,2,4-oxadiazoles (Eq. (182)) [918].



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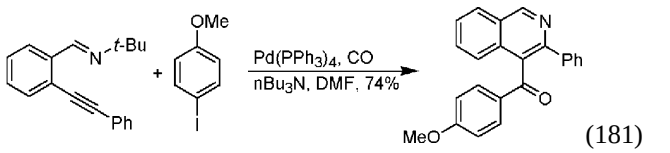

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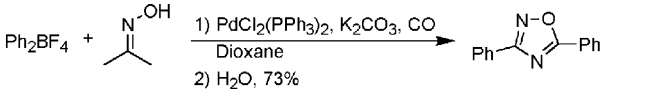

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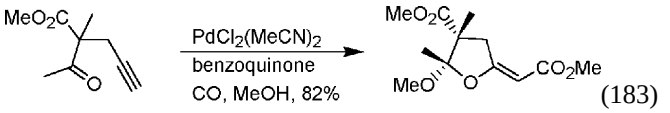
Intramolecular palladium-catalyzed hydroxy-palladation carbonylations of alkynes, for example, of *N*-2-propynylamides, was used in the synthesis of 5-(alkoxycarbonyl)methylene-3-oxazolines [919], of polymer-bound 2-hydroxyphenylalkynes to give 2,3-disubstituted benzo[*b*]furans [920], and in the synthesis of (+)-cystothiazole A [921]. An intramolecular alkyne hydroxy-palladation of in situ formed hemiketals followed by carbonylation furnished substituted α-methylenetetrahydrofurans (Eq. (183)) [922]. Cyclic ortho esters were formed in a related fashion from propargylic acetates [923]. Alkene hydroxy-palladation carbonylation was used in synthetic applications toward marine metabolites [924] and goniothalesdiol [925]. Pyrrole-2-acetic acid esters were prepared via a palladium-catalyzed cyclization-carbonylation of (*Z*)-(2-en-4-ynyl)amines (Eq. (184)) [926].



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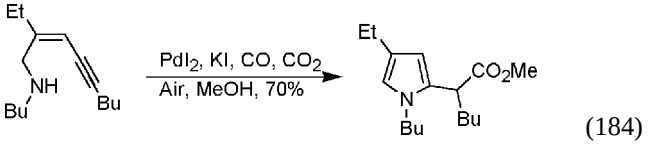

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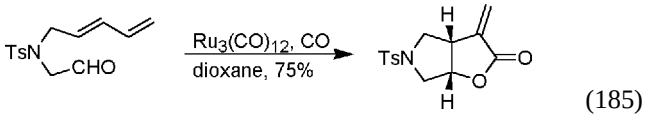

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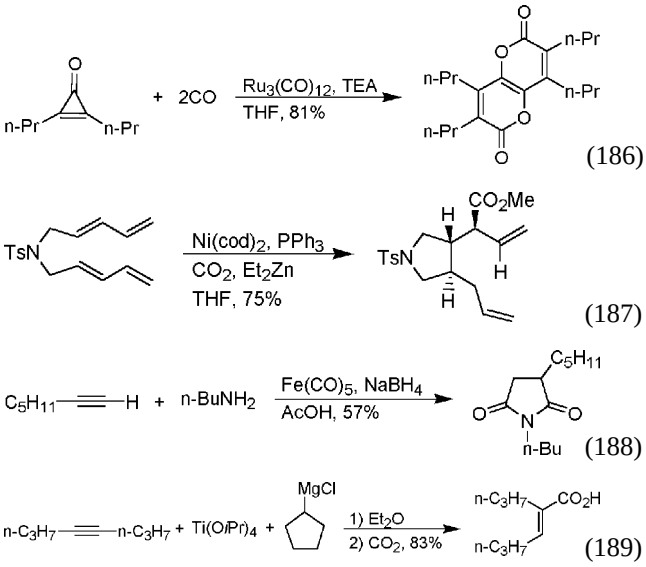
Rhodium catalyzed an interesting synthesis of α-methylene-γ-butyrolactones from allenyl aldehydes and carbon monoxide (Eq. (185)) [927]. Rhodium and iridium catalyzed the formation of fused cyclopentadienones from 1,6-diynes and carbon monoxide and fused cyclopentadienone imines from 1,6-diynes and isocyanides [928]. Iron-mediated cyclocarbonylation of silicon-tethered alkynes to give substituted cyclopentadienones was described [929]. Ruthenium catalyzed the synthesis of pyranopyrandiones from cyclopropenones and carbon monoxide or carbon monoxide and an alkyne (Eq. (187)) [930]. Nickel catalyzed a carbonylation of tetraenes using carbon dioxide to give cyclic compounds [931]. Tungsten catalyzed the oxidative carbonylation of tethered diamines to form cyclic ureas [932]. Iron mediated the formation of cyclic imides forming a primary amine and two equivalents of an alkyne

(Eq. (188)) [933]. Cyclic anhydrides were obtained in the absence of an amine. Carbonylation of titanacyclopropanes and propenes with carbon dioxide furnished carboxylic acids (Eq. (189)) [934].

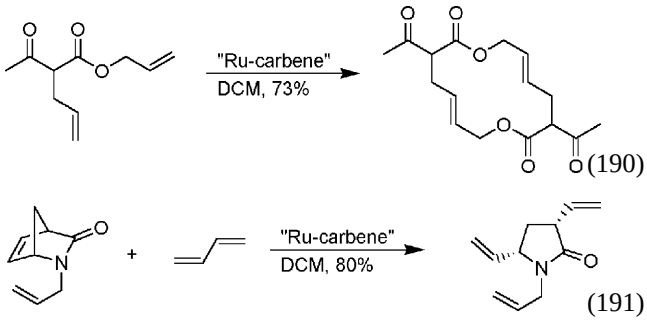


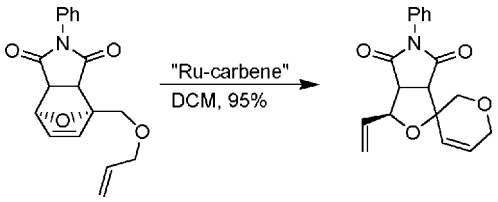
2.4. Metathesis reactions

A substantial number of reports on ring-closing metathesis reactions using Grubbs'-type ruthenium catalysts were published. New, highly efficient, and sometimes air stable ruthenium catalysts were developed [935–940]. A rhodium-catalyst for ring-closing metathesis or alkene isomerization was described [941]. Microwave-accelerated reactions were developed [942]. Polymer supported, recyclable catalysts were reported [943–945]. Ruthenium-catalyzed ring-closing metathesis in ionic liquids [946] and in protic media [947]. A ring-closing metathesis was used to cleave a polymer alkene linker [948], and this was used in synthesis of dysidiolide-derived protein phosphatase inhibitors [949]. Diastereoselective ruthenium-catalyzed ring-closing metathesis of trienes having a chiral ester functionality was reported [950]. A polymer-bound molybdenum-catalyst for asymmetric ring-closing metathesis was developed [951].

A large variety of ring-systems were prepared via alkene–alkene ring-closing metathesis using ruthenium catalysts. Ring-systems including cyclic phosphonamides [952], cyclic phosphinate [953], 2-vinyl-tetrahydropyridine, and -dihydropyrrole derivatives [954], five- to eight-membered cyclic amines [955,956], fused bicyclic and tricyclic amines [566,957–959], bridged bicyclic amines [960], 2,5-dihydropyrroles [961,962], six- to nine-membered lactams [963–966], fused bicyclic lactams [967], indole-fused lactams [968], fused indoles [26,969], macrocyclic lactams [970,971], 5–10-membered and macrocyclic-lactones [972–974], macrocyclic aza-lactones [975], cyclic peptides, dimeric peptides, and pseudopeptides [976–987], cyclic amino acids [988], cyclic sulfones [989], sultones [990], bicyclo[5.3.0]decanes, oxabicyclo[6.4.0]- and oxabicyclo[7.4.0]alkenes [991,992], five- to eight-membered cycloalkenes [993–998], macrocycles [999–1003], α,β-unsaturated cyclic ketones [484], bridged tricyclic carbon skeletons [1004], spiropiperidines [1005], carbocylic methyl and silyl ethers [1006], 2,5-dihydrobenzo[*b*]oxepines [1007], 3,6-dihydro-1*H*-benzo[*c*]oxocines [1008], spirocycles [1009,1010], six-membered unsaturated cyclic ethers [1011], fused unsaturated cyclic seven- to eight-membered ethers [1012], 3-oxo-1-oxa-4-cycloalkenes [1013], 6,8-dioxabicyclo[3.2.1]octane [1014], 5,5-fused thiophene γ-lactams [1015], 3′,4′-*trans*-linked bicyclic nucleosides [1016], bridged metallocenes [1017], ferrocene-substituted bis(2,5,8,9-tetrahydro-3*H*-imidazo[1,2-*a*]diazepin-3-ones) [1018], cobalt and iron sandwich complexes containing rings [1019], di- and trinucleotides [1020], bicyclic thymidine analogs [1021], a rhenium containing macrocycle [1022], polycyclic ethers [1023], and carbocyclic nucleosides [1024], were all prepared using ring-closing metathesis methodology.

A 14-membered bislactone was obtained in place of the expected seven-membered lactone (Eq. (190)) [1025]. A domino metathesis of 2-azanorbornenes affording 1-azabicyclic compounds was reported (Eq. (191)) [1026]. Domino metathesis reactions were also used to prepare functionalized pyranes (Eq. (192)) [1027]. Formation of a cyclohexene from a more sterically demanding alkene was observed upon attempted macrocyclization (Eq. (193)) [483]. Ring-closing metathesis of a variety of sulfur-containing dienes was reported [1028]. Indoles and 1,2-dihydroquinolines were prepared using a ring-closing metathesis reaction (Eq. (194)) [1029]. Depending on the length of the tether between an alkene and a diene, either of the diene, double bonds may react to form a ring-closed product [1030]. A catalyst system for tandem ring-closing metathesis alkene isomerization to give cyclic enol ethers or enamides was developed (Eq. (195)) [1031]. A tandem palladium-catalyzed allylic acetate isomerization ruthenium-catalyzed ring-closing metathesis was described (Eq. (196)) [1032]. 2-Trimethylsilyl-1,6- and 1,7-dienes were used to give a variety of silyl-substituted cyclopentenes- and -hexenes [1033]. Schrock-type molybdenum carbenes were used to prepare silyloxycyclo-hexenes and -heptenes [371], in enantioselective ring-closing metathesis to give silyloxycycloheptanes [1034], and to afford unsaturated cyclic amines [1035].





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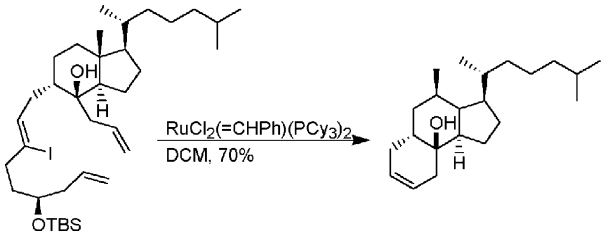

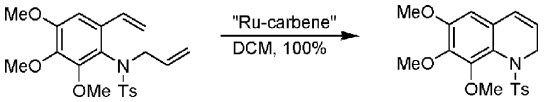

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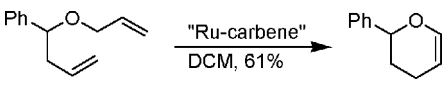

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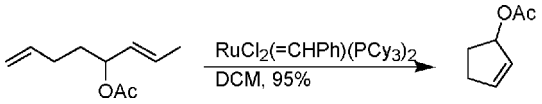
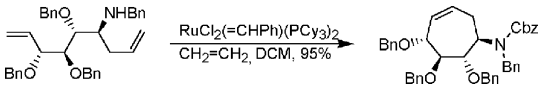


(196)

Synthetic targets of alkene–alkene ring-closing metathesis include motopurine analogs [1036], nor-1,6-germacradien-5-ols [1037], (+)-sedamine [1038], ciguatoxins [1039–1046], (+)-calystegine B_2 (Eq. (197)) [1047], prostaglandins [349], liverwort diterpenes [1048], cyclic peptomimetics [1049], (+)-boronolide [1050], carbocyclic nucleosides [1051], (2*S*,3*R*,4*S*)-3,4-dihydroxyproline [1052], (+)-goniodiol, (−)-8-epigoniodiol, and (+)-9-deoxygoniopyporone [1053], epothilones [61,63,1054,1055], (−)-swainsonine, (+)-1,2-di-epi-swainsonine, and (+)-1,2,8-tri-episwainsonine [1056], silaic acid [1057], the izidine alkaloid skeleton [1058], (+)-fostriecin [271], toward guanacatepene A [1059], (−)-adaline (Eq. (198)) [1060], amphidinolide A (Eq. (199)) [1061], mycoepoxydiene [1062], (−)-pinolidoxin [1063], dihydrocorynanteol[1064], a guanacasterpene A fragment (Eq. (200)) [1065], microcarpalide [1066], a neplanocin A analog [1067], (+)-boronolide [1068], the C17–C28 fragment of spongistatin A [1069], fostriecin [88,1070], 1,14-herbertenediol and 11-epi-herberteneolide [1071], octalactin A [1072], C15–C18 fragment of laulimalide [1073], stemoamide [1074], woodrosin I (Eq. (201)) [1075], coleophomones B and C (Eq. (202)) [1076], amphidinolide T4 (Eq. (203)) [185], (+)-puraquinonic acid [90], (−)-osmundalactone and (−)-muricatacine [1077], cycloaraneosene and ophiobolin [122], (+)-lentiginosine [1078], halicholactone [1079], salicylihalamide A [1080], aspidosperma alkaloids [1081], (−)-laulimalide [1082–1084], (+)-methynolide [1085], (*S*)-homopipecolic acid, (*S*)-homoproline, (*S*)-coniine [1086], geldanamycin analogs (Eq. (204)) [1087], prelaureatin [671], herbertanes [1088], (−)-argentilactone [1089], (+)- and (−)- cacospongionolide B [1090], dysinosin A [1091], (+)-goniodiol [1092], solanoeclepin A [1093], okilactomycin [1094], the triptoquinone-triptinin A ring-system [901], (+)-epifricanol [603], (−)-isolaurallene [1095], (+)-methynolide [1096], phorboxazoles [1097], (−)-salicylhalamide A [1098], pterulone [1099], (+)-tanikolide and (−)-malyngolide [1100], baikiain [1101], d4T [1102], (−)-acaterin [1103], [7*R*,6*S*,5*S*,4*R*]-triacetoxy-(−)-goniotriol [1104], toward guanacatepene A [1105], CVS 1778 analogs [1106], and migrastatin [747]. Schrock's molybdenum catalyst was used in syntheses of (−)-laulimalide [1107], (+)-brasilenyne [373], and (−)-gleenol [1108].



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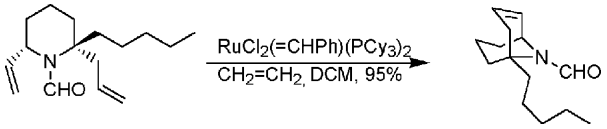

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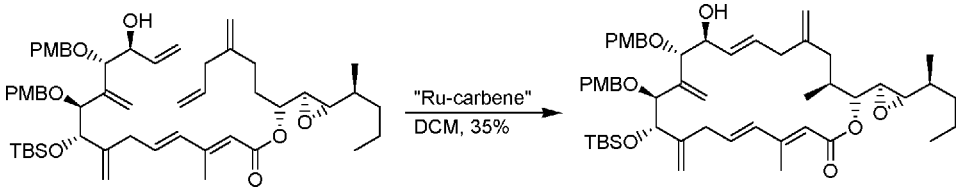

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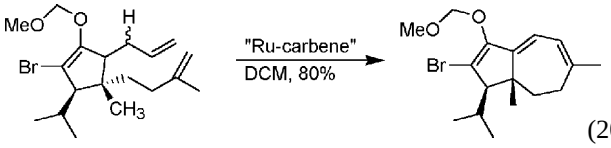

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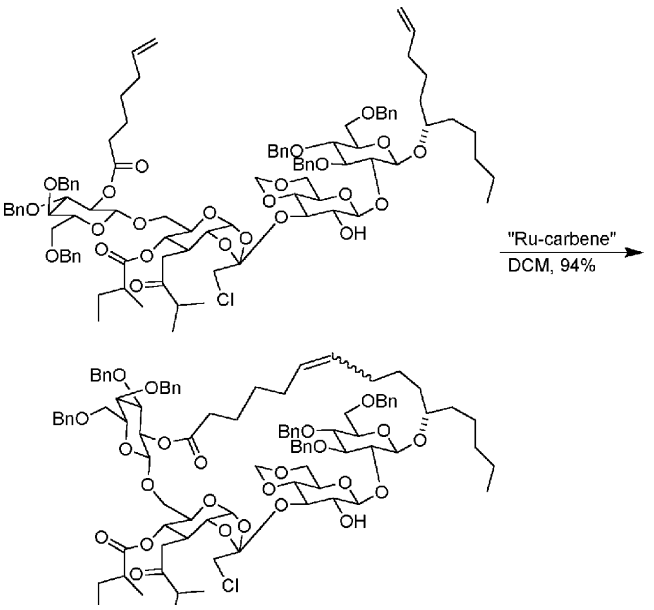

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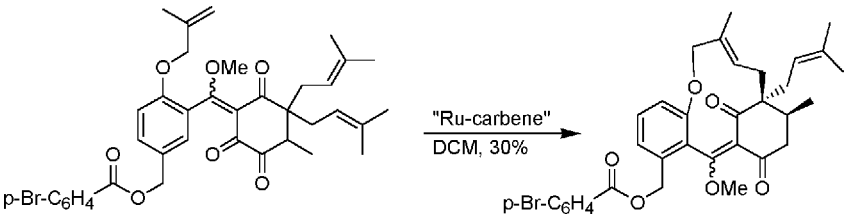

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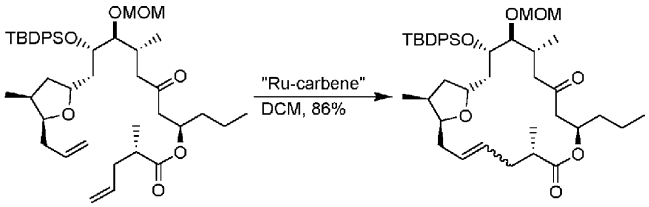

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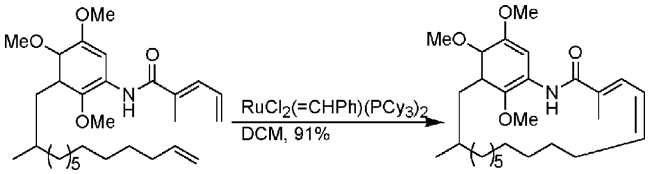
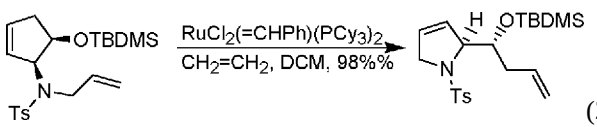


(204)

Ring-opening alkene–alkene cross-coupling metathesis of cyclooctadiene and trisubstituted cyclic alkenes was reported [1109]. A related reaction of cyclobutenes with alkenes was described affording 1,5-dienes with high regioselectivity [1110]. 2-Azabicyclo[2.2.1]hept-5-en-3-one [1111], norbornadienes and norbornenes [1112,1113], and a 1,3-cyclopentadiene-heterodienophile cycloadduct [1114] all participated in ring-opening cross-metathesis reaction with allyl trimethylsilane. A catalyst for asymmetric ring-opening cross-coupling metathesis was developed [1115]. A ring-opening cross-coupling metathesis was used in the synthesis of 15-F_2 isoprostanes [1116].

A ring-opening ring-closing (ring rearrangement) methodology [1117] was used to prepare (−)-swainsonine (Eq. (205)) [1118], (+)-dihydrocuscohygrine and cuscohygrine [1119], bicyclic phophonates [586], (−)-α-pinene [1120], (−)-anaferine [1121], the dendrobatid alkaloid 251F [1122], bicyclo[n.3.0]cycloalkenes [1123], and (−)-indolizidine 167B [1124]. Molybdenum catalyzed the formation of chiral tertiary cyclohexenyl ethers and spirobicyclic ethers via ring rearrangement reactions (Eq. (206)) [1125].



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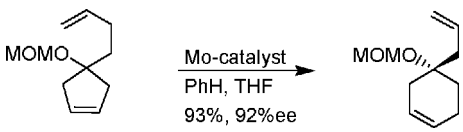
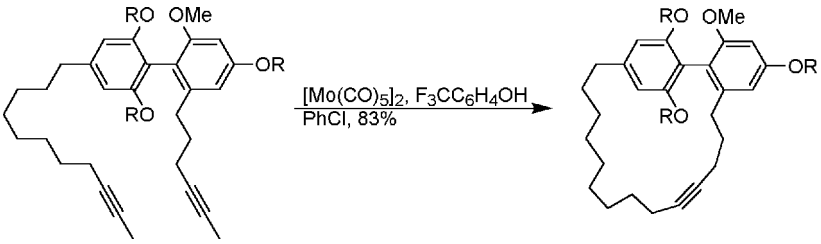


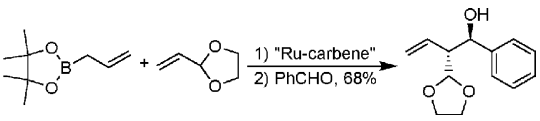
(206)

Alkyne–alkyne ring-closing and cross-metathesis using a preheated mixture of $Mo(CO)_6$/4-chlorophenol/3-hexyne [1126] or $Mo(CO)_6$-2-fluorophenol [1127] were reported. Alkyne–alkyne metathesis was used in a synthesis of turrianes (Eq. (207)) [1128]. A tandem dienyne metathesis was used to prepare bicyclic lactones [1129] and guanacastepen related ring-system [1130].



(207)

A ruthenium catalyst for alkene–alkene cross-coupling of acrylonitrile was developed [1131]. A ruthenium catalyst for tandem cross-metathesis-hydrogenation was described [1132]. Cross-metathesis on a solid support was reported [1133]. Acetals formed from 1,2-dihydroxy-3-butene were better suited for cross-metathesis compared to the corresponding diethers and diesters [1134]. Cross-metathesis of allyl- and vinyl-silanes with unsaturated ethers and epoxides was described [1135,1136]. Alkene–alkene cross-metathesis of unsaturated Fischer carbenes was reported [1137]. A one-pot cross-coupling metathesis of an allyl boronic ester followed by benzaldehyde allylation was developed (Eq. (208)) [1138]. Related metathesis reactions of alkenes with allyl pinacol boronate were described [1139]. Alkene metathesis between a cycloalkene and a divinyl ketone was used to prepare macrocyclic diketones (Eq. (209)) [1140].



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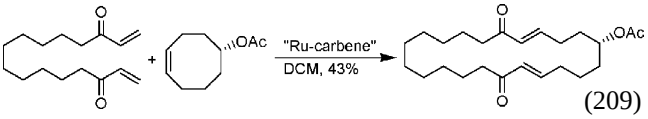
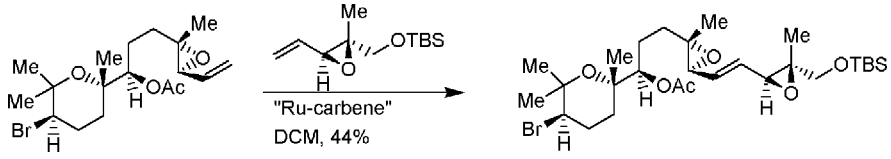


(209)

Alkene–alkene cross-metathesis [1141–1143] was used in organic synthesis to prepare dipeptide alkene isosteres [1144], ABC tristetrahydropyran of thyrsiferol and venustatriol (Eq. (210)) [1145], symmetrical trisubstituted alkenes [1146], the bicyclo[3.3.1]nonane core of garsubellin A [1147], cyclosporin A derivatives [1148], alkene-substituted sugars [1149,1150], glycosyl amino acids [1151], (+)-brefeldin A [1152], C-neoglycopeptides [1153], K252a dimers [580], callipeltoside A (Eq. (211)) [103], high-affinity mu opioid receptor ligands [1154], of (−)-prosophylline [1038], and epothilone analogs [299].



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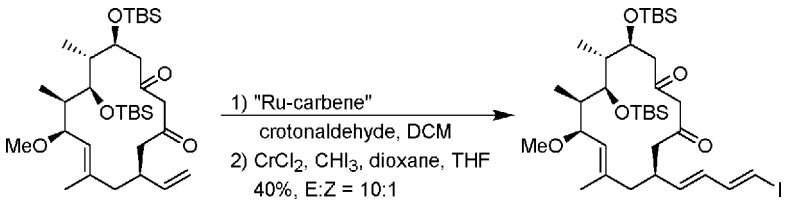
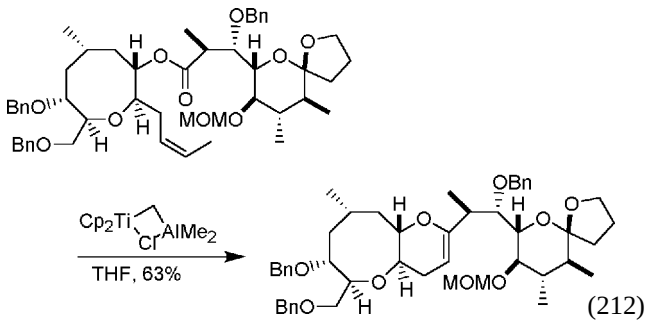


(211)

An interesting metathesis reaction using a titanocene was employed in a synthesis toward ciguatoxin CTX3C (Eq. (212)) [1044]. Titanium promoted the reaction of thioacetals with ethene resulting in either one-carbon homologation or two-carbon homologation via metathesis (Eq. (213)) [1155].



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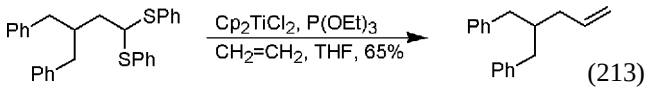


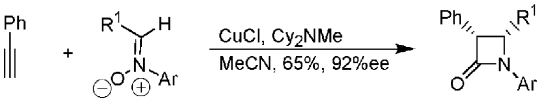
(213)

2.5. Rearrangements

2.5.1. Cycloisomerizations

Rhodium and iridium catalyzed a reductive intermolecular imine-acrylate [2 + 2]cycloaddition to form β-lactams (Eq. (214)) [1156]. Iron catalyzed an enantioselective [2+2]cycloaddition of imines and ketenes to give β-lactams [1157]. A copper catalyst was used to prepare β-lactams from alkynes and nitrones (Eq. (215)) [1158]. Molybdenum catalyzed an intramolecular [2 + 2]cycloaddition of 1,2-diene-7-ynes (Eq. (216)) [1159] Intramolecular copper-catalyzed [2 + 2]photocycloadditions were used in a synthesis of kelsoene [1160] and (+)-herbertene [1161]. Palladium catalyzed a tandem [2 + 2]cycloaddition–allylic rearrangement of ketene with α,β-unsaturated aldehydes and ketones (Eq. (217)) [1162]. Chromium catalyzed a [6 + 2]cycloaddition of 1,3,5-heptatriene with dienophiles [1163].

(214)



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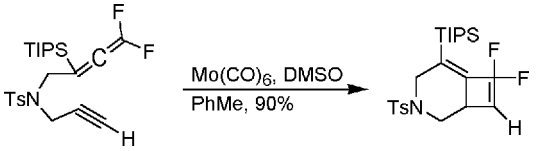

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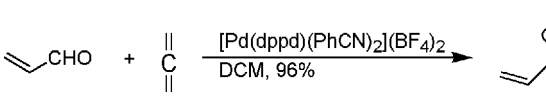
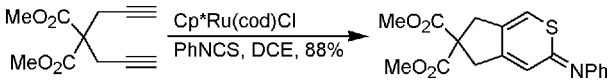


(217)

Methylidynetricobalt nonacarbonyl catalyzed the cyclotrimerization of alkynes [1164]. Cobalt catalyzed the intermolecular [2+2+2]cyclobenzannulation of nitriles with ethyne to give 2-substituted pyridines [1165]. The ABC core of taxoids was prepared via a sequence of cobalt-mediated [2 + 2 + 2] and [4 + 2]annulations [1166]. An intramolecular, cobalt-mediated, [2 + 2 + 2]cyclobenzannulation was used in the synthesis of angular [4]phenylene [1167]. Intermolecular alkyne cyclotrimerization in aqueous media at room temperature was described [1168].

Ruthenium catalyzed [2 + 2 + 2]annulation of 1,6-diynes with isothiocyanates or carbon disulfide (Eq. (218)) [1169] and of 1,6-diynes with tricarbonyl compounds to give functionalized cyclopentenes (Eq. (219)) [1170]. Ruthenium also catalyzed the annulation of two acetylene dicarboxylates with allylic alcohols to give functionalized benzenes [1171]. Benzo[*b*]thiophenes were prepared by a ruthenium-catalyzed cyclobenzannulation of bis(3-thienyl)ethyne [1172]. Ruthenium was used for stereocontrolled [2 + 2 + 2]cyclobenzannulation of 1,6,11-triynes [1173].

Chiral 3-substituted phthalides were prepared by rhodium-catalyzed [2 + 2 + 2]cycloaddition (Eq. (220)) [1174]. A titanium-mediated [2+2+2]cyclobenzannulation of two different alkynes and a nitrile to give pyridines was developed [1175]. Wilkinson's catalyst was used for [2+2+2]cyclotrimerization of polyynes with ethyne [1176]. The illudalane sequiterpene alcyopterosin E was prepared using an intermolecular rhodium-catalyzed cyclotrimerization (Eq. (221)) [1177]. Carbazoles were prepared in a similar fashion (Eq. (222)) [1178]. A related reaction of an alkyne with a propargylic bromide was used in a synthesis of alcyopterosin A (Eq. (223)) [1179].



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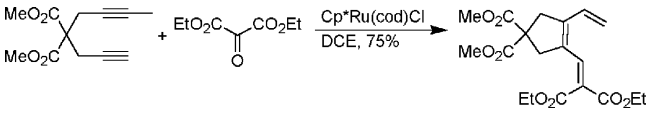

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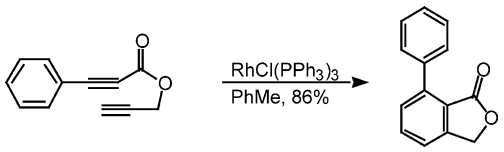

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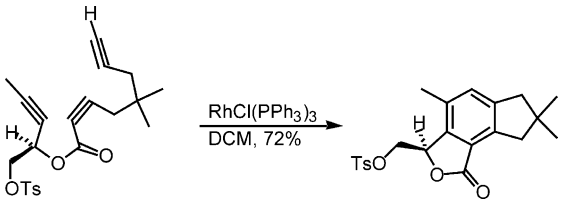

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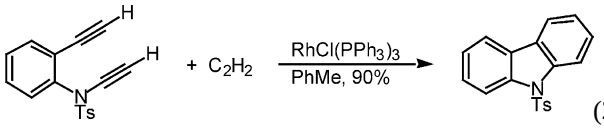

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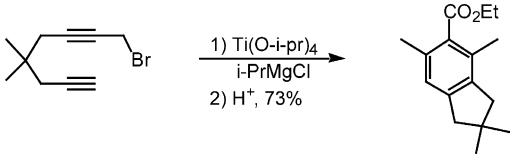
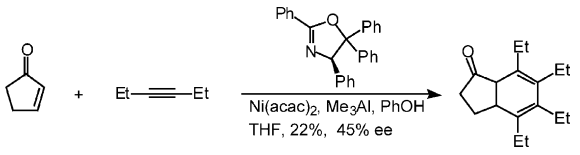


(223)

Polysubstituted benzenes were prepared via nickel-catalyzed chemo- and regioselective formal [2 + 2 + 2]cycloaddition of electron-deficient 1,6- and 1,7-diynes with allenes (Eq. (224) [1180]. Nickel catalyzed cyclotrimerizations of nonconjugated diynes with 1,3-diynes [1181], of dienetriynes to give helicenes [1182], and of 1,6-diynes with carbon dioxide (Eq. (225)) [1183]. A nickel-catalyzed asymmetric intermolecular [2 + 2 + 2]cycloaddition of an alkene and two alkynes was reported (Eq. (225)) [1184].



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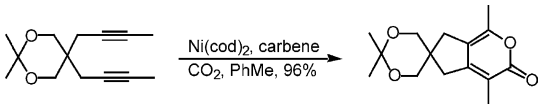

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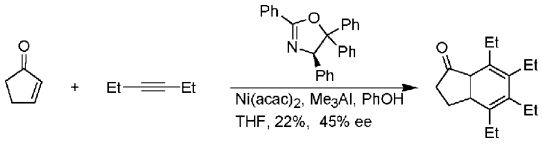
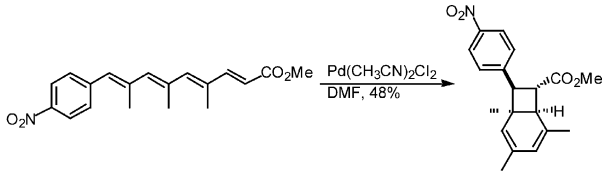


(226)

A palladium-catalyzed cycloisomerization of 1,3,5,7-tetraenes was used in the synthesis of the core of SNF4435 C and D (Eq. (227)) [1185]. Rhodium catalyzed an intermolecular [4 + 2 + 2]cycloaddition of 6-ene-1-ynes with conjugated dienes forming eight-membered rings (Eq. (228)) [1186]. A related reaction of dienynes with alkynes was also described (Eq. (229)) [1187]. A cobalt-catalyzed [4 + 2 + 2]cycloaddition of norbornadienes with butadiene was reported [1188].



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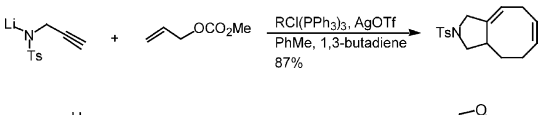

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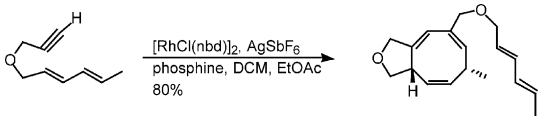
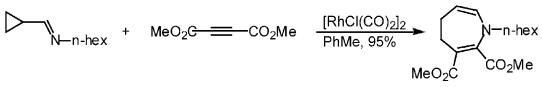


(229)

Nickel catalyzed a [3 + 2]cyclodimerization and linear dimerization of ethyl cyclopropylidenacetates [1189]. Rhodium catalyzed the intramolecular [5 + 2]cycloaddition of alkynes-tethered vinylcyclopropanes to give [5.3.0]ring-systems [1190]. A rhodium-catalyzed hetero[5 + 2]cycloaddition of cyclopropyl imines with alkynes was also described (Eq. (230)) [1191]. Rhodium catalyzed a [5+2+1]cycloaddition of vinylcyclopropanes with alkynes and carbon monoxide to give bicyclo[3.3.0]ring-system [1192]. Rhodium catalyzed a [4 + 2]annulation of 4-alkynals with alkynes to give cyclohexenones (Eq. (231)) [1193].



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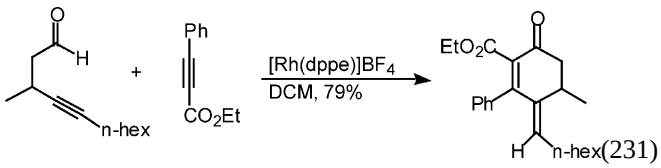
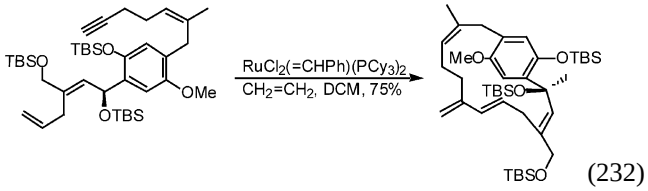


(231)

An in situ generated ruthenium catalyst was used for cycloisomerization of terpenoid derived 1,6-enyne ethers to 3-vinyl-2,5-dihydrofuranes [1194]. Ruthenium-catalyzed 1,6- and 1,7-enyne ring-closing metathesis reactions were reported forming conjugated vinylcyclohexenes [1195], fused indoles [26], and chiral oxacyclic dienes [1196]. This enyne cycloisomerization was used to prepare (−)-longithorone A (Eq. (232)) [189] and cyclic ethers [1197], and a rhodium-catalyzed reaction was used in a synthesis of (+)-pilocarpine (Eq. (233)) [1198]. Ruthenium catalyzed ene–yne ring-opening ring-closing metathesis reactions [1199]. Rhodium and iridium catalyzed intramolecular allenic Alder-ene reactions to give cross-conjugated trienes (Eq. (234)) [1200]. Palladium-catalyzed 1,6-enyne cycloisomerization were used in a synthesis of cyclic farnesyl analogs [1201] and kelsoene (Eq. (235)) [1202], and the corresponding 1,7-enyne cycloisomerization was used in a synthesis of 7-*O*-methyldehydropinguisenol [1203]. Phthalides and 3,4-dihydroisocoumarins were prepared by intramolecular palladium-catalyzed enyne–enyne benzannulation [1204]. Titanium-mediated cyclization of 1-carboethoxy-1,6-enynes was used to prepare chiral bicyclic ketones (Eq. (236)) [1205]. A variety of transition metals catalyzed the cyclization of 1-silyl- or 1-stannyl-2-ene-7-ynes to give 1-vinyl-2-methylene substituted cyclopentanes [1206]. Platinum catalyzed cyclizations of dienynes to form a variety of complex carbocycles (Eq. (237)) [1207].



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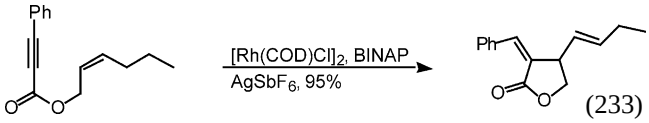

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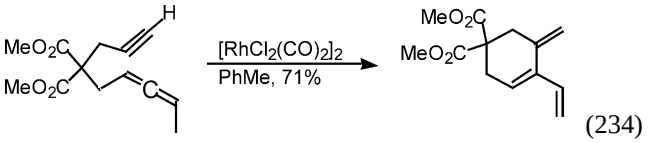

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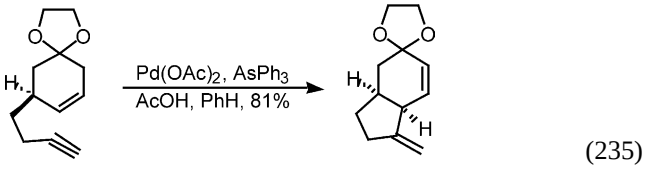

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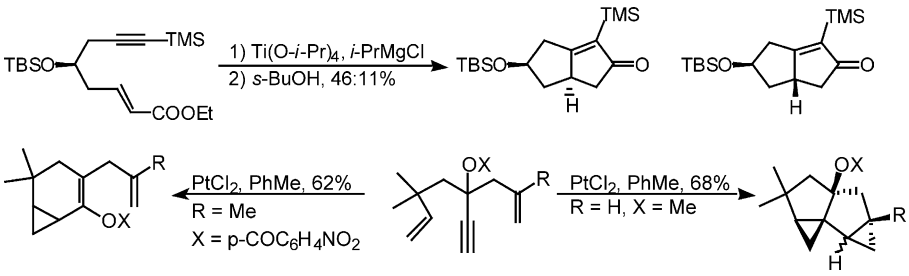

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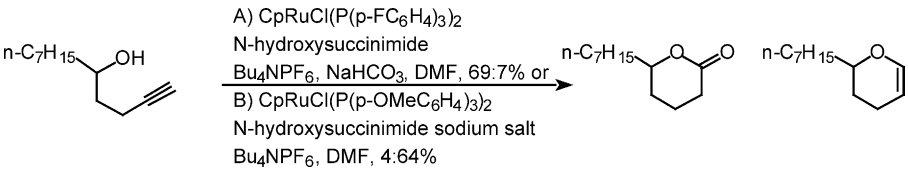
Depending on the catalyst, either oxidative cyclization or cycloisomerization of 5-hydroxy-1-alkynes can be obtained (Eq. (238)) [1208]. A computational study of the tungsten-catalyzed cycloisomerization of 5-hydroxy-1-alkynes was published [1209]. Rhodium catalyzed the cycloisomerization of 8-hydroxy-1,6-diynes and 9-hydroxy-1,7-diynes to give dienylketones (Eq. (239)) [1210]. Rhodium catalyzed the cycloisomerization of 1,2,7-trienes to give cyclic 1,3- or 1,4-dienes [1211]. Iron catalyzed a cyclization of 1,3,8-trienes to give a vinyl-substituted cyclopentane, and this was used in a synthesis of (−)-gibboside (Eq. (240)) [1212]. Zirconocene mediated the cyclization of 1,3,6-heptatrienes to form predominantly *trans*-1,2-dimethylcyclopentenes [1213].



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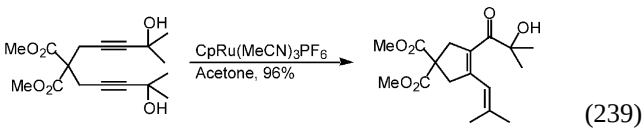

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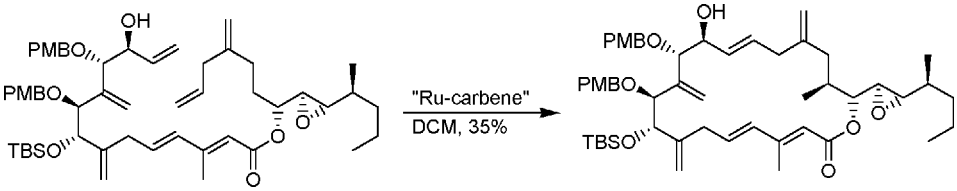

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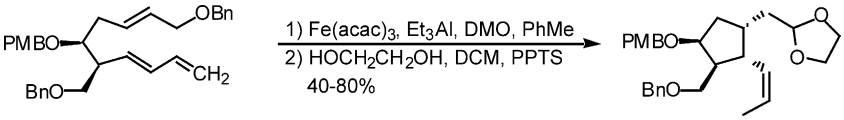

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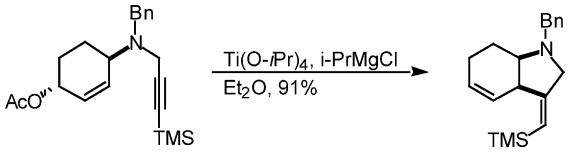
Titanium mediated the formation of carbo- and azbicyclo[4.3.0] and -[4.4.0] compounds from 2,7- or 2,8-enynyl-1-ol acetate or phosphonate derivative (Eq. (241)) [1214]. Gold catalyzed a benzannulation of 2-(1-alkynyl)benzaldehydes with alkynes to give naphthyl ketone derivatives (Eq. (242)) [1215]. An iridium-catalyzed benzannulation of aroyl chlorides with internal alkynes forming substituted naphthalenes and anthracenes was reported (Eq. (242)) [1216]. Gallium catalyzed the cycloisomerization of 5-aryl-1-alkynes to give naphthalene derivatives [1217]. A ruthenium-catalyzed annulation of enynes with alkenes to give cyclohexadienes was developed [1218].



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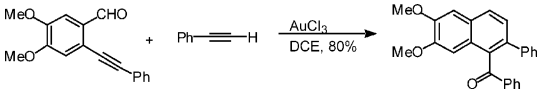

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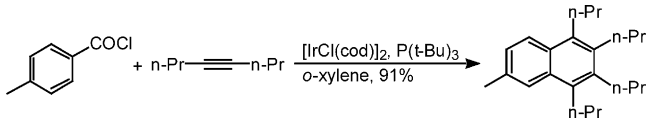
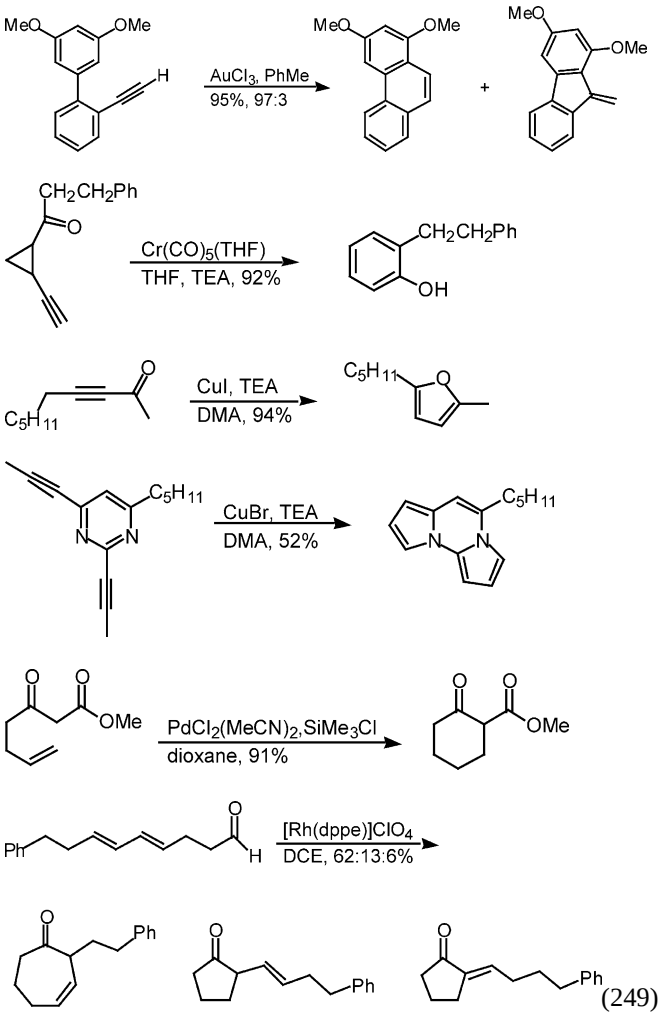


(243)

Gold and a number of other transition metals catalyzed the cycloisomerization of 2-alkynyl substituted biphenyls to afford phenanthrenes (Eq. (244)) [1219]. Chromium mediated the cycloisomerization of *cis*-1-acyl- or -1-vinyl-2-ethynylcyclopropanes to give phenols and cycloheptatrienes, respectively (Eq. (245)) [1220]. A tungsten-catalyzed cycloisomerization of a 5-hydroxy-1-alkyne to give a dihydropyran was used in a synthesis of vancosamine and saccharosamine [1221]. Ruthenium catalyzed the cycloisomerization of epoxyalkynes to give furanes [1222]. A

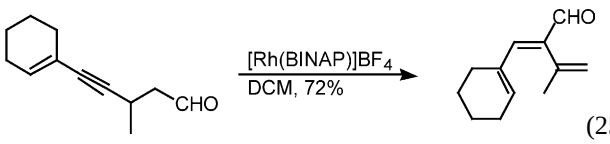
copper-catalyzed cycloisomerization of alkynyl-substituted ketones to give substituted furanes was developed (Eq. (246)) [1223]. Copper-mediated double cycloisomerization of bis-alkynylpyrimidines was used as a key step in a synthesis of tetraponerine T6 (Eq. (247)) [644]. Palladium catalyzed the cycloisomerization of 6-alkenyl-3-ketoesters to give substituted cyclohexanones (Eq. (248)) [1224]. Rhodium catalyzed an intramolecular hydroacylation of 4,6-dienals to give cycloheptenones as the major product (Eq. (249)) [1225]. Sulfur-containing cyclohepta- and cyclooctanones were prepared in a similar fashion [1226].



(249)

2.5.2. Alkene and alkyne isomerizations

A palladium–imidazolium system catalyzed a regio- and stereoselective reaction of terminal alkynes forming 1,3-enynes [1227]. Grubb's catalyst was used in alkyne–alkyne dimerization to give conjugated enynes [1228,1229]. A new rhodium-catalyzed rearrangement of 4-alkynals to 3-formyl-substituted-1,3-dienes was reported (Eq. (250)) [1230].



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Alkene–alkyne couplings (Alder-ene reactions) to give 1,4-dienes were catalyzed by ruthenium [1231,1232]. Substituents were shown to have a significant influence on product formed from the ruthenium-catalyzed Alder-ene reaction (Eqs. (251) and (252)) [1233]. Ruthenium-catalyzed alkyne–ethene cross-metathesis [1234] and ene–yne cross-metathesis of sulfur-substituted alkynes with ethene [1235] to give dienes were described. Ruthenium-catalyzed Alder-ene reactions were used in the synthesis of *cis*-solamine [1236], callipeltoside A [103], anolignans A and B

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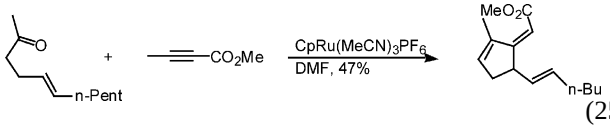
(Eq. (253)) [1237], the proposed structure of amphidinolide A (Eq. (254)) [1238], and of trisubstituted alkenes with defined geometry [1239]. A tandem ruthenium-catalyzed Alder-ene reaction followed by an intramolecular asymmetric amino- or alkoxypalladation was used to prepared a variety of heterocyclic compounds (Eq. (255)) [1240]. Rhodium-catalyzed intermolecular asymmetric Alder-ene reactions were reported [1241,1242]. Chromium catalyzed asymmetric hetero-ene reactions of aldehydes with enol ethers [1243]. Ene–yne coupling using a cobalt catalyst was described (Eq. (256)) [1244].



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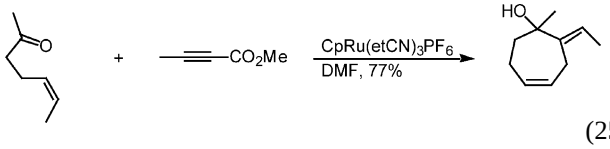

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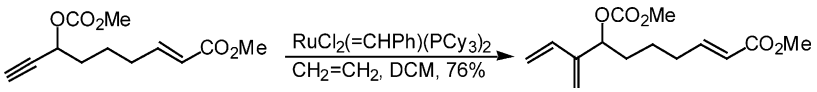

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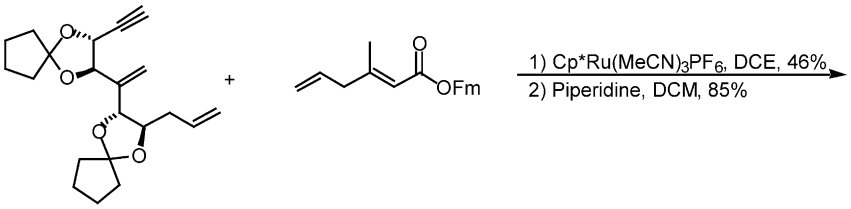

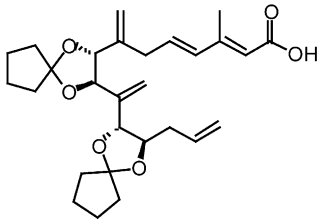

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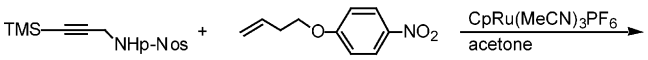

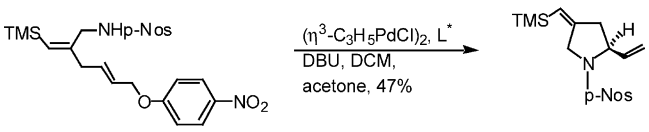

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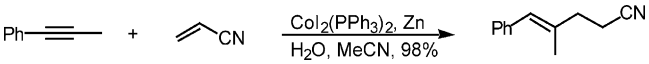
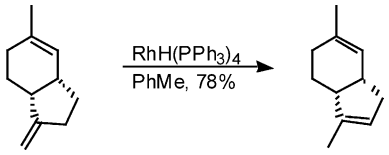


(256)

Rhodium-catalyzed double bond isomerizations were used in syntheses toward stachyflin [1245] and naturally occurring lactones (Eq. (256)) [1246]. Rhodium catalyzed the isomerization of allylic ethers to vinyl ethers [1247] and this reaction was used in a synthesis toward ciguatoxin [1248]. Ruthenium catalyzed the isomerization of allylic to vinylic ethers and allylic amines to enamines [1249]. Palladium catalyzed the isomerization of a 3-phenyl-1-propene to a 1-phenyl-1-propene [1250]. Iron catalyzed the isomerization of allylic amides to enamides [1251]. A polymer-bound iridium catalyst was used for isomerization of allyl- to vinylbenzenes and of allyl ethers to vinyl ethers [1252]. An iridium-catalyzed isomerization of an allylic ether to an enol ether was used in a synthesis of sphingofungin E [352]. A ruthenium-catalyzed exo- to endo-cyclic enol ether isomerization was used in a synthesis toward gambierol [187]. Isomerization of terminal alkenes to internal alkenes was achieved using a ruthenium carbene catalyst in the presence of an enol ether [1029]. Palladium catalyzed the *cis-trans* isomerization of styrenes [1253]. Iridium-catalyzed isomerization of an allylic alcohol to an aldehyde was used in a synthesis of the F-ring of halichondrin B [1254]. Ruthenium catalyzed the isomerization of propargylic alcohols to α,β-unsaturated ketones [1255].

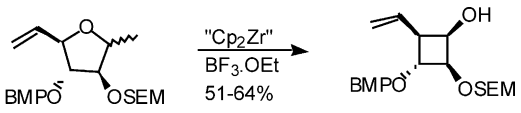


(257)

2.5.3. Rearrangements of allylic and propargylic compounds

Palladium catalyzed an enantioselective rearrangement of *O*-allylic thiocarbamates to *S*-allylic thiocarbamates [1256]. Palladium catalyzed the rearrangement of a 5-vinyloxazolinone to a 5-vinyloxzolidine, and this was used in a synthesis of sphinganines [354]. Palladium also catalyzed the asymmetric rearrangement of allylic imidates to give allylic amides [1257]. Palladium-catalyzed 1,3-allylic acetate transposition was used in the synthesis of prostaglandin E_1 methyl ester [1258], (+)-quebrachamine [1259], and achilleol [1260]. Palladium catalyzed the rearrangement of silyl-substituted vinyl epoxides to silylated unsaturated aldehydes which can be further functionalized [1261]. Palladium-catalyzed isomerization of vinyl epoxides to dienols was used in the synthesis of a Vitamin D_3 building block [1262] and as an entry to the ingenane diterpenes [1263]. A diastereoselective, zirconium-mediated

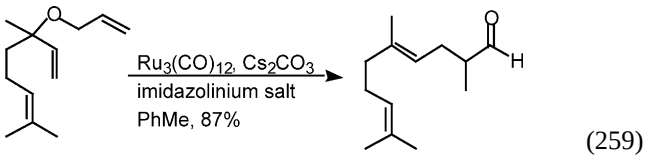
ring-contraction of 4-vinylfuranosides to cyclobutanes was described (Eq. (258)) [1264].



(258)

2.5.4. Skeletal and miscellaneous rearrangements

Rhodium catalyzed a diastereoselective ester enolate Claisen rearrangement [1265]. A tandem isomerization-Claisen rearrangement was reported using a ruthenium catalyst (Eq. (259)) [1266,1267]. Platinum in the presence of trialkylsilane catalyzed the ring-opening isomerization of methylenecyclopropanes to give 1,3-dienes [1268]. Palladium-catalyzed the ring-expansion of hydroxy methoxyallenyl-substituted compounds (Eq. (260)) [1269]. Cobalt mediated the rearrangement of 4-isoxazolines to acylaziridines (Eq. (261)) [1270]. Cobalt also mediated the rearrangement of alkynyl-substituted cyclic enol ethers to ketones (Eq. (262)) [1271,1272]. Ruthenium catalyzed the rearrangement of 3-benzyl-1-butynyl ethers to give 1,3-dienes and benzaldehyde (Eq. (263)) [1273].



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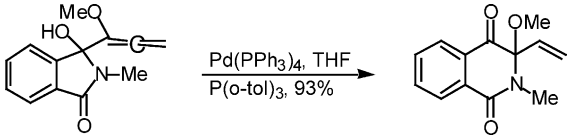

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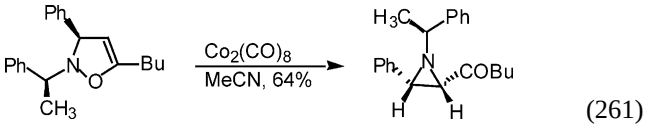

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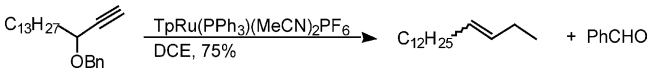
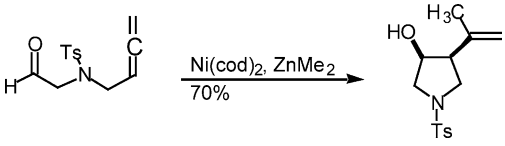


(263)

3. Miscellaneous cyclizations

A nickel-catalyzed cyclization of 5,6-dieneals in the presence of dialkyl zinc reagents furnished *cis*-1-hydroxy-2-vinylcyclopentanes (Eq. (264)) [1274], and this reaction was used in a synthesis of (+)-testudinarol A (Eq. (265)) [1275]. Nickel catalyzed the reaction of allenyl–aldehydes and -ketones with organozinc reagents to give homoallylic cyclopentanols and related five-membered heterocycles (Eq. (266)) [1276]. Related reactions of 6,8-dien-1-ynes with aldehydes and dimethylzinc in the presence of a nickel catalyst to give polyfunctionalized five-membered rings were reported [1277]. A nickel-catalyzed cyclization of a 8-yne-2-enamide in the presence of dimethylzinc was used in the synthesis of the isogeissoschizoid skeleton (Eq. (267)) [1278]. Stryker's reagent, $(Ph_3PCuH)_6$, catalyzed the reductive coupling of 7-oxo-2,8-dienylesters to give substituted cyclohexanones (Eq. (268)) [1279]. Reaction of 2,7-dien-1-ones, 2-ene-7-yne-1-ones, and 2-yne-7-en-1-ones with acylzirconocenes in the presence of a palladium catalyst and a Lewis acid gave stereoselectively bicyclo[3.3.0]compounds (Eq. (269)) [1280]. Palladium catalyzed the cyclization of allene–yne substituted β-lactams to give tricyclic compounds (Eq. (270)) [880]. Annulation of 1,6-enyne mediated by allylic chromate or magnesium species was reported (Eq. (271)) [1281]. Nickel catalyzed an addition–cyclization of 2-iodobenzaldehydes to give optically active phthalides (Eq. (272)) [1282].



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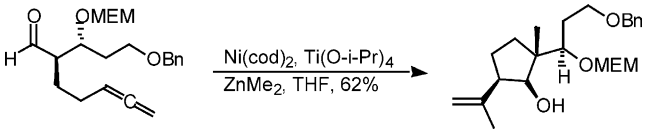

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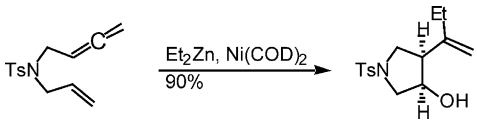

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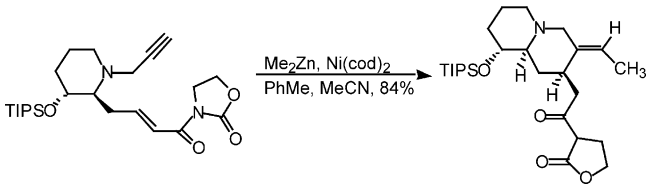

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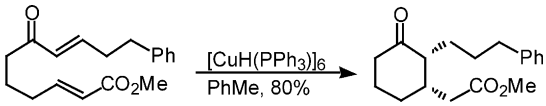

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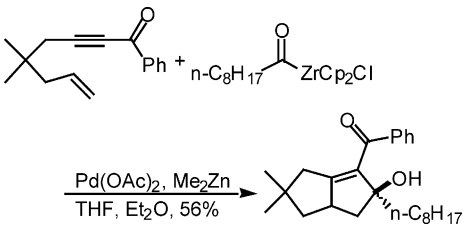

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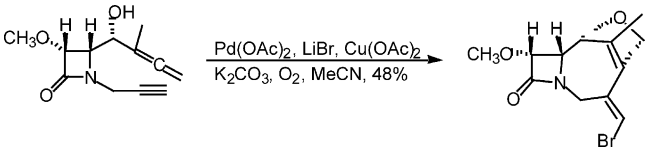

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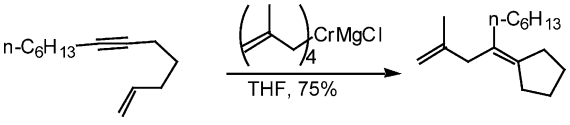

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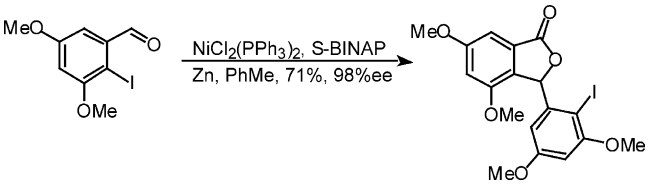
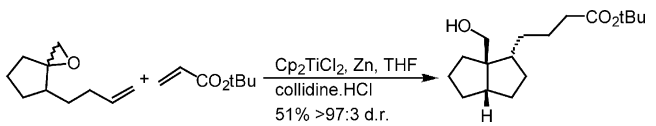


(272)

A titanocene-catalyzed anullation-Michael addition of epoxyalkynes with α,β-unsaturated substrates was developed (Eq. (273)) [1283]. A titanium-mediated intramolecular coupling of vinyl ethers with pendant alkene or alkyne followed by an electrophilic quenching was described (Eq. (274)) [1284]. Palladium catalyzed the cyclization of alkynes having a pendant aldehyde, ketone, or nitrile functionality. An initial acetoxypalladation was suggested (Eq. (275)) [1285]. Palladium catalyzed the formation of indene derivatives form acetals of 2-(1-alkynyl)benzaldehydes (Eq. (276)) [1286]. Palladium catalyzed the cyclization–alkylation of alkynes having a tethered stabilized carbanion in the presence of an allylic halide or ester (Eq. (277)) [1287]. A cyclization-coupling reaction of conjugated enynes having a tethered stabilized nucleophile was described (Eq. (278)) [1288]. Palladium catalyzed the formation of polycyclic compounds from 2-iodobenzyl propargyl ethers and trimethylsilyl ethyne (Eq. (279)) [1289].



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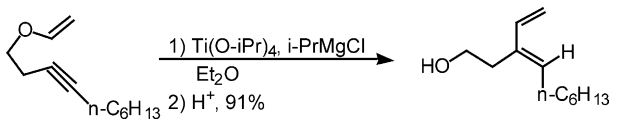

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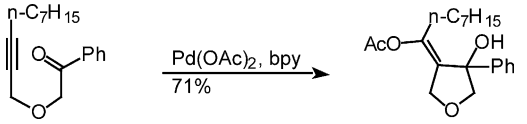

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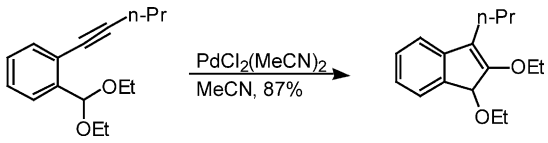

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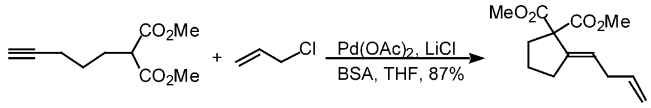

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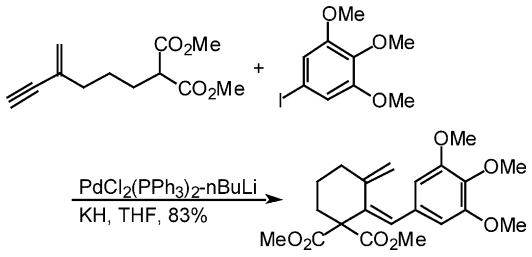

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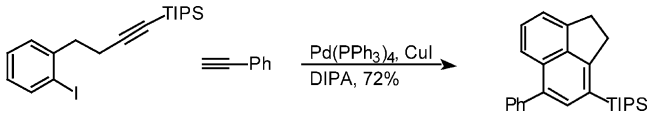

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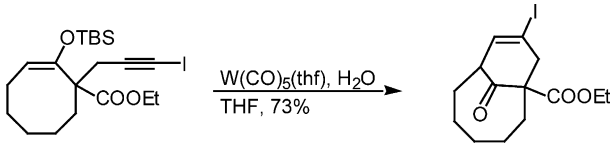
Tungsten catalyzed the cyclization of silylenol ethers tethered 1-iodo-5-en-1-ynes to give five- or six-membered rings (Eq. (280)) [1290]. Tungsten catalyzed the annulation of 7-silyloxy-6-en-1-ynes (Eq. (281)) [1291] and 6-silyloxy-5-en-1-ynes [1292] to give polycyclic compounds. Ruthenium catalyzed a cycloaddition of propargylic alcohols with phenol derivatives (Eq. (282)) [1293]. A palladium-catalyzed [3 + 2]cycloaddition of trimethylenemethane to an alkene was used in a synthesis of (+)-brefeldin A (Eq. (283)) [1294]. Indium mediated an intramolecular alkylation of allylic bromides having a pendant terminal alkyne (Eq. (284)) [1295].



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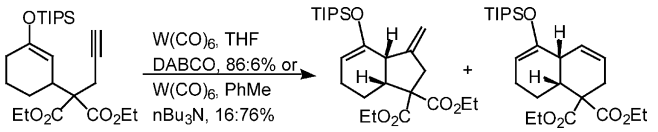

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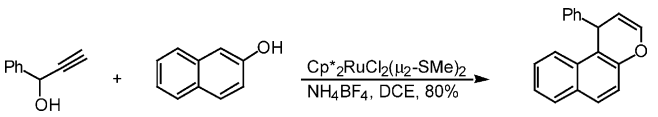

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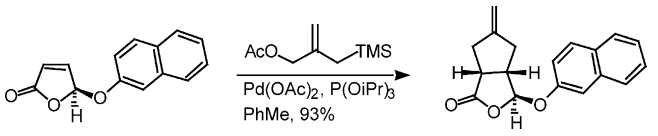

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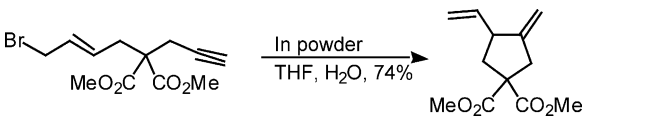

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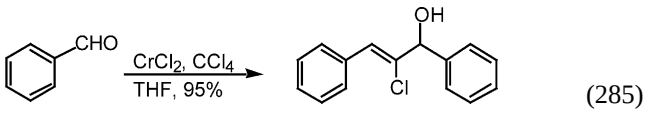
4. Functional group preparation

4.1. Halides, amides, nitriles, azides, imines, ureas, and amidines

Vinyl iodides were prepared by the chromium(II)-mediated Takai olefination of aldehydes using iodoform. This reaction was used in the synthesis of mucocin [658], rhizoxin D [64], callipeltoside A [103], (−)-motuporin [195], pseudorubrenoic acid A analog [674], sphingofungin E [352], and toward superstolide [194]. An interesting variation using tributyltindiiodomethane introducing a vinyl stannane was employed in the synthesis of (+)-crocacin D [68]. Chromium mediated the reaction between carbon tetrachloride or bromide with two molecules of aldehyde forming 2-halo-2-(*Z*)-en-1-ols (Eq. (285)) [1296]. A related reaction of trichloromethylcarbinols with chromium in the presence of an aldehyde was also described (Eq. (286)) [1297].



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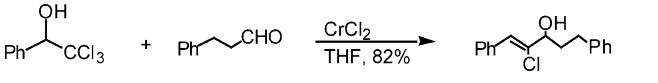
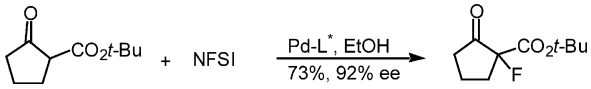


(286)

An enantioselective fluorination of β-ketoesters using a palladium complex was described (Eq. (287)) [1298]. Copper catalyzed the formation of aryl and vinyl iodides from aryl and vinyl bromides, an aromatic Finkelstein-type reaction (Eq. (288)) [1299]. Copper also catalyzed the formation of mono- and diahaloalkenes from aromatic hydrazones (Eq. (289)) [1300]. Rhodium catalyzed the decomposition of iodonium ylids in the presence of benzyl and acid halides to give β-substituted α-haloenones (Eq. (290)) [1301]. Ruthenium catalyzed a three-component coupling of a terminal alkyne, an α,β-unsaturated ketone, and an halide to give vinyl halides [1302]. A palladium-catalyzed bromo- or chloro-allylation of alkynes with allyl bromides and chlorides affording 1-bromo (or chloro)-1,4-dienes was described [1303]. This reaction was used in one pot tandem with Wacker oxidation, Sonagashira, or Suzuki couplings (Eq. (291)) [1304].



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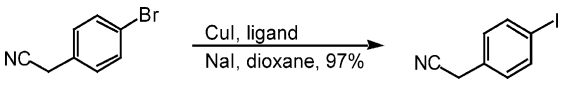

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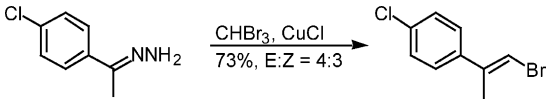

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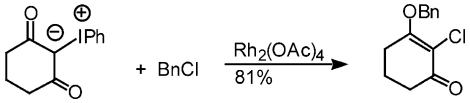

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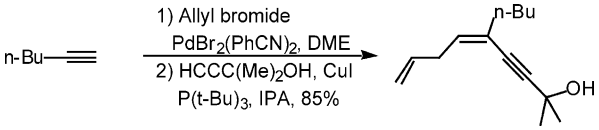

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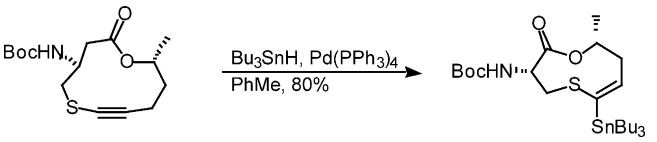
Hydrozirconation of a terminal alkynes followed by addition of iodine to produce vinyl iodides was used in synthetic applications toward *trans*-epothilone A [190], halicholactone [1079], CP-molecules [1305], fostriecin [1306], and C292 and hypothemycin [294]. Hydrozirconation of a silyl-substituted alkyne followed by addition of iodine produced a silyl-substituted vinyl iodide that was used in the synthesis of C1–C14 fragment of discodermolide [1307].

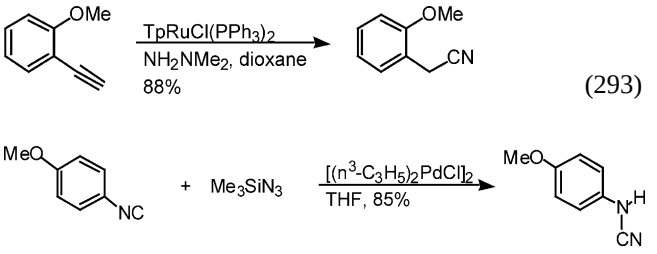
Methylzirconation of a terminal alkyne using trimethyl aluminum and $ZrCp_2Cl_2$, followed by treatment with iodine to give a vinyl iodide, was used in synthesis of (−)-bafilomycin A [283], bafilomycin V_1 [56], β-carotene and (3*R*,3*R*′)-zeaxanthin [58], rhizoxin D [64], coenzyme Q_{10} [182], vicenistatin [286], (+)-curacin A [288], and fragments of aurisides and callipeltosides [1308]. Palladium-catalyzed hydrostannation–iodination of a terminal alkyne

was used in the synthesis of callystatin A [348], toward orevactaene [266]. A regioselective hydrostannation–iodination of a cyclic alkyne was used as an approach to griseoviridin (Eq. (292)) [894]. A related regioselective reaction was used to prepare a subunit of callystatin A [664].



(292)

Palladium catalyzed the cyanation of aromatic halides with zinc dicyanide [1309], and of a 2-bromoquinoline derivative with copper cyanide in the synthesis of luotonin [1310]. Nickel catalyzed the cyanation of vinyl triflates [1311]. Ruthenium catalyzed the formation of nitriles from terminal alkynes and hydrazine (Eq. (293)) [1312]. A palladium-catalyzed azidation of allylic acetate was used in a synthesis of the C_{11}–C_{26} fragment of griseoviridin [1313]. Cyanamides were prepared via palladium-catalyzed reaction of isonitriles with TMS-azide (Eq. (294)) [1314].



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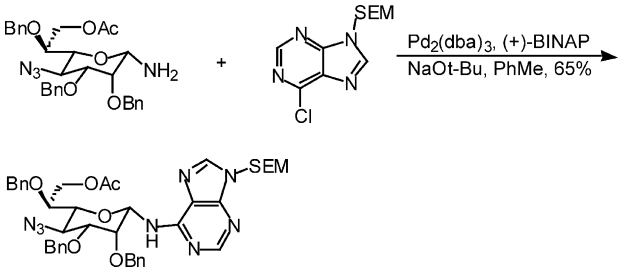
4.2. Amines and amides

A variety of aryl halides and triflates were aminated or amidated using palladium or copper catalysts. Aromatic chlorides were aminated using nickel- and palladium-based catalyst system [223,1315,1316]. The mechanism of the palladium-catalyzed amination reaction was probed under synthetically relevant conditions [1317]. The use of air-stable ferrocenyl dialkylphosphines as ligands [218], alkalimetal hydroxides and Pd(P*t*-Bu_3)$_2$ [1318], an air-stable palladium-*N*-heterocyclic carbene complex [1319], palladium and bulky, electron-rich ligand [1320], palladium nanoparticles [1], a arylpalladium halide phosphine complex [201], and palladium on KF-alumina surface were used [1321]. Microwave-induced palladium-catalyzed aminations of aryl bromides were described [1322]. Palladium catalyzed the amination of aryl chlorides and bromides with boronic acids in minutes at ambient temperature [1323]. Palladium-catalyzed amination of aminostannanes was used in the synthesis of 7-aminotetracyclines [1324].

Palladium catalyzed the *N*-arylation of 2-aminopyridines [1325], polymer-supported amines [1326], aminosugar in the synthesis of spicamycin (Eq. (295)) [1327], a 2-chloroquinazoline derivative in the synthesis of iodoazidoaryl prazosin [1328], pyrroles, indole, carbazole, and phenothiazine with vinyl bromides [1329], aminopyridins, 2-aminopyrimidine, 2-aminopyrazine, an aminothiophene, 2-aminothiazoles, 2-amino-1,3,4-thiadiazole, and 2-aminobenzimidazole [1330], (2S,5S)-2,5-dimethylpiperazine [1331], cyclam, cyclen, and azacrown ethers [1332], aryl benzophenonehydrazones [1333], sequential *N*-arylation of piperazine [1334], *O*-methylamidoximes (Eq. (296)) [1335], and benzophenone imine [1336]. Palladium catalyzed the sequential amination intramolecular Heck-type reaction to give carbazolles [1337]. Palladium-catalyzed amination was used in the synthesis of the kappa-agonist CJ-15,161 [1338].



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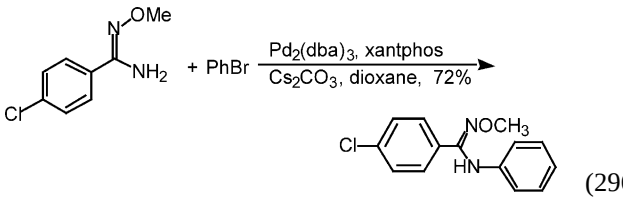

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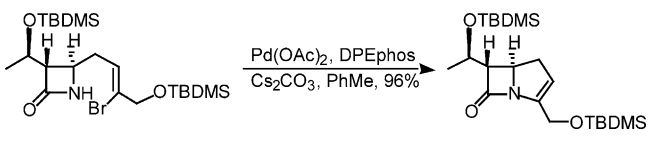
Aminations of 8-bromo-2′-deoxyguanosine [1339], polymer-bound chloro-*N*-heterocycles [233], 3-iodo-6-arylpyridazines [315], and 2-chloro-3-iodo- and 2-chloro-5-iodopyridine [1340] were described. Enamines were prepared from alkenyl bromides and triflates and secondary amines [1341,1342]. Amidations of aryl halides using palladium and copper catalysts were reported [1343,1344]. Intramolecular palladium-catalyzed amidation was used to prepare 3-alkoxycarbonyl-1β-methylcarbapenems (Eq. (297)) [1345], benzimidazoles (Eq. (298)) [1346], indoles on solid support [443]. A related intramolecular amination was used to prepare 2-substituted indolines [1347]. Intramolecular aminations using a hydrazine group was used to prepare indolo[1,2-*b*]indazines (Eq. (299)) [1348].



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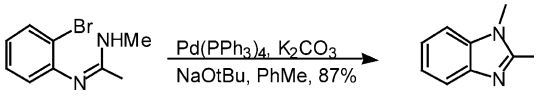

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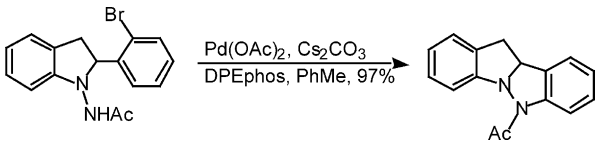
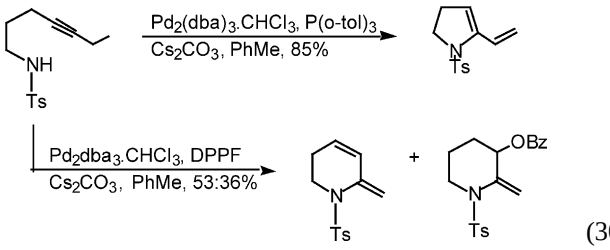


(299)

Transition metals other than palladium, mainly copper, also catalyzed amination reactions. Copper-catalyzed aminations and amidations of aromatic and heteroaromatic halides using *N*-heterocycles, amines, and amides [739,1349–1353] and amidation of diaryiodonium salts [1354] were described. Copper-catalyzed amidations of aryl bromides were accelerated by microwave irradiation [1355]. Copper catalyzed the reaction of β-aminoalcohols with aryl iodides [1356]. Copper catalyzed intramolecular aminations of amino tethered aromatic halides [1357]. Nickel catalyzed the amination of aryl chlorides [1358]. Copper promoted the amination of boronic acids with ethyl pyrrole-3-glyoxalate [1359]. Copper mediated couplings of amides with phenyl trimethyltin [1360]. Copper mediated the coupling between *N*-stannyl-5-substituted tetrazoles and diaryliodonium salts [1361]. Palladium and copper-catalyzed regioselective 2-arylation of 5-aryltetrazoles and diaryl iodonium salts [1362]. Rhodium catalyzed the formation of spirocyclic heterocycles by reaction of indolyl-substituted carbamates with iodobenzene diacetate [1363].

Palladium catalyzed a variety of aminations of allylic substrates via η^3-allyl intermediates. A dendrimer-bound catalyst was used [1364]. A regio- and stereoselective allylic amination of α-fluoroalkyl substituted allylic mesylates to give γ-fluoroalkyl substituted amines was reported [1365]. Palladium catalyzed the direct allylic amination of allylic alcohols [1366]. The ring-size obtained from intramolecular palladium-catalyzed amination of propagylic esters can be controlled by the choice of ligand (Eq. (300)) [1367]. Palladium-catalyzed allylic amination was used to prepare 4′-ethoxy-2′,3′-didehydro-2′,3′-dideoxynucleosides [1368], carbocyclic nucleosides [1051,1369], aminocyclitols [1370], α-dehydro-β-amino acid esters [1371], carbocyclic nucleoside analogs [869], 1,2,4-trisubstituted pyrroles (Eq. (301)) [1372], and tetrahydrocarbazoles [1373]. Palladium catalyzed the double amination of 1,4-dihydoxy-2-butene affording 2-vinyl-1,2,3,4-tetrahydroquinoxalines [1374]. An intramolecular palladium-catalyzed allylic amination forming a cyclic urea was used in a synthesis of (+)-biotin [1375].



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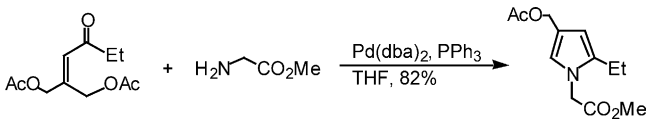
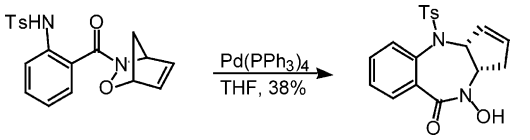


(301)

A palladium-catalyzed regio- and diastereoselective cyclization of allylic *N*-tosylcarbamate was used in the synthesis of 1,4-dideoxy-1,4-imino-L-xylitol [1376]. A palladium-catalyzed regio- and stereoselective allylic amination was used in synthesis of (+)-dihydrocuscohygrine and cuscohygrine [1119]. Intramolecular enantioselective allylic aminations were used to prepare vinyl-substituted 2-oxazolidinones, 2-imidazolidinones, and 2-pyrroliinones [1377], indolopyrrolocarbazolles [1378], and tetrahydroquinoline derivatives [1379]. Rhodium catalyzed an enantioselective asymmetric allylic amination of azabicyclic alkenes to give diamines [1380]. Iridium catalyzed an regio- and enantioselective allylic amination of allylic esters [1381]. An interesting synthesis of 1,4-benzodiazepines via ring-opening intramolecular amination was developed (Eq. (302)) [1382]. An iron-catalyzed allylic amination of alkenes using nitroarenes was described (Eq. (303)) [1383].



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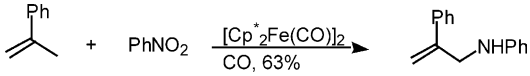

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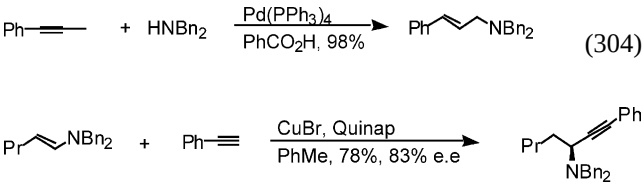
Titanium catalyzed an intermolecular hydroamination of 1,1-disubstituted hydrazines with alkynes affording hydrazones [1384] and of alkynes with primary amines [1385]. Anti-Markovnikov hydroaminations of terminal alkynes catalyzed by titanium was reported [1386]. Titanium-catalyzed intermolecular hydroaminations of alkynes using secondary amines [1387,1388] and intramolecular hydroaminations of amino-tethered 1,2-allenes [1389] and alkynes [1390–1392] were reported.

Palladium- and rhodium-catalyzed intramolecular oxidative amination of alkenes to give five-membered heterocycles was described [1393,1394]. Dramatic rate enhancement was observed using 2-hydroxybenzenamine in intermolecular palladium-catalyzed hydroaminations of alkynes [1395]. Nickel catalyzed intermolecular hydroaminations of 1,3-dienes [1396]. An interesting palladium/benzoic acid-catalyzed amination of internal alkynes forming allylic amines was developed. Intramolecular reactions furnished 2-alkenyl-substituted tetrahydropyrroles and piperidines (Eq. (304)) [1397]. Copper catalyzed the asymmetric addition of alkynes to enamines forming propargylic amines (Eq. (305)) [1398].



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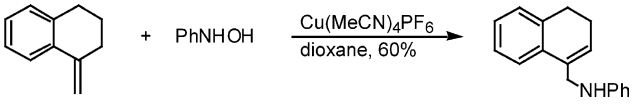
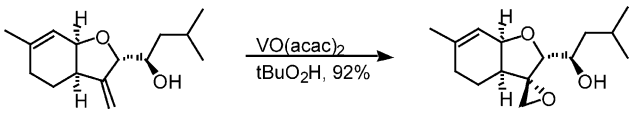
Diastereoselective yttrium- and neodymium-catalyzed intramolcular hydroaminations were reported using chelating diamine ligands [1399]. The mechanism of the palladium-catalyzed hydroamination of alkenylarenes with benzenamines was examined and η^3-benzyl complexes were suggested as intermediates [1400]. Copper catalyzed an amino-palladation elimination sequence forming allylic amines (Eq. (306)) [1401]. Palladium catalyzed the formation of indoles from 2-(1-alkenyl)benzenamines via an amino-palladation elimination sequence [127].



(306)

4.3. Alcohols, ethers, esters, lactones, and acids

Reaction of terminal alkynes with trimethylaluminum and a catalytic amount of zirconocene dichloride followed by reaction with formaldehyde was used in a synthesis of LY426965 [1402]. Vanadyl acetylacetonate-catalyzed epoxidations of allylic and in some cases homoallylic alcohols using peroxides continued to be an invaluable tool in organic total synthesis. Synthetic applications include (+) cheimonophyllon E and (+)-cheimonophyllal (Eq. (307)) [1403], kedarcidin chromophore aglycon (Eq. (308)) [677], pectenotoxins-4 and -8 [1404], phomactin A [1405], the ABC-ring-system of paclitaxel [1406], and (−)-semburin and (−)-isosemburin [1407]. Microencapsulated $VO(acac)_2$ was used in allylic epoxidations [1408]. Asymmetric epoxidation of alkenes using $VO(OiPr)_3$ and a chiral ligand was described [1409].



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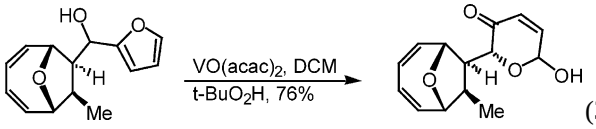
Vanadyl acetylacetonate-catalyzed epoxidations-rearrangement of furyl alcohols to give pyranones was used in synthesis [1410–1413] of fumagillin analogs [1414] and (+)-isoaltholactone [1415]. A related reaction was used in a synthesis of mycoepoxydiene (Eq. (309)) [1062]. Epoxidation of alkenes in the presence of a tethered phenol resulted in the formation of five- and six-membered *O*-heterocycles [1416]. Palladium catalyzed the epoxidation of alkenes using *t*-butylhydrogen peroxide [1417]. Epoxides were prepared from aldehydes using tosylhydrazone salts and rhodium catalysts [1418,1419].



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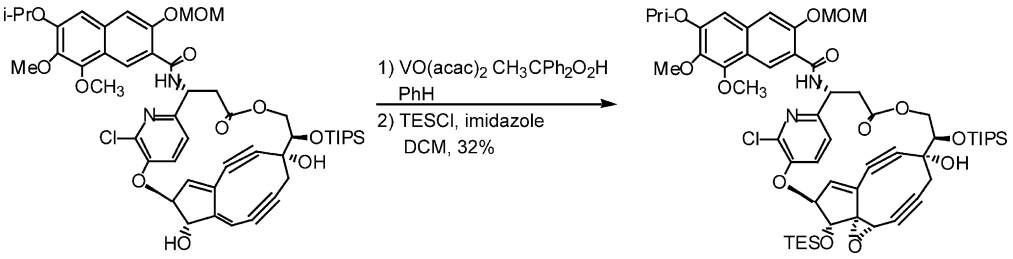
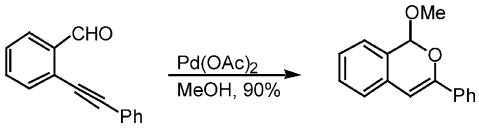
Sharpless enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continued to be extensively used in organic synthesis. For example, this reaction was used in synthetic approaches to the ABC tristetrahydropyran of thyrsiferol and venustatriol [1420], ancistrocladidine [1421], amphidinolide A [1061], the ABCDEF-ring-system of yessotoxin and adriatoxin [1422], fostriecin [1070], C15–C18 fragment of laulimalide [1073], pectenotoxins-4 and -8 [1404], leucascandrolide A [764], neocarzinostatin chromophore [1423], (−)-laulimalide [1007,1082], (+)-methynolide [1424], an EP2-recetor agonist [1425], epothilone A [1426], (−)-gambierol [79], chroman-2-ylmenthol [1427], okilactomycin [1094], (20*R*)-homocamptothecin [108], cigautoxin CTX3C [1044], (+)-tanikolide and (−)-malyngolide [1100], strongyldiol A [1428], kalihinene X [1429], (*S*)-dimethyl-2-oxo-6-octene-1,3-diol [1430], oxygenated taxane precursors [1431], C292 and hypothemycin [295], to pumilotoxin analogs [1432], (+)-lactacystin [1433], and muconin [1434]. Epoxidation using polymer-bound tartrates was reported [1435]. A renewable tertiary furyl hydroperoxide was used in asymmetric allylic epoxidations [1436].

Palladium catalyzed the oxidation of alkenes using *t*-butylhydrogen peroxide to give allylic perethers and α,β-unsaturated ketones [1417]. A palladium-catalyzed annulation of alkyne tethered aldehydes to give cyclic alkenyl ethers was developed (Eq. (310)) [1437]. Palladium catalyzed the formation of ethers using polymer-bound



(308)

chloro-*N*-heterocycles [233].



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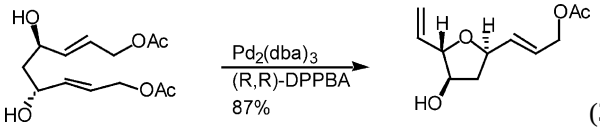
A regio- and stereoselective palladium-catalyzed allylic alkoxylation of α-fluoroalkyl substituted allylic mesylates to give γ-fluoroalkyl substituted ethers was reported [1365]. Allylic sulfones [1438] and 1-ethenyl-cyclopropyl sulfonates [1439] were alkoxylated in a related fashion using palladium catalysts. An intramolecular palladium-catalyzed alkoxylation was used in the synthesis of chiral 2-vinyl-tetrahydrofuran and pyran derivatives [1440] and of the F-ring of halichondrin B (Eq. (311)) [1254]. Intramolecular palladium-catalyzed formation of an oxazoline from a 4-*N*-benzoylamino-3-acetoxy-1-ene was used in a synthesis of sphingofungin F [184]. A related formation of oxazolidines from intermediately formed dicarbamates was described [1441].



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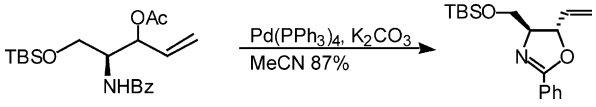
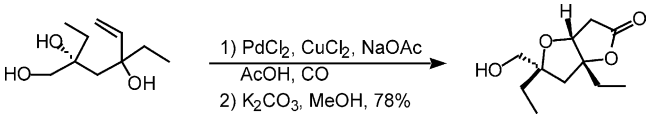
Palladium-catalyzed asymmetric allylic additions of alkoxides were used in the synthesis of (−)-galanthamine [490], (+)-brefeldin A [1294], callipeltoside A [103], furiquinocin E [1442], (−)-codein and (−)-morphine [492], and (+)-calanolide A [1443]. Intramolecular asymmetric allylic alkoxylation was used to prepare a *cis*-2,6-substituted tetrahydropyrane [1444]. A dynamic kinetic asymmetric allylic alkoxylation was used in a synthesis of tipranavir [709]. A diastereoselective palladium-catalyzed alkoxylation using in situ-formed zinc alkoxide was reported [1445]. Palladium catalyzed the intramolecular alkoxylation of *N*-benzoyl-substituted allylic acetates, and this was used in a synthesis of muriocin (Eq. (312)) [193]. A regio- and enantioselective rhodium-catalyzed allylic alkoxylation using copper alkoxides was described [1446].



(312)

Palladium and copper-catalyzed intramolecular reaction of phenols with tethered aryl halides was used to prepare dibenzoxepino[4,5-*d*]pyrazoles [1447]. The use of air-stable ferrocenyl dialkylphosphines as ligands was reported [218]. An arylpalladium halide phosphine complex was used for alkoxylation of aryl halides [201]. An intramolecular aromatic substitution of ruthenium chloroarene with a pendant alcohol was used in a synthesis of macrocyclic ethers [1448]. Copper catalyzed the intermolecular alkoxylations of aryl iodides with alcohols [1449]. A copper-catalyzed alkoxylation of an aromatic bromide was used in a synthesis of verbenachalcone [1450].

Palladium-catalyzed "hydroxy-palladation–carbonylation–lactonization" sequence was used in a synthesis of plakortone D (Eq. (313)) [1451]. An interesting palladium-catalyzed hydroalkoxylation of conjugated alkynes using phenols to give 2-alkoxy-1-en-3-ynes was developed (Eq. (314)) [1452]. The mechanism for intramolecular lactonization of acetic acid tethered cyclopentenes was shown to proceed via acyloxy-palladation-β-hydride elimination. When this route was inaccessible, an η^3-mechanism was suggested [1453].



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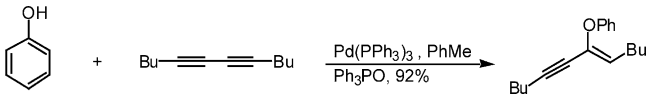
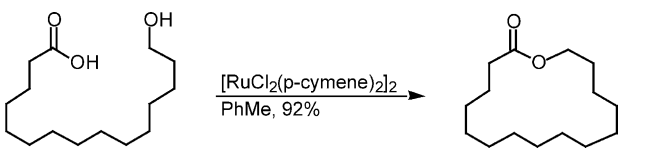


(314)

Palladium catalyzed the formation of monosaccharide vinyl ethers from monosaccarides and butyl vinylether [1454]. Vinyl ethers and vinylsulfides were prepared by an iridium-catalyzed reaction of alcohols and thiols with vinyl acetate [1455]. A ruthenium-ethoxyethyne catalyst system followed by acid treatment was developed for macrolactonizations (Eq. (315)) [1231]. Palladium catalyzed the addition of alcohols and acids to alkynes to give allylic ethers and esters [685]. Ethoxyvinyl esters were prepared by addition of aromatic acids to ethoxyethyne in the presence of a ruthenium catalyst [1456]. Zirconium mediated an intramolecular ester transfer reaction, and this reaction was used to prepare α-substituted-γ-aminobutyric acid derivatives (Eq. (316)) [1457]. Palladium catalyzed the allylation–alkoxylation of alkynylaldehydes to give cyclic ethers (Eq. (317)) [1458].



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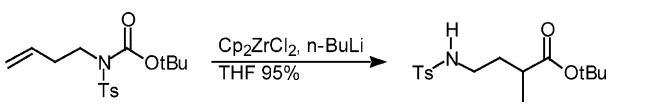

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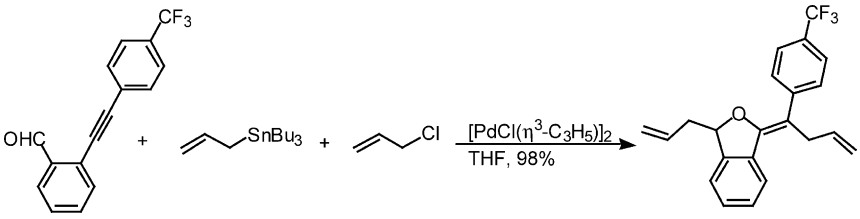
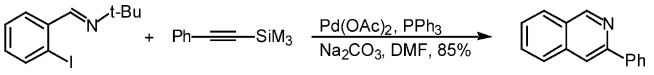


(317)

4.4. Heterocycles

Decumbenine B was prepared via a palladium–copper-catalyzed alkyne-imine cyclization (Eq. (318)) [1459]. Copper catalyzed the annulation of 3-(1-alkynyl)-indole-2-carboxaldehyde imines and 2-(1-alkynyl)-indole-3-carboxaldehyde imines to give β- and γ-carbolines, respectively [1460]. Palladium catalyzed a related annulation of 2-haloindole-3-carboxaldehyde imines and 3-haloindole-2-carboxaldehyde imines with alkynes forming ss- and γ-carbolines [1461]. Annulated γ-carbolines were obtained in a related fashion (Eq. (319)) [1462].



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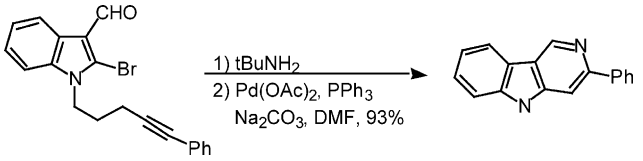
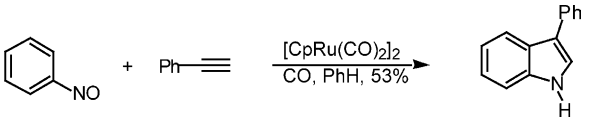


(319)

Ruthenium catalyzed an interesting annulation of aromatic nitroso compounds with alkynes forming indoles (Eq. (320)) [1463]. Indoles were also prepared by reductive annulation of aromatic nitro compounds and alkynes [1464]. Rhodium catalyzed the decomposition of iodonium ylids in the presence of ketones, thioketones, carbon disulfide, alkynes, or nitriles affords a variety of five-membered heterocycles [1465]. Palladium catalyzed a reductive *N*-heteroannulation of 2-nitroaniline derived enamines to give 1,2-dihydroquinoxalines and 3,4-dihydroquinoxalinones (Eq. (321)) [1466]. 1,2-Dihydro-4(3*H*)-carbazolones were obtained by reductive *N*-heteroannulation of 2-(2-nitrophenyl)-2-cycloalken-1-ones using a palladium-catalyst and carbon monoxide [111]. Ruthenium catalyzed the formation of quinolines via heteroannulation of nitroarenes with 3-amino-1-propanols [1467] and related cyclizations of nitroarenes with trialkylamines [1468].



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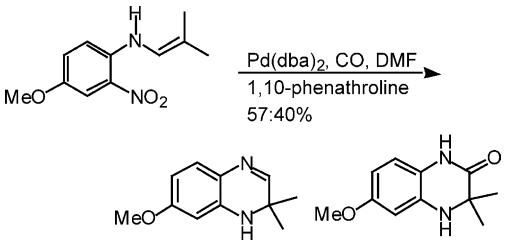
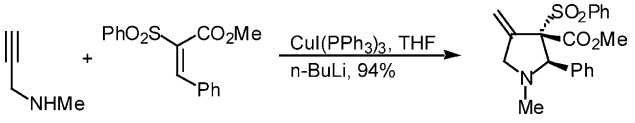


(321)

Copper catalyzed the annulation of propargylic amines with activated alkenes to give pyrrole derivatives (Eq. (322)) [1469]. Copper catalyzed the formation of quinolines from an aromatic amine, an aldehyde, and an alkyne (Eq. (323)) [1438]. Indoles were obtained upon reaction of 2-(1-alkynyl)arylisocyanates and allylic carbonates in the presence of a palladium–copper catalyst system (Eq. (324)) [1470]. 6-Trifluoromethyl-12-acylindolo[1,2-*c*]quinazolines were prepared via a palladium-catalyzed reaction of bis(2-trifluoroacetamidophenyl)ethyne in the presence of aryl or vinyl halides and triflates and carbon monoxide (Eq. (325)) [1471]. Palladium catalyzed the formation of 2,3-disubstituted indoles from reaction of aromatic iodide, 2-(1-alkenyl)phenylisocyanide, and an amine (Eq. (326)) [1472]. A three component reaction of a nitrile, allyl methyl carbonate and trimethylsilyl azide using a palladium catalyst produced 2-allyltetrazoles (Eq. (327)) [1473]. Related reactions using an alkyne–nitrile were also described [1474].



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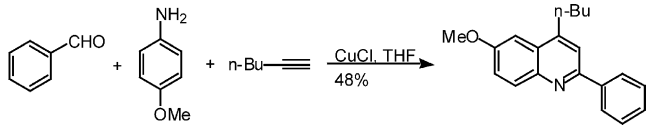

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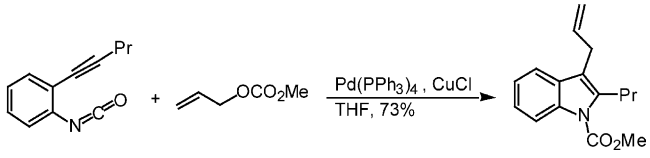

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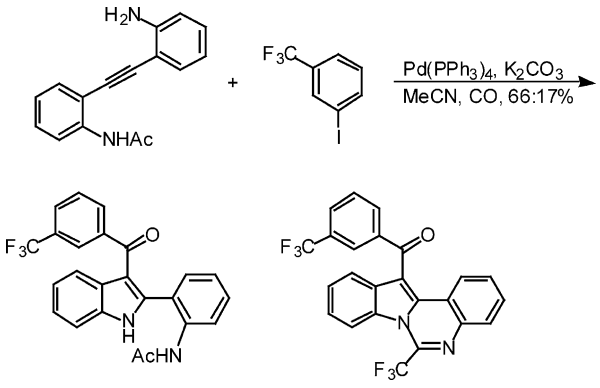

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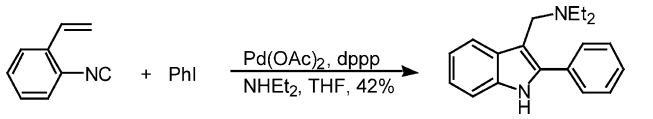

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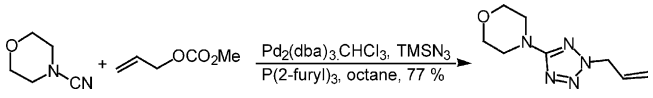
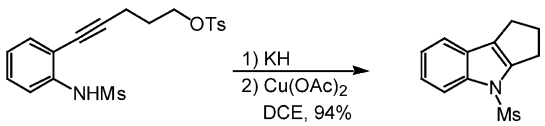


(327)

2,3-Disubstituted indoles were prepared from substituted 2-iodoanilines and internal alkynes and a palladium catalyst [1475]. Palladium catalyzed the formation of 5-carbomethoxy-*N*-acyltryptamine from a 2-iodobenzenamine and internal alkynes under microwave irradiation [1476]. Copper catalyzed the intramolecular amination of 2-(1-alkynyl)benzenamines to give indoles [127]. The copper intermediate can be alkylated in an intermolecular fashion (Eq. (328)) [1477]. Copper catalyzed the formation of 1,9-diaza-1*H*-cyclopent[*a*]azulene by intramolecular cyclization of 2-mesylamino-3-trimethylsilylethynyl-1-azaazulene [647]. An intramolecular amino-palladation carboxylation of *O*-alkenyl hydroxylamines to give *cis*-substituted isoxazolidines was described [1478]. Palladium-catalyzed hydroarylation of alkynes with aromatic iodides in ionic liquids was described [1479].



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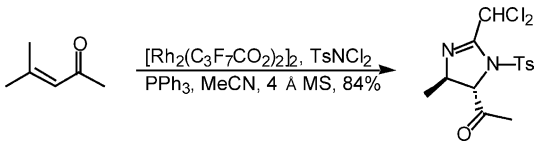
Imidazolines were prepared via an iron-phosphine-catalyzed cyclization of α,β-unsaturated esters with *N*, *N*-dichloro-*p*-toluenesulfonamide (Eq. (329)) [1480,1481]. Palladium catalyzed the addition of amines to 5,5-dimethyl-4-methylene-1,3-dioxolan-2-one to give 3-alkyl-4-hydroxy-4,5,5-trimethyloxazolidin-2-ones [1482].



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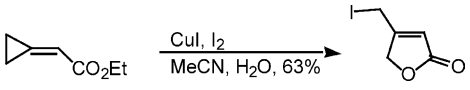
Rhodium catalyzed intramolecular aziridinations of sulfonamides [1483]. Rhodium catalyzed aziridination of alkenes using sulfamate esters and $PhI(OAc)_2$ [1484].Copper-catalyzed intramolecular aziridination of unsaturated sulfamates mediated by PhI=O was reported [1485].

Palladium catalyzed the reaction of iodo-hydroxypyridines with terminal alkynes to give furo[2,3-*c*]- and [2,3-c]pyridines [1486]. Cyclopropylideneacetic acids and esters were transformed into 4-halomethyl-2(5*H*)-furanones and 4-halo-5,6-dihydro-2*H*-pyran-2-ones using copper catalysts (Eq. (330)) [1487]. An intramolecular hydroxy-palladation of 2,3- and 3,4-dienols followed by allylation was described (Eq. (331)) [1488]. Intramolecular hydroxy-palladation was used in a synthesis of the 18-epi-tricyclic core of garsubellin A [70]. Palladium catalyzed an asymmetric reaction of benzene-1,2-diols with propargylic carbonates to give 2-alkylidene-1,4-benzodioxanes [1489]. An asymmetric hydroxy-palladation using polymer bound chiral ligand [1490] and enantioselective hydroxy-chlorinations of alkenes using a chiral palladium complex [1491] were described. A palladium-catalyzed annulation was used to prepare the furan-containing C1–C18 segment of lophotoxin and pukalide (Eq. (332)) [183].



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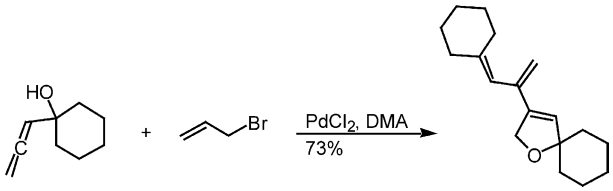

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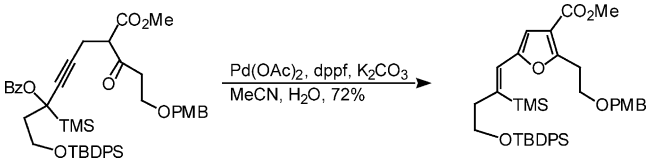

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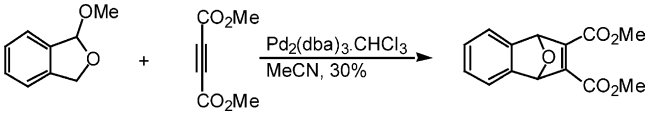
4.5. Alkenes, allenes, alkanes, alkynes, and arenes

A variety of functional groups were replaced by a hydrogen atom using palladium-catalyzed methodologies. Palladium(0) complexes, together with a hydride source, such as tributyltin hydride, dimethylamine borane complex [1492], or ammonium formates catalyzed the reduction triflates

and halides. Synthetic applications include magellanine [518], (−)-laulimalide [1493], sphinosines [1494], (−)-histrionicotoxin 235A [1495], (−)-blestriarene C [1496], and the deoxypumiloside skeleton [1497]. A 2-alkoxy-1-iodoalkane was reduced using palladium and tributyltin hydride in a synthesis of vancomycin analogs [1498]. Palladium-catalyzed regioselective ammonium formate or sodium borohydride reductions of allylic carbonates and ester were used in synthesis of colletodiol, colletol, and grahamimycin A [1499], epothilone B and D [1500], and (−)-tuberostemonine [1501].

Monodebromination of 1,1-dibromoalkenes was achieved using tributyltin hydride in the presence of a palladium catalyst forming *cis*-substituted bromoalkenes and this reaction was used in the synthesis of prelaureatin [671] toward oxazolomycin antibiotics [101], (−)-codein and (−)-morphine [492], (−)-gambierol [79], and AK-toxins [106].

Palladium-catalyzed transformation of allylic esters to conjugated dienes was used in a synthesis of podolactone-related compounds [1502]. Isobenzofurane was generated by reaction of a benzylic acetal with palladium catalyst (Eq. (333)) [1503]. A palladium–diethyl zinc system was used to prepare allenes from 2-bromo-1-alkenes having a leaving group in the 3-position (Eq. (334)) [1504]. Titanocene dichloride catalyzed the hydroalumination of internal alkynes affording Z-alkenes [1505]. Wilkinson's catalyst was used to remove an aldehyde functionality (deformylation) in the synthesis of the tetracyclic erythrinane core [1506], (+)-goniodiol [1092], and 12-epiverticillol [1507]. Vinyl sulfides, sulfones, and sulfoxides reacts with a zirconacyclopropane followed by an electrophile to give substituted alkenes (Eq. (335)) [1508].



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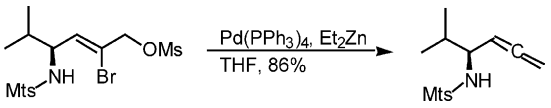

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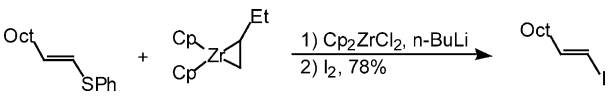

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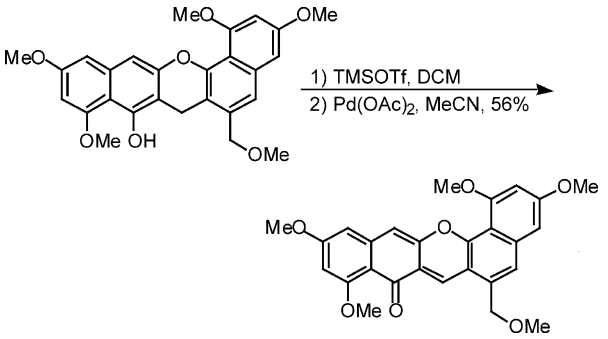
4.6. Ketones and aldehydes

A number of synthetic applications of the Saegusa reaction were published. Synthetic applications include decahydroquinoline type dendrobatid alkaloids [1509], hypnophilin [1510], gambierol [78], hypoxyxylerone (Eq. (336)) [1511], (−)-xialenone [1512], 9,12-anhydroerythronolide aglycone [91], temonamide and isostemonamide (Eq. (337)) [1513], and (+)-aspidospermidine [1514]. A related palladium-catalyzed reaction of an enol acetate was used in the synthesis of tetrahydrocannabinols [1515]. Palladium-catalyzed oxidation of a trimethylsilyl enol ether to an α,β-unsaturated enone in the presence of diallylcarbonate was used in a synthesis of (−)-strychnine [104]. An α,β-unsaturated enone was prepared from an allylester in the synthesis of ingenol (Eq. (338)) [1516].



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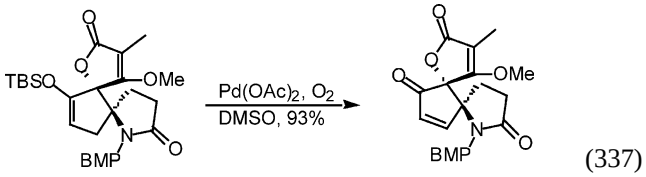

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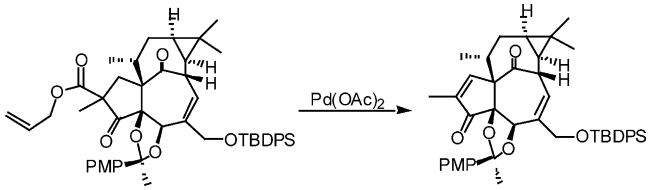

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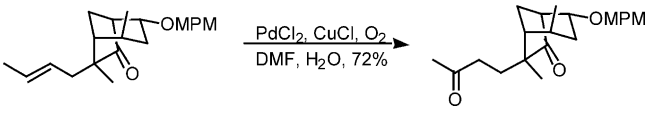
Wacker-type oxidation, i.e., reaction of monosubstituted terminal alkenes with palladium(II) and water to afford methyl ketones, was utilized in a number of syntheses [1517,1518]. For example, the spirocyclic skeleton of pathylacton A [1519], hypnophlin, coriolin, ceratopicanol [1520], pheromone of the banded cucumber beetle [1521], ABE tricyclic analogs of methyllyaconitine [1522], and the proposed structure of pathylactone A [1523] were prepared using a Wacker-type reaction in one of the steps. Regioselective Wacker oxidation of a 1,2-disubstituted alkene was used in a synthesis of leucascandrolide A [1524], and steroid C–D ring synthesis (Eq. (339)) [1525]. Polyethylene glycol-$H_5PV_2Mo_{10}O_{40}$-palladium [1526] and NPMoV/O_2 oxidation systems [1527] were developed. Reversal of regioselectivity of the water addition to the alkene was observed in special cases [1528]. Gold catalyzed the formation of ketones from alkynes via the addition of water [1529].



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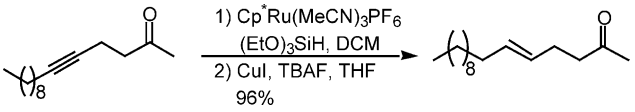
4.7. Organoboranes, silanes, germanes, and stannanes

Ruthenium catalyzed the hydrosilylation of cyclic alkynes to give cyclic vinylsilanes [1530]. Depending on the ruthenium catalyst, *E*- or *Z*-alkenylsilanes was obtained from terminal alkynes [1531,1532]. A ruthenium catalyst for regioselective hydrosilylation of terminal alkynes to give 2-silyl-1-alkenes was developed [1533]. Rhodium and iridium catalyzed the hydrosilylation of 1,3-dienes [1534]. Palladium catalyzed asymmetric hydrosilylations of conjugated dienes [1535,1536], ethenylsilanes [1537], and styrenes [1538]. Regioselective palladium-catalyzed hydrosilylation of terminal alkynes was described [1539]. Rhodium and platinum catalysts together with dimethyl(2-pyridyl)silane and dimethyl(2-thienyl)silane were used for hydrosilylation of alkenes [367,1540]. Platinum catalyst systems were used in hydrosilylations of terminal alkenes [1541,1542], alkynes [1543], and 2,2-diaryl-1-methylenecyclopropanes [1544]. Rhodium-catalyzed hydrosilylation of alkenes in a fluorous ionic liquid was described [1545]. A enantioselective double hydrosilylation of norbornadiene, catalyzed by palladium, was used in a synthesis of (+)-sparteine [1546]. Related intramolecular ruthenium-catalyzed anti-hydrosilylation of alkynes was also described [368]. Selective reduction of alkynes to *trans*-alkenes was obtained via a ruthenium-catalyzed hydrosilylation followed by protodesilylation (Eq. (340)) [1547]. Nickel catalyzed the hydrosilylation of propargyl chloride to give 1,2-propanedienylsilane, or using a copper catalyst gave propargyl silane [1548]. Palladium-trimethylsilyl triflate catalyzed the bissilylation of α,β-unsaturated carbonyl compounds [1549].



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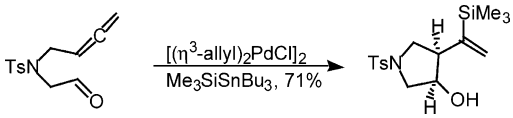
Palladium catalyzed a tandem silastannylation alkylation of 1,2-heptadien-7-als and -7-ones and 1,2-heptadien-8-als and -8-ones to give *cis*-cyclopentanols and cyclohexanols, respectively (Eq. (341)) [1550]. A palladium-catalyzed ring-opening, carbocyclization, hydrosilylation sequence of 1-cyclopropyl-1,6-heptadienes was developed (Eq. (342)) [1551]. Rhodium and palladium catalyzed the cyclization–silylation of 1,6-diynes affording silylated cyclopentanes [1552–1554]. Nickel catalyzed the cyclization of 5,7- and 6,8-dienals in the presence of silanes affording five- and six-membered rings (Eq. (343)) [1555].



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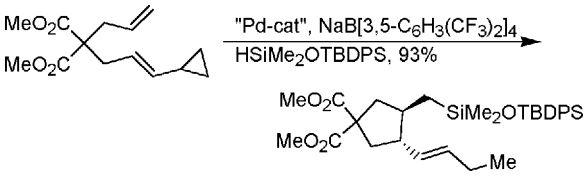

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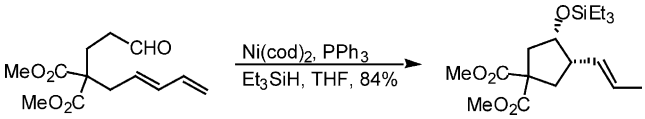

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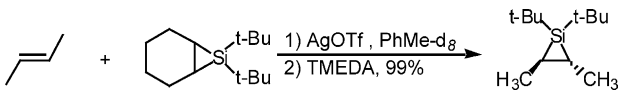
Reaction of aromatic bromides and iodides with triethoxysilane in the presence of a rhodium catalyst gave aryltriethoxysilanes [1556]. A polymer-bound trialkylsilane was reacted in a similar fashion with an aryl iodide [1557]. Silver catalyzed the silacyclopropanation of alkenes (Eq. (344)) [1558].



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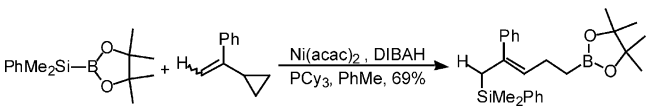
A direct, regioselective, aromatic, and heteroaromatic borylation catalyzed by iridium was developed [1559]. Palladium catalyzed the cross-coupling of aromatic or vinylic halides and triflates, and benzyl halides with dipinacolyldiborane, [2,2′]bi[[1,3,2]dioxaborinanyl], or pinacoleborane producing aromatic, vinylic, or benzylic pinacolyl boronic esters [277,1560–1567], and this reaction was used in synthetic applications toward halitulin [287], clusiparalicoline [114]. 3-Bromopyrroline nitroxides were also used as substrates [324]. Aryl chlorides were used as substrate employing microwave irradiation [1568]. A related, iridium-catalyzed, borylation was described [1569]. Rhodium and ruthenium catalyzed a regioselective oxidative borylation of alkenylarenes with pinacolborane to give alkenylboronic esters [1570]. A nickel-catalyzed silaboration of small-ring vinyl cycloalkenes was reported (Eq. (345)) [1571].



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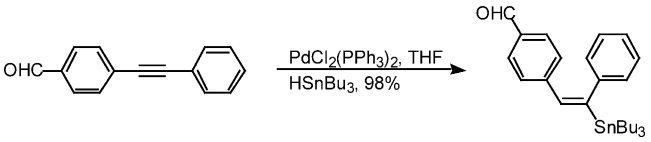
Palladium catalyzed the reaction of bromopyridines and hexamethylditin to give trimethyltin substituted pyridines [1572]. Palladium-catalyzed reaction of hexaalkylditins with

vinyl iodides or triflates to give vinyltin reagents was employed in the synthesis of rhizoxin D [64], SNF4435 C and SNF4435 D [76], and gambierol [187]. 6-Bromoazulene was stannylated using the same type protocol [14]. Regioselective stannylation in the 3-position of 3,5-dibromo-2-pyrone was reported [653].

Palladium-catalyzed hydrostannation of terminal alkynes, using tributyltin hydride, forming a vinylstannane, was used in total synthesis of amphidinolide A diastereomer [117], (+)-herbarumin I [1573], and the core of apicularen A [115]. Palladium-catalyzed hydrostannation of bromoalkynes to give vinylstannanes was used in a synthesis of 4,4a-didehydrohimbacine and 4,4a-didehydrohimandravine [73], amphidinolide A [1061], rhizoxin D [119], (−)-macrolactin A [94], (−)-sanglifehrin A [95], and lobatamide C [96]. Trineophyltin hydride was also used in alkyne hydrostannations catalyzed by palladium [1574]. Regioselective hydrostannation of propargylic alcohols to give 3-hydroxy-1-stannyl-1-alkenes was reported [1575]. *ortho*-Substituents direct the hydrostannation of unsymmetrical diaryl ethynes (Eqs. (346) and (347)) [1576]. Regioselective molybdenum-catalyzed hydrostannation of terminal alkynes was reported [1577]. Palladium and a number of other transition metals catalyzed hydrostannation, distannation, or silastannation of cyclopropenes (Eq. (348)) [1578].



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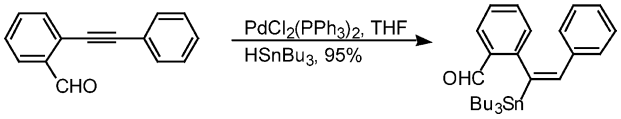

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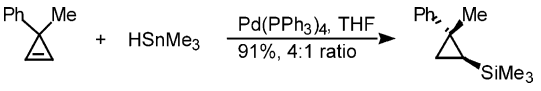

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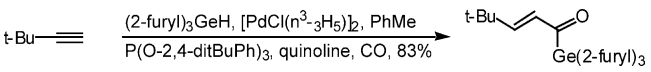
Palladium catalyzed a regio- and stereospecific silylstannylation of allenes [1579] and a cyanostannylation of activated alkynes [1580]. A silylstannylation cyclization of 1,6-enynes was described affording substituted cyclopentanes [1581]. A palladium-catalyzed synthesis of acylgermanes via addition of trifurylgermane and CO to terminal alkynes was described (Eq. (349)) [1582].



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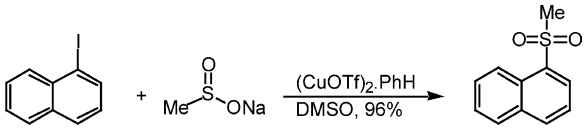
4.8. Organophosphorous, selenium, and sulfur compounds

Aryl diethylphosphonates were prepared from aromatic halides and triflates using palladium catalysts and diethylphosphite [1583–1586]. Aromatic halides and triflates were transformed into (arylphosphinyl)methylphosphonates using a palladium catalyst and (diethylphosphinyl)methylphosphonate [1587]. Diarylphosphine oxides were used in related reactions to give triarylphosphine oxides [1588]. Nickel and palladium catalyzed the formation of triarylphosphines from aryl triflates and halides and diphenylphosphine [1589–1591]. A microwave assisted palladium-catalyzed coupling of diphenylphosphine with aryl iodides was reported [1592]. Solvent-free conditions were also described [1593]. Palladium catalyzed the phosphination of aryl iodides using diphenylphosphine tin compounds [1594]. Palladium catalyzed an enantioselective formation of diaryl methylphosphine chiral on phosphorous using an arylmethylphosphine and an aryl iodide [1595].

Palladium and nickel catalyzed the anti-Markovnikov hydrophosphination of alkynes [1596] and palladium catalyzed the bishydrophophination of alkynes [1597]. Palladium also catalyzed hydrophosphinations of alkenes [1598] and the formation of vinyl phosphates from vinyl bromides and diethylphosphite [1599].

Palladium-catalyzed coupling of vinyl chlorides with thiols to give thioethers was described [146]. Coupling of an aromatic iodide with *t*-butylthioxy tributyltin in the presence of a palladium catalyst was reported [1600]. Copper catalyzed the coupling of aryliodides and aromatic thiols [1601,1602]. Copper catalyzed the coupling of aryl and alkenyl boronic acids with *N*-thio(alkyl, aryl, heteroaryl)imides to give thioethers [1603]. Copper and palladium catalyzed the formation of sulfones from sulfinic acid salts and aromatic halides (Eq. (350)) [1604,1605]. Palladium catalyzed the coupling of dibromoarenes with sulfoximines to give sulfur-containing heterocycles (Eq. (351)) [1606]. Molybdenum catalyzed the transformation of isonitriles to isothiocyanates using elemental sulfur or methylthiirane as the sulfur donor [1607].



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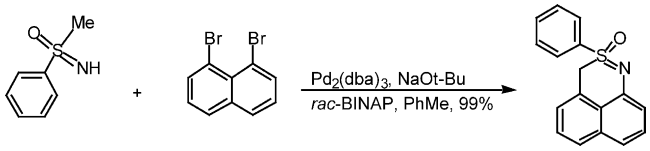

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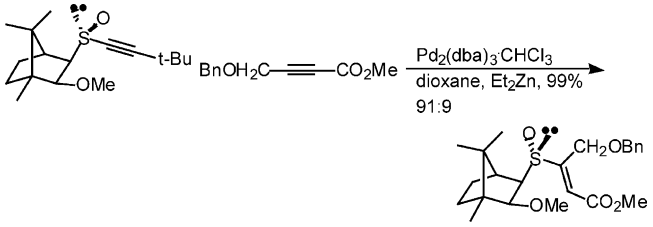
A palladium-catalyzed asymmetric sulfonylation of allylic substrates was developed [1608]. Ruthenium catalyzed the

formation of propargylic sulfides from propargylic carbonates [1609]. Palladium catalyzed an asymmetric sulfinylzincation of alkynyl sulfoxides (Eq. (352)) [1610].



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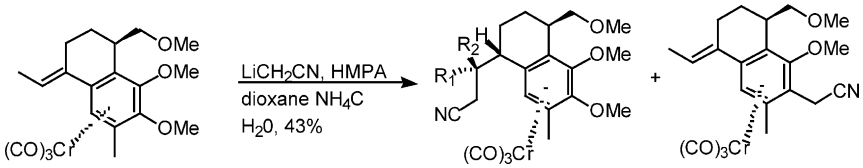
5. Preparations and reactions of isolated metal complexes and metal intermediates

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. Arene tricarbonyl chromium complexes continued to be extensively used as templates for organic reactions. For example, as templates for enantiotopic deprotonation of benzylic positions [1611,1612], Michael additions of chiral chromium tricarbonyl complexed benzylic anions [1613], diastereoselective alkylation of benzaldimine complexes [1614], β-aminoalcohols were prepared by samarium-mediated reductive coupling of benzylideneamines with tricarbonyl chromium complexed benzaldehydes [1615]. Intramolecular indium mediated reactions of chromium tricarbonyl arene complexes having a pendant imine were used to prepare tricyclic compounds related to marine diterpenoids [1616]. Samarium mediated a coupling of ketones with fluoroarene chromium tricarbonyl complexes [1617]. An interesting annulation of 2-trimethylsilylbenzaldehyde tricarbonyl chromium complex with ester enolates to give indoline derivatives was reported (Eq. (353)) [1618]. Tricarbonyl chromium complexed benzaldehydes participated in Ugi reactions [1619]. Regioselective nucleophilic additions to the 1-position of 3-methoxycyclobenzocyclobutendiones chromium tricarbonyl complex were reported [1620].

(353)

Nucleophilic addition to a 1-ethylidene-tetralin tricarbonyl chromium complex was used as a key step toward serrulatane diterpenes (Eq. (354)) [1621]. Polyarylated products were observed upon Heck reaction of dihydronaphthalene chromium complexes [1622]. Depending on the counterion, either silicon-aryl or silicon methyl bond cleavage was observed upon nucleophilic addition of allyl lithium to a chromium complexed 2-TMS-aryl ketone (Eq. (355)) [1623].



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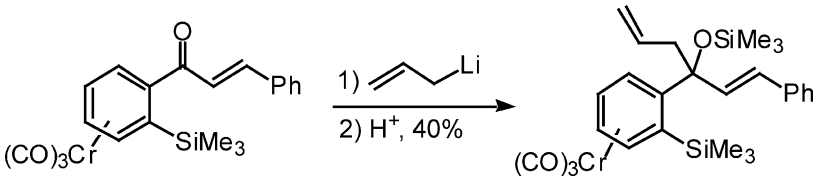
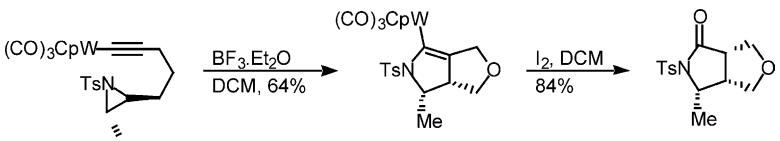


(355)

Tungsten-σ-alkyne complexes were used to prepare *cis*-fused bicyclic lactams indolizidine via [3 + 2]cycloaddition with a tethered aziridine (Eq. (356)) [1624]. Related reactions of tungsten-σ-alkyne complexes with aldehydes furnished 2,5-dihydrofuran tungsten-σ complexes (Eq. (357)) [1625,1626].



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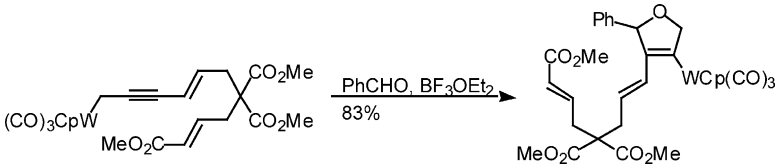
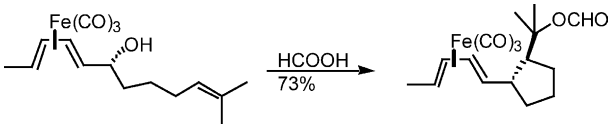


(357)

Palladium catalyzed the introduction of nitrogen, sulfur, and phosphorous substituents employing η^5-1-chlorocyclohexadienyl manganese complex [1627]. Stereospecific cationic cyclizations of tricarbonyl iron 1,3-diene complexes having pendant alkenes and arenes were described (Eq. (358)) [1628]. Chiral pentadienol iron tricarbonyl complexes participated in polyene cyclizations (Eq. (359)) [1629]. Intramolecular cyclization of 1,3-diene complexes having a pendant alcohol was used to prepare conformationally locked phosphocholines (Eq. (360)) [560]. An iron η^3-allyl lactone complex was used in a synthesis of taurospongin A (Eq. (361)) [1630] and α- and β-zearalenol [105]. Stereodefined 1,7-diols can be obtained by reductive decomplexation of iron η^3-allyl lactone complexes using lithium naphthalenide (Eq. (362)) [1631].



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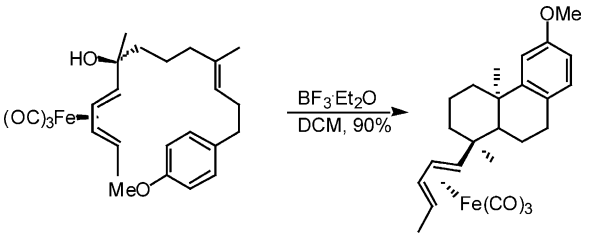

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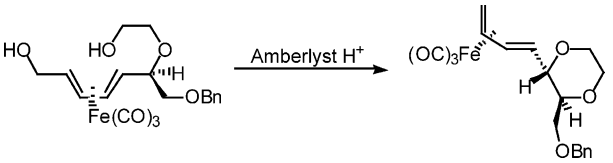

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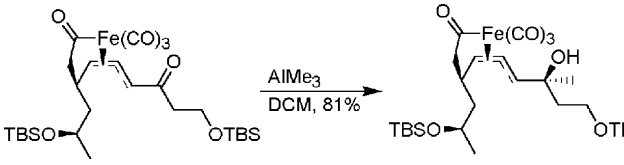

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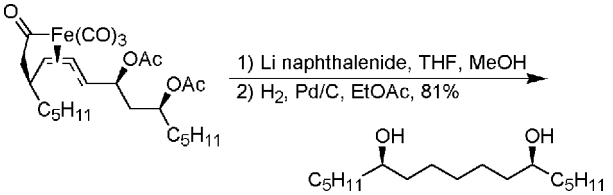
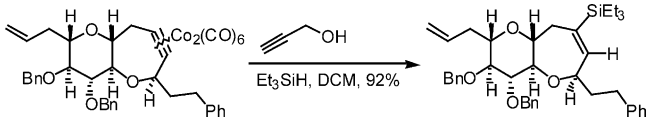


(362)

Nicholas type reactions on a solid support were reported [1632], and reaction of cobalt alkyne complex stabilized cations were used toward ciguatoxins [1633] and oxaspiro[m.n]skeletons [1634]. Removal of the dicobalt hexacarbonyl fragment from a cyclic alkynyl ether with triethylsilane furnished a vinyl silane with high regioselectivity (Eq. (363)) [1635]. Protection of an alkyne as a hexacarbonyl dicobalt complex was used as an approach to the E′FGH′ ring fragment of ciguatoxin 1B [1636]. An interesting [5 + 2]cycloaddition of a cobalt-complexed alkyne with enol silyl ethers forming cycloheptanone derivatives was developed (Eq. (364)) [1637]. Reaction of a hexacarbonyl dicobalt complexed 2-ynal with diazocompounds and an alkene in the presence of a rhodium-catalyst produced tetrahydrofuran derivatives (Eq. (365)) [1638]. Enatioselective alkylation of phenylpropynal dicobalt hexacarbonyl complex using diethyl zinc and a chiral auxiliary was used in a synthesis of (+)-incrustoporin [882].



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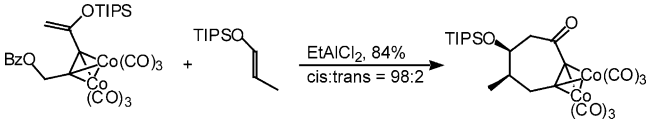

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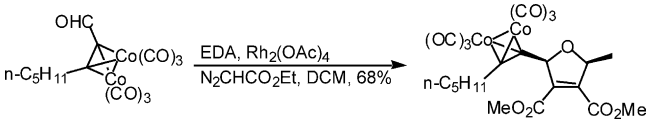
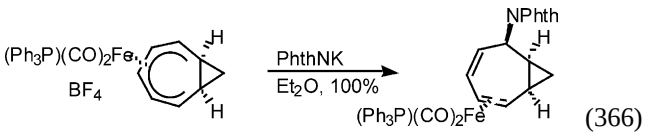


(365)

The bicyclo[5.1.0]octadienyliron cation was used as a template in a synthesis of *cis*-2-(2′-carboxycyclopropyl)glycine (Eq. (366)) [1639]. A cationic η^5-iron cyclohexadienyl complex was used in a synthesis of carazostatin [1640]. Intramolecular coupling of cyclohexadiene iron tricarbonyl complexes with pendant alkenes produced azspiro[5.5]undecane derivatives (Eq. (367)) [1641]. Iron tricarbonyl diene complexes served as chiral templates for asymmetric synthesis toward amphtotericin B [1642] and soraphen A_{1a} [1643].



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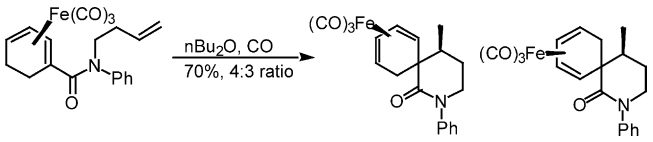
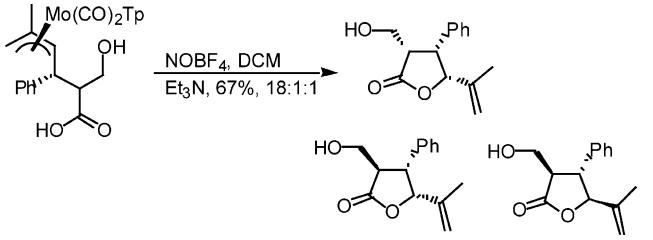


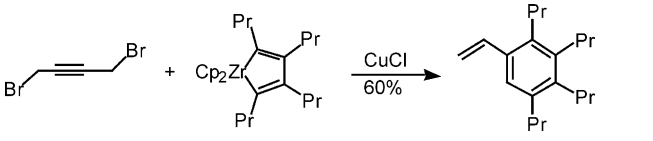
(367)

Cyclic η^3-allyl molybdenum complexes were oxidatively decomplexed using PDC and were used in enantiocontrolled synthesis of unsaturated ketones and lactones [1644]. η^3-Allylmolybdenum complexes were also used as scaffolds for diasteroselective synthesis of 3,4,5-trisubstituted γ-butyrolactones (Eq. (368)) [1645]



(368)

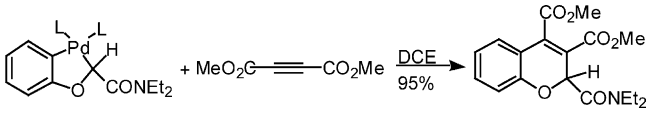
Zirconacycles undergo a variety of reactions to give organic products. Intermediately formed zirconacyclopentadienes couple with dihalonaphthalenes and dihalopyridines to give anthracenes, quinolines, and isoquinolines [1646]. Reactions of zirconacyclopentadienes with carbon–oxygen, carbon–nitrogen, and nitrogen–nitrogen double bonds furnished six-membered heterocycles [1647]. Azazirconacyclopentenes afforded pyridines, pyridones, and iminopyridines in related reactions [1648]. A regioselective synthesis of 2-(hydroxymethyl)vinylphosphonates were obtained via insertion of ketones into zirconacyclopropene phophonates [1649]. Zirconacyclopentadienes underwent coupling reactions with dihalides in the presence of a copper catalyst to form substituted styrenes and vinylcyclohexadienes (Eq. (369)) [1650].



(369)

6. Miscellaneous reactions

A polymer-supported zirconocene was used in a reaction of phenylethyne with trimethylaluminum affording α-methylstyrene [1651]. 2*H*-1-benzopyrans were prepared by reaction of activated alkynes with oxapalladacycles (Eq. (370)) [1652]. Molybdenum mediated the cleavage of isoaxazoline rings fused to bicyclic frameworks [1653]. Cobalt catalyzed the triethylsilylperoxidation of alkenes [1654]. Rhodium catalyzed an interesting reduction–oxidation reaction of 4-alkynals to give *cis*-4-alkenoates (Eq. (371)) [1655]. Iridium catalyzed a coupling of 1-methylimidazole with aldehydes in the presence of diethylmethylsilane (Eq. (372)) [1656]. Tungsten hexacarbonyl mediated the formation of tungsten containing azomethine ylides from 2-(1-alkynyl)benzaldehyde imines. The ylides reacted with alkenes to give polycyclic indole derivatives (Eq. (373)) [1657].



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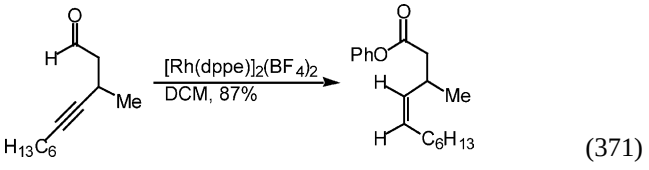

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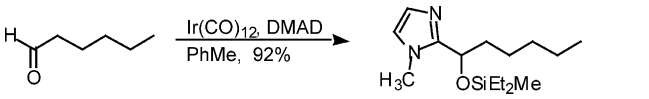

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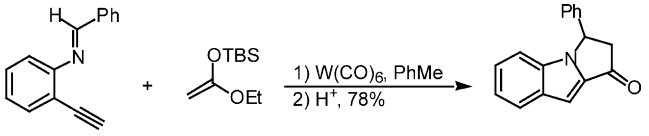
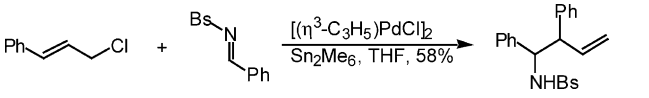


(373)

Reactions of allylic chlorides with aldehydes, imines, or benzylidenmalonitrile in the presence of hexamethylditin and a palladium catalyst were described (Eq. (374)) [1658]. Nickel catalyzed the coupling of vinylzirconium reagents with alkynes in the presence of an aldehyde to give a variety of products [1659]. Intramolecular reactions of ynals were also reported. Nickel catalyzed a four-component coupling of a α,β-unsaturated aldehyde, a terminal alkyne, a stannylalkyne, and a dialkyl zinc to give functionalized cyclohexenols (Eq. (375)) [1660].



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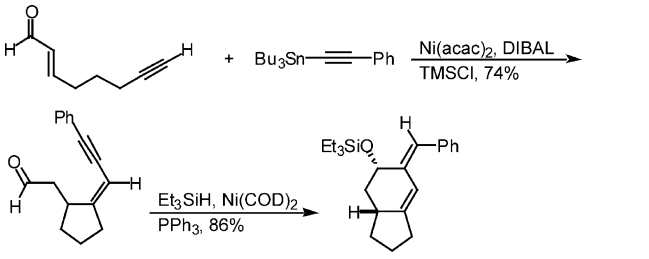
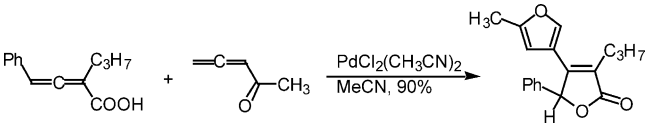


(375)

A rhodium-catalyzed hydroarylation of alkynes using arylboronic acids to give trisubstituted alkenes was reported [1661]. Ruthenium catalyzed hydroarylations and -alkenylations of alkynes using silanediols [1662]. Palladium catalyzed an oxidative cyclization–dimerization of 2,3-allenoic acids with 1,2-allenyl ketones (Eq. (376)) [1663]. Rhodium catalyzed the coupling of norbornene, sodium tetraphenylborate, and anhydrides to give 2-acyl-3-phenylnorbornanes [1664]. A synthesis of homoallylic ketones using an alkyne, an alkene, and an aldehyde in the presence of zirconocene was described (Eq. (377)) [1665]. A rhodium–copper catalyst system was used to prepare propargylic amines from a terminal alkyne, an aromatic amine, and an aldehyde (Eq. (378)) [1666]. Palladium catalyzed a three-component coupling of alkynes with acrolein or methyl vinyl ketone and acetic acid (Eq. (379)) [1667]. Palladium catalyzed the coupling of allylic carbonates, TMS-azide, and 2-(1-alkynyl)benzenisocyanide to give *N*-cyanoindoles at high temperature and allyl cyanamides at lower temperatures (Eq. (380)) [649]. Activated alkenes react with allylic acetates and tri-*n*-butyltin hydride in the presence of a palladium catalyst (Eq. (381)) [1668]. A palladium-catalyzed, three-component coupling of a propargylic amine, an aromatic iodide, or vinyl triflate, and an activated alkene was described (Eq. (382)) [1669]. A nickel-zinc-promoted coupling of enones and 1,6-enynes to give functionalized six-membered rings was developed (Eq. (383)) [1670]. Rhodium catalyzed an intramolecular insertion of an alkene into a cyclobutane carbon–carbon single bond (Eq. (384)) [1671].



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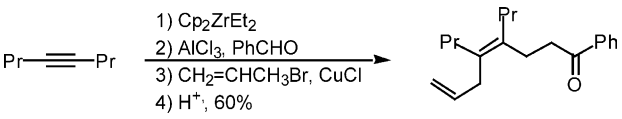

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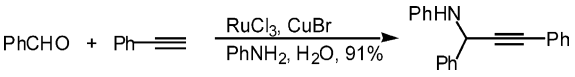

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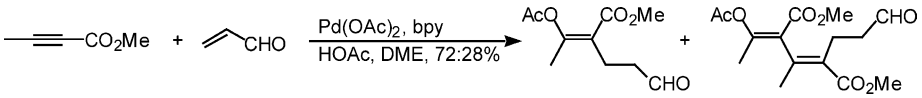

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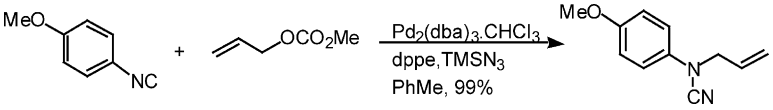

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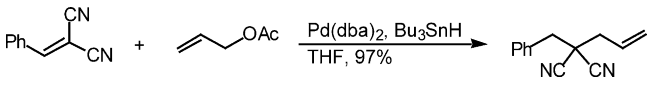

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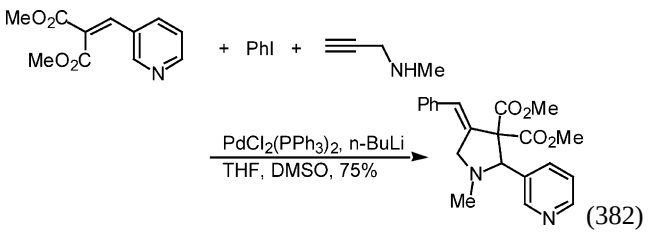

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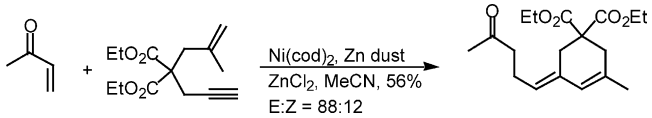

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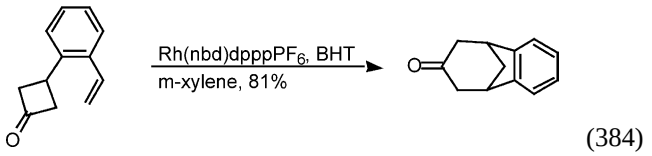

(384)

7. Reviews

The following reviews appeared in 2002:

- Halide effects in transition metal catalysis (114 references) [1672].
- *N*-Heterocyclic carbenes: a new concept in organometallic chemistry (131 references) [1673].
- Palladium-catalyzed coupling reactions of aryl chlorides (347 references) [1674].
- Transposition of allylic alcohols into carbonyl compounds mediated by transition metals (108 references) [1675].
- Rhodium-catalyzed carbon–carbon forming reactions of organometallic compounds (120 references) [1676].
- Palladium-assisted routes to nucleosides (245 references) [1677].
- Palladium-catalyzed alkynylation (269 references) [1678].
- Asymmetric hydrovinylation reaction (48 references) [1679].

- Catalytic enantioselective C–H activation by means of metal–carbenoid-induced C–H insertion (251 references) [1680].
- Enantioselective catalytic aziridinations and asymmetric nitrene insertions into CH bonds (130 references) [1681].
- The behaviour of 1,*n*-enynes in the presence of transition metals (137 references) [1682].
- Aryl–aryl bond formation one century after the discovery of the Ullmann reaction (765) references) [1683].
- Ru-, Rh-, and Pd-catalyzed C–C bond formation involving C–H activation and addition on unsaturated substrates: Reactions and mechanistic aspects (136 references) [1684].
- Transition metal-catalyzed carbostannylation of alkynes and dienes (41 references) [1685].
- Chelation-assisted carbon–hydrogen and carbon–carbon bond activation by transition metal catalysts (31 references) [1686].
- Transition metal-catalyzed carbon–heteroatom three-component cross-coupling reactions: A new concept for carbothiolation of alkynes (28 references) [1687].
- New strategies for the synthesis of carbocycles based on transition metal-catalyzed cyclization reactions of organostannanes and organosilanes (87 references) [1688].
- The aminopalladation/reductive elimination domino reaction in the construction of functionalized indole rings (41 references) [1689].
- Pauson–Khand reactions of electron-deficient alkenes (41 references) [1690].
- Palladium catalysis in the construction of the benzo[*b*]furan and furan rings from alkynes and organic halides or triflates (38 references) [1691].
- Nitrogen-containing heterocycles via palladium-catalyzed reaction of alkynes with organic halides (45 references) [1692].
- Development of palladium-catalyzed cycloalkenylation and its application to natural product synthesis (42 references) [1693].
- Iridium complex-catalyzed highly selective organic synthesis (52 references) [1694].
- Homogenous catalytic isomerization of allylic alcohols to carbonyl compounds (164 references) [1695].
- Catalytic cross-coupling reactions mediated by palladium/nucleophilic carbene systems (71 references) [1696].
- Cross-coupling reactions via organoboranes (27 references) [1697].
- Pyridylsilyl group-driven cross-coupling reactions (47 references) [1698].
- Synthesis of functionalized alkenes by transition metal-catalyzed carbostannation of alkynes and dienes following cross-coupling reactions (12 references) [1699].
- New cascade and multiple cross-coupling reactions for the efficient construction of complex molecules (30 references) [1700].
- The directed ortho metalation cross-coupling symbiosis. Regioselective methodologies for biaryl and heterobiaryls. Deployment in aromatic and heteroaromatic chemistry (47 references) [1701].
- Multiple arylations of carbonyl compounds via palladium-catalysis (19 references) [1702].
- Carbon–carbon cross-coupling reactions for the combinatorial synthesis of novel organic materials (36 references) [1703].
- Palladium-catalyzed carbon–nitrogen and carbon–carbon cross-couplings as versatile, new avenues for modification of purine 2′-deoxynucleosides (64 references) [1704].
- The intramolecular Stille reaction in some target natural product synthesis (48 references) [1705].
- Application of cross-coupling reactions at Merck (20 references) [1706].
- Some applications of the Grignard cross-coupling reaction in the industrial field (11 references) [1707].
- The lactone concept—a novel approach to the metal-assisted atropselective construction of axially chiral biaryl systems (75 references) [1708].
- The directed synthesis of axially chiral ligands, reagents, catalysts, and natural products through the ‘lactone methodology’ (89 references) [1709].
- Transition metal-catalyzed oxidations of bishomoallylic alcohols (94 references) [1710].
- Applications of stoichiometric transition metal complexes in organic synthesis (115 references) [1711].
- Titanium reagents for the alkylidenation of carboxylic acid and carbonic acid derivatives (186) [1712].
- Copper-catalyzed allylic oxidation with peresters (107 references) [1713].
- Palladium-catalyzed reactions of aryl halides with soft non-organometallic nucleophiles (272 references) [1714].
- The Nicholas reaction: the use of dicobalt hexacarbonyl-stabilized propargylic cations in synthesis (115 references) [1715].
- Tandem cyclization–cycloaddition reactions of rhodium generated carbenoids from a-diazo carbonyl compounds (123 references) [1716].
- Recent applications of the Suzuki–Miyaura cross-coupling reaction in organic synthesis (352 references) [1717].

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