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Review

Transition metals in organic synthesis: Highlights for the year 2004

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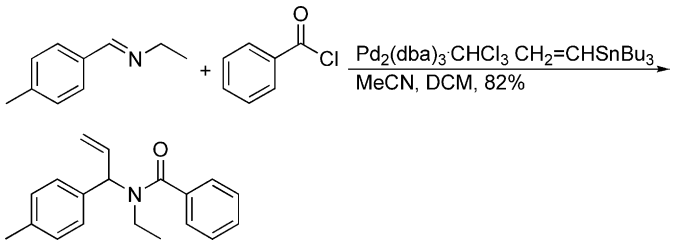
Abstract

A review with 1479 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 2004. Comprehensive literature coverage with extensive citations is presented. The field of transitionmetal chemistry continued to expand in 2004 and have a significant role in functional group transformations and organic total synthesis. 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. Emphasis was put on reactions used in total synthesis of biologically active compounds. Alkylations and most Michael type reactions of intermediately formed copper reagents have not been reviewed. 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.



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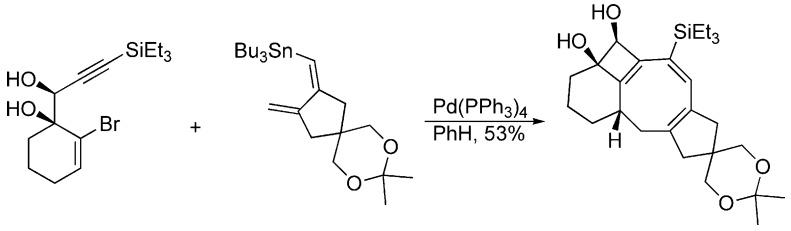

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2. Reactions of alkyl, alkenyl, alkynyl, benzyl, and aryl halides halides, sulfonates, alcohols, and nitriles

2.1. Carbon–carbon bond-forming reactions via transmetallation

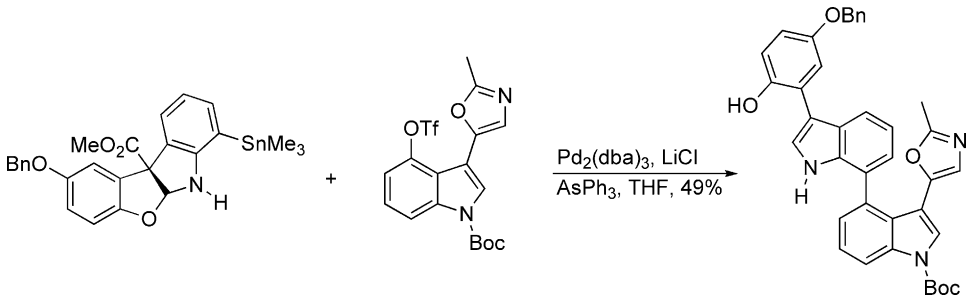
Palladium-catalyzed cross-coupling reactions of organotin reagents (Stille couplings) with a large variety of electrophiles continued to be developed and extensively used in organic synthesis. Palladium catalysts for cross-coupling of aryl chlorides with organotin reagents were described [1,2]. Addition of cesium fluoride to the catalyst system accelerated Stille couplings [3]. A Stille-type coupling of imines with organotin reagents in the presence of an acid chloride was described (Eq. (1)) [4]. A tandem Stille coupling 8π-electrocyclization was reported (Eq. (2)) [5]:

Intramolecular Stille coupling of 1,2-distannylated allylic ethers [6] and intermolecular couplings of α-sulfonamidoorganostannanes [7], 2-stannylcyclopropylamines [8], and 2-silyl-1-stannyl-1-arylethene [9] were reported. A variety of heterocyclic stannane derivatives such as 2-stannylindole [10], 3,4-distannylfuran [11], 4-stannylquinoline [12], 2-stannylthiazole [13], and 5-stannylisoxazole [14] were used in Stille-type couplings with aromatic halides and triflates.

Ethyl chloromethanoate, in a synthesis of difluorinated (hydroxymethyl)conduritol analogs [15], arenesulfonyl chlorides [16], benzylbromides [17], and 3-methylsulfanyl-1,2,3-triazines [18] were used as electrophiles in Stille-type couplings.

A large variety of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and alkenyltin reagents. Examples of ringsystems include, 2-iodopyrroles [19], dibromomethylenebutenolides [20], 4-chloroquinazolines and 5-trifloxyquinazoline [21], 6-bromo-2(3*H*)benzothiazolone [22], 5-iodo-3(2*H*)-pyridazinone [23], 4-bromoimidazole [24], 4-iodo- or 4-bromo-5,10-dihydropyrido[2,3-*b*:3,2-*e*]pyrazines [25], 3- and 5-1*H*-pyrazolo[3,4-*b*]pyridines [26,27], and 4-bromomethyl-2-chlorooxazole [28].

The intermolecular Stille reaction continued to be extensively used in organic total synthesis. Synthetic targets include, pancratistatin [29], tartrolone B [30], diazonamide (Eq. (3)) [31], spirofungin B [32], 13-methoxy-15-oxozoapatlin [33], (−)-antofine and (−)-cryptopleurine [34], (+)-puraquinoic acid [35], lignans [36], polycyclic meroterpenoids [37], epiuleine [38], (−)-apicularene [39], guanacastepene [40], cochleamycin A [41], kavalactones [42], cystothiazoles A and B [43], basiliskamides A and B [44], 15,16-dihydrominquartynoic acid [45], C11–C29 fragment of amphidinolide F [46], erogor-



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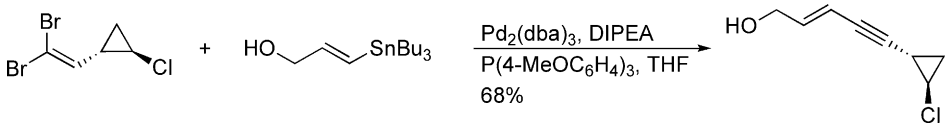
giaene [47], (−)-SNF4435 C and (+)-SNF4435 D [48], RK-397 [49], (+)-SCH 351448 [50], pateamine A [51], macquarimicins [52], steroids [53], oximidine III [54], xerulinic acid [55], apicularen A [56], 6β-hydroxyeuryopsin [57], cytostatin [58], gambierol analogs [59], subarine [60], arenarene B [61], 3-methoxyolivacine and olivacine [62], C10–C18 analogs of mycalamides and theopederins [63], a naturally occurring cyclopentenone [64], a number of isoquinoline alkaloids [65], strobilurin G, M, and N [66], (−)-strychnine [67], furocaulerpin [68], (+)-ochromycinone [69], (*E*)-13,14-dihydroxyretinols [70], *N*-protected staurosporines [71], tyrosine derivatives [72], C12–C19 fragment of amphidinolide E [73], and the tricyclic core of guanacastepene A [74]:

Polyunsaturated pyrrole metabolites were prepared from an acid chloride and a 2-stannylpyrrole derivative [75]. A Stille-type coupling of a 1,1-dibromoalkene to give an alkyne was used in a synthesis of callipeltoside A (Eq. (4)) [76]. Intramolecular Stille-type macrocyclization were used in synthesis of mangicols [77], dynemicin analog (Eq. (5)) [78], toward bafilomycin A_1 [79], and leinamycin related compounds [80]. Copper promoted Stille-type couplings were used in synthesis of formamicin [81], apoptolidin (Eq. (6)) [82], (−)-dictyostatin [83], and macrolactin analogs [84]:



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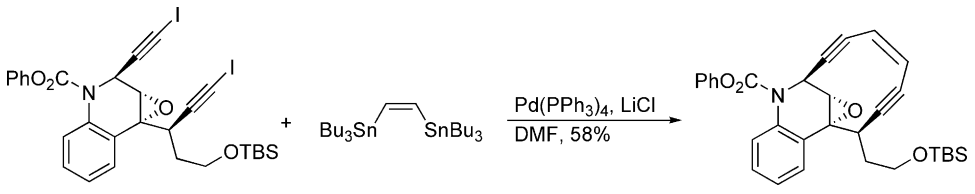

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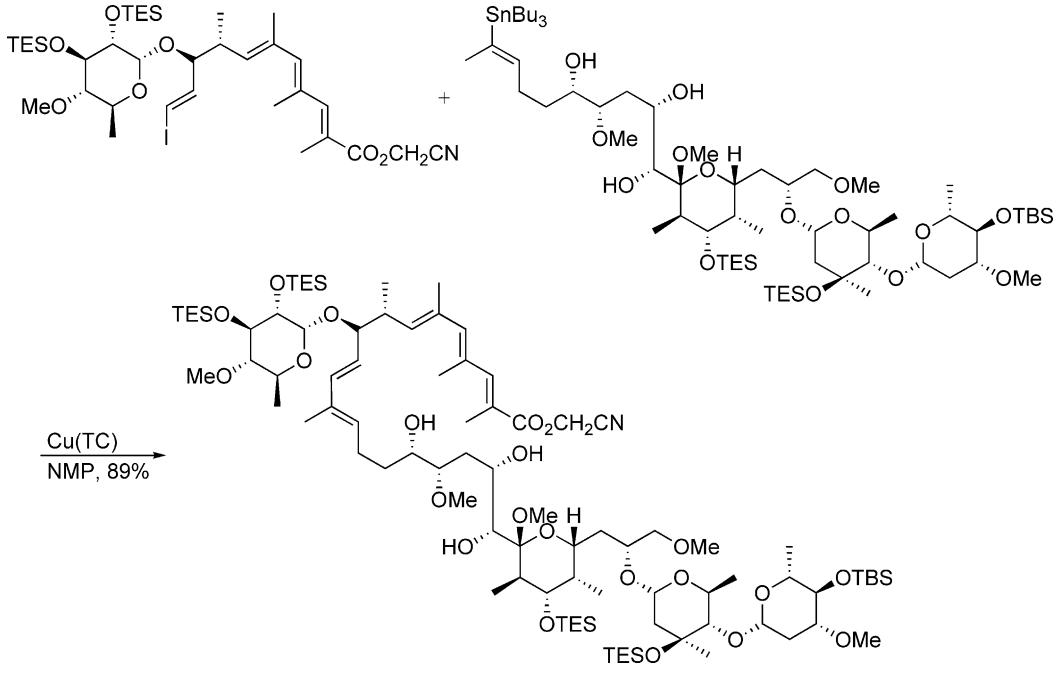
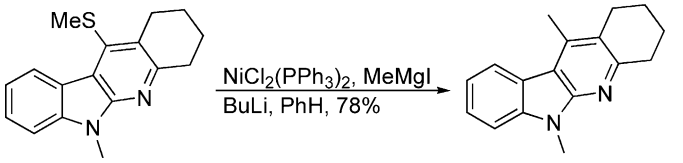


(6)

Nickel catalyzed the coupling of alkenylthioethers [85], aryl alkyl ethers [86], and neopentyl biphenylsulfonates [87] with Grignard reagents. A nickel-catalyzed coupling of an aryl thioether with methylmagnesium iodide was used in a synthesis

of cryptotachienine (Eq. (7)) [88]. Nickel complex-catalyzed cross-couplings of Grignard reagents with triflates were used in synthesis of 3-methoxyolivacine and olivacine [62]. Nickel catalyzed the coupling of aromatic sulfonamides with Grignard reagents [89]. Nickel-catalyzed asymmetric couplings of Grignard reagents with dibenzothiophens to give axially chiral biaryls (Eq. (8)) [90]:



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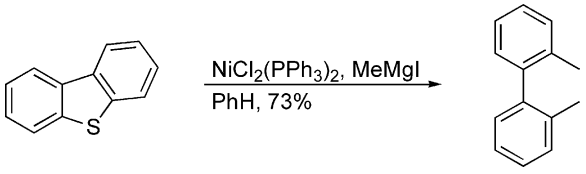

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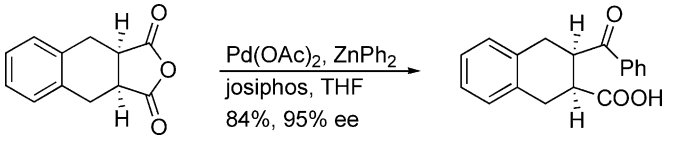
Palladium catalyzed the coupling of 4-iodo-2-bromopyridine [91] with Grignard reagents. Palladium catalyzed the formation of atropisomeric biaryls [92] by coupling of an aromatic triflate and phenylmagnesium bromide. Palladium-catalyzed asymmetric coupling reactions of alkenyl bromides with 1-phenylethylmagnesium chloride [93]. Palladium catalyzed the cross-coupling of alkynyl Grignard and lithium reagents with alkyl bromides and iodides [94].

Cobalt catalyzed the coupling of *N*-heteroaromatic chlorides with Grignard reagents [95]. Cobalt catalyzed couplings of allylic and benzylic Grignards with alkyl halides [96]. Cobalt catalyzed the mono-coupling of 1,2-dihaloethenes with trimethylsilylmethyl magnesium chloride [97].

Iron catalyzed cross-couplings of alkyl bromides, enol triflates, acid chlorides, and dichloroarenes with Grignard reagents [98–102]. Iron catalyzed the coupling of 1,1-dichloro-1-alkenes with Grignard reagents to give trisubstituted alkenes as the major and 1,2-disubstituted alkenes as the minor product [103]. Iron also catalyzed the coupling of 2,6,8-trichloropurine with a Grignard reagent [104]. This type of coupling was used in a synthesis of FTY720 [105] and agarospirol, hinesol, and α-vetispirene [106].

Palladium- or nickel-catalyzed couplings of organic halides and triflates with organozinc reagents (Negishi couplings) continued to grow. Catalysts and reaction conditions for facile coupling of organozincs with acid fluorides, chlorides, anhydrides, and thioesters were developed [107–109]. A palladium-catalyzed enantioselective coupling of meso-succinic anhydrides with organozinc reagents was described (Eq. (9)) [110]. 1-Chloro-2-iodoethene, 1-bromo-2-iodoethene, and 1,2-dibromoethene were used in mono-couplings with organozinc reagents [111]. 1,2-Dienylzinc reagents were coupled with aryl iodides [112]. Successive couplings of 1,1-dibromo-1-alkene with organozinc reagents were reported [113]. Dimethylzinc was coupled with aromatic bromides [114]:

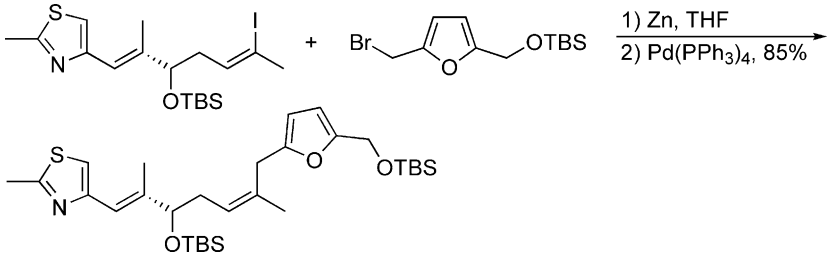


(9)

Palladium-catalyzed couplings of alkyl bromides and tosylates [115], of dodecanthiol esters [116], 5-iodo-3-benzyloxyisothiazole [117], and iodophenols [118] with alkylzinc reagents were reported. Alkenyl- and aryl-tellurides were used in place of halides and triflates [119]. Chiral 2,2′-diiodo-1,1′-binaphthyl was coupled with organozinc reagents [120]. Pyrrolylzinc [121] and 2-thiophenyl zinc and 2-thiazolyl zinc [13] reagents were coupled with aryl halides. Alkynylamide zinc reagents were used in Negishi type couplings [122].

Copper catalyzed the coupling of alkylzinc halides with α-chloroketones [123]. Nickel catalyzed the coupling of alkynylzinc reagents with aromatic cyanides [124].

Synthetic targets using the palladium-catalyzed Negishi coupling include, MetAP-2 inhibitors [125], an ant venom [126], tartrolone B [30], (+)-phomopsidin [127], aromatic bisabolenes [128], capensifuranone [129], (−)-kazusamycin A [130], (−)-callystatin A [131], 6-(hydroxymethyl)purines [132], 6,7-dehydrostipiamide [133], scyphostatin side chain [134,135], (+)-scyphostatin [136], furano-epothilone D (Eq. (10)) [137], 4-aza epothilone analogs [138], difluorinated (hydroxymethyl)conduritol analogs [15], GE 2270 D2 [139], and (+)-tanikolide [140]. Hydrozirconations of terminal alkynes followed by transmetallation to zinc and cross-coupling were used in syntheses of (−)-callystatin A [131] and zincophorin [141]. Alkenyl aluminum and zirconium reagents prepared in situ via hydro- or alkylmetallation of terminal alkynes were coupled with alkenyl iodides in the presences of a palladium-indium catalyst system [142]:

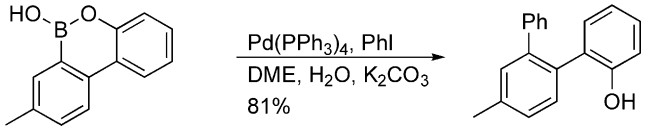


(10)

The Suzuki cross-coupling, i.e., palladium-catalyzed coupling of organoboronic acids and esters with organic halides and triflates, continue to see substantial use in organic chemistry. New and very versatile ligands or catalyst systems were reported for Suzuki couplings [143–161]. Bis(dibenzylidenacetone)palladium complexes can be tuned using substituents on the aromatic rings. Electron donating groups on the ring accelerated Suzuki reactions [162]. Selective Suzuki-coupling at an sp^2-carbon was observed using 2-bromo-3-acetyloxy-1-alkenes [163]. Enantioselective Suzuki couplings of meso-ditriflates were described [164].

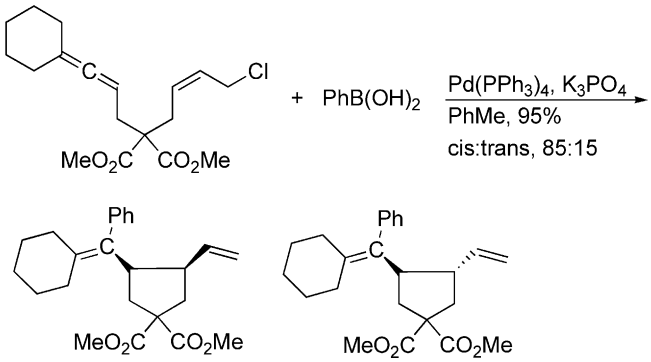
2,6-Disubstituted aryl chlorides were coupled with 2,6-disubstituted arylboronic acids [165]. 1-Chloro-2-iodoethene [111], arylsulfonyl chlorides [166], and benzylic halides [167] were coupled with organoboron reagents. Nitrogen heterocycles can also be used in place of halides and triflates. Palladium-catalyzed Suzuki reactions of 1-aryltriazenes, 6-(imidazol-1-yl)-, 6-(benzimidazol-yl)-, 6-(1,2,4-triazol-4-yl)purine nucleosides [168,169], and 3-methylsulfanyl-1,2,3-triazines were described employing boronic acid derivatives [18].

β-Chloroalkylidene and arylidene malonates [170], aryl perfluorooctylsulfonates [171], 2-(bromoalkylidene)-tetrahydrofurans and -pyrrolidines [172,173], and haloaromatic phosphine oxides [174] were coupled with organoboronic acids. 3,4-Dihydropyrimidine-2-thiones were coupled with aromatic boronic acids in the presence of a palladium–copper catalyst system (Eq. (11)) [175]. Thiol esters were also coupled with alkylboronic compounds to afford unsymmetrical ketones [176] and 2-pyridyl esters were coupled with aryl boronic acids in a related fashion [177]. Sulfonyl chlorides were coupled with aryl- and alkenyl-boronic acids with loss of sulfur dioxide (Eq. (12)) [178]. Palladium catalyzed Suzuki couplings of boroxarenes (Eq. (13)) [179]:



(13)

Palladium catalyzed a sequential cyclization-Suzuki coupling of allylic substrates containing a 1,2,7-triene (Eq. (14)) [180]. Palladium catalyzed a direct coupling of propargylic alcohols with aryl boronic acids to give 1,2-dienyl- substituted aromatic compounds (Eq. (15)) [181]:



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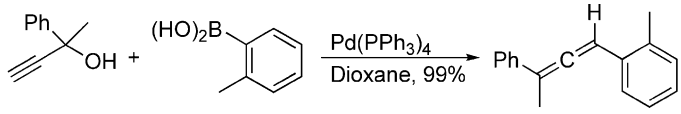

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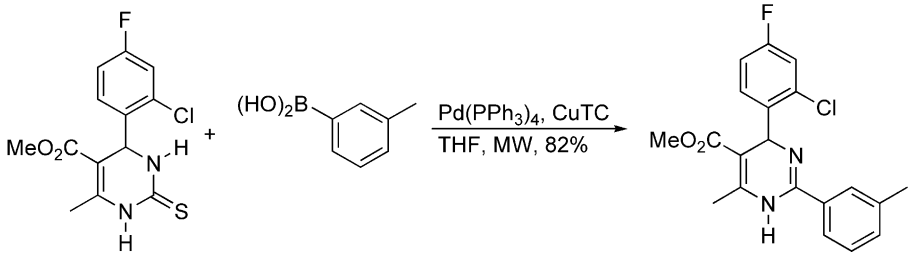

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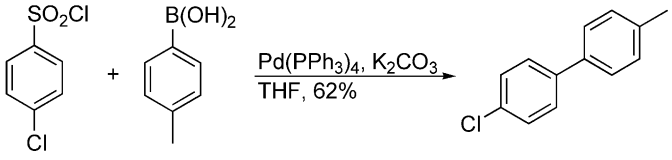
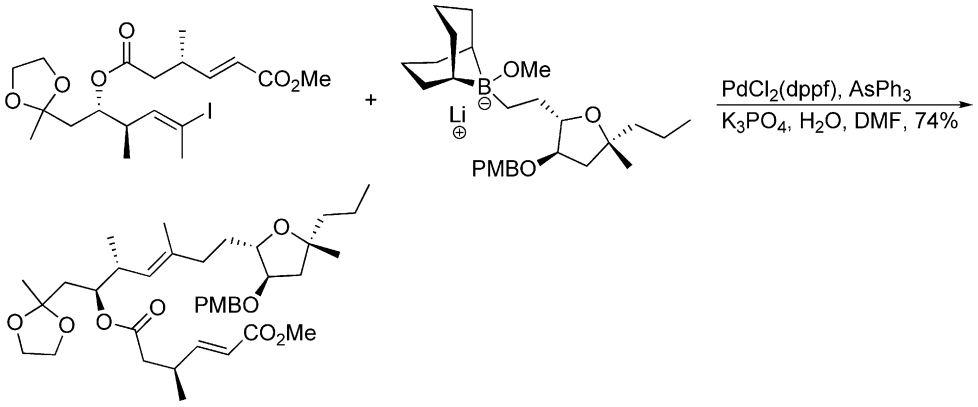


(12)

Suzuki couplings of potassium cyclopropyltrifluoroborates were described [182]. Potassium aryl trifluoroborates and lithium alkynyltriisopropoxyborates were coupled with aryl chlorides [183,184] and with 4-bromocoumarine and tetronic acid bromide [185]. Couplings of alkyl borates were used in syntheses of amphidinolide X (Eq. (16)) [186], kendomycin [187], and coupling of an alkenyltrifluoroborate was used in a synthesis of oximidine II (Eq. (17)) [188]:



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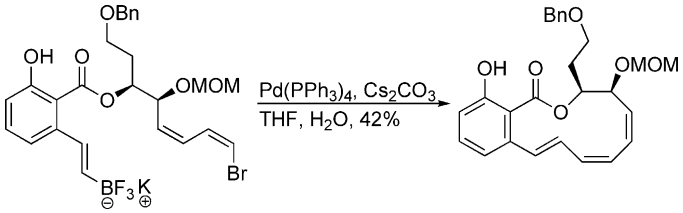


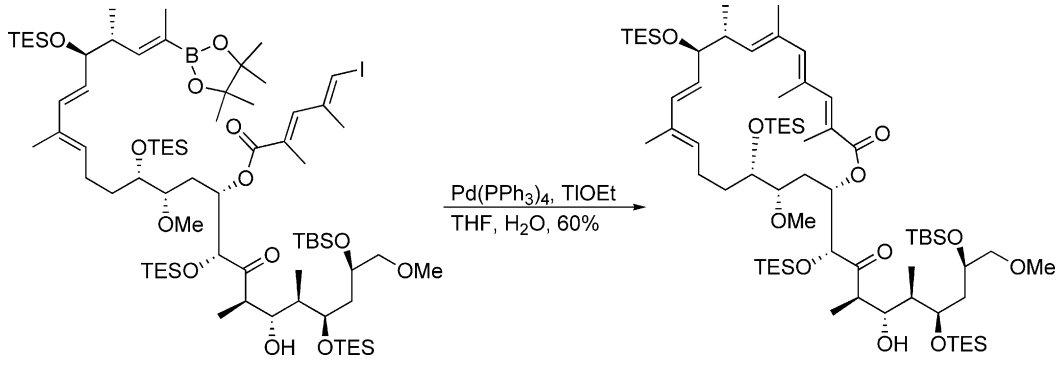
(17)

A large variety of functionalized heterocyclic halides and sulfonates were employed in the palladium-catalyzed Suzuki reaction. Heterocyclic ring-systems such as, 5-iodo-3-benzyloxyisothiazole [117], 2,6-substituted 4-chlorobenzimidazole [189], a variety of bromo and iodoindoles [190–192], 6-bromo-1-phenyl-3,4-dihydropyrrole[1,2-*a*]pyrazine [193], 2-trifloxypyrazine [194], 3- and 5-nonafloxypyrazoles [195], 5-iodo-3(2*H*)-pyridazinone [23,196], 4-trifloxycoumarins [197], 2,3-dichlorquinoxaline [198], 8-bromoadenosine [199], 2,6,8-trichloropurine [104], 6-chloropurine [200], 4-chloroquinazolines and 5-trifloxyquinazoline [21], 4-bromoimidazole [24], 4-iodo- or 4-bromo-5,-10-dihydropyrido[2,3-*b*:3,2-*e*]pyrazines [25], 3- and 5-iodo-1*H*-pyrazolo[3,4-*b*]pyridines [26,27], 4-bromomethyl-2-chlorooxazole [28], 5-bromo-thiazole, -oxazole, -1,3,4-thiadizole, and -1,3,4-oxadiazole [13], and 7-chloro- and 7-iodo-3,4,5,6-tetrahydro-2H-pyrido[4,5-*b*]- and [2,3-*b*]-1,5-oxazine-6-ones [201] were used in Suzuki type cross-couplings.

A 4,5-dichloropyrazin-3-one was selectively coupled in the 5-position [202], 3,5-dichloropyrazin-2-one in the 3-position [203], 2,4-dichloropyrimidine in the 4-position [204], and 3,5-dibromo-2-pyrone in either the 3- or 5-position depending on the conditions [205] using organoboronic acids.

A variety of heteroaryl boronic acid derivatives were used such as, 2-, 5-, 6-, and 7-indolyl [10,190,192], 1-phenyl-3,4-dihydropyrrole[1,2-*a*]pyrazin-6-yl [193], 5-pyridyl [206], 2-pyridyl [207], 7-[1,2,3]triazolo[1,5-*a*]pyridyl and [1,2,3]triazolo[5,1-*a*]isoquinyl [208], 5-(4-oxo-3,4-dihydroquinazolinyl) [−21], 3-pyrrolyl [209], 3-benzothienyl used in synthesis of benzo[*b*]thienyl dehydrophenylalanines [210], and 4-oxazolyl [211]. A 2-azulenylboronic ester was also used in coupling reactions [212].

A number of syntheses wherein aryl or alkenyl boronic acids or esters were coupled with an aryl-, heteroaryl-, alkenyl-halides, -triflate, and phosphonates were reported. Synthetic targets included, an ant venom [126], hippadine [213], lysergic acid [214], a fused pyrano[2,3-*d*]pyrimidin-7-one [215], eupomatilone-6 [216], (+)-phomopsidin [127], panomifene [217], lamellarin G trimethyl ether [218], vulpinic acid [219], korupensamines A and B [220], eupomatenoid 6 [221], *cis*- and *trans*-bupleurynol [222], ent-EI-1941-2 and epi-ent-EI-1941-2 [223], rocaglaol analogs [224], (+)-murisolin [225], formamicin [81], dragmacidin F [226], diazonamide A [227–229], panomifene [230], apoptolidinone [231], apogalanthamine analogs [232], spiro-B-norestradiol [233], 6-chlorohyellazole [234], tricyclic fragment of vindoline [235], machaeriol A and B [236], TAK-779 [237], atropisomeric biaryls [92,238], dehydroaltenusin [239], aporphinoids [240], lamellarins Q and O [241], pyridovericin [242], a CCR5 antagonist [243], 18-, 19-, and 20-HETE [244], ABCDE ring fragment of ciguatoxins [245], 6,8-heneicosadien-11-ones [246], a gem-difluorinated biflavonoid [247], (+)-laurenyne [248], and (+)-bongkrekic acid [249]. An intramolecular coupling was used in a synthesis of apoptolidinone (Eq. (18)) [231]:

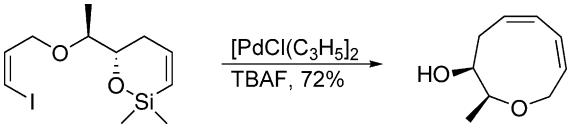


(18)

Alkyl boron reagents derived from hydroboration of terminal alkenes with 9-BBN were used in synthesis of for example clavulaones [250], (+)-(2′*S*,3′*R*)-zoapatanol [251], taxane skeleton [252], quinine and quinidine [253], salicylhalamide [254], unsaturated 1-monoacyl glycerols [255], C1-alkyl glycals [256], and phenylalanine analogs [257]. Acrolein diethylacetal was hydroborated and the product used in Suzuki couplings [258].

Copper catalyzed the coupling of aryl sulfonates and halides with arylboronic acids [259]. Nickel and palladium catalyzed the coupling of alkylbromides [260]. Nickel catalyzed the coupling of arylsulfonates with aryl boronic acids at room temperature [261]. Platinum catalyzed the coupling of aryl iodides with alkynyl triisopropoxy borates [262].

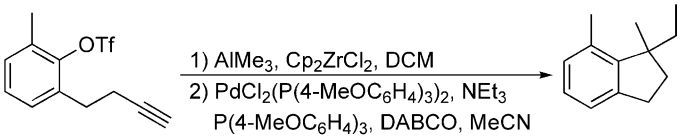
Silicon reagents continue to be used in coupling reactions using palladium catalysts. Palladium-catalyzed cross-couplings of aryl triethylammonium bis(catechol) silicates with aryltriflates [263], aryl siloxanes with aryl bromides and chlorides [264,265], alkenyl siloxanes with alkenyl and aryl halides (Eq. (19)) [266–268], 2-indolyldimethylsilanols with aryl halides [269], aryl triethylammonium bis(catechol) silicates with aryl bromides [270], a silalactone with iodobenzene [271], and aryl triallylsilanes with aryl chlorides [272]. High ipso-selectivity was observed using 2-dimethylphenylsilyl-1-alkenes and iodobenzene [273]. Nickel catalyzed the coupling of organosilicone compounds with alkyl bromides [274]. Copper catalyzed couplings of 2-(1-hydroxyalkyl)arylsilanes with alkyl halides [275]:



(19)

Some more unusual cross-coupling reactions employing organometallic reagents were described. Alkenylzirconium reagents were coupled with alkyl bromides [276] or aromatic bromides [277] using palladium or nickel catalysts. An alkenyl zirconium species derived from hydrozirconation of a terminal alkyne was used in a synthesis of *cis*- and *trans*-bupleurynol [222].

Palladium catalyzed the coupling of dimethylalkynylaluminum reagents with aryl halides and triflates [278]. Palladium catalyzed the coupling of alkenylaluminum reagents, prepared by zirconium-catalyzed methylalumination of terminal alkynes, with tethered iodides and triflates with concurrent migration of a methyl group (Eq. (20)) [279]. Ruthenium catalyzed the coupling of organobismuth compounds and aryl and alkenyl chlorides [280]. Rhodium catalyzed the coupling of esters with organoboron compounds forming unsymmetrical ketones (Eq. (21)) [281]. Ruthenium catalyzed an interesting coupling of aromatic ketones having adjacent methoxy groups (Eq. (22)) [282]:



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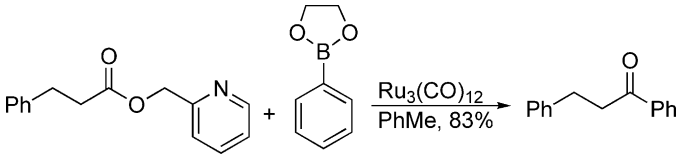

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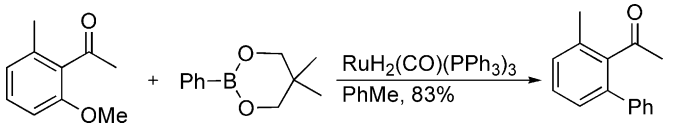


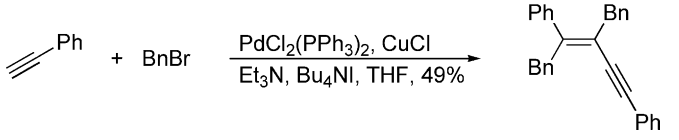
(22)

2.2. Carbon–carbon bond-forming reactions using terminal alkynes and other carbon nucleophiles

The palladium-catalyzed coupling between terminal alkynes and aryl and alkenyl halides or triflates, usually called the Sonogashira coupling, was one of the more frequently utilized carbon–carbon coupling reaction. A number of new and effective catalyst systems for the Sonogashira coupling have been described [156,283–285].

Palladium catalyzed the coupling of aromatic acid chlorides [286] and *N*-methoxy-*N*-methylcarbamoyl chloride [287]. Selective Sonogashira-coupling at the sp^2-carbon was observed using 2-bromo-3-acetyloxy-1-alkenes [163]. A β,β-dibromodehydroalanine derivative was used in double Sonogashira couplings [288]. Aryl perfluoroalkanesulfonates [289], dodecanethiol esters [116] alkenyltellurides were used [290–292], and 1-chloro-2-iodoethene [111] were used in Sonogashira couplings.

Ynamides were used as the alkyne component [293]. Highly substituted enynes were unexpectedly obtained upon reaction of benzyl halides with terminal alkynes (Eq. (23)) [294,295]. Palladium catalyzed a coupling of aromatic halides with propargylic alcohols to give alkenone intermediates. Further reactions in the same pot with an aldehyde furnished 1,4-diketones (Eq. (24)) [296]:



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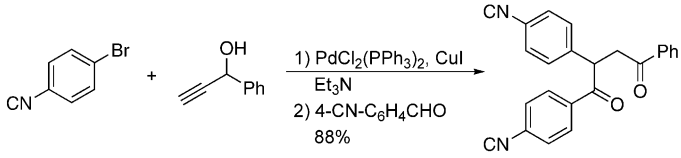


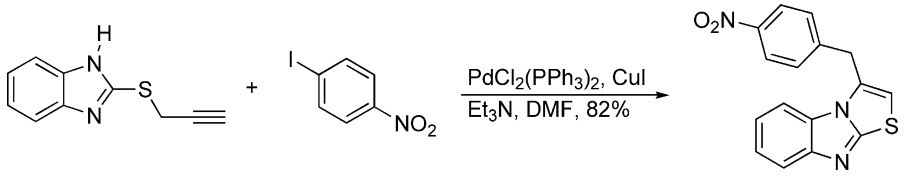
(24)

Heterocyclic ring systems were shown to readily participate in Sonogashira-type couplings. For example, reactions of

bromoquinolizium cations [297], 2′-deoxy-5-iodocytidine [298], 5-iodocytocine [299], 7-deaza-7-iodo-purines [300], 6-chloropurine, 2-chloro-, and 8-bromoadenine [301], 5-iodouridine, 8-bromoguanosine, 8-bromoadenosine, and iodouracil [302], 5-iodo-2′-deoxyuridine [303], 3-iodoindole [190], 4-iodophosphaisocoumarins [289], 3- and 4-halopyrazine [304–306], 1-chloropyrido[3,4-*d*]pyridazine [305], 5-iodo-3(2*H*)-pyridazinone [23], 4,6-dichloro-2-aminopyrimidine [307], 4-halopyrrolo[2,3-*d*]pyrimidine [308], 6-iodoisocoumarine [309], 4-bromoimidazole [24], 4,5-diiodoimidazole [310], 4-iodo- or 4-bromo-5,-10-dihydropyrido[2,3-*b*:3,2-*e*]pyrazines [25], and 3- and 5-iodo-1*H*-pyrazolo[3,4-*b*]pyridines [26,27] derivatives were reported.

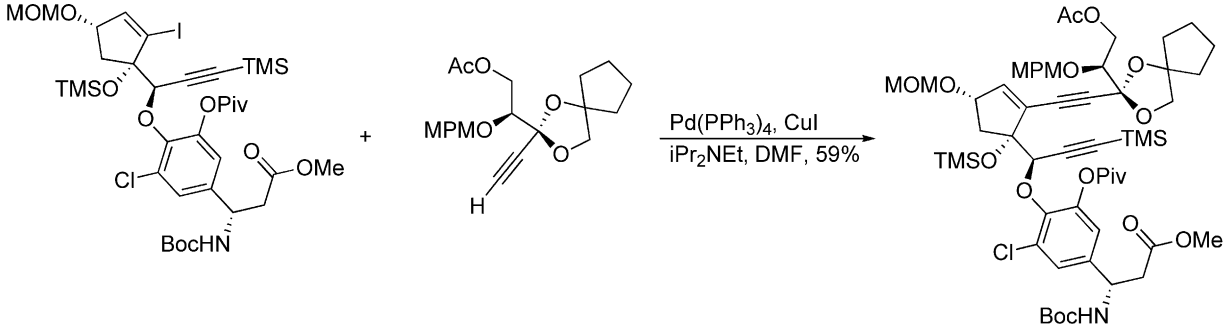
Sonogashira coupling of 2-trifloxybenzene amides followed by indolization was reported [311]. Tandem Sonogashira coupling of 2-iodoarenes with 2-aminophenylethynes followed by intramolecular indole formation was reported [312]. A related tandem Sonogashira coupling-annulation of 3-bromo-2-amino-quinoxaline and -pyrazine derivatives with terminal alkynes to give pyrrolo[2,3-*b*]quinoxalines [313] and pyrrolo[2,3-*b*]pyrazines [306,314] was developed. In situ formed propargylic amines were reacted with 2-iodophenol or 2-iodobenzeneamide to give 2-aminomethylsubstituted benzofurans or indoles, respectively [315].

Aminopropargylpyridines were prepared by Sonogashira reaction of bromo-pyridines with propargyl bromide in the presence of a secondary amine [316]. A tandem Sonogashira coupling followed by intramolecular alkyne-hydroamination was reported (Eq. (25)) [317]:



(25)

The Sonogashira reaction continued to be extensively used in organic synthesis. Examples of synthetic targets include, pancratistatin [29], an ant venom [126], hippadine [213], tetracenomycin A_2 [318], frondosin B [319], (−)-frondosine [320], (−)-(*E*)-15,16-dihydrominquartynoic acid [321], tetraponerin T6 [322], (−)-cochleamycin A [323], defucogilvocarcin M [324], duocarmycin SA [325], 15,16-dihydrominquartynoic acid [45], (−)-disorazole C_1 [326], (−)-gigantecin [327], azaphilones [328], C-1027 chromophore (Eq. (26)) [329,330], murisolin [331], reticulatain-1 [332], *cis*-solamine [333], dihydroxerulin and xerulin [334], (+)-diplyne [335], leinamycin related compounds [80], carbohydrate derived enynes [336], diyne-core of the C1027 chromophore [337], 12(*S*),20-DiHETE [338], and resolvin D2 [339]. An intramolecular Sonogashira coupling was in an synthesis toward maduropeptin chromophore (Eq. (27)) [340]:



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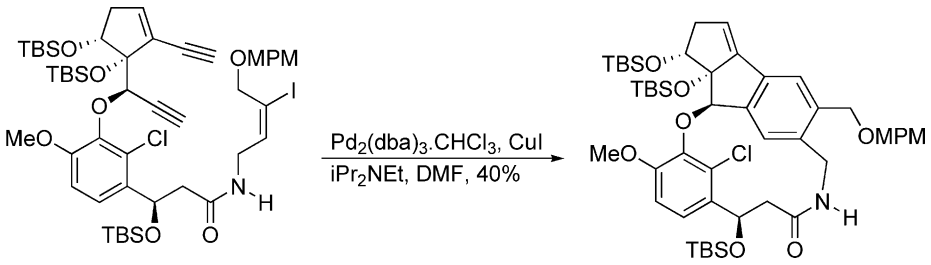
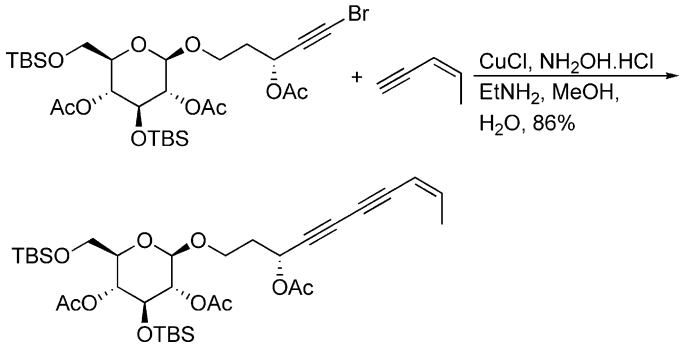


(27)

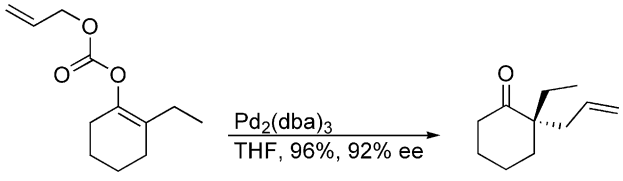
Related coupling reactions using copper catalysts have also been reported. Copper catalyzed the coupling of terminal alkynes with alkenyl iodides [341] and aryl halides [342,343]. Copper-catalyzed couplings of alkynyl bromides with terminal alkynes, Cadiot–Chodkiewicz-type reaction, were used in synthesis of (−)-nitidon [344], panaxydol [345], bidensyneosides A_2 and C (Eq. (28)) [346], and (+)-diplyne [347]:



(28)

A copper-catalyzed coupling of propargylic halides with terminal alkynes was used in synthesis of anandamide analogs [348]. Copper catalyzed a sequential coupling of 2-bromo-3-trifluoroacetamidoquinoxaline with terminal alkynes followed by intramolecular hydroamination to form pyrrolo[2,3-*b*]quinoxalines [349]. Copper catalyzed the formation of alkynyl selenides, sulfides, and tellurides from terminal alkynes and diselenides, disulfides, and ditellurides, respectively [350]. Nickel catalyzed the coupling of alkenyltellurides and terminal alkynes [351].

Other carbon nucleophiles were employed in palladium-catalyzed carbon–carbon bond-forming reactions employing organohalides, esters, and triflates. Palladium catalyzed a benzylation of activated methine compounds using benzylic carbonates in the presence of 1,5-cyclooctadiene [352]. Palladium catalyzed the arylation of *N*-(diphenylmethylidene)glycinate with 6-iodopurines [353]. Silyl enol ethers were used as nucleophiles in palladium-catalyzed reactions with aryl bromides [354]. Palladium catalyzed α-arylations of ester-derived TMS-enol ethers and enolates of esters and amides [355–357], of acetoacetate esters [358], and diethyl malonate [359]. An asymmetric alkylation of ketone enolate derived allyl alkenylcarbonates was reported (Eq. (29)) [360]. Palladium-catalyzed asymmetric alkylations of 2-phenyl-2-oxazoline-4-carboxylic acids *t*-butyl ester [361]. The influence of added base on the alkylation of sulfones, cyanoacetic ester and malononitrile with aryl bromides was examined [362]. Molybdenum catalyzed asymmetric regioselective alkylations of allylic carbonates using 5*H*-oxazol-4-ones as the pronucleophile (Eq. (30)) [363]. Rhenium catalyzed direct arylation of propargylic alcohols and this reaction was used in a synthesis of *O*-methyldetrol, mimosifoliol, podophyllotoxin and β-apopicropodophyllin (Eq. (31)) [364]. Iridium catalyzed the coupling of aromatic iodides and benzene to give biphenyls [365] and copper catalyzed the coupling of aroyl halides with activated methylene compounds [366]:



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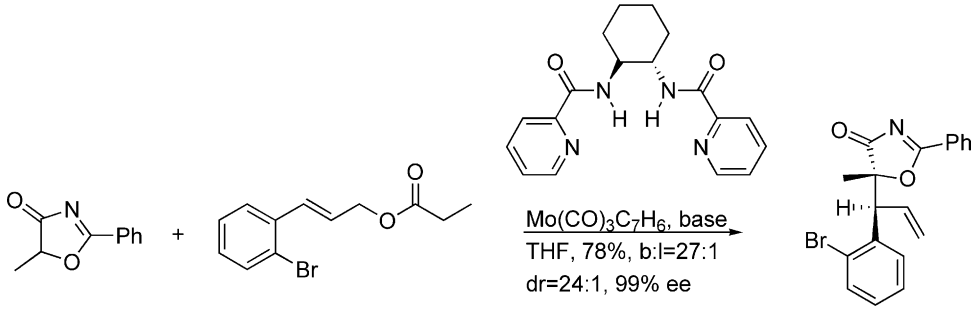

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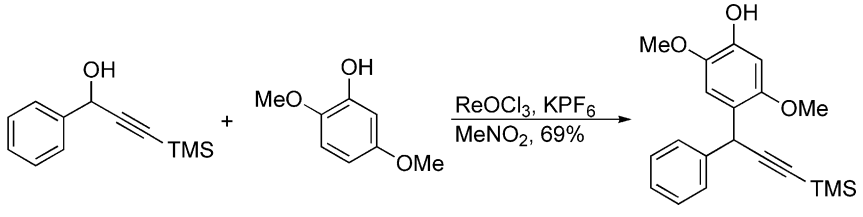
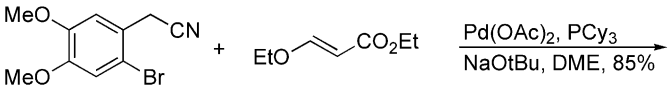
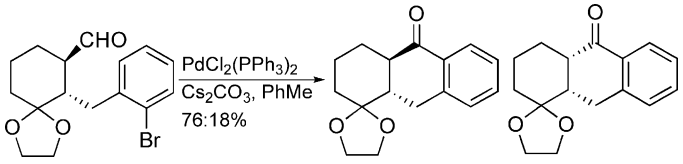


(31)

Palladium catalyzed an intramolecular coupling of ketone enolates with a pendant pyridinyl bromide to form tropane analogs [367]. Intramolecular reactions of alkenyl- and arylbromides with β,γ-unsaturated nitronates were described [368]. An intramolecular palladium-catalyzed cyclization of in situ formed enolates was developed (Eq. (32)) [369]. α-Arylations of aliphatic ketones, aldehydes, and nitro groups were described. In some cases using aliphatic aldehydes, carbonyl arylation was observed (Eq. (33)) [370]. Intramolecular palladium-catalyzed α-alkenylations was used to prepare (+)-12-methoxy-N_a-methylvellosimine, (+)-12-methoxyaffisine, and (−)-fuchsiaefoline [371] and cytisine [372]. Palladium catalyzed the formation of indoles from 2-chlorobenzenamines and ketones (Eq. (34)) [373]:





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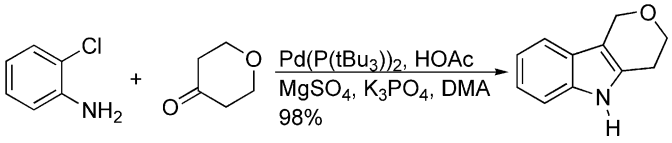
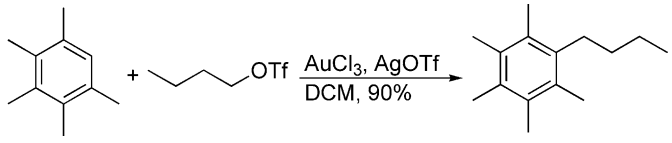


(34)

Heterocyclic aromatic substrates were also used as carbon nucleophiles, at least in a formal sense, without initial deprotonation. Rhodium catalyzed the reaction between heterocyclic compounds and aryl iodides [374]. Gold catalyzed direct alkylation of arenes with primary sulfonates (Eq. (35)) [375]. Palladium catalyzed the coupling of thiophenes and thiazole [376], in the 2-position of indoles [377], C3-arylation of indolizines [378], and in the 2-position of 3-substituted benzothiophens [379,380] with aryl halides. Intramolecular couplings in the 2- and 3-position of indoles and the 3-position of furans and thiophenes were reported and this reaction was used in a synthesis of (−)-frondosine (Eq. (36)) [381]:



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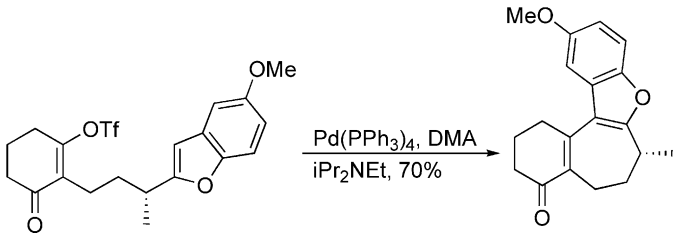
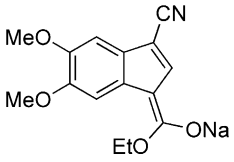


(36)

The palladium-catalyzed intramolecular variation using aromatic carbon nucleophiles and halides and triflates was used to prepare carbazoles and dibenzofurans [382], biaryls [383], aporphine alkaloids [384], phenanthridones [385], benzonaphthazepines [386], anhydrolycorinone, anhydrolycorin-7-one, assoanine, and oxassoanine [387], 5*H*-pyridazino[4,5-*b*]indoles [196], ancistrotanzanine B and ancistroealaine A [388], 8,9-dihydroimidazo[4,5-*c*]pyrrolo[3,2-*g*]quinolin-4(5*H*)-one [389], amaryllidaceae alkaloids [390], luotonins A and B and rutecarpine [391], toward stegane type compounds [392], and aporphine [393].

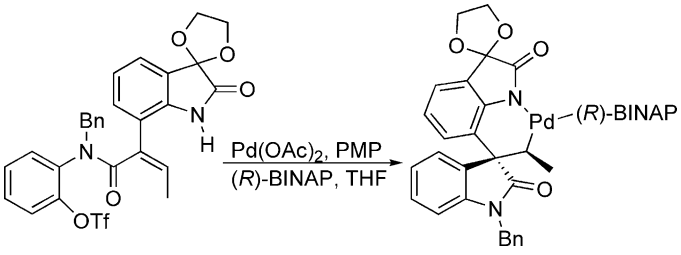


(32)

Palladium catalyzed the cyanation of aromatic halides with zinc dicyanide, potassium cyanide, or hexacyanoferrate [394]. The addition of zinc diacetate increased the yield of cyanation [395]. This reaction was also catalyzed by organotin compounds [396]. A palladium-catalyzed cyanation of a 1-iodo-1-alkene was used in a synthesis of (−)-borrelidin [397]. A palladium-catalyzed cyanation of an aromatic iodide was used in a synthesis of [3,4,8-^{13}C3]daidzein [398]. Palladium catalyzed the cyanation of halothiophens [399]. Copper catalyzed the cyanation of aryl diazonium salts using potassium cyanide [400].

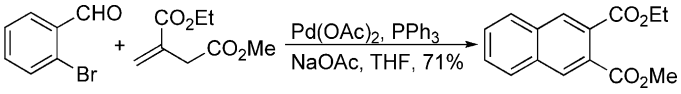
2.3. Carbon–carbon bond-forming reactions via insertion of alkenes

The Heck (or Mizoroki–Heck) reaction remained one of the most versatile methods for the alkylation of alkenes. A number of new and efficient catalyst systems was reported [156,401,402]. The alkene insertion product from a Heck reaction was isolated and characterized although easily accessible β-hydrogens were present (Eq. (37)) [403]. Enol esters formed from aromatic acids underwent a decarbonylative Heck reaction [404]. Aromatic carboxylic acids were used in place of halides via an initial decarboxylation [405]. Benzyl trifluoroacetates [406] and aryltelluronium salts [407], tetraphenylantimony carboxylates [408], were used in place of halides and triflates. α-Haloalkenyl ethers [409] and 1,2-ethanediol acetal of propenal [410] were used as the alkene in Heck reactions. α-Arylation of *N*-ethenylacetamide was observed using aromatic triflates [411]:



(37)

A double Heck reaction of *t*-butyl acrylate using aryl iodides in water was reported [412]. A tandem Heck reaction-aldol condensation of 2-bromobenzaldehydes to give benzannulated products was developed (Eq. (38)) [413]. 2,4-Diiodo-1-butenes were used in Heck reactions resulting in coupling at the 2-position and elimination at the 4-position [414]. Palladium catalyzed the formation of 1,2,3,4-tetrahydroisoquinolines and 2,3,4,5-tetrahydro-1*H*-2-benzazepines via tandem ortho-alkylation, Heck reaction, and Michael addition (Eq. (39)) [415]. An intermolecular Heck reaction of 1-bromo-3-hydroxy-1,5-dienes resulted in the formation of substituted cyclopentenones (Eq. (40)) [416]:



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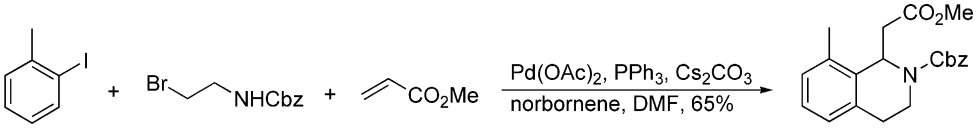

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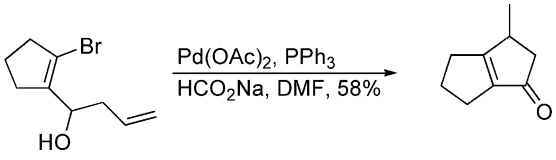
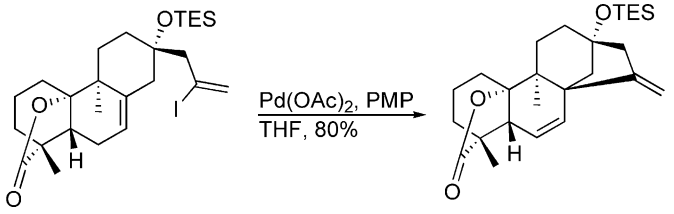


(40)

Halides and triflates of a variety of heterocyclic ring systems were used, such as 3-iodoindole [190], 6-iodoisocoumarin [309], 5-iodo- and 5-bromo-3(2*H*)-pyridazinones [23], 4-bromoimidazole [24], 3,5-dichloropyrazin-2-one in the 3-position [203], 4-iodo- or 4-bromo-5,-10-dihydropyrido[2,3-*b*:3,2-*e*]pyrazines [25], and 3- and 5-iodo-1*H*-pyrazolo[3,4-*b*]pyridines [26,27].

The intermolecular Heck reaction was used in organic synthesis of for example, dihydrochalocones [417], securinine and (−)-allonorsecurinine [418], thymidine analogs [419], steroids [53], clavicipitic acid [420], flavonoids [421], 8-alkenyl-substituted dihydrofuroangelicins [422], glycocinnasperimicin D [423], 2,3-fused indoles [424], virideno-mycin [425], methylated reservatrol and analogs thereof [426], tropinone derivatives [427], (−)-ephadradine A [428], camptothecin [429], oestrone [430], cryptophycin-24 [431], guanine analogs [432], (*R*)-(+)-6-(1,4-dimethoxy-3-methyl-2-naphtyl)-6-(4-hydroxyphenyl)hexanoic acid [433], and cinacalet analogs [434].

Intramolecular Heck reactions were used as the key step toward an array of synthetic targets for example, 13-methoxy-15-oxozoapatlin (Eq. (41)) [33], hamigeran B [435], carbocyclic core of mensacarcin [436], cyclopenta[*b*]indol-1-ones and carbazole-4-ones [437], nitrogen-heterocycles [438], putative structures of homopumilotoxins 235C and 233F (Eq. (42)) [439], lycoramine [440], benzofurans [441], 22-hydroxyacuminatine [442], bicyclic compounds [443]. Intramolecular reductive Heck reactions were used in syntheses of (−)-incarvillateine, (+)-incarvine C, and (−)-incarvilline (Eq. (43)) [444]. Reductive Heck reactions were reported using 6-halopurines [445]:



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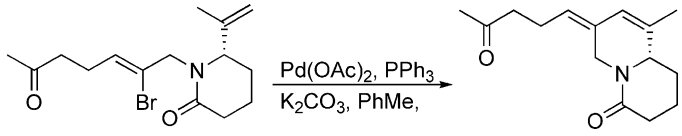

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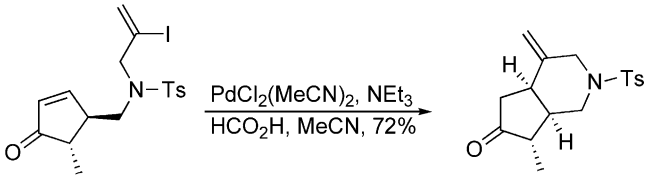
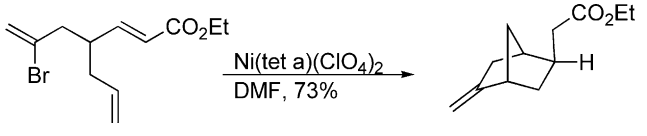


(43)

A nickel-catalyzed intramolecular Heck-type reaction of bromo-trienes was developed (Eq. (44)) [446]. Electrochemical intermolecular reductive Heck-type reactions were described [447]. Iron catalyzed a cyclization of 2-chloro-1,6-dienes in the presence of a trialkylaluminum reagent (Eq. (45)) [448]:



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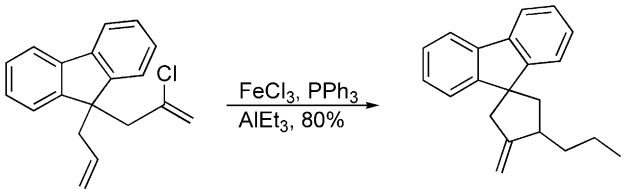
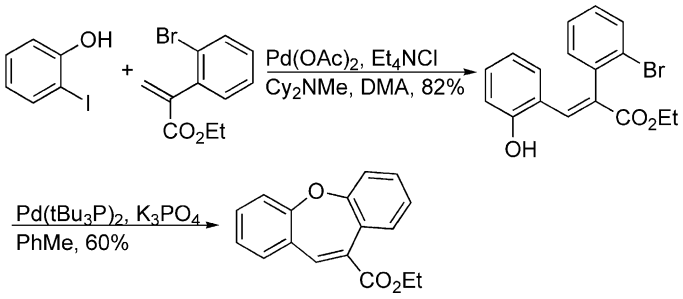


(45)

Reactions involving an oxidative addition of palladium to organic halides and triflates followed by alkene insertion and a termination step, different from the β-hydrogen elimination observed in the Heck reaction, were extensively examined. Palladium catalyzed a Heck-reaction-hydroamination using aryl bromides and 1-amino-4-alkenes (Eq. (46)) [449]. Sequential intermolecular Heck reaction followed by intramolecular amination or alkoxylation was used to prepare medium-sized heterocycles (Eq. (47)) [450] and pyrroles fused to an aromatic ring (Eq. (48)) [451]. An oxidative addition-1,4-migration-annulation sequence furnished fused polycyclic compounds (Eq. (49)) [452]. A palladium–norbornene system was used for the preparation of 6-phenanthridinones from aryl iodides and 2-bromo-arylamides (Eq. (50)) [453]. The same type of catalyst system was used in a coupling of an aryl iodide, an aryl bromide and an electron deficient alkene (Eq. (51)) [454]. An oxidative addition-intermolecular insertion-intramolecular amination sequence was used in a synthesis of (−)-pumilotoxin C [455]. Oxidative addition and insertion of 2-allylbenzeneamines followed by intermolecular amination and *N*-arylation produced *N*-aryl-2-benzylindolines (Eq. (52)) [456]. Palladium catalyzed the coupling of allylamines, an activated alkene, and an unsaturated halide or triflate to give pyrrolidines (Eq. (53)) [457]:

(46)



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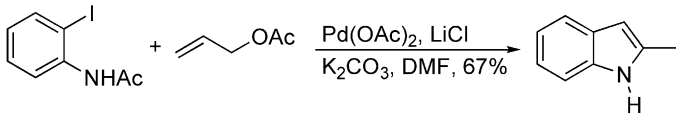

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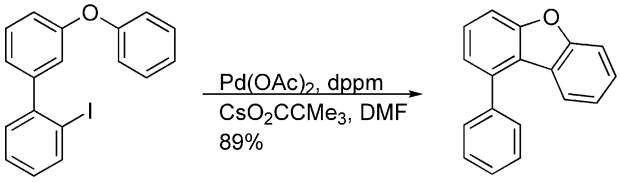

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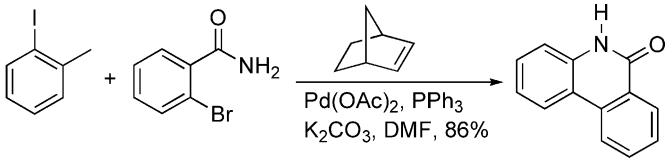

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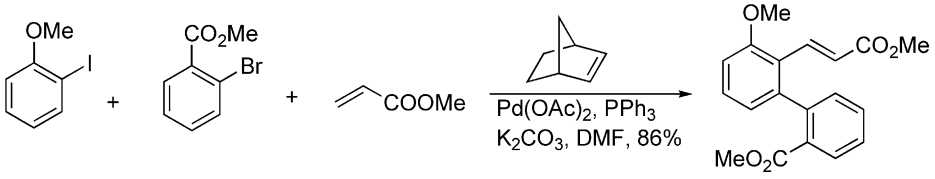

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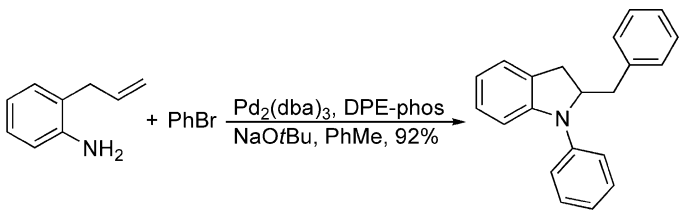

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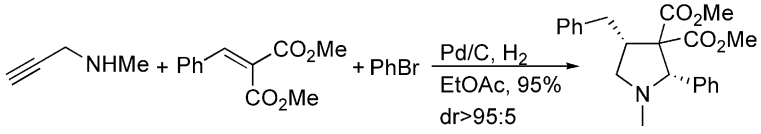
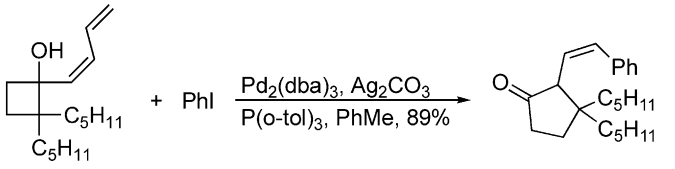


(53)

Oxidative addition-1,3-diene insertion followed by an intramolecular ring-expansion was used to prepare functionalized cyclopentanones (Eq. (54)) [458]. An oxidative addition-intermolecular 1,3-diene insertion and intramolecular alkoxylation sequence was used to prepare dihydrofuroflavonoids (Eq. (55)) [459]. An oxidative addition-alkene insertion-nucleophilic addition sequence using 1,5-cyclooctadienes was reported (Eq. (56)) [460]. The regio- and stereoselectivity of the oxidative addition-intramolecular alkene insertion-intramolecular carbocyclization was examined [461]. Palladium catalyzed an oxidative addition-intermolecular alkene insertion-zinc enolate intramolecular alkylation to give optically active 3-benzylprolines [462]:



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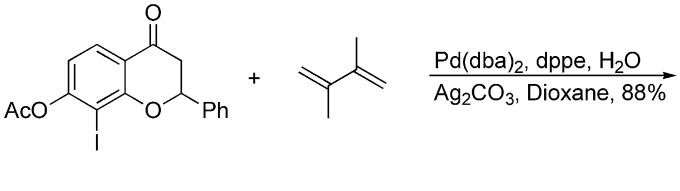

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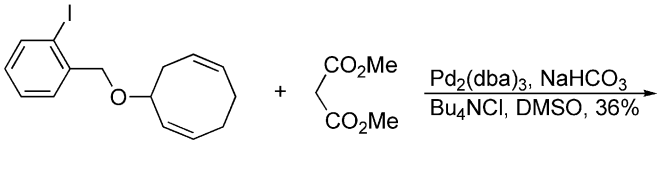
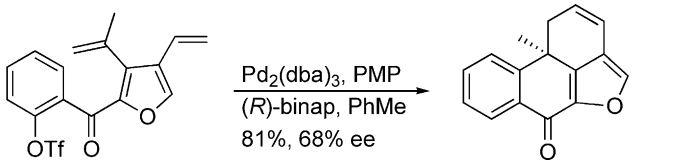


(56)

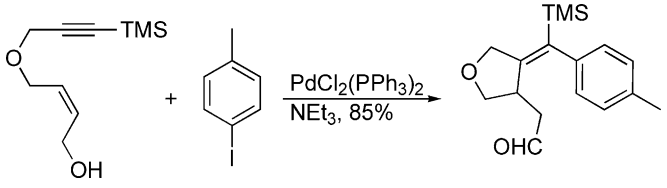
Oxidative addition followed by two sequential intramolecular alkene insertions and β-hydride elimination was used in an asymmetric synthesis of a polycyclic compound (Eq. (57)) [463]. A palladium-catalyzed oxidative addition, intramolecular alkene insertion, and carbonylation sequence was used in an approach to avenaciolide [464]:



(57)

2.4. Carbon–carbon bond formation via insertion of alkynes

Reactions wherein σ-palladium complex intermediates are formed by oxidative addition of palladium(0) to aryl halides and triflates followed by sequential insertion of an alkyne and a termination step such as a nucleophilic addition or carbonylation are discussed in this section. Palladium catalyzed the reaction of 1,2-diiodoaromatic compounds with two equivalents of an alkyne to form a new benzene ring [465]. Palladium catalyzed the reaction of 7-en-1-yne-9-ols and 6-en-1-yne-8 ols with aromatic bromides and iodides to form substituted five- and six-membered rings (Eq. (58)) [466]. Palladium catalyzed an interesting reaction of aryl iodides and 2-alkynylbenzaldehydes to give two products (Eq. (59)) [467]:



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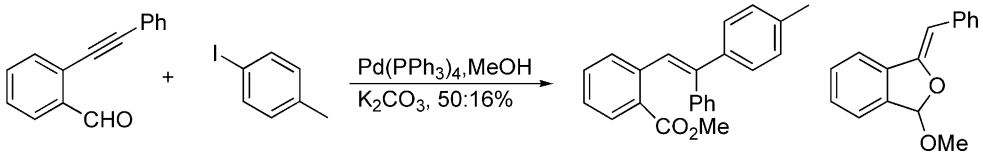
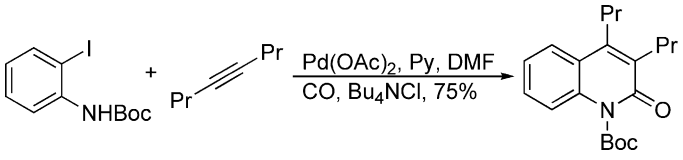


(59)

Palladium catalyzed the formation of 2-quinolones from 2-iodobenzenamines, an internal alkyne, and carbon monoxide (Eq. (60)) [468]. Oxidative addition, intermolecular alkyne insertion, and Sonogashira coupling of 3-iodothioflavones and flavones was reported [469]. An oxidative addition, intermolecular alkyne insertion, and aryl boronic acid termination was used to prepare 3-(arylmethylene)isoindolinones [470]. An oxidative addition, alkyne insertion, and Stille coupling termination of 5-bromo-3,4-dihydroxy-5-en-1-ynes to give fused carbocycles was described (Eq. (61)) [471]. Alkyne insertion was not observed in some cases depending on the starting bromo-enyne:



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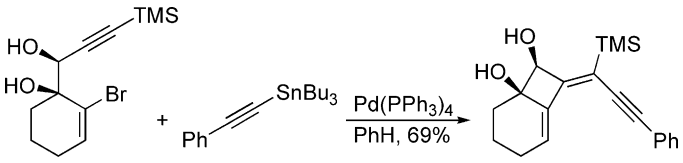
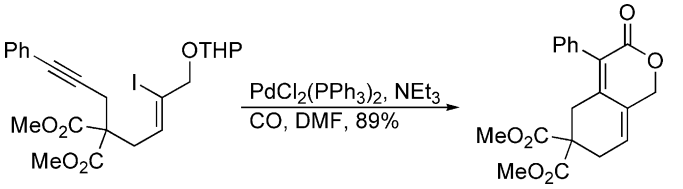


(61)

A sequence consisting of an oxidative addition, intramolecular alkyne insertion, and carbonylation–lactonization was reported (Eq. (62)) [472]. Cobalt catalyzed an oxidative addition, alkyne or alkene insertion, and carbocyclization sequence of 2-iodobenzaldehydes and 2-iodophenylketones to give indene derivatives [473]:



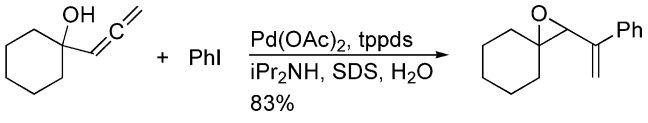
(62)

Palladium-catalyzed oxidative addition to 2-iodobenzamine derivatives followed by alkyne insertion and intramolecular amino-palladation producing indoles [474] were used to prepare (+)-12-methoxy-N_a-methylvellosimine, (+)-12-methoxyaffisine, and (−)-fuchsiaefoline [371], 2- and 3-fluoroalkylated indoles [475], duocarmycin SA [325], 2- and 3-fluoroalkyl-substituted indoles [476], and substituted tryptophans [477]. Related reactions of 2-bromo- and 2-chlorobenzenamines with internal alkynes [478] and reactions in water [479] were described. 7-Deazapurine derivatives were prepared from 5-iodocytocine [299].

Cobalt–rhodium catalyzed oxidative addition followed by insertion of an alkyne and carbonylative cyclization of 2-iodophenols furnished coumarins [480]. Palladium catalyzed the formation of 3-fluoroalkylated benzofurans from 2-iodophenols and fluorine-containing internal alkynes [481].

2.5. Carbon–carbon bond-forming reactions via insertion of allenes

Oxidative addition of palladium to organic halides and triflates followed by insertion of an allene usually leads to η^3-allyl palladium complexes that undergo a number of different termination reactions. Oxidative addition followed by insertion of a 1,2-diene and intramolecular alkoxylation was used to prepare alkenyl oxiranes (Eq. (63)) [482]. A related oxidative addition followed by insertion of a 1,2-diene and intramolecular carbocyclization was used to prepare dihydrofurocoumarins and dihydrofurodihydropyrid-2-ones [483]. Oxidative addition, intermolecular allene insertion followed by intramolecular nucleophilic addition was used to prepare substituted cyclopropanes (Eq. (64)) [484]. A stereoselective oxidative addition, intermolecular allene insertion, and intramolecular alkene insertion sequence to give pyrrolidines was reported (Eq. (65)) [485]. Allylic carbonates furnished 3-azabicyclo[3.1.0]hexanes in a related fashion (Eq. (66)):



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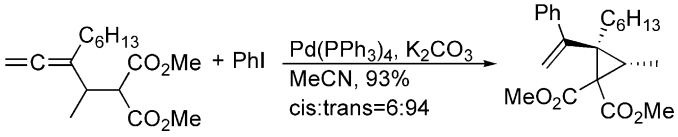

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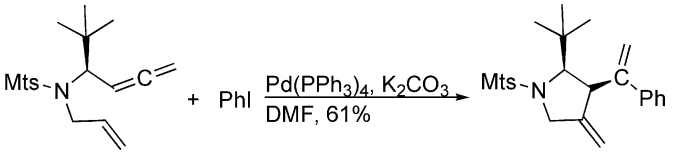

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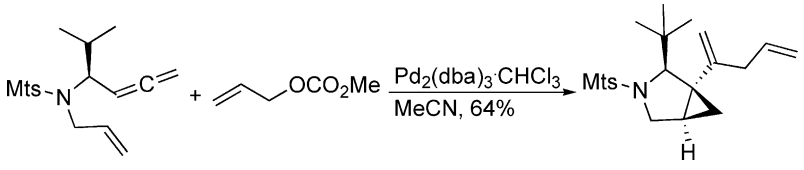
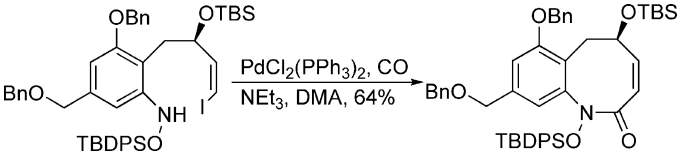


(66)

2.6. Carbon–carbon bond formation via insertion of carbon monoxide

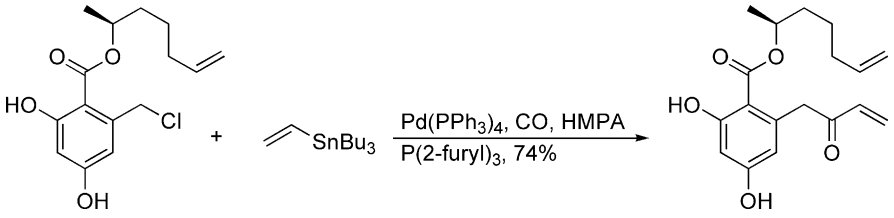
A variety of palladium complexes and catalyst systems catalyzed the carbonylation of alkenyl and aryl halides and triflates to afford carboxylic acids and derivatives. Bromobenzeneamines and -anisoles, and heterocyclic chlorides were transformed into the corresponding methyl esters [486]. DMF or $Mo(CO)_6$ was used as the carbon monoxide source in palladium-catalyzed carbonylations of 2-bromobenzylalcohols to give phthalides [487].

4-Iodo- or 4-bromo-5,-10-dihydropyrido[2,3-*b*:3,2-*e*]pyrazines were transformed into the corresponding esters [25]. The palladium-catalyzed alkoxy-carbonylation of organic halides and triflates was used in synthetic applications toward TAN-1085 [488], 12-oxygenated tremetones [489], tetrahydrocannabinol analogs [490], TAK-779 [237], alkaloids 223A and 205B [491], and 6,6-difluoroshikimic acid [492]. A nickel-catalyzed intramolecular alkoxy-carbonylation of a 4-hydroxy-2-bromo-1-ene was used in a synthesis of (−)-tuliparine [493]. Palladium catalyzed the formation of amides from *N*-unprotected haloindoles [494]. Formation of a lactam was used in a synthesis toward FR900482 (Eq. (67)) [495]:



(67)

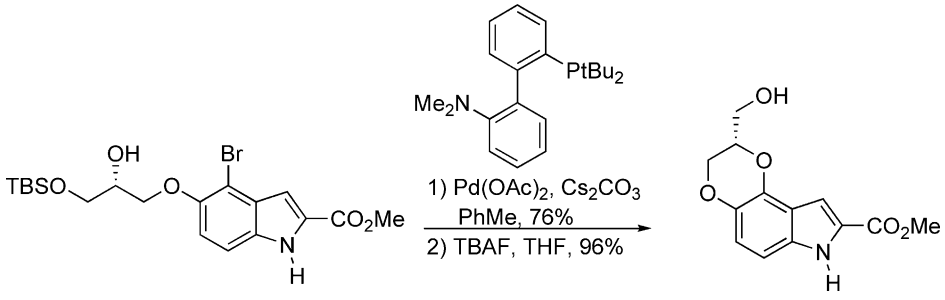
Added copper salts improved the yield of the palladium-catalyzed carbonylative coupling of alkenylstannanes with alkenyltriflates to form unsymmetrical ketones [496]. A palladium-catalyzed carbonylative coupling of a benzylic chloride with tributyl alkenyltin was used in a synthesis of *trans*- and *cis*-resorcylide (Eq. (68)) [497]. A palladium-catalyzed carbonylative coupling of an alkenylstannane and an aryl iodide was used in a synthesis of lapidilectine B [498]. ^{13}C- and ^{11}C-labelled ketones were prepared from aryl iodides, labeled carbon monoxide and organoboronic acids [499]. C1-acyl glycals were prepared in a similar carbonylative coupling [256]. Palladium catalyzed the carbonylative coupling of tetraorganoindiums with aromatic iodides to give unsymmetrical ketones [500]:



(68)

2.7. Carbon–oxygen, –nitrogen, and –sulfur bond-forming reactions

Intramolecular palladium-catalyzed alkoxylation of organic halides were used in synthesis of U86192A (Eq. (69)) [501] and 3,4,5,6-tetrahydro-2*H*-pyrido[4,5-*b*]- and [2,3-*b*]-1,5-oxazine-6-ones [201]. A palladium catalyzed intramolecular *O*-alkylation of enolates with a pendant aryl halide was used to prepare benzofurans [502]. A palladium-catalyzed double alkoxylation of 1,2-dibromoarenes using 1,2-diols was used in a synthesis of isoamericanol A and isoamericanini A [503]. Palladium catalyzed an intramolecular alkoxylation or amination of 2-(2-hydroxy- or 2-aminophenyl)-1,1-dibromoethene followed by reaction with an aromatic boronic acid or diethylphosphite to give 2-substituted benzofurans or indoles (Eq. (70)) [504]:



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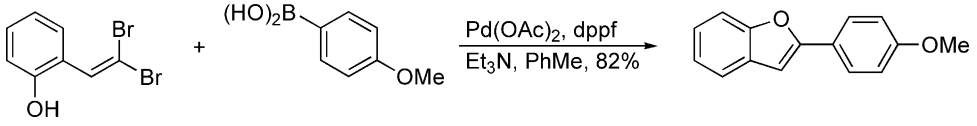
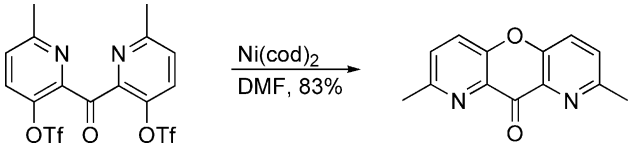


(70)

Copper-catalyzed intermolecular alkoxylations of aryl bromides were used in synthesis of elacomine and isoelacomine [505], renieramide [506], and spirotryptostatin A [507]. A copper-catalyzed synthesis of alkoxy ferrocenes via coupling of alcohols with iodoferrocene was described [508]. Nickel and palladium catalyzed an unusual intramolecular coupling of bispyridyltriflates (Eq. (71)) [509]:



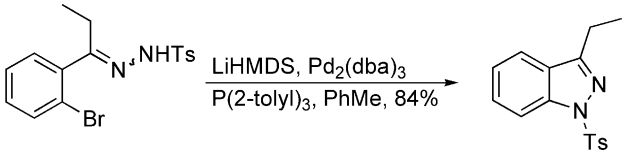
(71)

A variety of aryl and alkenyl halides [510–516] and sulfonates [517] were aminated or amidated using a number of palladium or copper catalysts [518,519]. Palladium catalyzed the intermolecular *N*-arylation or *N*-amidation of aza-crown ethers [520,521], 3-aminopiperidines and 3-aminopyrrolidines [522], sulfamides [523], silylamines with the loss of the silicon group [524], fluorous benzophenone imine [525], *N*-Boc protected aromatic hydrazides [526,527], chiral pyrrolidones [528], sultams [529], a sulfoximine [530], and hydrazones [531]. Palladium-catalyzed intermolecular *N*-alkenylations of sulfoximines using alkenyl-bromides and triflates [532]. This type of reaction using an aryl bromide was employed in a synthesis toward pseudopteroxazole [533]. Palladium-catalyzed coupling of *N*-trialkylsilylimines with aryl- and alkenyl bromides to give imines and azadienes [534].

Palladium catalyzed the amination of 6-bromo-1-phenyl-3,4-dihydropyrrole[1,2-a]pyrazine [193], 3-bromobenzothiophenes [535], 5-bromopyrroles [536], and 2-chloro-3-fluoropyridine [537]. An intermolecular amidation–cyclization of 2-halobenzaldehydes was used to prepare naphthyridinones [538]. Intermolecular amination of piperazines to give *N*-aryl- or *N*-heteroaryl-*N*′-(arylalkyl)piperazines was reported [539].

Intramolecular palladium-catalyzed aminations or amidations were used in the synthesis of diindolecarbazoles [540], *N*2-deoxyguanosine adduct of the mutagen IQ (Eq. (72)) [541], duocarmycin and CC-1065 analogs [542], and a variety of benzofused five- to seven-membered nitrogen heterocycles [543]. Palladium-catalyzed intramolecular aminations of *N*-tosylhydrazones to give indazoles (Eq. (73)) [544]. Indazoles were also obtained from 2-bromobenzaldehydes and arylhydrazines (Eq. (74)) [545]. A double amination was used to prepare tricyclic compounds (Eq. (75)) [546]:

(72)



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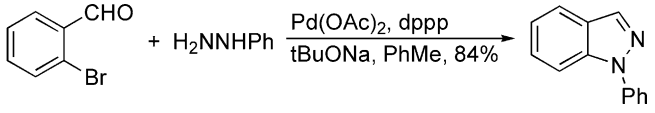

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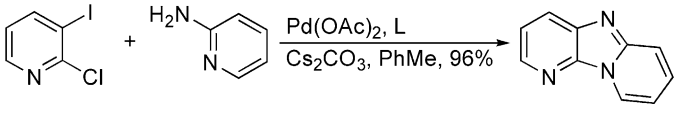
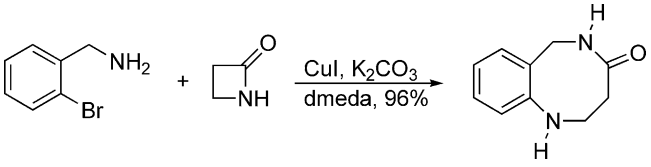


(75)

Transition metals other than palladium, mainly copper, also catalyzed amination and amidation [366,547–551] reactions. Copper-catalyzed couplings of amides with aryl or alkenyl halides were used in syntheses toward cytotrienins [552],

oximidines I–III, salicylhalamides A–B, and lobatamides A and D, and CJ-12,950 [553]. Copper catalyzed the *N*-alkynylation of alkyl and aryl sulfonamides and carbamates using bromoalkynes [554,555], of *N*-BOC protected hydrazine with aromatic iodides [501], of *N*-BOC mono- and diprotected aryl hydrazines with aryl iodides [556], of sulfoximes with aryl halides [557], of sultams with aryl halides [529], of pyrazoles, pyrroles, pyrazoles, indazoles, imidazoles, and thiazoles with aromatic halides [558,559], and of urea with aryl iodides [560].

Copper catalyzed an intermolecular amidation followed by a ring expansion of amino-substituted aromatic bromides with lactams (Eq. (76)) [561]. Copper catalyzed the formation of 1*H*-pyrazolo[3,4-*b*]pyridines from 2-chloro-3-cyanopyridine and hydrazines (Eq. (77)) [26]. Copper-catalyzed intermolecular amidations of alkenyl and aromatic halides were used in syntheses of the antibiotic CJ-15,801 [562], (−)-apicularene [563], 3-aryl-*b*-carboline-1-ones [564], and oximidine III [54]. Copper-catalyzed amination of *O*-acyl hydroxylamine derivatives with diorganozinc reagents (Eq. (78)) [565]. Copper catalyzed azidation of aromatic and alkenyl iodides (Eq. (79)) [566]:



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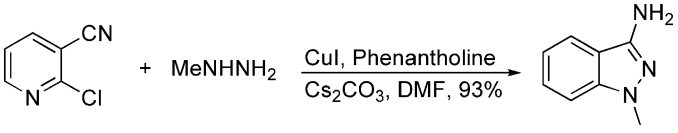

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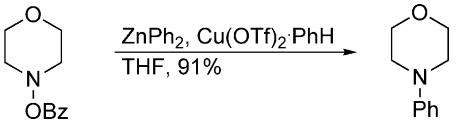

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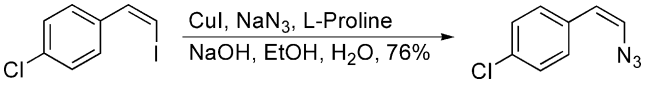
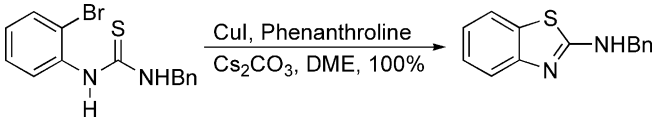


(79)

Palladium catalyzed the coupling of aryl halides and triflates with thiols to form thioethers [567,568]. Palladium catalyzed the coupling of aryl and alkenyl halides and triflates with sulfinic acid salts [569]. Palladium and copper-catalyzed intramolecular couplings of 2-halo-arylthioureas (Eq. (80)) [570]. Aryl iodides were coupled with disulfides to give unsymmetrical sulfides in the presence of zinc and a nickel catalyst. Copper catalyzed the coupling 8-mercaptoadenine with aryl iodides [571] and thiols with ethenyl iodides [572] or aromatic bromides and iodides [573]:



(80)

2.8. Carbon–tin, –silicon, –phosphorous, –arsenic, –antimony, –selenium, and –boron bond-forming reactions

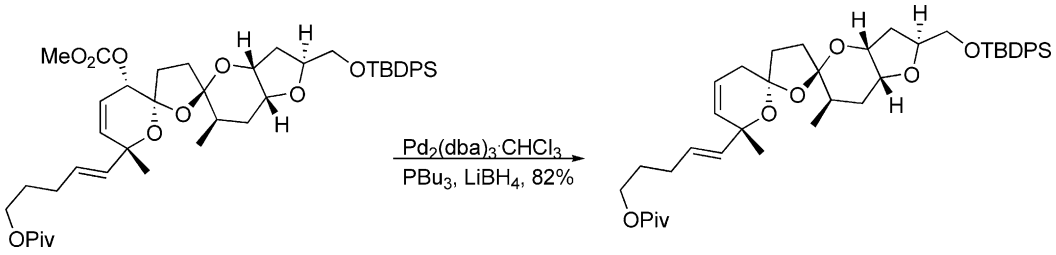
Palladium-catalyzed reactions of hexaalkylditins with alkenyl triflates or aryl bromides to give tin reagents were employed in the synthesis of lapidilectine B [498], a 3,3′-biindole a putative natural product [11], *N*-protected staurosporines [71], 5-IA-85380 precursor [574], and acromelic acid analogs [575]. Palladium catalyzed the formation of propargyl and allenyl stannanes from propargylic chlorides and mesylates and hexamethylditin [576].

Palladium catalyzed the coupling of aryl bromides with triarylphosphines to give unsymmetrical triarylphosphines [577,578]. Palladium-catalyzed couplings of a manno-iodoimidazole and dialkylphosphonates were used to prepare manno-tetrahydroimidazopyridine-2-phosphonates [579]. A related reaction was used to prepare α,α-disubstituted 4-phosphonophenylalanine analogs [580].

Palladium catalyzed the cross-coupling of aryl iodides with dipinacolyldiborane or pinacolborane producing aromatic pinacolyl boronic esters [212]. The products were used in Suzuki couplings toward TMC-95-A and -B [581], diazonamide A [227,228], and dehydroaltenusin [239]. Palladium catalyzed the coupling of an alkenyltriflate with dipinacolyldiborane followed by Suzuki coupling with aromatic bromides [582]. Reaction of arylhalides with dipinacolylborane gave homo-coupled biaryls [583].

2.9. Carbon–hydrogen bond-forming reactions (including decarbonylation of aldehydes)

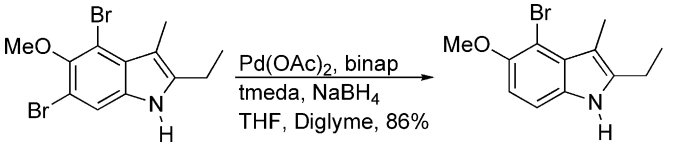
A variety of functional groups were replaced by a hydrogen atom using palladium-catalyzed methodologies. Palladium complexes, together with a hydride source, such as ammonium formats, tributyltin hydride, lithium borohydride, and triethylsilane catalyzed the reduction of organic triflates and halides including allylic acetates and carbonates. Synthetic applications include, (−)-borrelidin [584], (−)-mycalamide A [585], erogorgiaene [47], (−)-galanthamine [586], azaspiracid 1 (Eq. (81)) [587], calothrixin B [588], DEF-ring fragment of FR182877 [589], xerulinic acid [55], nominine [590], and aporphine [393]:



(81)

Monodebromination of 1,1-dibromo-1-alkenes was achieved using tributyltin hydride in the presence of a palladium catalyst forming *cis*-bromoalkenes. This type of reaction was used in the synthesis of oximidine II [188], gambierol analogs [59], and 18-, 19-, and 20-HETE [244]. Palladium catalyzed regioselective monodebromination of 4,6-dibromoindoles (Eq. (82)) [501]. Allenes were formed via a palladium-catalyzed reduction of propargylic amines (Eq. (83)) [591].

Palladium catalyzed the reduction of allylic silylsulfinates to alkenes (Eq. (84)) [592]. Wilkinson's catalyst was used in a decarbonylation of aldehydes in synthesis of taiwanine C [287] and infuscol A and cuprenelol [593]:



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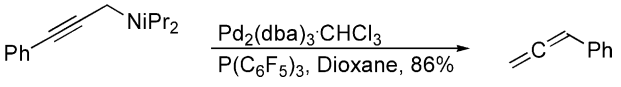

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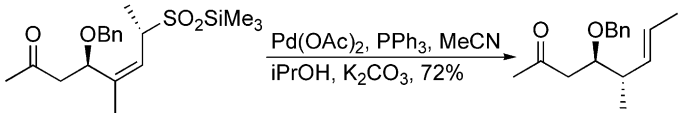


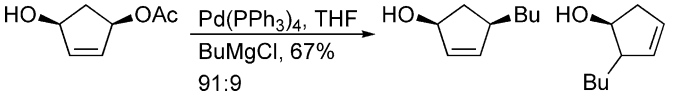
(84)

3. Reactions of allyl, propargyl, and allenyl halides, sulfonates, alcohols, oxiranes, and nitriles

3.1. Carbon–carbon bond-forming reactions using carbon nucleophiles

Palladium continued to dominate as the catalyst of choice for allylic alkylation reactions. Although the least substituted terminus of the intermediate η^3-allyl–palladium complex is usually substituted, the more substituted product was obtained using some catalyst systems [594]. Palladium catalyzed alkylation of allylic alcohols with stabilized carbon pronucleophiles [595,596]. Allylic alkylations using 2-arylsulfonyloxy-3-chloropropenes [597] and 2,5-diacetoxy-2,5-dihydrofuran [598] were described.

Palladium catalyzed the reaction of stabilized nucleophiles, alcohols, and amines with allylic alcohols in aqueous solutions [599]. Palladium-catalyzed allylic alkylation using allylic alcohols and organoboronic acids [600,601]. Palladium-catalyzed alkylations of 2-cyclopentene-1,4-diol monoacetate with Grignard reagents to give the 1,2-disubstituted product (Eq. (85)) [602]. Palladium catalyzed an asymmetric allylic alkylation of allyl-β-ketoesters (Eq. (86)) [603]. A related ruthenium-catalyzed reaction was reported [604]. An optically active enolate [605], α-isocyanoacetoacetates [606], and indoles [607] were used as the nucleophile. Trisubstituted allenes were used as the nucleophile in intermolecular allylic alkylation of alkenyl oxiranes (Eq. (87)) [608]:



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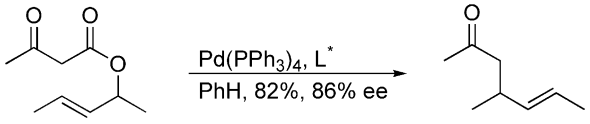

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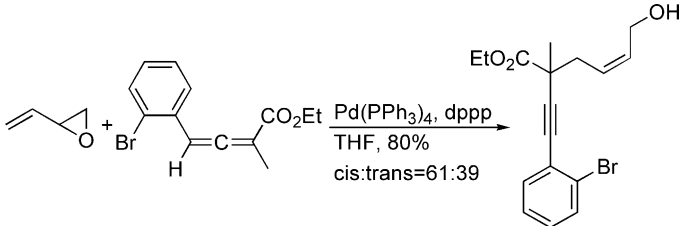
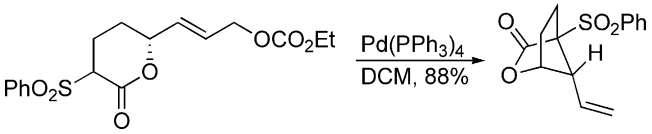


(87)

Palladium-catalyzed allylic alkylations were used in synthetic applications toward bacillariolide III (Eq. (88)) [609], (−)-enterolactone [610], carbocyclic (+)-uracil polyoxin C [611], (+)-α-allokainic acid (Eq. (89)) [612], (−)-strychnine (Eq. (90)) [613], sordaricin (Eq. (91)) [614], isoretronecanol [615], α,α-disubstituted amino acids [616], acetomycin bislacton analogs [617], quinine and quinidine [618], and a tetracyclic sesterterpene [619]:



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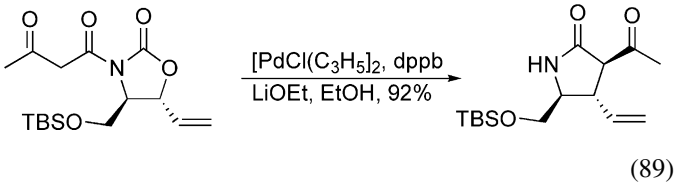

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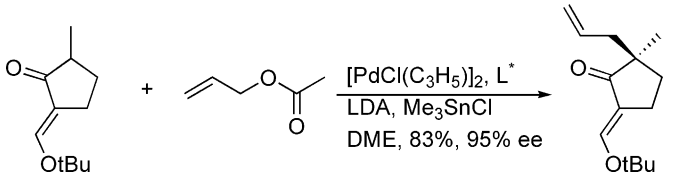

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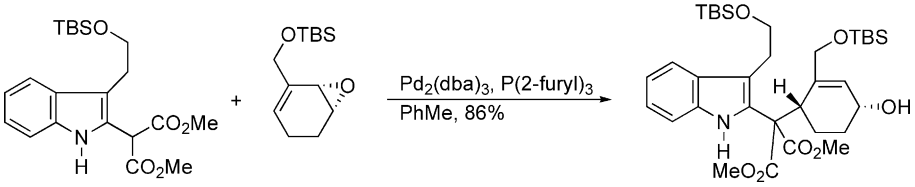

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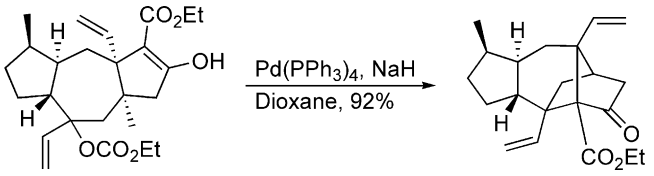
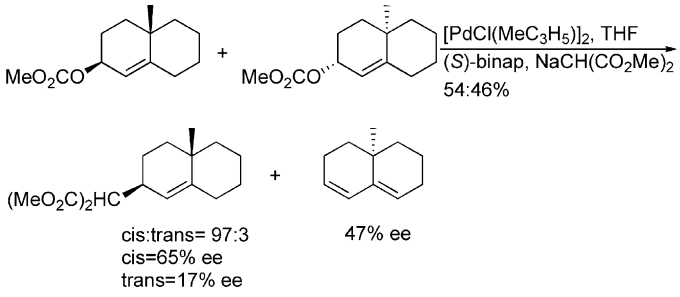


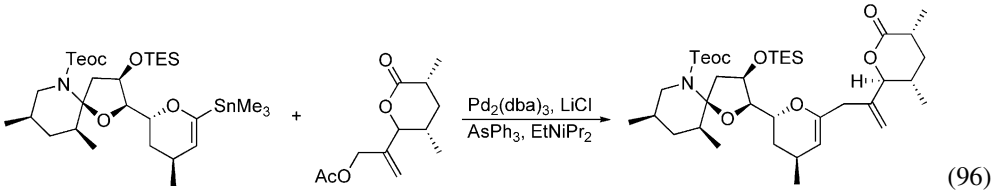
(91)

Substrate directed regio- and stereoselective palladium-catalyzed nucleophilic substitutions were described (Eq. (92)) [620]. A palladium-catalyst system for the asymmetric alkylation at the more substituted position of allylic acetates was developed [621]. Palladium-catalyzed asymmetric allylic alkylations were used in the synthesis of (−)-pumilotoxin C [455]. A palladium-catalyzed allylic alkylation-Michael addition sequence was used in a synthesis of TMC-69-6H (Eq. (93)) [622]. An enantioselective α-allylation of a ketone enolate was used in a synthesis of hamigeran B (Eq. (94)) [435]. Enolates derived from *N*-protected glycine esters were used as the nucleophile [623]. A kinetic resolution of racemic bicyclic allylic acetates was described (Eq. (95)) [624].



(95)

Palladium catalyzed the coupling of allyl bromides with tin reagents [17]. An alkenyltin reagent was used as the nucleophile in a synthesis of azaspiracid 1 (Eq. (96)) [587,625] and 7-epi-goniodiol and leiocarpin [626]. Palladium catalyzed the allylative ring-opening of oxabicycloalkenes using organozinc reagents [627]. Palladium catalyzed the coupling of allenic tin reagents, an allylic chlorides and activated alkenes (Eq. (97)) [628]:



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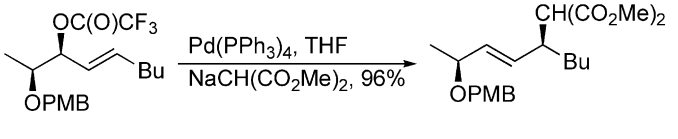

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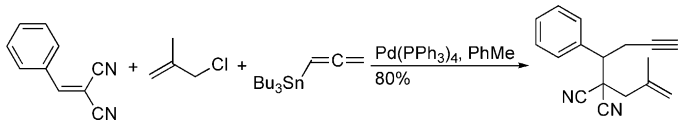

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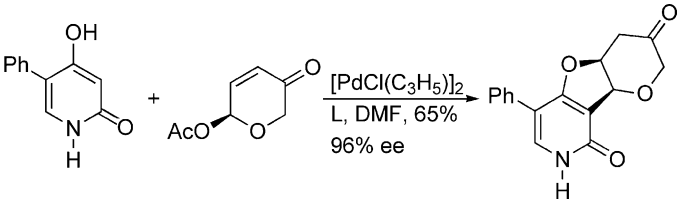
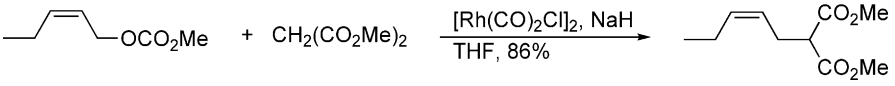


(93)

Copper-catalyzed enantioselective allylic alkylations of allylic phosphonates using alkylzinc reagents [629,630]. Copper also catalyzed asymmetric allylic alkylations of optically active allylic esters using Grignard reagents as the nucleophile [631] and of allylic chlorides with 2-(1-hydroxyalkyl)arylsilanes [275]. Regioselective iridium-catalyzed asymmetric allylic substitutions were reported [632,633]. Iridium catalyzed allylic alkylation of 1,2-disubstituted-2,3-butadienyl acetates to give

allenic products having a quartenary center in the α-to the diene [634].

A rhodium-catalyst for allylic alkylation affording products derived from alkylation of allylic carbonates at the site of the leaving group was described (Eq. (98)) [635]. Rhodium catalyzed an allylic alkylation cycloisomerization reaction [636]. A regio- and diastereoselective rhodium-catalyzed allylic alkylation of α-alkoxysubstituted copper enolates was described (Eq. (99)) [637]. Ruthenium catalyzed the direct propargylation of arenes with allylic alcohols [638]. A nickel-catalyzed allylic alkenylation was used in a synthesis toward ciguatoxin (Eq. (100)) [639]. Cobalt catalyzed allylic alkylation of Grignard reagents using allylic ethers as the substrate [640]:



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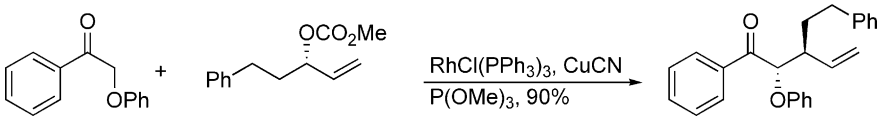

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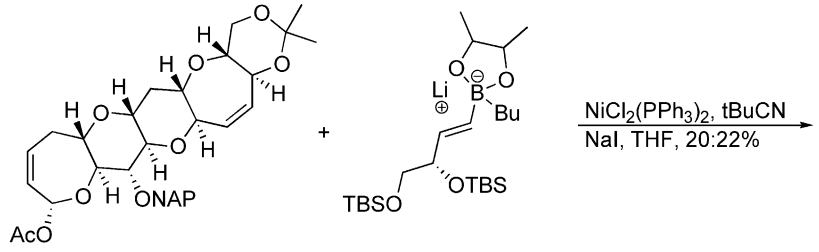
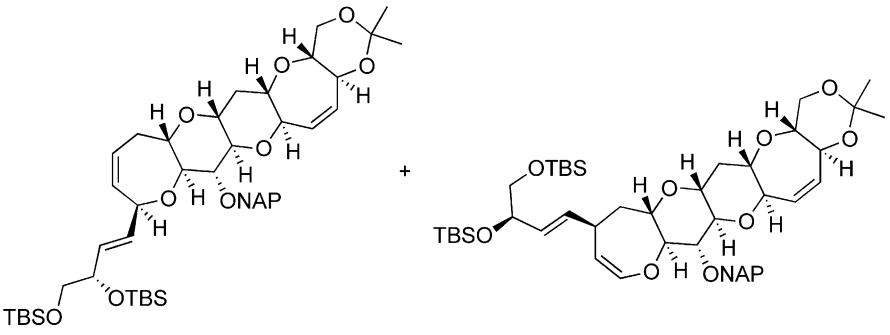
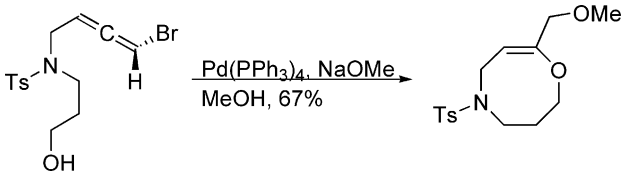




(100)

3.2. Carbon–oxygen, –nitrogen, and –sulfur bond-forming reactions using oxygen-, nitrogen-, and sulfur-nucleophiles

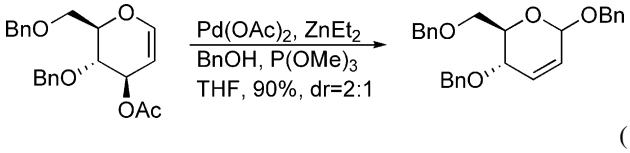
Palladium-catalyzed intramolecular alkoxylations of an alkenyl oxirane were used in syntheses of C-furanosides [641] and toward azaspiracid [642]. Related intramolecular reactions of allylic carbonates were used in synthesis of 2-alkenyl-4-methylene tetrahydropyranes [643]. 2,3-Dihydrofurans and benzofurans were prepared by palladium-catalyzed intramolecular alkoxylation of propargylic carbonates [644]. 6,11-*O*-Bridged bicyclic ketolides were prepared via a palladium-catalyzed bis-allylation of a diol [645]. Palladium catalyzed the formation of nitrogen- and oxygen-containing rings from amino- or hydroxy-tethered-1-bromo-1,2-dienes (Eq. (101)) [646]. 2-Alkylidene-pyrrolo[*c*]-1,4-dioxanes were prepared from a 3,4-dihydroxypyrrole and propargylic carbonates [647]:



(101)

Palladium-catalyzed asymmetric allylic alkylations of alkoxides or alcohols were used in the synthesis of ustiloxin D [648], (+)-clusifoliol, (−)-siccanin, and (+)-rhododaurichromanic acid [649], (−)-siccanin [650], (+)-brefeldin [651], and allylated dinucleotides [652].

An intermolecular palladium-catalyzed allylation of an alcohol was used in the synthesis of (+)-viroallosecurinine [653]. Palladium-catalyzed stereoselective *O*-glycosidations using glycols (Eq. (102)) [654] and alcohols [655]. Intramolecular alkoxylation of an allylic acetate was used in a synthesis of (−)-mycalamide A [585]. A stereoselective cyclization of an intermediately formed hemiacetal [656] was used in a synthesis of 5-epi-prelactone C (Eq. (103)) [657]. Palladium catalyzed the ring-opening of allylic oxiranes to give 3,4-dihydroxy-1-enes with retention of stereochemistry [658]:



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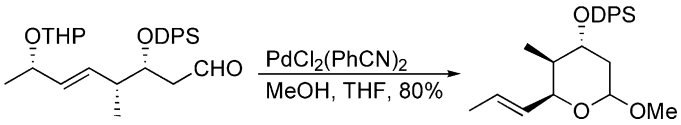
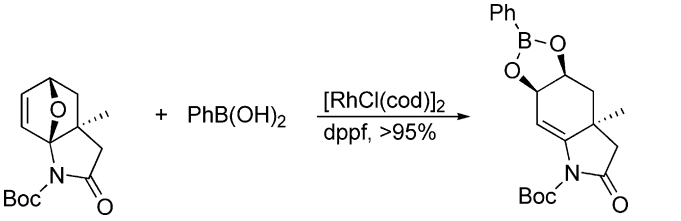


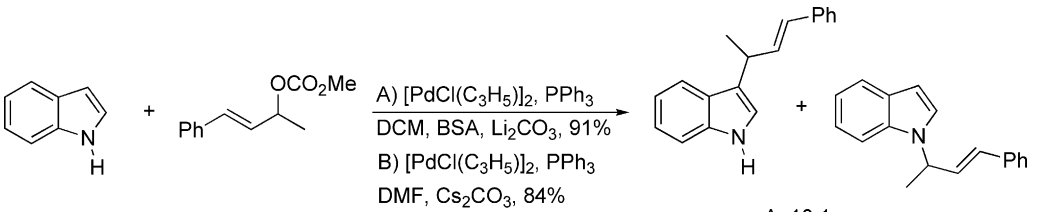
(103)

Iridium catalyzed *O*-allylations of alcohols using allylic acetates [659]. Iridium catalyzed kinetic resolution of allylic carbonates using phenol [660]. Iridium catalyzed asymmetric allylic alkoxylations using allylic phosphonates, carbonates, and oximes to give the more substituted product [661,662]. A regio- and enantioselective rhodium-catalyzed allylic alkoxylation was used in a synthesis of gaur acid [663]. Ruthenium catalyzed propargylic alkoxylation of 1-yne-3-ols [664]. Ruthenium catalyzed the direct formation of allylic ethers from an allylic alcohol and a second alcohol [638]. A rhodium-catalyzed ring-opening of oxabicyclic compounds was developed and used in a synthesis of epi-zephyranthine (Eq. (104)) [665]:



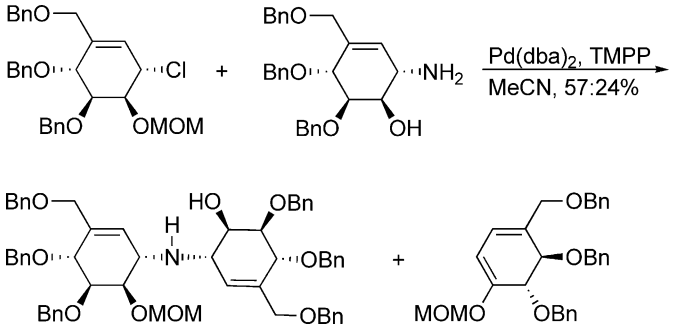
(104)

A number of reports dealing with amination of allylic substrates appeared in the literature. Palladium-catalyzed allylic aminations of aziridines gave predominately the more substituted product [666]. Palladium-catalyzed *N*-allylation using allylic alcohols [595]. The regioselectivity of the alkylation of indoles was controlled by proper choice of solvent and base (Eq. (105)) [667]. A palladium-catalyzed asymmetric allylation of meso-vicinal diamines was reported [668]. Palladium catalyzed a double allylation of 1,2-diamines with *cis*-1,4-diacetoxy-2-butene to give 2-ethenyl-pyrazine derivatives [669]. Related asymmetric reactions of 1,2-benzenediamine, 2-aminophenol, and 1,2-dihydroxybenzene derivatives with *cis*-1,4-diacetoxy-2-butene were developed [670]:



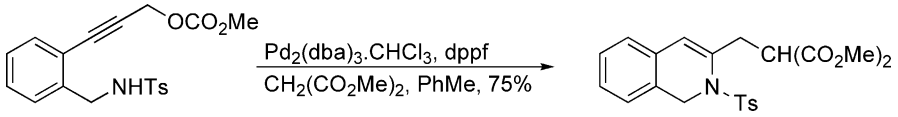
(105)

Palladium-catalyzed allylic aminations were used to prepare novel nucleoside analogs [671–673], martinellin pyrrolo[3,2-*c*]quinoline skeleton [674], and 6-ethenyl and 5-ethenylproline analogs of ascomycin [675]. Substrate directed regio- and stereoselective palladium-catalyzed allylic aminations were described [620]. Palladium-catalyzed regio- and enantioselective allylic intermolecular aminations were used in synthesis of crinamine, haemanthidine, and pretazzettine [676], 2-deoxystreptamines [677], and pseudodisaccharides (Eq. (106)) [678]. Intramolecular enantioselective allylic amidations of allylic benzoates were used to prepare imidazolidin-2-ones [679]:



(106)

Palladium catalyzed the reaction of propargylic esters having a tethered amine with nucleophiles to give fused heterocycles (Eq. (107)) [680]. Oxidative addition to cyclobutanol-substituted propargyl halides followed by nucleophilic addition and ring-expansion furnished substituted cyclopentanones (Eq. (108)) [681]. Palladium catalyzed the amination of alkenylcyclopropylbisphosphonates (Eq. (109)) [682]:



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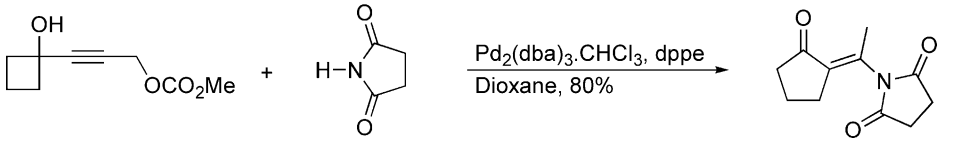

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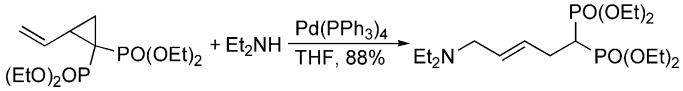


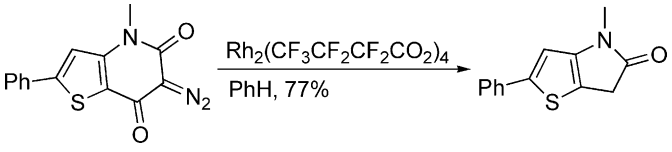
(109)

Asymmetric intramolecular nickel-catalyzed allylic aminations were described [683,684]. Iridium catalyzed asymmetric allylic aminations using allylic phosphonates or carbonates to give the more substituted product [633,661,685]. Intramolecular asymmetric allylic aminations catalyzed by iridium was also reported [686].

Rhodium catalyzed an asymmetric nucleophilic ring-opening of oxabicyclic alkenes using sulfur nucleophiles [687]. Thiocarboxylate ions were used as nucleophiles in dynamic kinetic resolution of allylic esters [688]. Palladium catalyzed the formation of propargylic and allenic sulfides from propargyl halides or mesylates and thiols [689]. Palladium catalyzed the coupling of allylic esters with bis(pinacolato)diboron to form allyl boronic esters [690].

4. Metal-catalyzed diazo decompositions (including other cyclopropanations)

Rhodium catalyzed the methylenation of ketones using trimethylsilyldiazomethane [691,692]. Rhodium or copper-catalyzed decomposition of aryldiazoacetates in the presence of enamines furnished γ-keto esters [693]. Tandem palladium-catalyzed oxidation of alcohols and rhodium catalyzed methylenation or methylenation followed by ring-closing metathesis, both using TMS-diazomethane, was described [694]. Rhodium catalyzed a ring contraction of 6-diazo-4*H*-thieno[3,2-*b*]pyridine-5,7-diones (Eq. (110)) [695]. Patulolide B was prepared via a ruthenium-catalyzed decomposition of a bisdiazo compound (Eq. (111)) [696]. Rhodium-catalyzed cyclizations via decomposition of diazo compounds were used to prepare cyclic amino acids [697] and α-quarternary pipecolic acid derivatives [698]:



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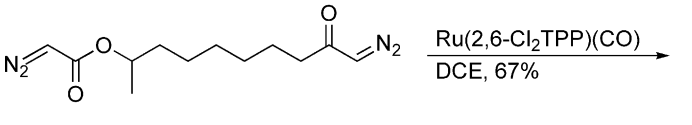
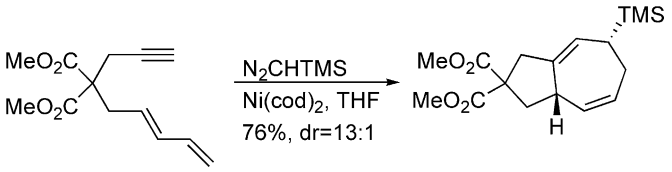


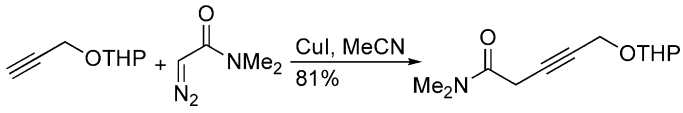
(111)

Nickel catalyzed the formation of bicyclo[5.3.0]ring systems from 1,3-dien-8-ynes and TMS-diazomethane (Eq. (112)) [699]. Chromium catalyzed the decomposition of diazo compounds in the presence of 2-substituted furans to give (2*E*,4*Z*)-2-aryl-hexadienedioic acid diesters [700]:



(112)

Copper-catalyzed intermolecular carbon–hydrogen bond insertions of terminal alkynes using diazo esters and amides (Eq. (113)) [701]. Silver catalyzed intramolecular carbon–hydrogen bond insertions of carbamates to form five-membered heterocycles in the presence of $PhI(OAc)_2$ as the oxidant (Eq. (114)) [702]. Rhodium catalyzed a related reaction of sulfamate esters to give 1,2,3-oxathiazinane-2,2-dioxides in the presence of $PhI(OAc)_2$ [703]. Ruthenium catalyzed amidations of heteroaromatic compounds using iminoiodanes or *N*-nosylamide and $PhIOAc_2$ [704]:



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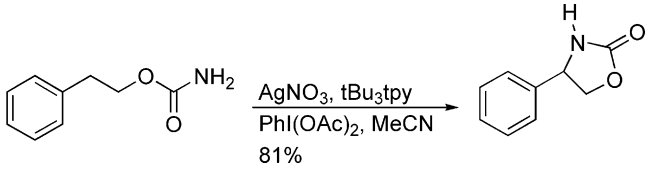
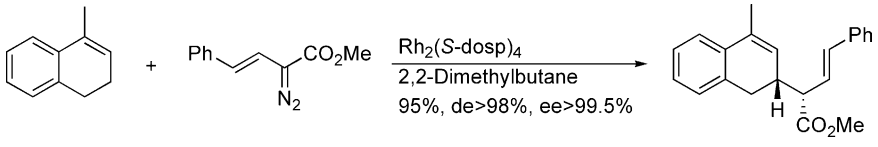


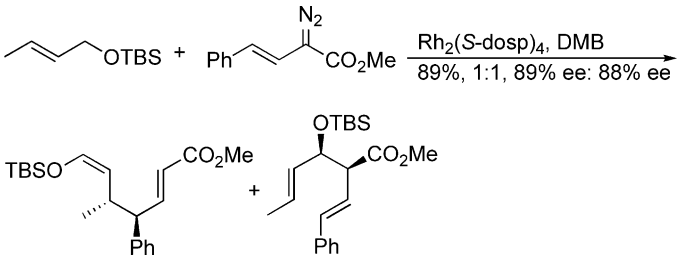
(114)

Rhodium catalyzed a diastereo- and enantioselective carbon–hydrogen bond insertion using 1,2-dihydronaphthalenes (Eq. (115)) [705]. Intramolecular rhodium-catalyzed carbon–hydrogen bond insertions via decomposition of diazo compounds were used in a number of synthetic applications forming five- and six-membered rings for example, toward endo–exo-furofuranones [706], thienofuranones [707], azaspiracid-1 [708], pregabalin [709], 2,3-fused indoles [710], (+)-preussin [711], (−)-ephadradine A [428], and cyclopentanone containing amino acids [712]:



(115)

Rhodium catalyzed an asymmetric carbon-hydrogen insertion silyloxy-Cope rearrangement (Eq. (116)) [713]. A double carbon-hydrogen bond insertion was used to prepare C_2-symmetric benzenamines (Eq. (117)) [714]. Asymmetric intramolecular carbon–hydrogen-bond insertions using in situ formed nitrenes from a sulfamate or carbamate ester using a rhodium-catalyst was described [715]:



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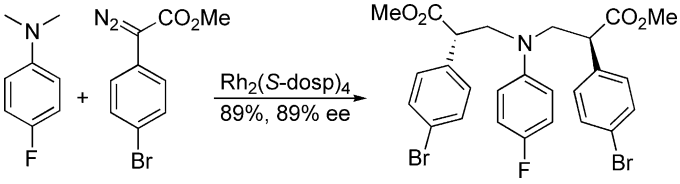
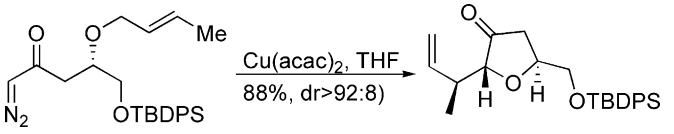


(117)

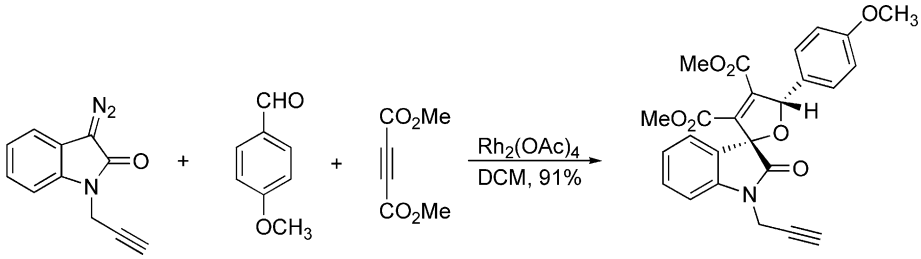
The carbenoids derived from rhodium-catalyzed decomposition of diazo compounds insert into nitrogen–hydrogen bonds. Intramolecular nitrogen–hydrogen bond insertions to give substituted piperidines [716], and azetidines [717], and intermolecular insertions to afford imidazoles and imidazolones [718] were reported. An intermolecular nitrogen–hydrogen-bond insertion was used to prepare 1,4-azines [719]. Copper catalyzed the formation of *N*-alkylimidazoles from α-diazocarbonyl compounds and imidazoles [720]. A rhodium-catalyzed oxygen-hydrogen bond insertion was used in a synthesis of (+)-(2′*S*,3′*R*)-zoapatanol [251] and 4″-alkoxy avermectin analogs [721].

Depending on the substrate and catalyst, a preference for the formation of a five- or six-membered oxonium ylide from diazoketones was observed [722]. A copper-catalyzed oxonium ylide formation [2,3]rearrangement was used to prepare the A-ring of gamberic acid (Eq. (118)) [723]:



(118)

A variety of cycloaddition reactions of carbonyl ylides formed by rhodium- or copper-catalyzed decomposition of α-diazocarbonyl compounds were described. Spirofurooxindoles were prepared from 3-diazoindole-2-ones, an alkene or alkyne and an aromatic aldehyde (Eq. (119)) [724]. An intramolecular carbonyl ylide-alkene cycloaddition was used to prepare polycyclic indoles [725]. Cycloaddition with aldehydes was used to prepare 2,4,5-triaryl-1,3-dioxolanes [726]. Intermolecular carbonyl ylide-alkene [3 + 2]cycloadditions were used to prepare irofulvene (Eq. (120)) [727], nemorensic acids [728], and mono- and bis-decahydrobenzocarbazoles (Eq. (121)) [729]. A rhodium catalyzed [3 + 2]cyclorevision was described (Eq. (122)) [730]:



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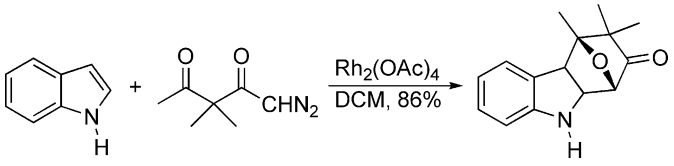

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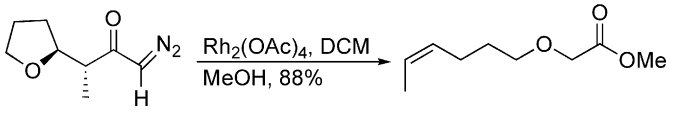
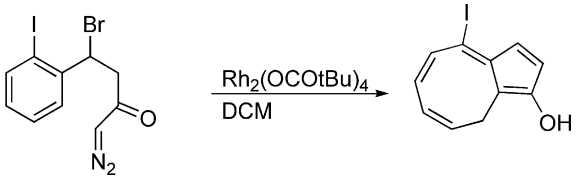


(122)

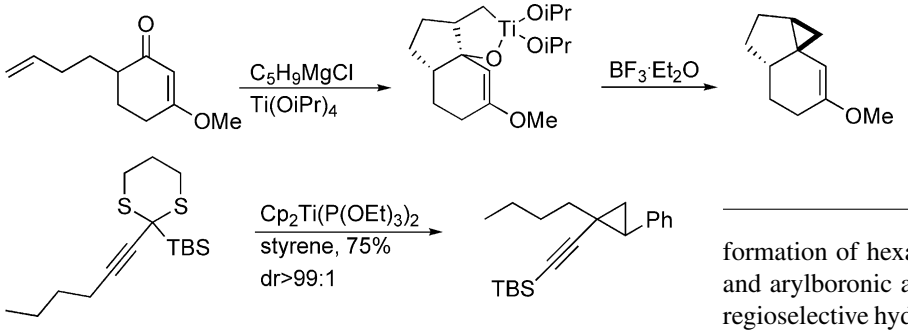
Cyclopropanations of alkenes via transition metal-catalyzed decomposition of diazo compounds continued to be developed. Rhodium catalyzed an enantioselective intermolecular cyclopropanation of styrenes with silyloxyethenyl diazoacetates [731]. Rhodium catalyzed intermolecular cyclopropanation was used in a synthesis toward zincophorin [732]. A rhodium-catalyzed intramolecular alkene cyclopropanation using a diazo compound was used in a synthetic application toward 2-C-branched glyco-amino acids [733]. Intramolecular cyclopropanation followed by ring expansion gave substituted azulenes (Eq.

(123)) [734]. Copper catalyzed intermolecular cyclopropanation of indoles and dihydropyridines using diazo compounds [735]:



(123)

Titanium mediated inter- and intramolecular cyclopropanations of vinylogous esters (Eq. (124)) [736] and alkene tethered amides [737]. A titanium-mediated cyclopropanation of alkenes with alkynylthioketals was described (Eq. (125)) [738]. The stereochemistry of the titanium or zirconium mediated cyclopropane formation from esters, amides, and allylic ethers was examined [739]. Titanium mediated an intramolecular cyclopropanation using *N*-(3-alkene-1-yl)amides to form 1-azabicyclo[3.1.0]hexanes [740]:



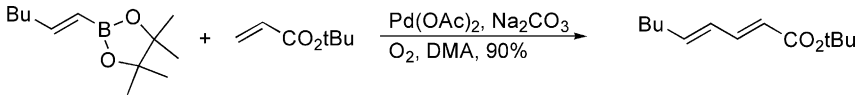
(124)

(125)

5. Additions to carbon–carbon, –oxygen, –sulfur, and –nitrogen multiple bonds

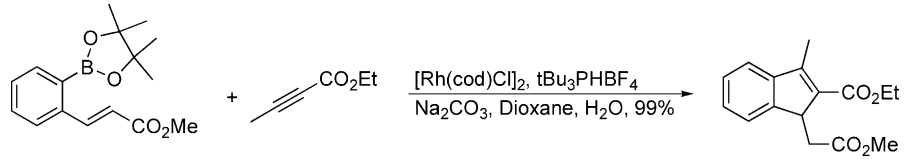
5.1. Addition of carbon-nucleophiles to alkene and alkynes

Palladium catalyzed a regioselective oxidative coupling of aromatic organoboron reagents with electron-deficient alkenes using oxygen or manganese diacetate to reoxidize the catalyst [741–744]. Palladium catalyzed an oxidative coupling of alkenyl boronic acids with alkenes to give dienes (Eq. (126)) [745]. Rhodium catalyzed a related oxidative coupling of aromatic boronic acids and activated alkenes [746]:



(126)

Palladium catalyzed asymmetric hydroarylations of activated alkenes using triarylbismuth compounds [747]. Rhodium catalyzed a cyclization of 2-(1-alkenyl)- or 2-(1-alkynyl)arylboronic esters with alkynes to give indenes and 1-alkylideneindanes (Eq. (127)) [748]. Palladium catalyzed the formation of hexasubstituted butadienes from internal alkynes and arylboronic acids (Eq. (128)) [749]. Rhodium catalyzed a regioselective hydroarylation of alkynes using arylboronic acids [750]:



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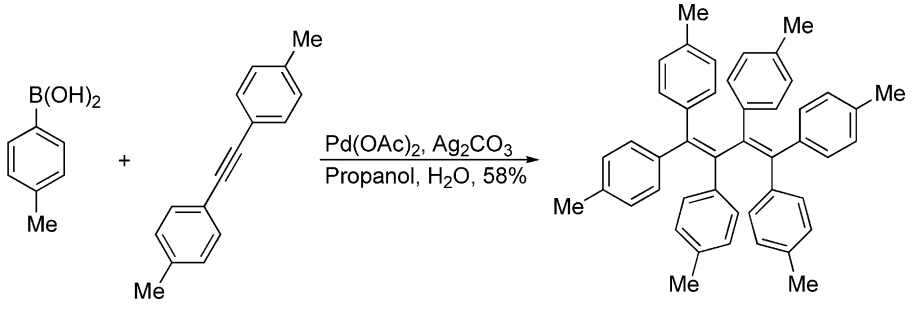
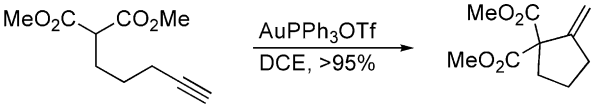


(128)

Palladium and platinum catalyzed oxidative hydroalkylations of ethene and propene using pronucleophiles [751]. Gold catalyzed intramolecular hydroalkylations of β-ketoesters having a pendent alkyne (Eq. (129)) [752]. Gold and silver catalyzed hydroalkylations of alkenes using activated methylene compounds (Eq. (130)) [753,754]. Palladium-catalyzed intramolecular hydroalkylations of 1-en-5-one derivatives to give cyclohexanes [755]. An oxidative variation was also described affording 2-cyclohexen-1-ones [756]. Palladium catalyzed the allylation of activated carbon nucleophiles with allenes [757]. Palladium catalyzed the allylation of 1,3-dicarbonyl compounds using alkynes (Eq. (131)) [758]:



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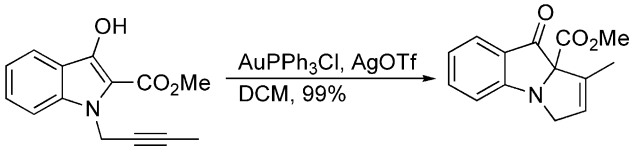

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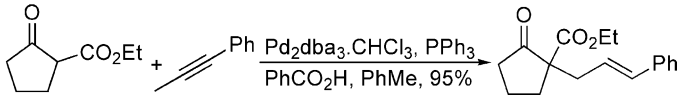
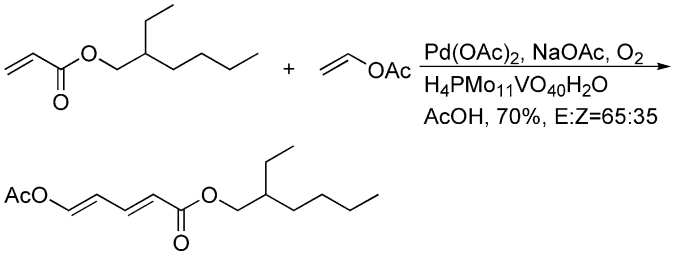


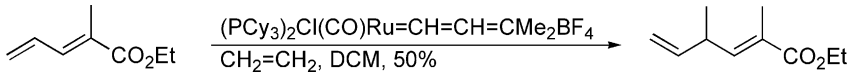
(131)

Other carbon nucleophiles were used to alkylate alkenes. A palladium-catalyzed cyclization of 3-allylsubstituted 2-silyloxy-1-cyclohexene was used in a synthesis toward garsubellin A [759]. Palladium-vanadomolybdophosphoric acid catalyzed oxidative cross-couplings of acrylates with alkenyl acetates (Eq. (132)) [760]:



(132)

An asymmetric nickel-catalyzed hydroalkenylation of styrenes was described [761]. This type of reaction was used in a synthesis of (−)-curcumene and (−)-ar-tumerone [762]. Rhodium catalyzed asymmetric hydroalkenylations of activated alkenes (Michael addition) using alkenylzirconium reagents [763]. A ruthenium-catalyzed hydroalkenylation was used toward the C10–C18 segment of ambruticin (Eq. (133)) [764]. Rhodium catalyzed the hydroalkenylation of alkenes using 4,4-dimethyl-2-oxazoline (Eq. (134)) [765]. Rhodium [766,767] and palladium [768] catalyzed hydroalkynylation of electron deficient alkenes were developed. A copper and palladium-catalyst system was used for hydroalkynylation of electron deficient alkynes [769]:



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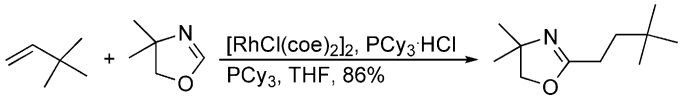
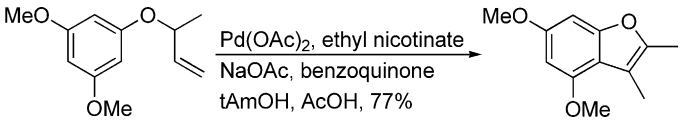
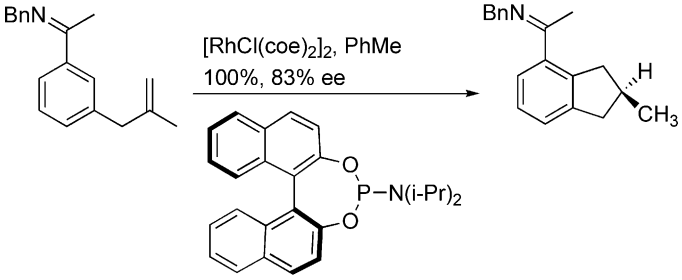


(134)

Palladium catalyzed intermolecular oxidative hydroarylations of activated alkenes using benzenes [770]. Palladium catalyzed an oxidative intramolecular hydroarylation of aryl allyl phenols (Eq. (135)) [771]. A gold–silver catalyst system was used in intra- and intermolecular hydroarylations of alkynes under solvent free conditions [772]. A platinum–silver catalyst system for hydroarylations of alkenes was reported [773]. An asymmetric rhodium-catalyzed intramolecular alkene hydroarylation of alkene containing aromatic imines was reported (Eq. (136)) [774]. Ruthenium catalyzed intermolecular hydroarylations of alkene-substituted aromatic compounds to give fused ring systems (Eq. (137)) [775]. Hydroarylation of electron-deficient alkenes were achieved using aromatic ketimines and a rhodium catalyst [776]:



(135)



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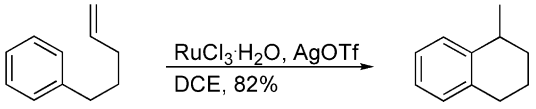
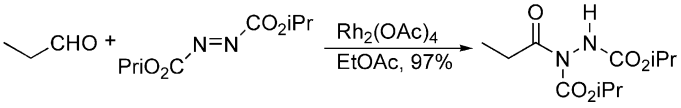


(137)

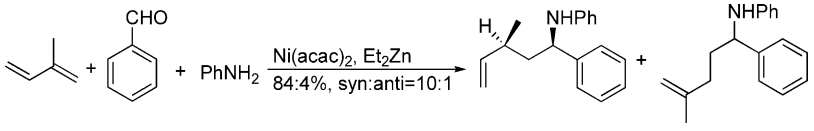
A rhodium-catalyzed hydroesterification lactonization sequence of allylic or homoallylic alcohols was reported (Eq.

(138)) [777]. Rhodium catalyzed regio- and stereoselective reductive hydroacylations of conjugated enynes with aldehydes to give 1,3-diene-5-ols (Eq. (139)) [778]. Rhodium catalyzed intermolecular hydroacylations of alkenes and alkynes [779]. Rhodium catalyzed chelation-assisted intermolecular hydroacylations of 2-hydroxy-substituted aromatic aldehydes with terminal nonconjugated dienes [780]. A rhodium-catalyzed intramolecular hydroacylation of 5- and 6-ynals was reported [781]. A rhodium-catalyzed hydroacylation was used to prepare the C16–C35 fragment of integramycin (Eq. (140)) [782]. Rhodium catalyzed hydroacylations of azodicarboxylates using aldehydes (Eq. (141)) [783]:



(141)

Nickel catalyzed a hydroacylation of dienes with aldimines in the presence of diethyl zinc (Eq. (142)) [784]. Pendant alkenes have a directing effect on the nickel-catalyzed reductive hydroacylations of alkynes using aldehydes to give allylic alcohols (Eq. (143)) [785,786]. Nickel-catalyzed reductive hydroacylations were used in syntheses of amphidinolide T1 (Eq. (144)) [787] and (−)-terestacin [788]:



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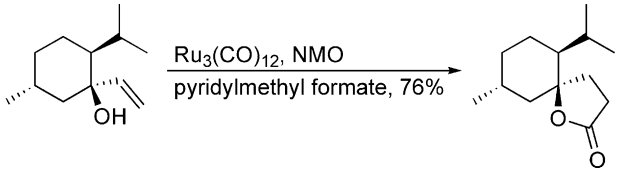

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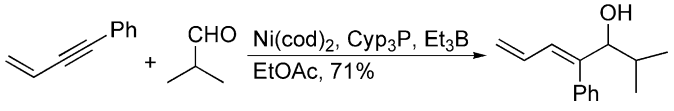

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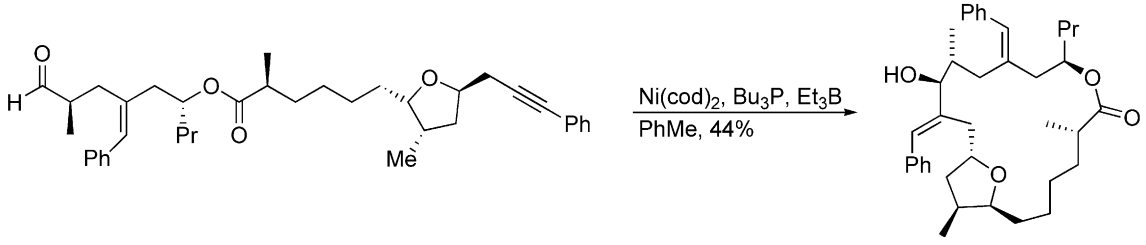

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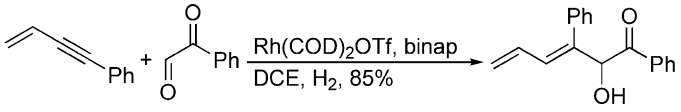

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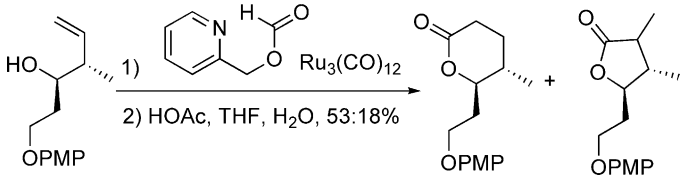
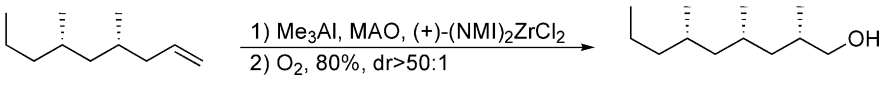


(140)

A zirconium-catalyzed asymmetric methylalumination–oxidation of terminal alkenes was used in syntheses of siphonarienolone (Eq. (145)) [789], 6,7-dehydrostipiamide [133], and scyphostatin side chain [134]. A titanium-mediated chelation-controlled hydromethylation of a propargylic alcohol using trimethylaluminum was used in a synthesis of (−)-borrelidin (Eq. (146)) [584]. Cobalt catalyzed regioselective allylzincations of 1-phenyl-1-alkynes (Eq. (147)) [790]. Nickel catalyzed an arylcyanation of alkynes (Eq. (148)) [791]:



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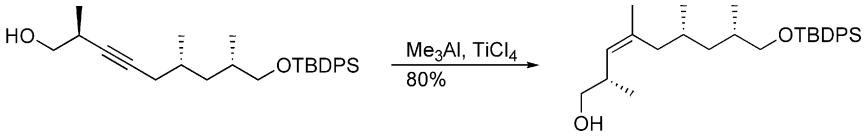

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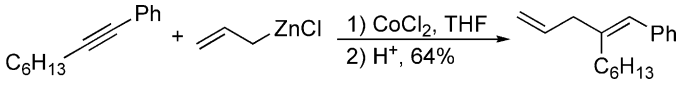
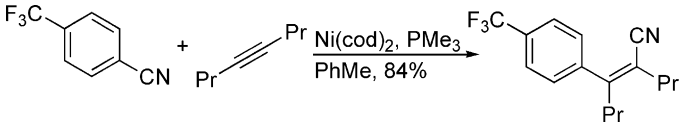


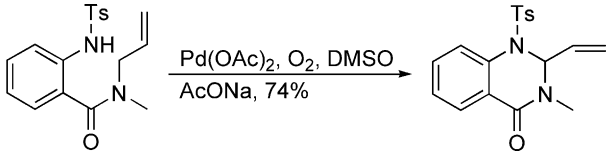
(147)



(148)

5.2. Addition of nitrogen-nucleophiles to alkenes, alkynes, and allenes

Palladium catalyzed the intermolecular amination of *N*-allyl anthranilamides to give six- or seven-membered rings depending on the conditions (Eqs. (149) and (150)) [792]. Titanium catalyzed intermolecular hydroaminations of alkynes with primary amines to give imine intermediates [793–796]. Palladium-catalyzed asymmetric intramolecular hydroaminations of pendant alkynes (Eq. (151)) [797]. Titanium and zirconium catalyzed intramolecular hydroaminations of 5- or 6-amino-1-alkenes [798,799] and of 6-amino-1,2-dienes [800]. Silver catalyzed the hydroamination of propargyl bromides with enaminones furnishing pyrroles (Eq. (152)) [801]:



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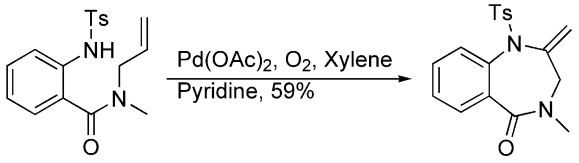

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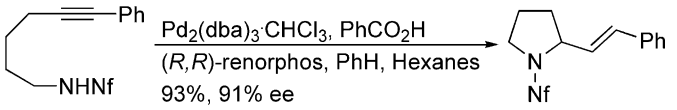

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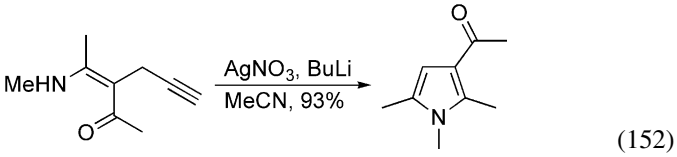
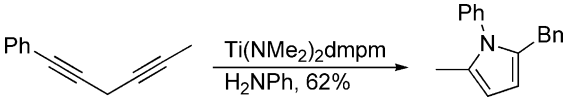


(152)

A titanium-catalyzed double hydroamination of 1,4- or 1,5-diynes was used to prepare 2,5-substituted pyrroles (Eq. (153)) [802]. A sequential titanium-catalyzed hydroamination-Heck reaction was used to prepare 2,3-disbstituted indoles (Eq. (154)) [803]. An intermolecular hydroamination of alkynes using aromatic hydrazines followed by a Fischer indole synthesis was developed (Eq. (155)) [804]:



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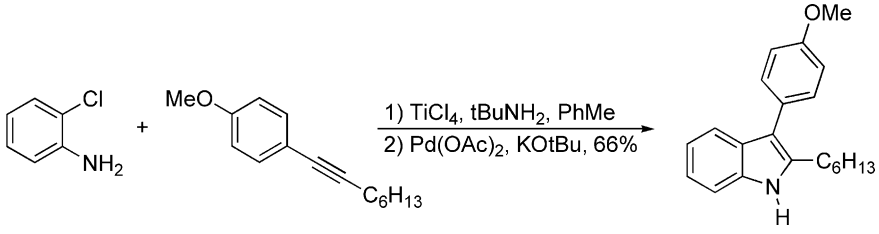

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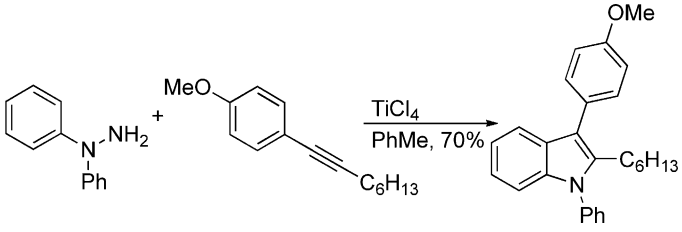
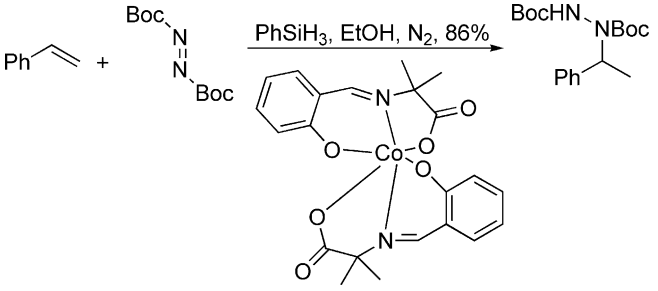


(155)

Cobalt catalyzed hydroazination of alkenes using azodicarboxylates in the presence of triethylsilane (Eq. (156)) [805]. A variety of hydrofunctionalizations of alkenes mediated by rhodium was reported [806]. Copper-catalyzed intramolecular hydroamidations of 1-amino-3-yne-2-ols to give dihydropyrroles [807]. Copper catalyzed the formation of indoles from 2-alkynyl-substituted benzenamines and this type of reaction was used to prepare hippadine [213]. A copper-catalyzed reaction of 2-allylarylsulfonylbenzenamides to give tetracyclic compound was described (Eq. (157)) [808]. Copper-catalyzed aminohalogenation of α,β-unsaturated ketones (Eq. (158)) [809]. Copper

catalyzed the formation of *N*-substituted indoles from 2-alkynylbenzaldehyde imines in the presence of an alcohol (Eq. (159)) [810]:



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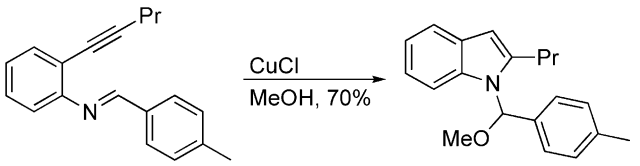
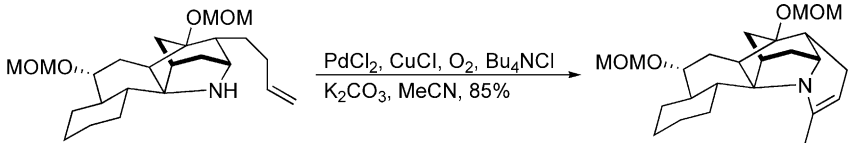


(159)

An oxidative palladium-catalyzed hydroamination of terminal alkenes was used in a synthesis of the himandrine skeleton (Eq. (160)) [811]. Ruthenium catalyzed anti-Markovnikov hydroaminations of arene substituted alkenes [812]. Palladium catalyzed the formation of enamides from alkenyl ethers using amide nucleophiles (Eq. (161)) [813]. A palladium-catalyzed hydroamidation was developed and used in a synthesis of quinolizidine 223A (Eq. (162)) [814]:



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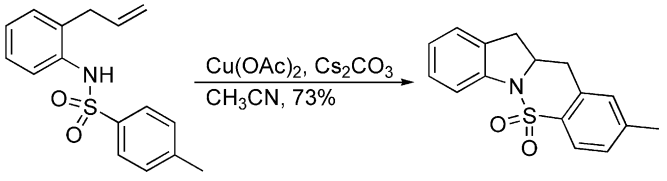

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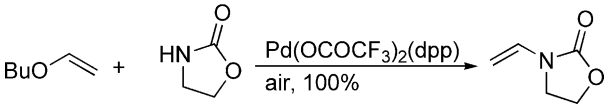

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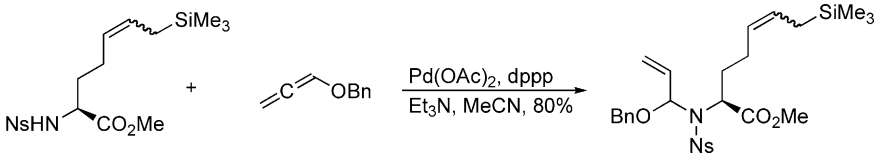

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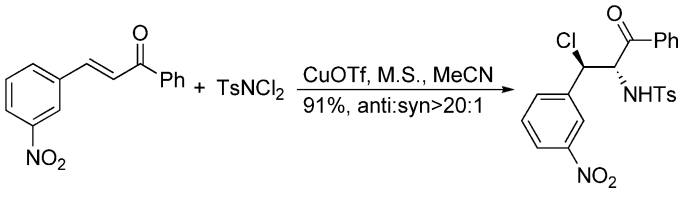
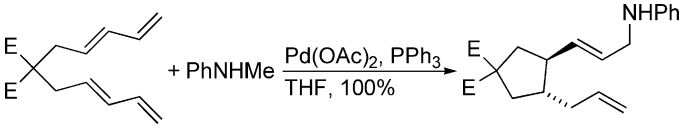


(158)

Palladium catalyzed a cyclization-amination of a 1,3,8,10-tetraene with benzenamines (Eq. (163)) [815]. Palladium-catalyzed intramolecular amidohalogenation of amides (Eq. (164)) [816] and carbamates [817] having a pendant unsaturation:



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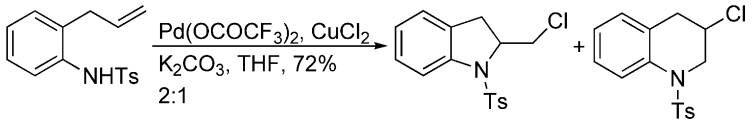
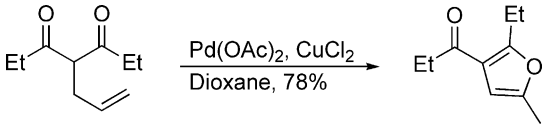


(164)

5.3. Addition of oxygen- and sulfur-nucleophiles to alkenes, alkynes, and allenes

Oxidative intramolecular hydroxy-palladation of a pendant alkene was used to form functionalized furans (Eq. (165)) [818], optically active pyrans [819], 2,3-dihydro-4*H*-pyrane-4-ones from 5-hydroxy-1-ene-3-ones [820], and ventiloquinone L [821]. A related oxidative intramolecular acyloxy-palladation of a pendent alkene was used in a synthesis of ent-EI-1941-2 and epi-ent-EI-1941-2 (Eq. (166)) [223]:



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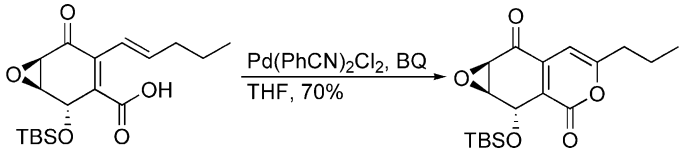
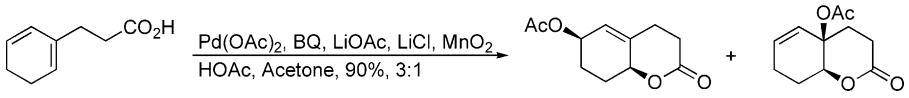


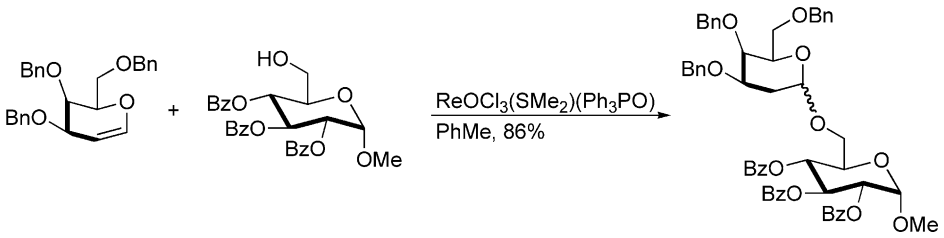
(166)

Intramolecular alkene hydroxy-palladations were shown to proceed with a high *syn*-selectivity between the palladium and the alcohol in the absence of chloride and in a predominately *anti*-fashion in the presence of chloride [822]. 2,3-Disubstituted furans were prepared by palladium-catalyzed reaction of 2-(1-alkynyl)phenols in the presence of aryl iodides [823]. Palladium catalyzed intramolecular 1,4-oxyacyloxylations of conjugated dienes (Eq. (167)) [824]. Intramolecular alkyne hydroalkoxylations were used to prepare (−)-muscone [825] and 10b-substituted hexahydropyrroloisoquinolines [826]:



(167)

Rhenium catalyzed intermolecular hydroalkoxylations to give 2-deoxy-α-glycosides (Eq. (168)) [827]. Platinum catalyzed intramolecular hydroalkoxylations to form cyclic ethers [828]. Copper catalyzed an intramolecular hydroalkoxylation using 2-(1-alkynyl)benzaldehydes and an alcohol (Eq. (169)) [829]. Iridium catalyzed the formation of ethenyl ethers from sugars and ethyl ethenyl ether [830]:



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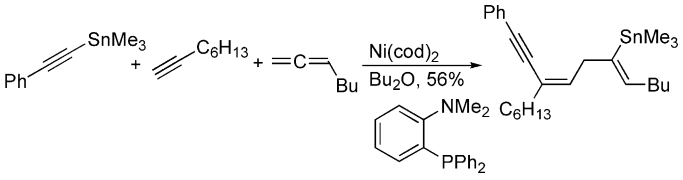
(169)

Ruthenium catalyzed a hydroacyloxylations of terminal alkynes forming Markovikov alkenyl esters [831]. This type of reaction was used in a synthesis of apicularen A [56]. Ruthenium catalyzed hydroacyloxylations of terminal alkenes in a Markovikov fashion [832]. Nickel-catalyzed hydrothioxylation of terminal alkynes [833].

5.4. Additions of hydrogen–boron, –tin, –zirconium, and miscellaneous heteroatom reagents to alkenes and alkynes

Palladium-catalyzed hydrostannations of terminal alkynes, using tributyltin hydride, to give alkenylstannanes were used in total syntheses of spirofungin B [32], (−)-apicularene [39], cystothiazoles A and B [43], C11–C29 fragment of amphidinolide F [47], (−)-disorazole C_1 [326], mangicols [77], macrolactin analogs [834], (*E*)-13,14-dihydroxyretinols [835], and the tricyclic core of guanacastepene A [74]. Regioselective hydrostannations of internal alkynes were used in syntheses of formamicin [81] and kendomycin [187]. Molybdenum was used as the catalyst for hydrostannation of an internal alkyne in a synthesis of (−)-borrelidin [397]. Palladium-catalyzed hydrostannation of bromoalkynes to give alkenylstannanes was used in a synthesis of macquarimicins [52].

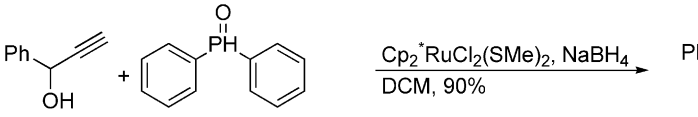
An asymmetric palladium-catalyzed hydrostannation of cyclopropenes was described [836]. Hydrostannation of an alkene was used in a synthesis of difluorinated (hydroxymethyl)conduritol analogs [15]. Palladium catalyzed the 1,2-distannation of benzynes using hexabutylditin [837]. Palladium-catalyzed distannylation of 1,4-dihydroxy-2-butyne [11]. Palladium-catalyzed germylstannation of bisallenes to give germylstannyl-substituted dialkenyl-substituted five-membered rings and [3.2.0]ring systems was developed [838]. Molybdenum catalyzed regioselective hydrostannylation or distannylations of propargylic ethers and esters [6]. Nickel catalyzed a carbostannylation of alkynes and 1,2-dienes (Eq. (170)) [839,840]:



(170)

Palladium catalyzed an asymmetric diboration of allenes [841]. Platinum catalyzed 1,4-diboration of conjugated dienes [842]. Palladium-catalyzed intramolecular disilylations of alkenes [843] and zirconium catalyzed oxidative silylation of alkenes [844]. Rhodium catalyzed a *trans*-hydrosilylations of cyclic alkynes [845]. Palladium catalyzed a tandem silastannylation–cyclization of 1,6-enynes to give dimetallated six-membered rings [846]. Silastannylation of arylethynes [9], propargylic alcohols [847], and ethyne [848] were reported.

Palladium catalyzed the hydrophosphination of alkenes and alkynes using hypophosphorous acid [849]. A double phophination of propargylic alcohols catalyzed by ruthenium was reported (Eq. (171)) [850]. Nickel-catalyzed hydrophosphination of terminal alkynes. The regioselectivity can be controlled by the catalyst system used (Eqs. (172) and (173)) [833]. A nickel-catalyzed enantioselective hydrophosphination of methacrylonitriles was described [851]:



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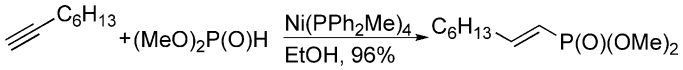

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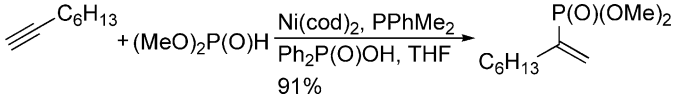
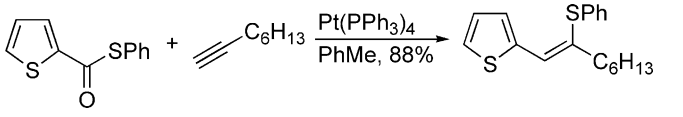


(173)

Platinum catalyzed a regio- and stereoselective thienylthiolation of alkynes (Eq. (174)) [852]. Ruthenium catalyzed the addition of sulfenamides to alkynes to give 1-thio-2-amino-substituted alkenes [853]. A zirconium-mediated haloamidation of alkynes was reported (Eq. (175)) [854]:



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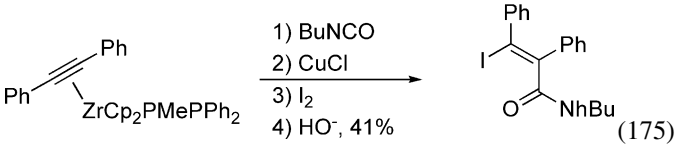
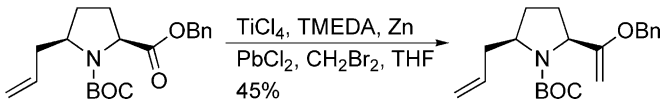


(175)

5.5. Alkylations of carbonyl compounds, imines, and azocompounds

A number of carbonyl to alkene transformations were reported. Reaction of dibromomethane- or diiodomethane–zinc–titanium tetrachloride and in some cases lead diiodide or lead dichloride with a ketone to give an alkene was used in the synthesis of (+)-acanthodoral [855], laulimalide [856], and mycalamide analogs [857]. A modified procedure consisting of dibromomethane–zinc–tetramethylethylenediamine–titanium tetrachloride–lead dichloride was used in a synthesis of ferruginine to selectively methylenate an ester (Eq. (176)) [858]. A related procedure was used to methylenate a lactone in a synthesis amphidinolide T1 (Eq. (177)) [787]. $Zn(CH_2ZnBr)_2$–titanium tetrachloride was used to methylenate a ketone in a synthesis of the A-ring of gamberic acid [723]:



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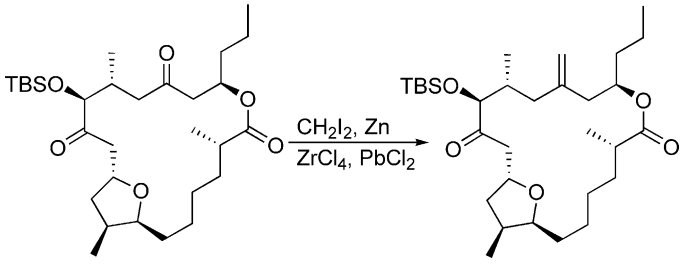
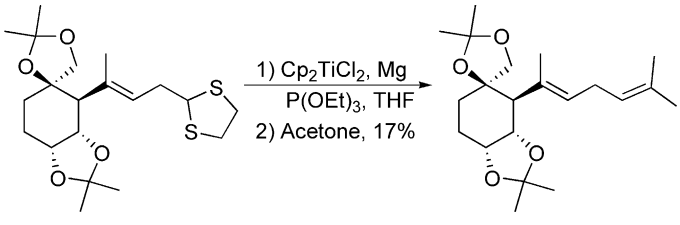


(177)

Tebbe's reagent was also used to transform esters to alkenyl ethers in synthetic approaches to scleophytin A [859] and cryptophycin-24 [431]. Petasis reagent, Cp_2TiMe_2, was used to selectively methylenate ketones in a synthesis of the C1–C17 segments of phorboxazole B, in the presence of an α,β-unsaturated ester [860], of EF fragment of (+)-spongistatin 1 [861], (+)-cuparenone [862], and epothilone analogs [863].

Titanium benzylidenes were used in solid-phase synthesis of 2-substituted benzo[*b*]thiophens [864]. A magnesium–titanium tetrachloride–dichloromethane system was used to methylenate ketones and esters [865,866]. A titanium carbene complex formed in situ from a dithioacetal was reacted with acetone in a synthesis of (−)-fumagillol (Eq. (178)) [867]. An interesting methylenation Claisen rearrangement to give eight-membered lactones mediated by titanium was described and used in a synthesis of ocatlactin B (Eq. (179)) [868]:



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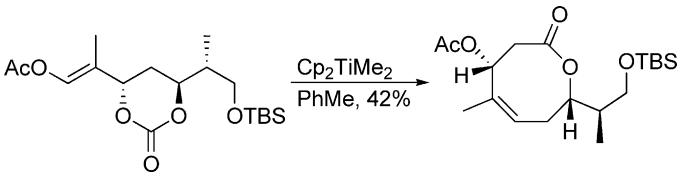

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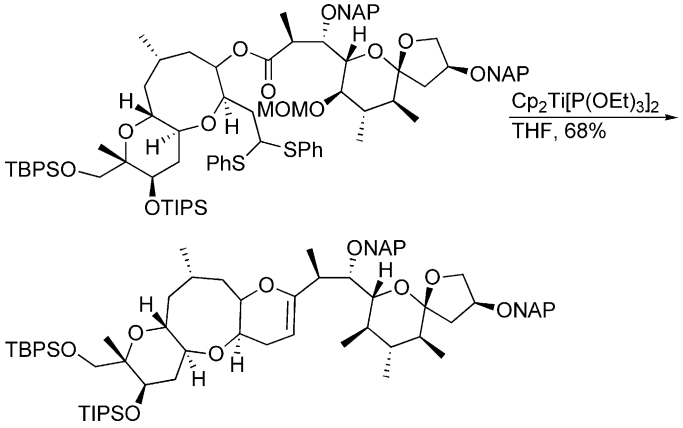
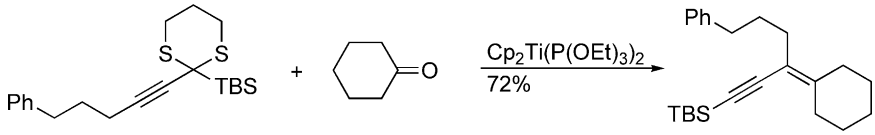


(181)



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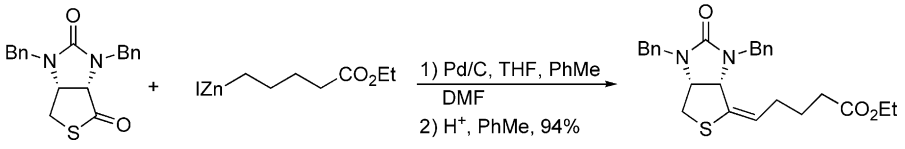
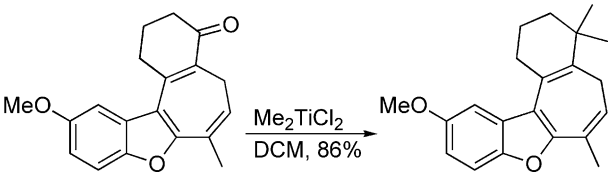


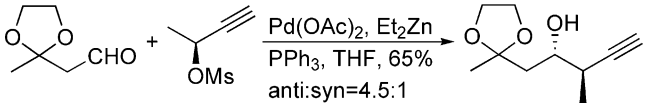
(183)

Transformation of a ketone to a gem-dimethyl group was achieved using Me_2TiCl_2 was reported in a synthesis of frondosin B (Eq. (180)) [319]. A titanium-mediated formation of a dihydropyrane from an ester and a thioacetal was used in a synthesis of ciguatoxin (Eq. (181)) [869]. Titanium-mediated alkenylation of ketones using alkynylthioketals was described (Eq. (182)) [870]. Palladium catalyzed the alkylation of thiolactones with zinc reagents. This was used in a synthesis of (+)-biotin (Eq. (183)) [871]:



(180)

Umpolung of η^3-allyl palladium complexes by transmetallation to indium or zinc was used in a number of cases. Palladium-catalyzed enantioselective allylation of benzaldehyde in the presence of diethylzinc was described [872]. Palladium-catalyzed diasteroselective alkylations of an aldehyde by a chiral propargylic mesylate in the presence of diethylzinc were used in syntheses of the C3–C13 fragment of cytostatin [873] and amphidinolide X (Eq. (184)) [186]. Palladium catalyzed a decarboxylative intermolecular aldol condensation of allyl β-ketoesters (Eq. (185)) [874]:



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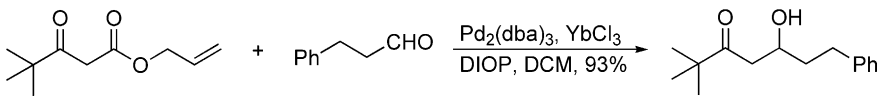
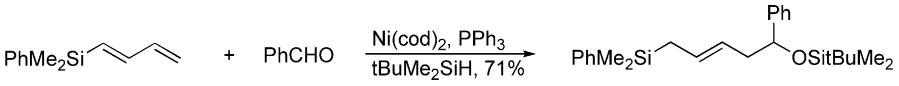


(185)

η^3-Allylnickel intermediates obtained from allylic alcohols were used in transmetallations to indium followed by alkylation of aldehydes and ketones [875]. Nickel catalyzed a reductive coupling of an aldehyde and an alkyne in the presence of triethylsilane to give allylic triethylsilyl ethers [876]. Depending on the ligand, either *E*- or *Z*-allylsilanes were prepared from dienes and aldehydes using a nickel catalyst (Eq. (186) and (187)) [877]. Rhodium catalyzed a reductive alkylation of 1,2-diketo compounds in the presence of hydrazine to give 3,5-disubstituted pyridazines (Eq. (188)) [878]. γ-Regioselective alkylation of aldehydes was observed using 2,4-pentadienylzirconium reagents [879]. Iron/chromium and cobalt/chromium mediated asymmetric allylation of aldehydes (Eq. (189)) [880]. Iron mediated allylations of aromatic aldehydes [881]:



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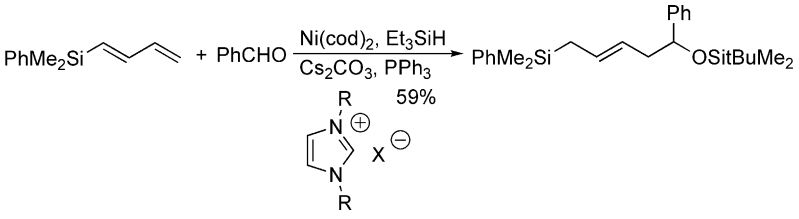

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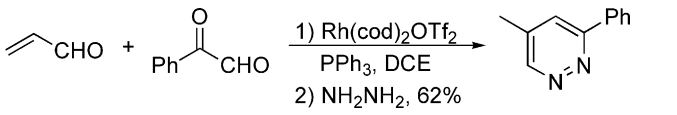
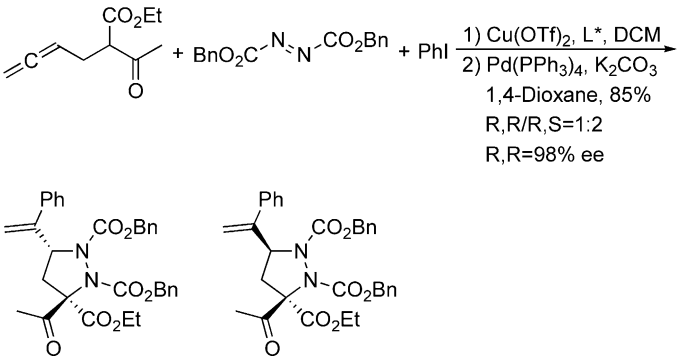


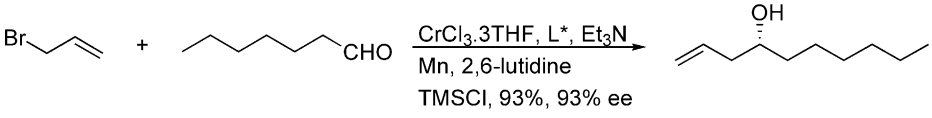
(188)

A three-component palladium-catalyzed reaction of arylboronic acids, an allene, and an aldehyde was described (Eq. (190)) [882]. A palladium–copper-catalyst system was used in an enantioselective addition–cyclization reaction of 2-(2′,3′-dienyl)-β-keto esters, organic halides, and dibenzyl azodicarboxylate (Eq. (191)) [883]:



(191)

Rhodium catalyzed an intramolecular aldol-type condensation of a metal enolate derived from an α,β-unsaturated aldehyde (Eq. (192)) [884]. Asymmetric rhodium-catalyzed reductive aldol reactions of α,β-unsaturated esters with aldehydes were reported (Eq. (193)) [885]. Rhodium catalyzed the allenylation of aryl and alkenyl aldehydes with propargylic bromides in the presence of tin oxide [886]:



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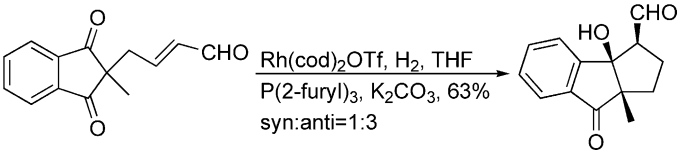
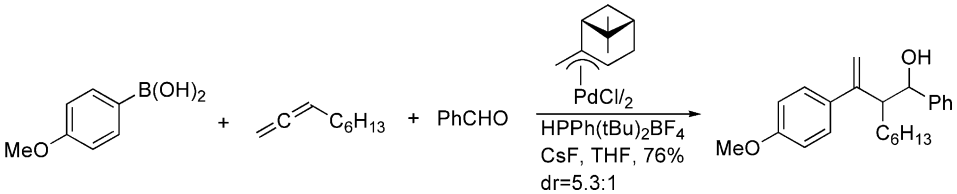
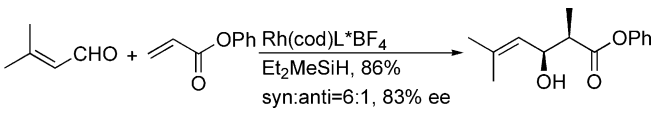


(192)

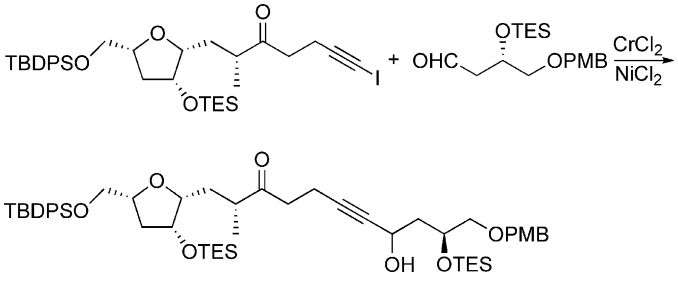


(190)



(193)

Chromium–nickel mediated Nozaki–Hiyama–Kishi type reactions were used in the syntheses of mangicols [77], ocatlactin B [868], azaspiracid-1 trioxadispiroketal (Eq. (194)) [887], and phomactin G [888]. Seven- and eight-membered rings were prepared by an intramolecular Nozaki–Hiyama–Kishi reaction of allylic phosphonates (Eq. (195)) [889]. An asymmetric nickel–chromium catalyzed Nozaki–Hiyama–Kishi reaction of alkenyl iodides with aldehydes was used in a synthesis of laulimalides (Eq. (196)) [856]:



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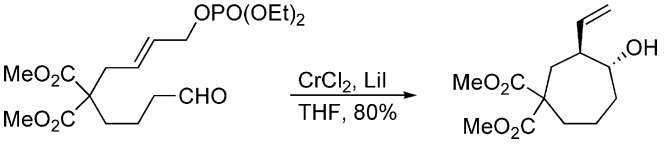

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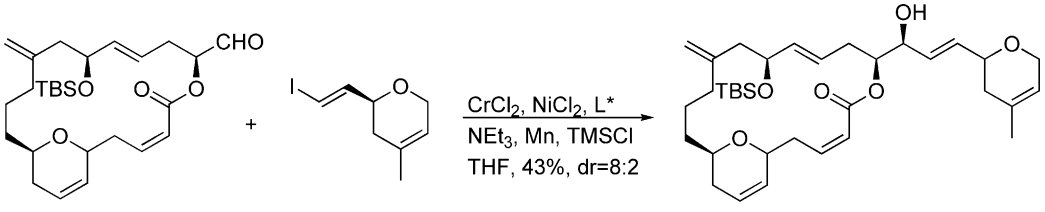
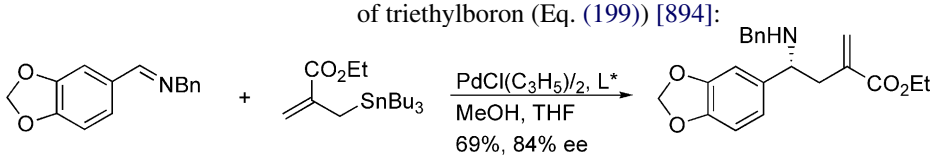


(196)

Palladium catalyzed an asymmetric carbalkoxyallylation of imines (Eq. (197)) [890]. Palladium catalyzed an asymmetric allylation of imines using tetrallylsilane–tetrabutylammonium fluoride [891]. Copper catalyzed the coupling of imines, acid chlorides, and terminal alkynes to give propargylamides [892]. A microwave-assisted addition of alkenylzirconocenes to aldimines in the presence of dimethylzinc to give allylic amines was reported (Eq. (198)) [893]. Nickel catalyzed an enantioselective coupling of internal alkynes with imines in the presence of triethylboron (Eq. (199)) [894]:



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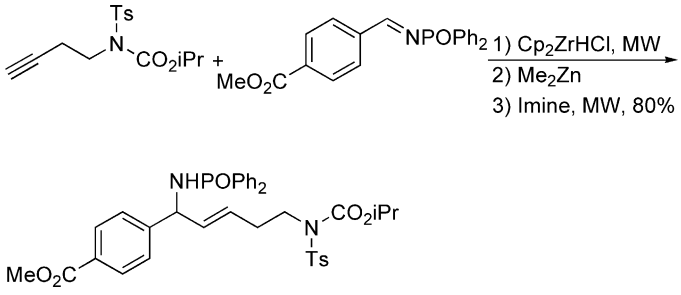

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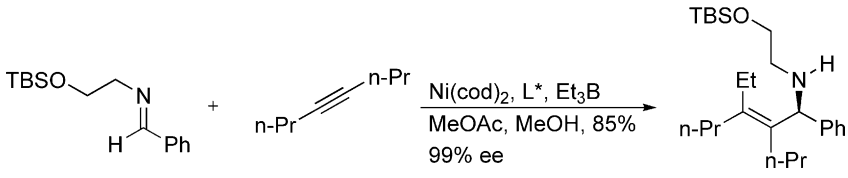
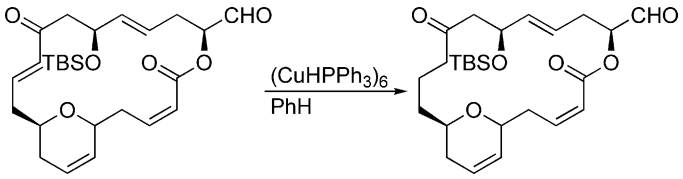


(199)

5.6. Reductions of alkenes, alkynes, carbonyl compounds, and imines

Selective conjugate reductions of electron-deficient alkenes using Stryker's reagent, $[CuHPPh_3]_6$, were used in syntheses of laulimalide (Eq. (200)) [856], migrastatins [895,896], (−)-dictyostatin [83,897], (+)-pinnatoxin A [898], and aliphatic musks [899]. Copper hydride and Stryker's reagent catalyzed asymmetric 1,4-reductions of α,β-unsaturated esters in the presence of polymethylhydrosiloxane and *t*-butanol [900]. Stryker's reagent was used in reductive aldol condensations of alkynediones (Eq. (201)) [901]. Reduction of α,β-unsaturated ketones with triethylsilane to give silylenol ethers using Wilkinson's catalyst was used in a synthesis of MetAP-2 inhibitors [125]:



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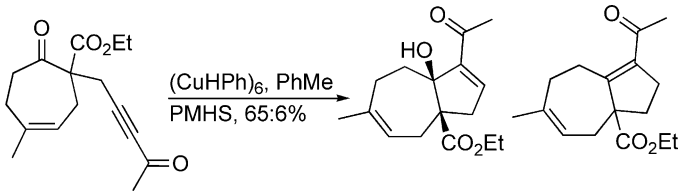
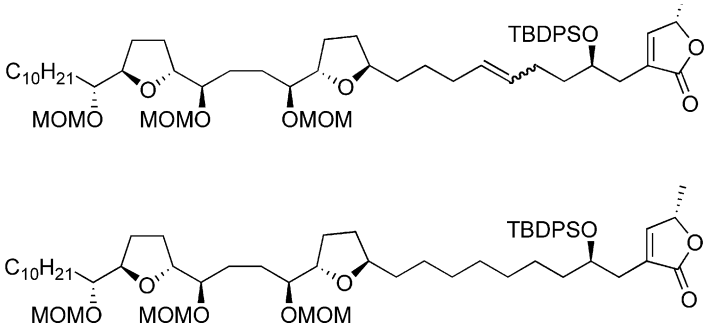


(201)

Selective carbon–carbon double bond reduction using Wilkinson's catalyst was used in a synthesis of bullatanocin (Eq. (202)) [902] and murisolin [331]. The same catalyst was used in a stereoselective chelation-controlled reduction in a synthesis of the A-ring of gamberic acid [723]. Selective reduction of an alkene in the presence of an α,β-unsaturated lactone using Wilkinson's catalyst was used in a synthesis of 8-epi-(+)-boronolide [903]. A rhodium-catalyzed chelation-controlled reduction was used in a synthesis of (−)-borrelidin [584]. A chemo- and diastereoselective rhodium-catalyzed reduction was used in a synthesis of cacospongionolides B and E (Eq. (203)) [904]. Crabtree's iridium catalyst was used in a synthesis of nemorensic acids [728], (−)-muscone [905], and dihydroagarofuran ring system [906]:



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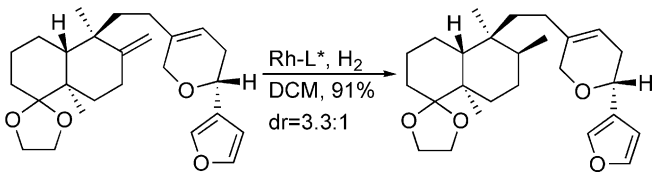
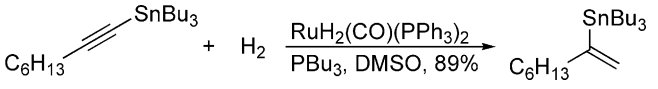


(203)

Rhodium catalyzed asymmetric reduction of alkenyl-1,2-bis(boronates) [907] and a cyclopentene carboxylic acid [908]. Ruthenium catalyzed hydrogenations of alkynylstannanes with migration of the stannyl group (Eq. (204)) [909]. Hydroxy-group directed rhodium- or ruthenium-catalyzed reductions of allylic alcohols were used in syntheses of the C29–C40 F/G segment of pectenotoxin-2 (Eq. (205)) [910] and an epothilone building block [911]. Asymmetric reduction of acetamidoalkenoates using rhodium as the catalyst was used in synthetic applications toward BILN 2061 and HCV NS3 [912] and the tryptophane residue of stephanotic acid [913]. A related reduction of an α,β-unsaturated acid was used to prepare (*R*)-(+)-6-(1,4-dimethoxy-3-methyl-2-naphthyl)-6-(4-hydroxyphenyl)hexanoic acid [433]:



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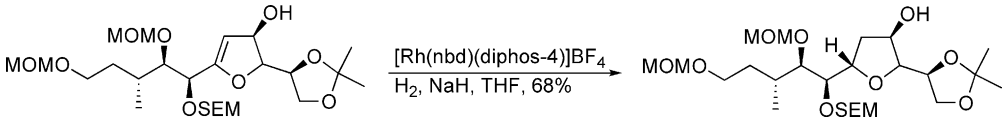
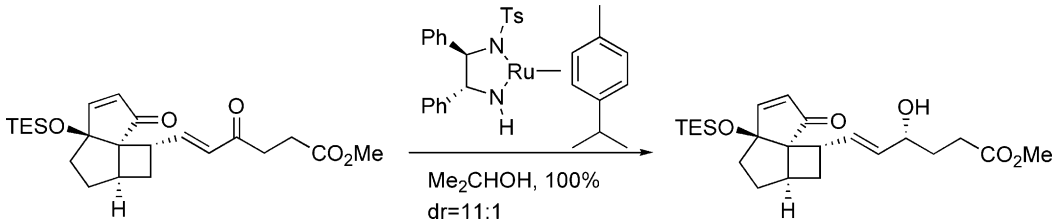


(205)

Asymmetric ruthenium-catalyzed Noyori-type reductions of ketones were used to prepare (+)-tricycloclavulone (Eq. (206)) [914], (+)-brasilenyne [915], and tetrahydrolipostatin [916], salicylhalamide [254], C3–C13 fragment of cytostatin [873], and (*R*)-rostrenol [917]. A rhodium-catalyzed enantioselective reduction of a ketone to an alcohol was used in a synthesis of eriolanin and eriolangin [918]. Dynamic kinetic resolution via ruthenium catalyzed hydrogenation of 2-substituted 3-keto esters were used to prepare Boc-dolaproine (Eq. (207)) [919] and sulfobactin A [920]:



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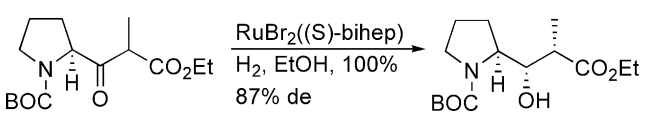
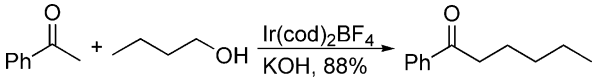


(207)

Ruthenium catalyzed an asymmetric reduction of 3,4-dihydro-β-carbolines in a synthetic route to arborescidines [921]. Iridium catalyzed reductions of quinolines to 1,2,3,4-tetrahydroquinolines [922] and this type of reaction was used in an asymmetry reduction of 2-alkylquinolines to give 2-alkyl-1,2,3,4-tetrahydroquinolines [923]. An iridium-catalyst was used in an asymmetric reduction of an imine in the synthesis of 1,2,3,4-tetrahydroharmine and 1,2,3,4-tetrahydroharmane [924]. An asymmetric rhodium-catalyzed reduction of indoles to indolines was described [925].

6. Miscellaneous carbon–carbon bond-forming reactions

Iridium catalyzed the α-alkylation of ketones with primary alcohols (Eq. (208)) [926]. Palladium catalyzed the reaction of 2-hydroxy-2-methylpropiophenone with aryl bromides to give complex multi-arylated products (Eq. (209)) [927]. Palladium catalyzed the coupling of allenic sulfoxides with allyl bromide (Eq. (210)) [928]. Palladium catalyzed the reaction of 3-methylsulfanyl-1,2,3-triazines with organozinc or Grignard reagents to give addition to the 5-position (Eq. (211)) [18]. Palladium catalyzed three component reactions of alkenyl- or aryl-bromide, an isonitrile, and an amine or alkoxide to give α,β-unsaturated amidines or imidates (Eq. (212)) [929,930]. Cyclic amidines and imidates were prepared in a similar fashion [931]. Palladium-catalyzed dehydroarylations of triarylmethanols. The intermediates can be coupled with aryl halides, alkenes, and alkynes (Eq. (213)) [932]:



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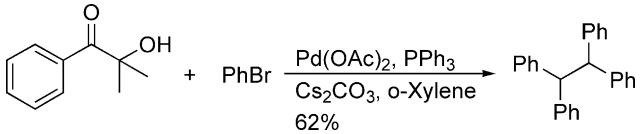
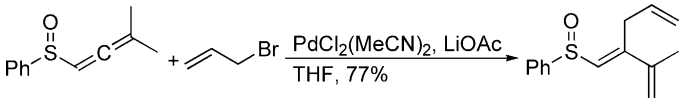
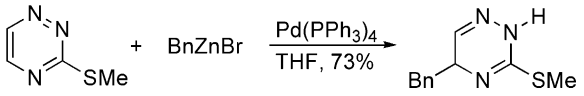


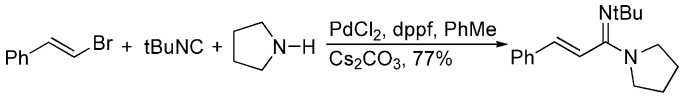
(209)



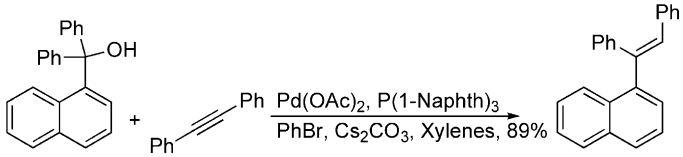
(210)



(211)

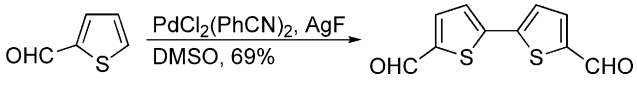


(212)



(213)

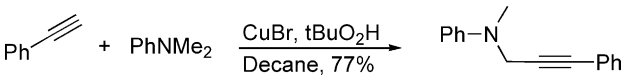
Nickel catalyzed the coupling of 4-mesylcoumarins with aryl halides [933]. Palladium-catalyzed homocoupling of furans to give 2,2′-bithiophenes (Eq. (214)) [934] was reported. Palladium catalyzed the coupling of a 3-iodoindole derivative to give a 3,3′-biindole in a synthesis of a putative natural product [11]. A palladium-catalyzed copper-mediated coupling of 1-bromo-2-nitrobenzenes with 3-halo-2-enals, enones, and esters was described [935]:



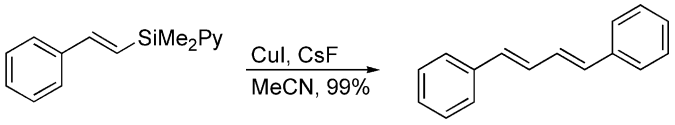
(214)

Copper-catalyzed alkynylation of an sp^3–carbon adjacent to the nitrogen of aryl amines in the presence of *t*-butylhydrogen peroxide (Eq. (215)) [936]. A copper-catalyzed homo-coupling of ethenyl(2-pyridyl)dimethylsilane was used to prepare substituted butadienes (Eq. (216)) [937]. Rhodium catalyzed a hydrative dimerization of terminal alkynes to give enones (Eq. (217)) [938]. Cobalt catalyzed a tail-to-tail reductive dimerization of electron-deficient alkenes in the presence of water and zinc [939]. Rhodium catalyzed oxidative arylations of cyclic amines (Eq. (218)) [940]. Palladium catalyzed the reaction of

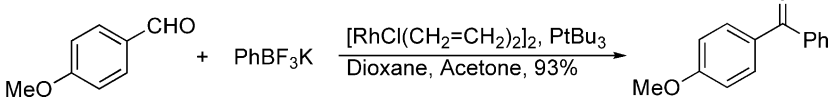
organoboronic acids with 1,2-dien-3-ols to give substituted 1,3-dienes (Eq. (219)) [941]:



(215)



(216)



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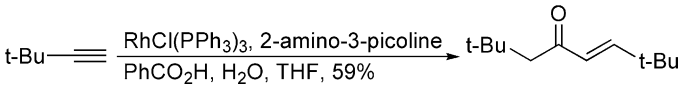
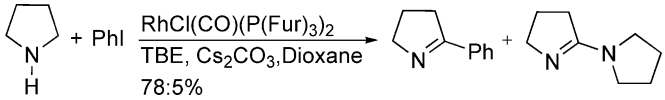
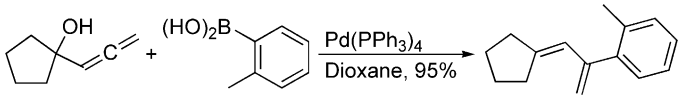


(217)

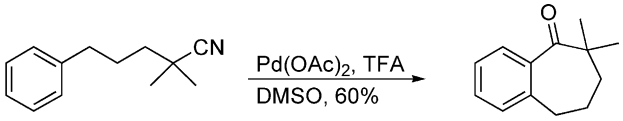


(218)

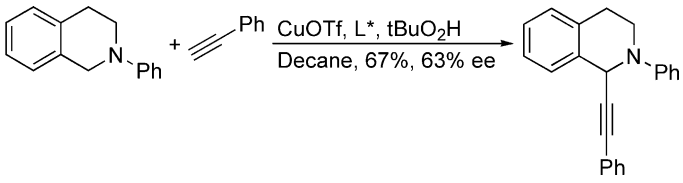


(219)

Unsymmetrical aryl ketones were prepared via a palladium-catalyzed coupling of aromatic compounds and aryl nitriles (Eq. (220)) [942]. Rhodium catalyzed the coupling of aromatic aldehydes with trifluoro(organo)borates to give unsymmetrical ketones (Eq. (221)) [943]. A copper-catalyzed intermolecular arene–arene coupling was used to prepare axially chiral biphenyls [944]. Copper catalyzed an asymmetric 1-alkynylation of tetrahydroisoquinolines (Eq. (222)) [945]. Copper catalyzed the coupling of an aromatic iodide with sodium trifluoroacetate introducing a trifluormethyl group in a synthesis of (*R*)- and (*S*)-2-trifluoromethylepinephrine [946]:

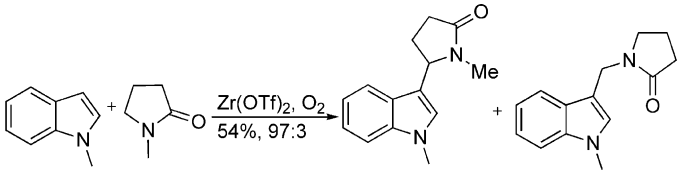


(220)

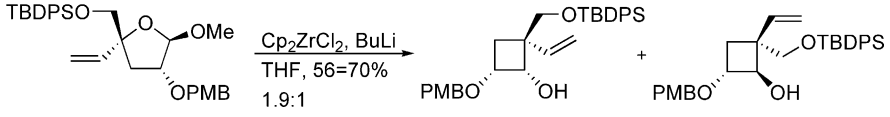


(222)

Zirconium catalyzed direct coupling of indoles, pyrroles, and thiophenes with cyclic amides (Eq. (223)) [947]. Zirconium promoted a ring contraction of 4-alkenylfuranosides to give alkenylcyclobutanols (Eq. (224)) [948]. Palladium catalyzed an allylalkynylation of intermediately formed benzyne (Eq. (225)) [949]:



(223)



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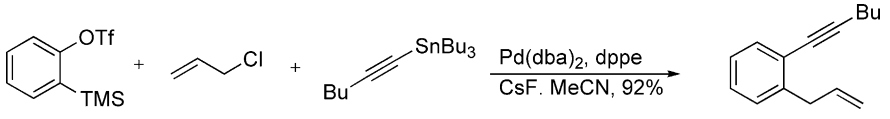
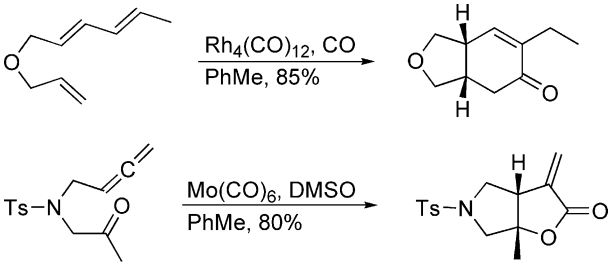


(225)

7. Carbonylations

7.1. Carbonylations of alkenes, allenes, and arenes

Rhodium-catalyzed a [3 + 2 + 1]cycloaddition of 1,3,8-trienes and carbon monoxide (Eq. (226)) [950]. Molybdenum mediated a carbonylative carbocyclization of 7-carbonyl-1,2-dienes to form bicyclic α-methylene-lactones (Eq. (227)) [951]. Rhodium catalyzed intra- and intermolecular [2 + 2 + 1]cycloadditions of 1,3-dienes, alkenes, and carbon monoxide (Eq. (228)) [952]:



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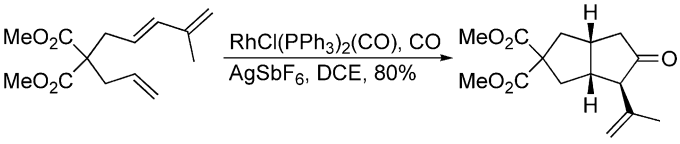
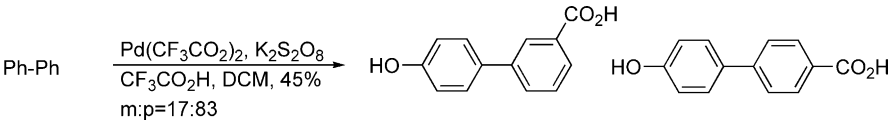


(228)

Palladium catalyzed an interesting hydroxylation carboxylation of biphenyl (Eq. (229)) [953]. Palladium-catalyzed carbo-carboalkoxylation of alkene-tethered indoles (Eq. (230)) [954]. Palladium catalyzed a direct carbonylation of ethyl- or methylamino-substituted aromatics (Eq. (231)) [955]. Palladium-catalyzed hydrocarbonylation of alkenes or 1,2-dienes having a tethered amino or hydroxy group produced lactones and lactams, respectively (Eq. (232)) [956]. Palladium catalyzed a related enantioselective formation of substituted 3,4-dihydroquinoline-2-ones from 2-alkenylbenzenamines (Eq. (233)) [957]. Palladium catalyzed an asymmetric hydrocarbonylation of 2-isopropenylphenol to give 4-methyl-3,4-dihydrocoumarine [958]. Cobalt catalyzed a hydrocarbonylation of enamides [959]:



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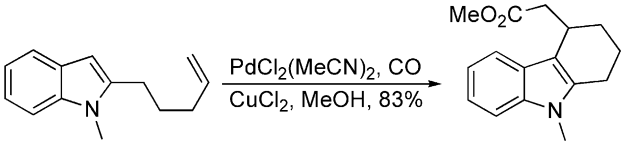

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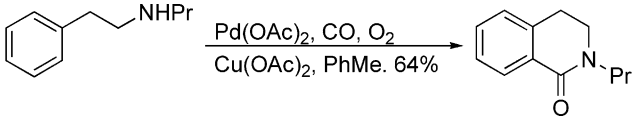

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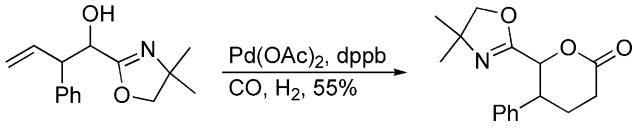

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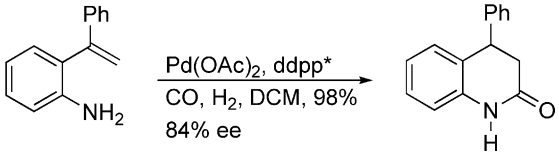
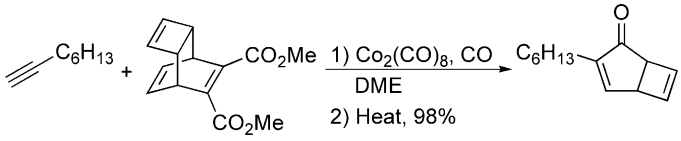


(233)

7.2. Carbonylations of alkynes (including the Pauson–Khand reaction)

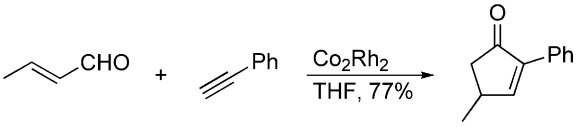
Metal-mediated [2 + 2 + 1]cycloadditions forming cyclopentenones continued to be extensively studied, in particular the cobalt-mediated or -catalyzed, Pauson–Khand reaction. The mechanism of the rhodium-catalyzed asymmetric Pauson–Khand reaction was examined [960]. A cyclobutadiene equivalent was developed (Eq. (234)) [961]. Pauson–Khand reactions in aqueous media was reported [962]. A double intermolecular Pauson–Khand reaction of macrocyclic diynes with ethene was described [963]. Some endo-product was isolated from reactions of electron-deficient alkynes and norbornadiene [964]:



(234)

A rhodium–cobalt catalyst was used together with an α,β-unsaturated aldehyde in Pauson–Khand reactions. The aldehyde served both as the alkene component and source of carbon monoxide (Eq. (235)) [965]. Formaldehyde was used as the carbon monoxide source in asymmetric rhodium-catalyzed intramolecular Pauson–Khand reactions [966]. Rhodium catalyzed intramolecular Pauson–Khand reactions of alkynes tethered to 1,3-cyclohexadienes [967]. Rhodium catalyzed inter-

molecular Pauson–Khand reactions using 1,3-dienes (Eq. (236)) [968]. Ruthenium catalyzed intermolecular Pauson–Khand reactions where the regioselectivity was directed by a pyridylsilyl or pyrimidylsilyl group [969]. Desymmetrization of meso dienynes was described using a chiral rhodium or iridium catalyst [970]:



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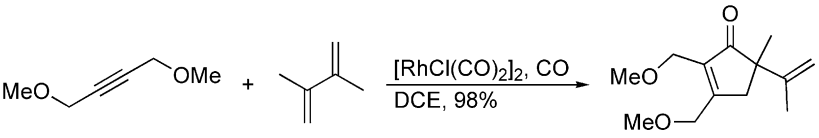
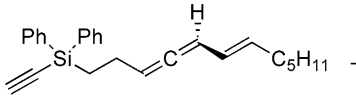


(236)

Asymmetric intermolecular cobalt-mediated Pauson–Khand reactions of chiral alkenylsulfoxide were used to prepare (−)-pentenomycin I and the (−)-aminocyclopentitol part of a hopane triterpenoid [971]. The intermolecular Pauson–Khand reaction was used in a synthesis of (−)-terestacin [788]. Intramolecular Pauson–Khand reactions of 2-aryl-1,6-enynes, 1-methyl- and 1-phenyl-1,7-enynes [972], and cyclic 3-(2-alkyne-1-yl)-1-alkylidenes [973] were reported. Cobalt mediated asymmetric Pauson–Khand reactions of electron-deficient alkenes [974].

Intramolecular Pauson–Khand reactions were used in synthetic applications toward 8α-hydroxystreptazolone [975], treprostinil [976], indole alkaloids [977], (+)-conessine (Eq. (237)) [978], nucleoside analogs [671], and $\Delta^{12,14}$-15-deoxy-PG-J_1 methyl ester and epi-Δ^{12}-15-deoxy-PG-J_1 [979].



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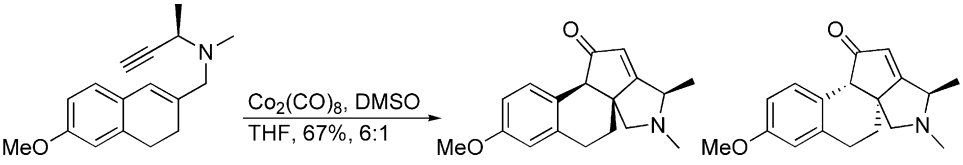
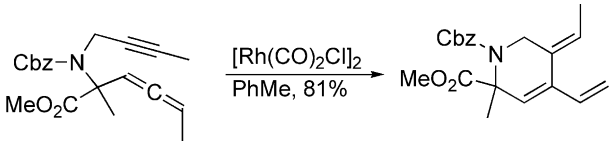
Intramolecular Pauson–Khand reactions of chiral 1,5-dien-7-ynes to give bicyclo[4.3.0]-1,7-nondiene-6-ones were described [980]. An intramolecular Pauson–Khand reaction of an enyne having a trimethylgermyl group on the alkyne terminus was described [981]:



(237)

An intermolecular cobalt-catalyzed allenic Pauson–Khand reaction was described [982]. Rhodium catalyzed intramolecular allenic Pauson–Khand reactions [983,984]. Depending on the catalyst, both the regio- and chemoselectivity of cyclizations of 1,2-diene-7-ynes were controlled (Eq. (238)–(240)) [985]. A molybdenum-catalyzed intramolecular allenic Pauson–Khand reaction was used to prepare 15-deoxy-$\Delta^{12,14}$-prostaglandin J_2 (Eq. (241)) [986]:



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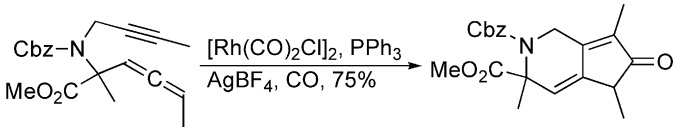

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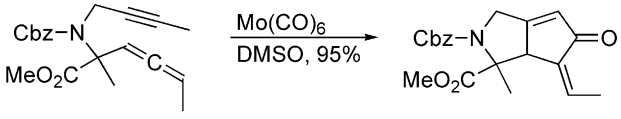
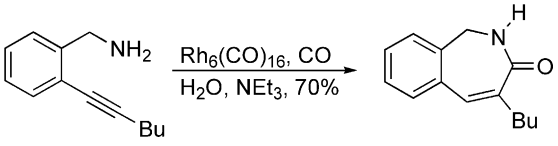


(240)

Rhodium catalyzed the formation of benzazepinones from 2-alkynylbenzylamines via a hydrocarbonylation (Eq. (242)) [987]. Rhodium catalyzed a [2 + 2 + 2 + 1]cycloaddition of 1-ene-6,11-diynes to give tricylic ring systems (Eq. (243)) [988]. Nickel mediated a hydrocarboxylation of alkynes using carbon dioxide to form α,β-unsaturated carboxylic acids [989]. Palladium catalyzed the formation of β-selenylacrylamides from reaction of terminal alkynes with sulfenamides, diphenyldiselenide, and carbon monoxide (Eq. (244)) [990]:



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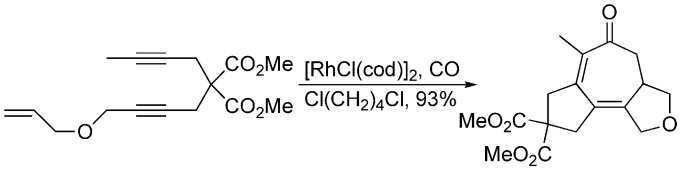

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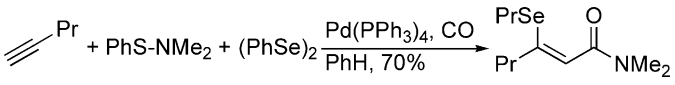

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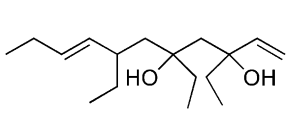
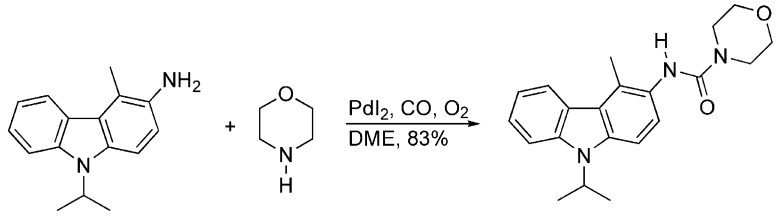


7.3. Miscellaneous carbonylations

Palladium catalyzed the carbonylative ring-expansion of *N*-alkylisothiazolidines to give tetrahydro-2*H*-1,3-thiazin-2-ones [991]. Palladium-catalyzed reactions of imines, allylic halides and carbon monoxide to form alkenyl-β-lactams [992,993]. Palladium catalyzed the formation of allylic amides from allylic halides and phosphonates, carbon monoxide and a titanium–nitrogen complex [994]. Palladium catalyzed the decarboxylation-carbonylation of 4-methoxycarbonyloxy-2-butyn-1-ols with phenols to give alkenyl-substituted cyclic carbonates [995]. A palladium-catalyzed oxidative carbonylation of amines to give ureas was developed and applied to the synthesis of NPY5RA-972 (Eq. (245)) [996]. Palladium-catalyzed carbonylations and couplings of boroxarenes and borazarene (Eq. (246)) [179]. This reaction was used in a synthesis of phenaglydon:



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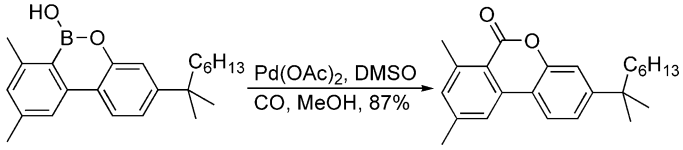
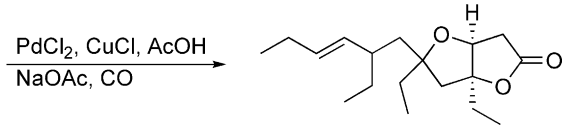


(246)

An intramolecular palladium-catalyzed hydroxy-palladation carbonylation of an ene-diol was used to prepare plakortone E (Eq. (247)) [997]. An interesting palladium-catalyzed cyclization–carbonylation was used in a synthesis of (+)-acanthodoral (Eq. (248)) [855]. Palladium catalyzed an annulation-carbonylation sequence of 2-ethynylbenzenamine amides or ureas to give 4*H*-3,1-benzoxazines, quinazolin-2-ones, and quinoline-4-ones (Eq. (249)) [998]. Palladium catalyzed a five-component coupling of a two different organozinc reagents, TMS-chloride, carbon monoxide, and propenoyl chloride to give 1,4-diketones (Eq. (250)) [999]:



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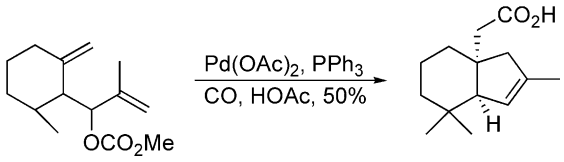

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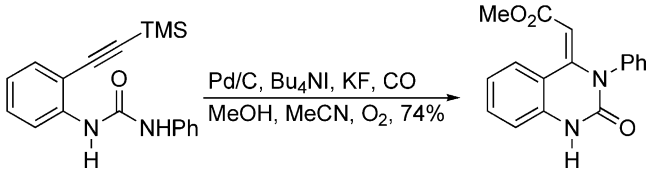

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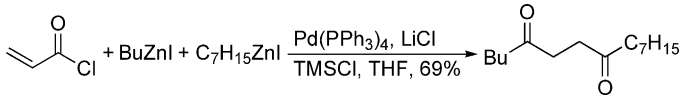
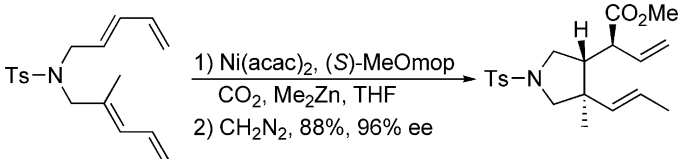


(250)

Nickel catalyzed an asymmetric cyclization of 1,3,8,10-tetraenes with diorganozincs in the presence of carbon dioxide (Eq. (251)) [1000]. Cobalt catalyzed a carbonylation of β-lactones to give succinic anhydrides (Eq. (252)) [1001]. Cobalt catalyzed hydroformylations of oxiranes to give β-hydroxyaldehyde acetals in the presence of trimethylorthoformate (Eq. (253)) [1002]. Iron mediated a carbonylative ring-expansion of alkenylcyclopropanes to give 2,5-dialkyl cyclohexenes (Eq. (254)) [1003]. Molybdenum catalyzed an interesting cyclocarbonylation of alkylidene cyclopropanes having a pendant aldehyde (Eq. (255)) [1004]:



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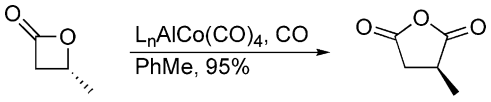

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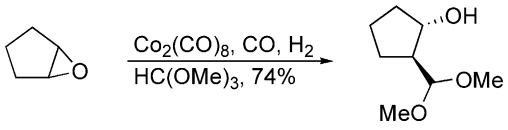

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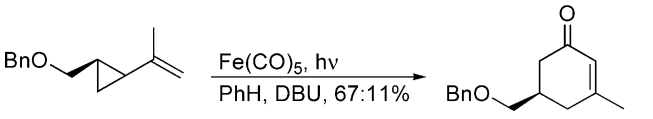

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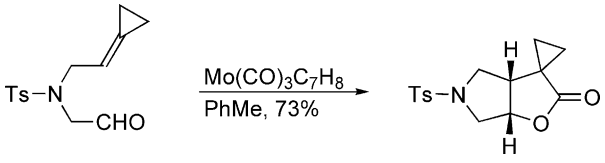
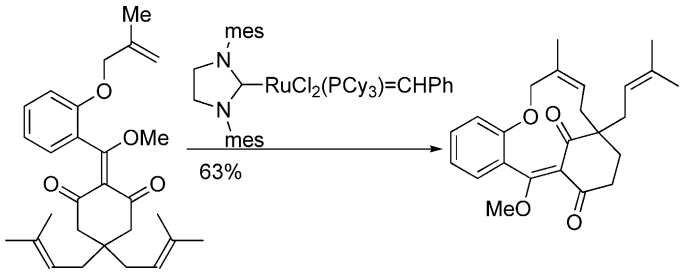


(255)

A number of transition metals catalyzed the formation of methylene substituted cyclic carbonates from propargylic alcohols and carbon dioxide in an ionic liquid [1005]. Ruthenium catalyzed a carbon–hydrogen bond insertion, carbonylation, and alkylation sequence of the benzene ring of *N*-arylpyrazoles [1006].

8. Metathesis reactions

A large number of reports on ring-closing metathesis (RCM) reactions using Grubbs'type ruthenium catalysts were published. A number of new ruthenium catalysts were developed [1007–1011]. Remote substituents affect the outcome of RCM (Eqs. (256) and (257)) [1012]. Ruthenium catalyzed a tandem cyclopropanation-ring-closing metathesis of dienynes (Eq. (258)) [1013]. Ruthenium catalyzed a ring-closing metathesis isomerization reaction (Eq. (259)) [1014]. Double ring-closing metathesis of nitrogen containing tetraenes was reported [1015]. 2-Chloro-, 2-trifluoromethyl-, or 2-fluoro-1,*n*-dienes were cyclized to form the corresponding chlorinated or fluorinated cycloalkene [1016]:



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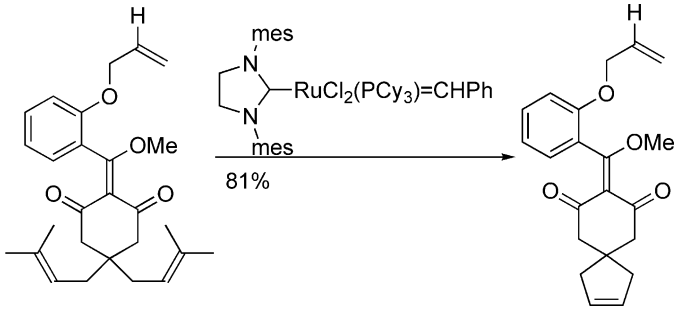

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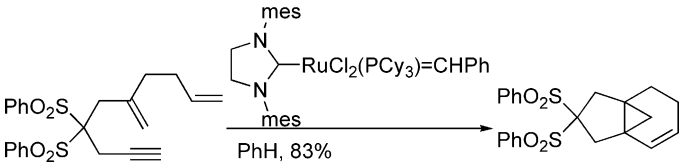

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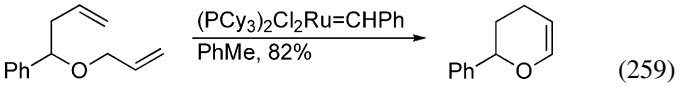
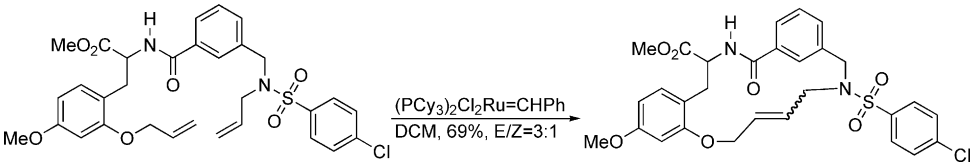


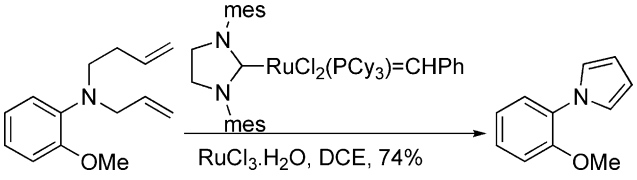
(259)

A large variety of ring systems were prepared via alkene–alkene RCM using ruthenium catalysts. Ring-systems including, cyclopentene [1017], substituted cyclopentenols [1018], cyclic β-hydroxyallylsilanes [1019], phenanthrenes [1020], fused 1,4-dihydrobenzenes [1021], bicyclo[4.2.1]nonanes [1022], 2,3,6,7-tetrahydro[1,2]oxasilepines [1023], 2*H*-1,5-benzodioxepins and 2,5-dihydro-1,6-benzodioxocins [1024], oxepene and oxocene [1025], 6-, 7-, 16-, 19-, and 20-membered lactams [1026,1027], 2,5-dihydropyrroles [1028,1029], 1,2,3,6-tetrahydropyridines [1030], bicyclic 1,2-fused-1,2,3,6-tetrahydropyridines [1031], 1,4-dihydrocarbazole [1032], azepines [1033], cyclic hydrazines [1034], 2,3-fused indoles [1035], cyclic peptides [1036], α-boronic acid

substituted cyclic amine [1037], macrocycles (Eq. (260)) [1038–1044], thiazocine-2-acetic acid derivatives [1045], cyclic amino acid derivatives [1046], cyclic β-amino acid esters [1047], cyclic peptidosulfonamides [1048], bicyclic heptapeptides [1049], vicinal *trans*-diaminocyclitols [1050], quinolizinium cations [1051], 1,2-dihydroquinolines, benzoazocine, and benzoazepine [1052], cyclic hydrazine derivatives [1053], 5,6-dihydro-2*H*-1,6-benzoazocines and 4*H*-1,4-benzooxazines, 1,2,5,6-tetrahydro-1,6-benzodiazocines, 5,6,9,10-tetrahydropyrido[2,3-*b*][1,4]diazocine, 5,6-dihydro-2*H*-1,6-benzothiazocine 1,1-dioxide, and 2,5-dihydro-1,5-benzothiazepine 1,1-dioxide [1054], nitrogen-containing spirocycles [1055], benzofurans [1056], bicyclic lactams [1057,1058], β-lactam-sulfonamides hybrids [1059], 4,4-difluoroglycosides [1060], carbohydrate-derived spirocycles [1061], azaspirocycles [1062], pyranopyran sugar amino acids [1063], polyfunctionalized pyrrolidines [1064], spirocyclic piperidines [1065,1066], spirocyclic lactams [1067,1068], spirocyclic ketals [1069], [4.3.0]bicylic nucleotide [1070], allylated dinucleotides [652], 1,4-dihydro-9,10-anthraquinones [1071], azepino[3,4-b]indole-1,5-diones [1072], eight-membered iminoalditol [1073], and sultones and sultams [1074] were all prepared using ring-closing metathesis methodology. Two ruthenium catalysts were used to form pyrroles from diallylamines via ring-closing metathesis and oxidation (Eq. (261)) [1075]:

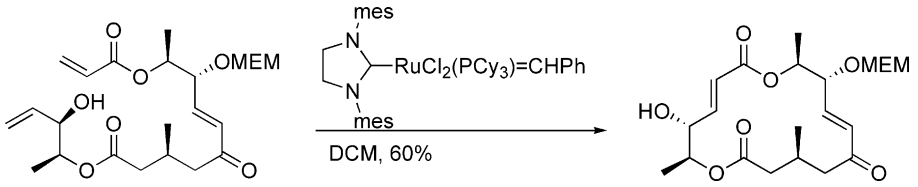


(260)

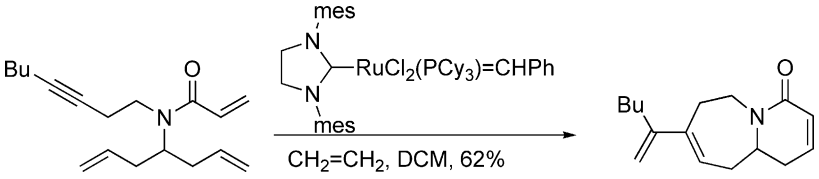


(261)

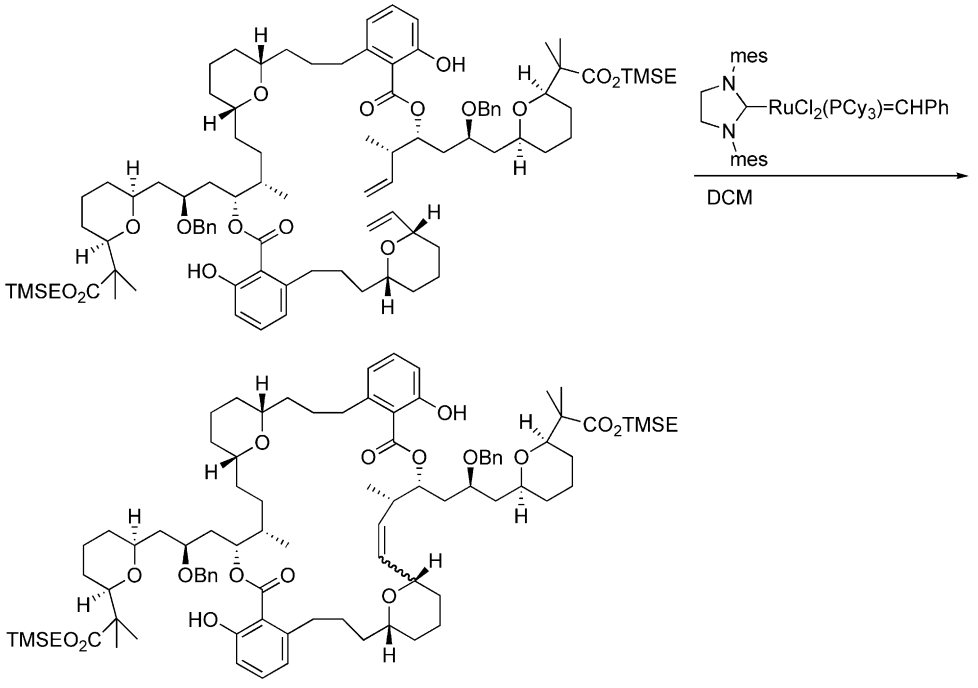
Synthetic targets using alkene-alkene ring-closing metathesis include, MetAP-2 inhibitors [125], macrosphelides (Eq. (262)) [1076], 1-*O*-β-D-glucopyranolsyl-5-deoxyadenophorine [1077], guanacastepenes and rameswaralide [1078], frondosin B [319], (−)-cytisine [1079], cytotrienins [552], ciguatoxin [639], pancratistatins [1080], 3-deoxy-8-oxatropanes [1081], halichlorine and pinnaic acid [1082], D-carbocyclic nucleosides [1083], carbocyclic nucleoside intermediates [1084], (−)-antofine [34], pumilotoxin precursors [1085], (−)-aristeromycin [1086], (+)-puraquinoic acid [35], cacospongionolides B and E [1087], solamine [1088], oncinotine and neooncinotine [1089], hepatitis C virus NS3 protease inhibitor [1090], subunits of (+)-discodermolide [1091], bicyclic quinolizidine alkaloids (Eq. (263)) [1092], meso-2,6-diamonopimelic acid and FK565 [1093], periplanone C [1094], (+)-4-amino-2-cyclopentene-1-one [1095], (+)-rhizoxin D [1096], securinine and (−)-allonorsecurinine [418], (−)-spongidepsin [1097], BILN 2061 and HCV NS3 [912], (−)-callystatin A [131], wiedemannic acid [1098], C1–C16 segments of bryostatin 1 [1099], ADE fragment of nakadomarin A [1100], (+)-migrastatin [895], ingenol [1101], (+)-SCH 351448 (Eq. (264)) [50], quinolizidine 223A [814], (+)-tricycloclavulone [914], bistramide A [1102], ophirin B [1103], (E)-dehydroepothilones [1104], migrastatins [896], (+)-brasilenyne [1105], (−)-gigantecin [327], nakadoamine [1106], spongidepsin [1107], pochonin C [1108], (+)-scyphostatin [136], gaur acid [663], (−)-aplystatin, (+)-palisadin A-B, 12-hydroxypalisadin B and the AB-ring system of adociasulfate-2 and toxicol A [1109], salicylhalamide [254], 1,13-herbertendiol [1110], (−)-fumagillol [867], epothilone analogs [863,1111], (−)-gloesporone [1112], (+)- and (−)-microcarpalide [1113,1114], 1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine [1115], epothilone [1116], furano-epothilone C [1117], analogs of methyllycaconitine [1118], (−)-cladospolide A [1119], phosphatidylinositol analogs [1120], enokipodins A–B [1121], croomine [1122], miharamycin [1123], (−)-stemoamide [1124], quinolizidine alkaloids [1125], C15–C24 fragment of discodermolide [1126], siastatin B analogs [1127], goniothalmin [1128,1129], huperzine B [1130], C1–C11 fragment of peloruside A [1131], 1,4-dideoxy-1,4-iminohexitols [1132], massoialactone [1133], *iso*-β-bisabolol [1134], –infusiol A and cuprenelol [593], anamarine [1135], (1*R*,2*S*,9*S*,9*aR*)-octahydro-1*H*-pyrrolo[1,2-*a*]azepin-1,2,9-triol [1136], (−)-allosedamine and (1*R*,3*R*)-HPA-12 [1137], pseudoheliotridanes [1138], (+)-1-epiaustraline [1139], (+)-allosedamine [1140], aziridinomitosenes [1141], 1-deoxynojirimycin [1142], toward ciguatoxins [1143–1145], L-cyclopentenyl nucleosides [672], martinellin pyrrolo[3,2-*c*]quinoline skeleton [673], toward palau'amine [1146], a V-ATPase inhibitor [1147], homopumiliotoxin 223G [1148], 6-epi-hyptolide [1149], (−)-salicylihalamide A and B [1150], segment of apratoxin A [1151], preclavulone-A methyl ester [1152], heliannuol D [1153], (5′-oxoheptene-1′*E*,3′*E*-dienyl)-5,6-dihydro-2*H*-pyran-2-one [1154], BC-ring system of taxanes [1155], pentalongin and psychorubin [1156], 2-deoxystreptamine [1157], C9–C19 subunits of dictyostatin-1 [1158], herbarumin III [1159], syributins [1160], taxuspine X [1161], hemibrevetoxin B [1162], cryptophycin-24 [431], campthotecin [1163], dihydroagarofuran ring system [906], (+)-lauthisan [1164], (+)-laurenyne [248], guaiane sequiterpenoids [1165], oximidine III [54], aigialomycin D [1166], and gem-fluorinated massoialactone [1167]:



(262)

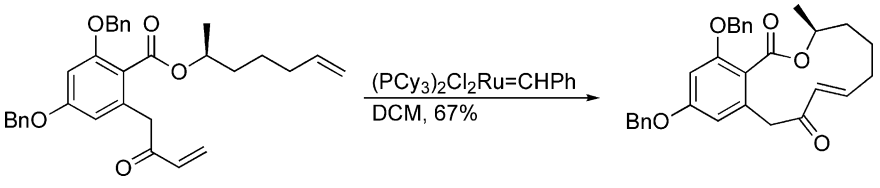


(263)

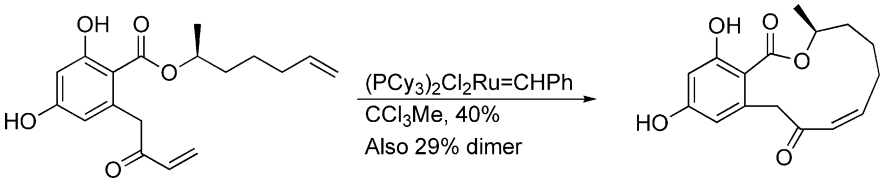


(264)

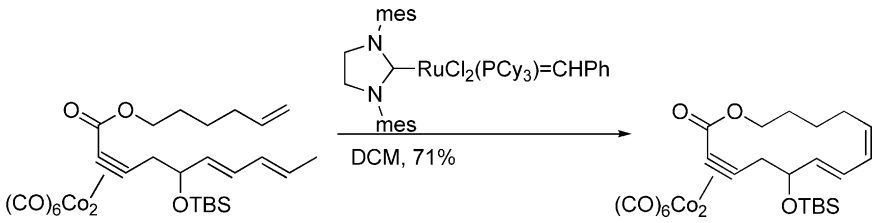
The double-bond geometry of the RCM was controlled by hydrogen-bonding in a synthesis of *trans*- and *cis*-resorcylide (Eqs. (265) and (266)) [497]. Complexation of an alkyne to cobalt facilitated the formation of macrolides (Eq. (267)) [1168]:



(265)

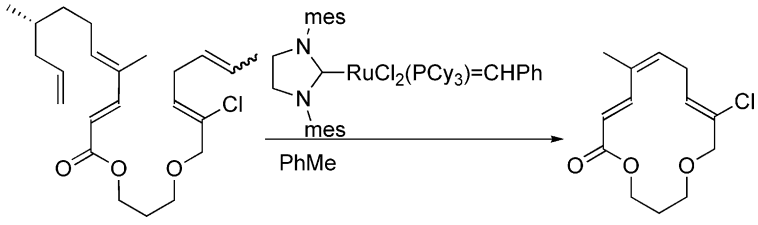


(266)

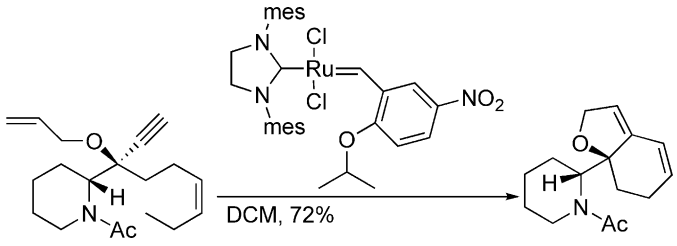


(267)

Dienynes were used in multiple relayed metathesis reactions (Eq. (268)) [1169]. Ruthenium catalyzed ene–yne–ene metatheses were used in approaches to angularly fused dioxatriquinanes [1170], (+)-viroallosecurinine [653], (−)-securinine (Eq. (269)) [1171], taxol-type ring systems [1172], and guanacastepene [40]:

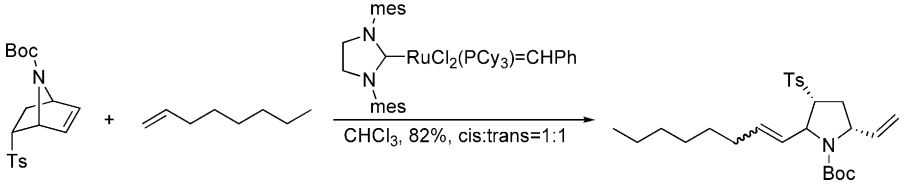


(268)

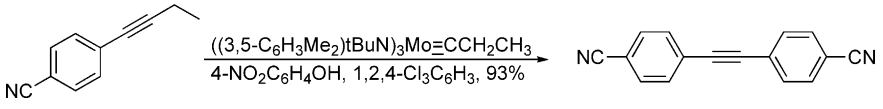


(269)

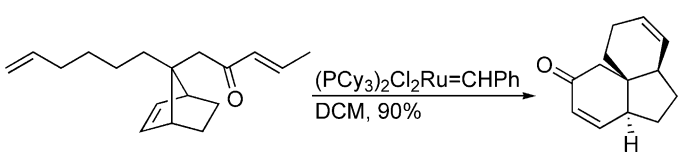
A ring-opening ring-closing metathesis of cycloalkenes having pendant alkynes was reported [1173]. Catalysts for ring-opening cross-metathesis were developed [1174]. A highly regioselective ring-opening alkene–alkene cross-metathesis was described (Eq. (270)) [1175]. Cyclohexa[*c*]indenes were prepared by a ring-opening ring-closing metathesis (Eq. (271)) [1176]. Related cascade reactions were described (Eq. (272)) [1177]:



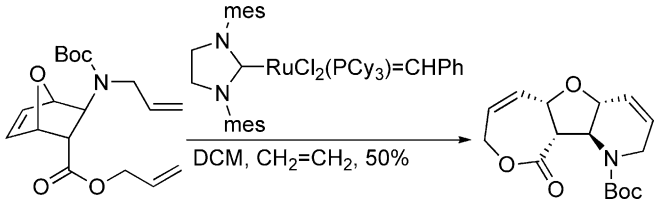
(270)



(273)



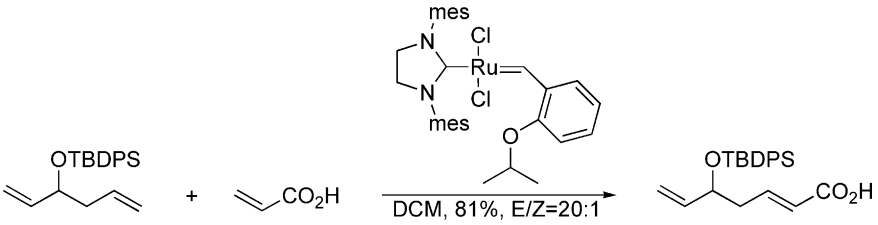
(271)



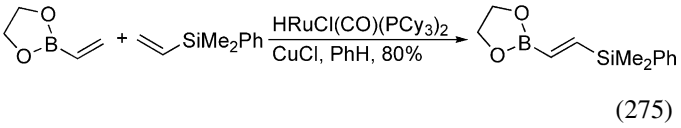
(272)

Cyclic allylboronates were prepared by a molybdenum-catalyzed asymmetric ring-closing metathesis [1178]. Molybdenum catalyzed the ring-closing metathesis to form cyclic siloxanes [268]. Chiral molybdenum complexes were used in asymmetric ring-closing metathesis [1179]. Molybdenum catalyzed intermolecular metathesis reactions of alkyl arylalkynes to give diaryl alkynes (Eq. (273)) [1180]. Tungsten-catalyzed alkyne–alkyne ring-closing metatheses were used in syntheses of macrocyclic alkynes [845] and cyclic β-turn mimetics [1181]:

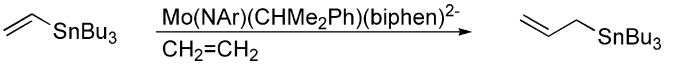
Alkene–alkene cross-metathesis of conjugated enynes [1182] and of allylic chlorides and bromides [1183] with terminal alkenes were described. Reactions of *n*-bromo-1-alkenes to give dibromo-alkenes were reported [1184]. Regioselective cross-metathesis was observed at the least hindered alkene of a series of dienes (Eq. (274)) [1185]. Allylic boronic esters were used in alkene–alkene cross-metathesis [1186]. Trisubstituted alkenylpinacolboronates were prepared by cross-metathesis of 1-substituted 1-alkenylboronates with alkenes [1187]. Ruthenium catalyzed the metathesis of alkenyl silanes with alkenylboronic acids esters (Eq. (275)) [1188]. Alkene–alkene cross-metathesis was used to couple sugars and fatty acids with lysin and cystein [1189]. *N*-Ethenylamides were used in cross-metathesis reactions with ethenyl triethoxysilane [1190]. Molybdenum catalyzed the homologation of alkenyltributyl stannane to allyltributylstannane in the presence of ethene (Eq. (276)) [1191]. An intermolecular ringopening-cross-metathesis of a cyclopropenone ketal was used in a synthesis of bistramide A (Eq. (277)) [1102]. An intermolecular molybdenum catalyzed alkyne–alkyne cross-metathesis was used in a synthesis of (−)-terestacin [788]:



(274)

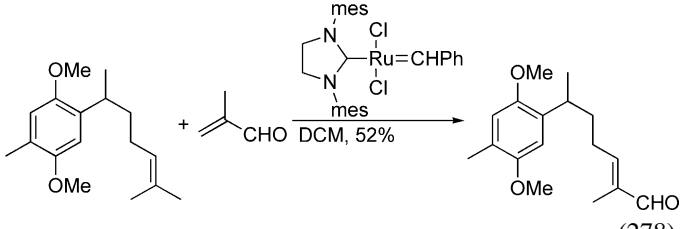


(275)

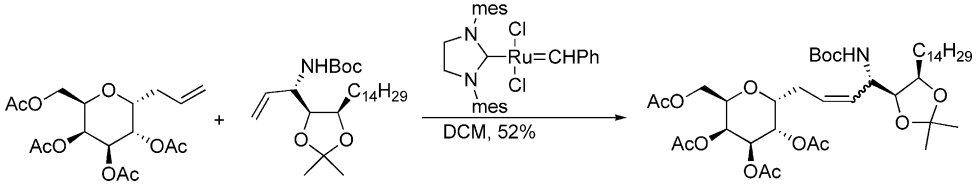


(276)

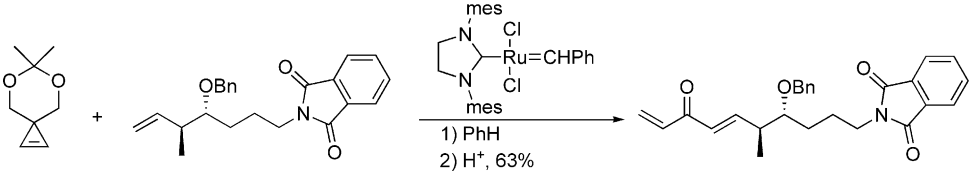
Alkene–alkene cross-metathesis was used in syntheses toward glandulone (Eq. (278)) [128], (−)-enterolactone [610], (−)-cryptopleurine [34], erythromycin derivatives [1192], optically active allylic hydroxy phosphonates [1193], fragment A of cryptophycins [1194], cochleamycin A [41], (−)-spongidepsin [1097], D-erythro-ceramide [1195], 2,6-dialkylpiperidines [1196], hybride inhibitors of type 1 17β-hydroxysteroid dehydrogenase [1197], lipid A derivative [1198], tetrafibricin fragments [1199], A,B,C-ring system of hexacyclinic acid [1200], C-glycolipids (Eq. (279)) [1201], (6*R*,7*S*)-7-amino-7,8-dihydro-α-bisabolene [1202], RK-397 [49], amphidinolide W (Eq. (280)) [1203], azaspiracid 1 [587], apoptolidinone [231], sphingomyelin [1204], D-erythro-sphingosine [1205], 8-alkenyl-substituted dihydrofuroangelicins [422], allylic fluorides [1206], martinellin pyrrolo[3,2-*c*]quinoline skeleton [674], (−)-lasubine II [1207], (+)-anthramycin [1208], (5′-oxoheptene-1′*E*,3′*E*-dienyl)-5,6-dihydro-2*H*-pyran-2-one [1154], C10–C18 segments of ambruticin [764], and 2-alkenyl-4-methylene tetrahydropyranes [643]:



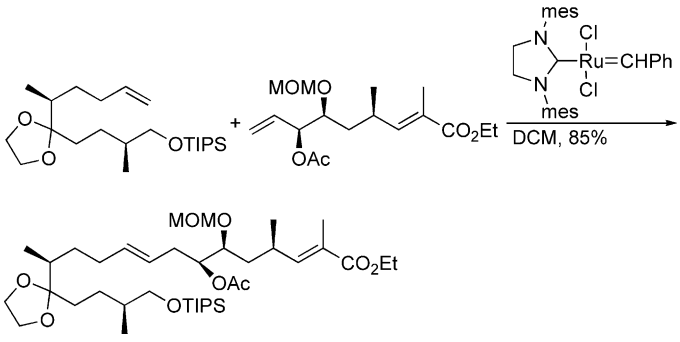
(278)



(279)

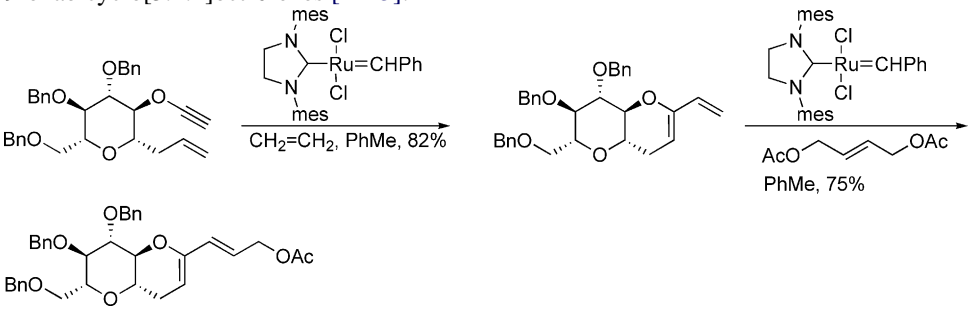


(277)

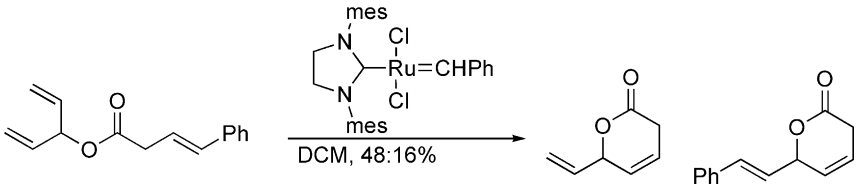


(280)

A domino ring-closing ene–yne and cross ene–ene metathesis was described (Eq. (281)) [1209]. Sequential ring-closing cross-metatheses were used in syntheses of (−)-muricatacin [1210] and butenolides and pyrones (Eq. (282)) [1211]. A relayed ring-opening metathesis, cross-metathesis, ring-closing metathesis was used in a synthesis of (+)-mycoepoxydiene and (−)-1893A [1212]. Functionalized tetrahydropyranes were prepared via ruthenium catalyzed ring-opening cross-metathesis of 9-oxabicyclo[3.2.1]oct-6-enes [1213]:

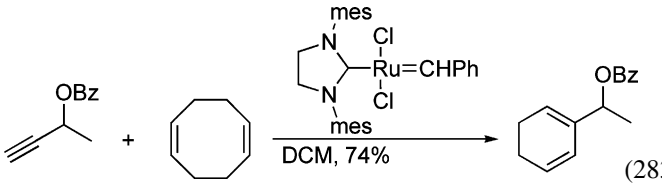


(281)

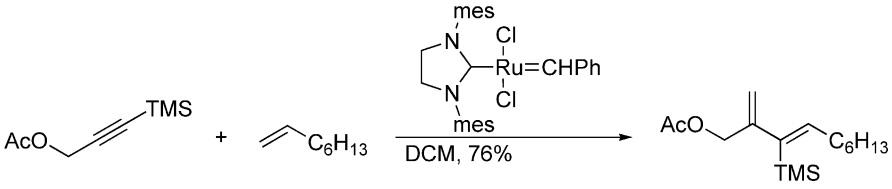


(282)

Ruthenium catalyzed ene–yne metathesis was described [1214]. An alkene–alkyne cross-coupling of 1,4-cyclooctadienes and an alkyne to give a 1,3-cyclohexadiene was developed (Eq. (283)) [1215]. A regio- and stereoselective enyne cross-metathesis was reported (Eq. (284)) [1216]. A sequential 1,8-enyne metathesis alkene cross-metathesis of sulfamide linked enynes was described [1217]:



(283)

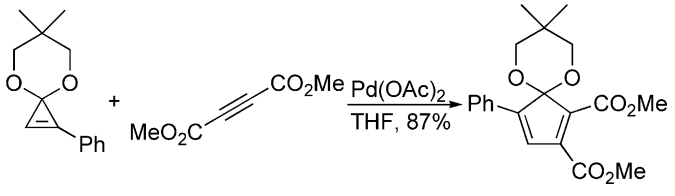


(284)

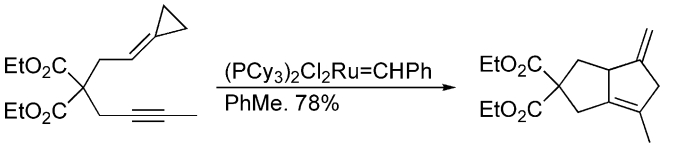
9. Cycloisomerizations

Reactions wherein the same number and type of elements in the starting material were found in a cyclic product are grouped in this section. Copper-catalyzed substrate-induced asymmetric intramolecular [2 + 2]alkene–alkene cycloadditions [1218]. Silver catalyzed [2 + 2]cycloadditions of silyloxyalkynes [1219]. Asymmetric ruthenium catalyzed [2 + 2]cycloisomerizations of bicyclic alkenes with chiral alkynyl acylsultams were reported [1220].

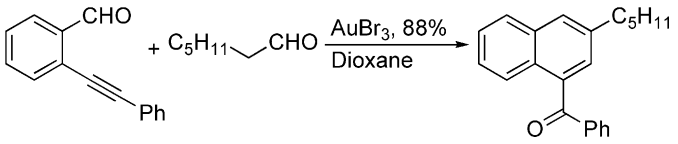
Palladium catalyzed a [3 + 2]cycloaddition of cyclopropenone ketals with alkynes to give cyclopentadienone ketals (Eq. (285)) [1221]. Ruthenium catalyzed an intramolecular [3 + 2]cycloaddition of alk-5-ynylidenecyclopropanes (Eq. (286)) [1222]. Zirconium-catalyzed enantioselective [3 + 2]cycloadditions of alkenes and hydrazones affording pyrazolidine, pyrazoline, and 1,3-diamine derivatives [1223]. Gold catalyzed intermolecular [4 + 2]cycloadditions of enynals with enols (Eq. (287)) [1224]. Rhodium catalyzed intramolecular [4 + 2]cycloisomerizations in an ionic micellar system [1225]:



(285)

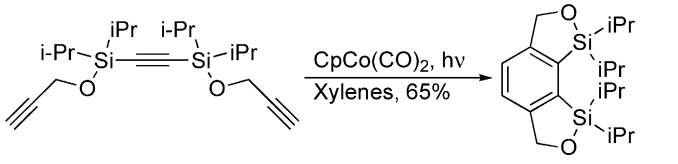


(286)

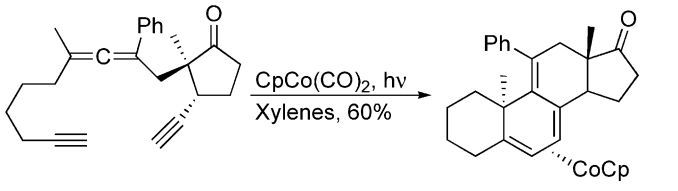


(287)

Cobalt mediated a chemo- and regioselective intramolecular [2 + 2 + 2]cycloaddition of three different alkynes linked with silyloxy bonds (Eq. (288)) [1226]. Cobalt-mediated cycloadditions of alkynylboronates with diynes [1227] and 1,*n*-diynes with cyanamides [1228] were described. A cobalt mediated [2 + 2 + 2]cyclization of 6,7-diene-1,13-diynes was use in a diastereoselective synthesis of 11-arylsteroid skeletons (Eq. (289)) [1229]. Cobalt catalyzed an asymmetric [2 + 2 + 2]cycloisomerization of diynes or two molecules of alkyne with nitriles to give pyridines [1230]:

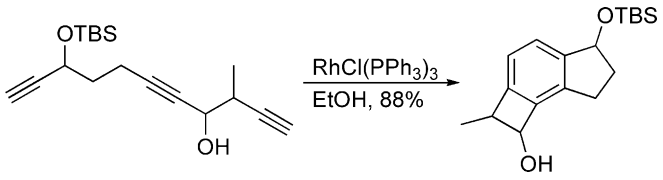


(288)

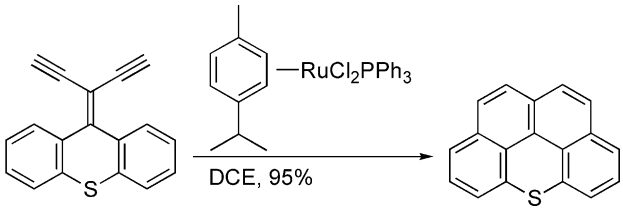


(289)

An intramolecular rhodium-catalyzed 1,6,10-triyne cycloisomerization was used in a synthesis of viridin (Eq. (290)) [1231]. Ruthenium catalyzed a double [2 + 2 + 2]annulation of an arene–ene–diyne to give two new aromatic rings (Eq. (291)) [1232]. Ruthenium catalyzed cycloisomerizations of two alkynes temporarily tethered via boron with alkynes [1233]. C-Arylglycosides were prepared by ruthenium-catalyzed diyne–yne cycloisomerization [1234]. Ruthenium catalyzed cycloisomerizations of amide- and ester-tethered 1,6-diynes with alkynes to give benzofused lactams and lactones [1235]. Iridium catalyzed enantio- and diastereoselective [2 + 2 + 2]cycloadditions to give axially chiral teraryls [1236]. Rhodium catalyzed enantioselective yne–diyne [2 + 2 + 2]cycloisomerization to give axially chiral phthalides [1237]:

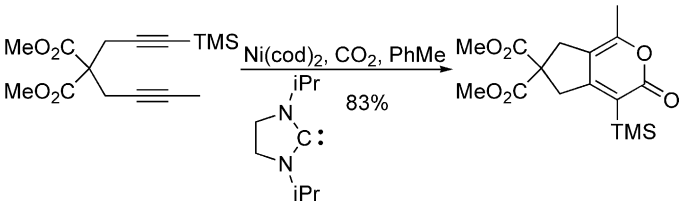


(290)



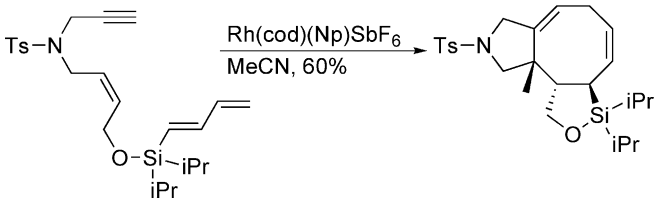
(291)

6-Alkynylpurines were used in nickel or cobalt catalyzed [2 + 2 + 2]benzannulations with 1,6-diynes [1238]. Nickel-catalyzed [2 + 2 + 2]cycloisomerizations of diynes with isocyanates to formed bicyclic pyrones [1239]. A nickel-catalyzed [2 + 2 + 2]cycloisomerization of 1,6-diynes with carbon dioxide to give bicyclic fused lactones was described (Eq. (292)) [1240]:

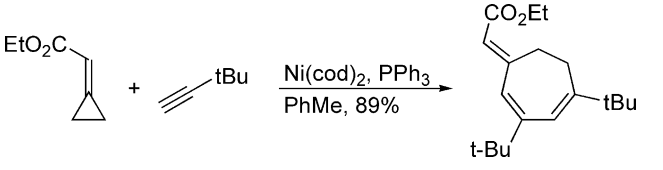


(292)

A rhodium-catalyzed [4 + 2 + 2]cycloisomerization of silicon tethered 1,3,8-trien-13-ynes was reported (Eq. (293)) [1241]. Nickel catalyzed an intermolecular [3 + 2 + 2]cycloaddition of ethyl cyclopropylideneacetate and alkynes (Eq. (294)) [1242]. Palladium-catalyzed cycloisomerizations of 2-en-4-ynals with dimethyl acetylenedicarboxylate (Eq. (295)) [1243]:



(293)



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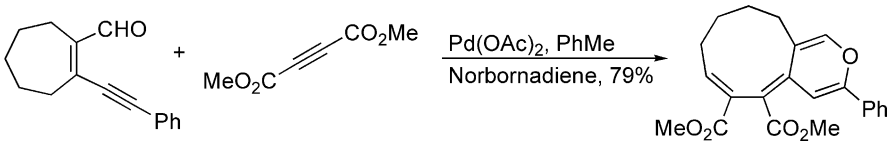
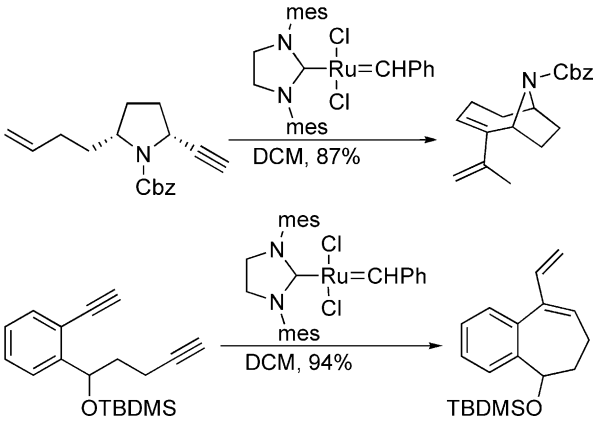


(295)

Ruthenium-catalyzed enyne ring-closing metathesis reactions were reported forming polyhydroxylated 1-ethenylhexenes [1244], toward the A ring of FR182877 [1245], ferruginine [858], tricyclic core of halichlorine [1246], dienynes [1247], (+)-anatoxin-a (Eq. (296)) [1248–1250], and (+)-anthramycin [1208], dysidiolide [1251]. Ruthenium catalyzed 1,6- 1,7-, and 1,8-enyne metathesis (Eq. (297)) [1252–1254] to give alkenylcycloalkenes. Reactions in the absence of solvent were shown to give a higher selectivity using silyloxy-tethered endienynes (Eq. (298)) [1255]. A ruthenium catalyzed 1,7-enyne metathesis was used in a synthesis of erogorgiaene (Eq. (299)) [47]. The endo/exo selectivity and stereoselectivity of intramolecular enyne cycloisomerizations was controlled using ethene:



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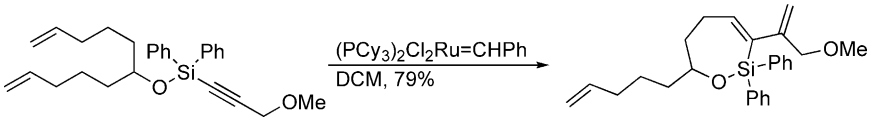
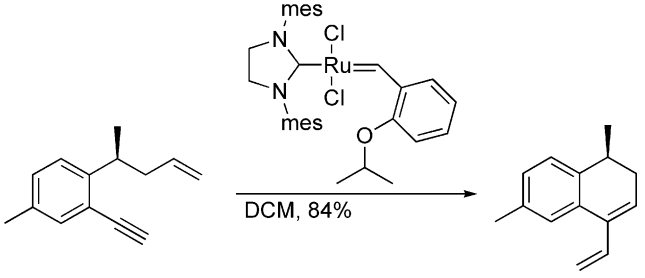
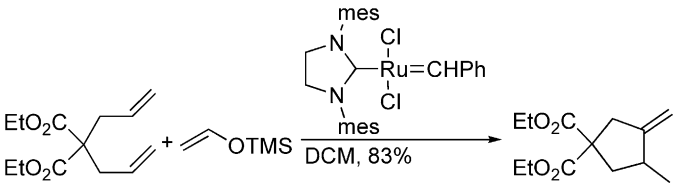


(298)

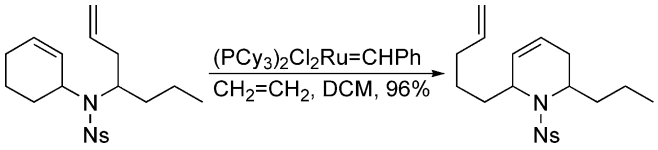


(299)

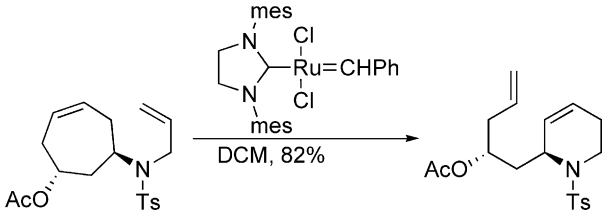
Ruthenium catalyzed an isomerization–cycloisomerization sequence in the presence of TMS-enol ether (Eq. (300)) [1256]. A ruthenium catalyzed 1,7-diene cycloisomerization in the presence of ethene was reported (Eq. (301)) [1257]. Ruthenium-catalyzed ring-rearrangements of 1,6- or 1,7-dienes were used in a syntheses of (−)-halosaline and (2*R*, 9a*R*)-(+)-2-hydroxyquinolizidine (Eq. (302)) [1258], and (−)-lasubine II [1207], and 251F (Eq. (303)) [859]:



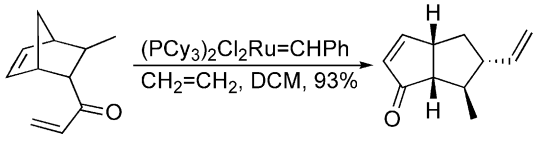
(300)



(301)

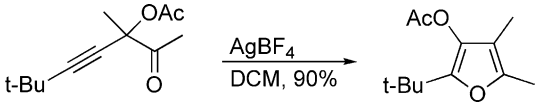


(302)

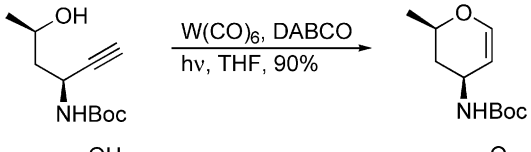


(303)

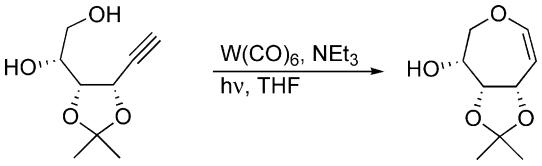
Silver catalyzed a 1,2-acylmigration–cycloisomerization of 4-acyloxy-, 4-tosyloxy-, or 4-phosphatyloxy-2-yne-1-ones to give furans (Eq. (304)) [1259]. A tungsten-catalyzed cycloisomerization of a 1-yne-5-ol was used in a synthesis of D-desosamine (Eq. (305)) [1260]. Tungsten catalyzed an endo-selective cycloisomerization of 1-yne-5,6-diols to give seven-membered rings (Eq. (306)) [1261]. Ruthenium catalyzed cycloisomerizations of 1-yne-5-ols to give dihydropyrans [1262]. Palladium catalyzed cycloisomerizations of in situ formed 2,3,4-triene-1-ols to give furans [1263]. Gold and platinum catalyzed cycloisomerizations of hydroxy-enynes (Eq. (307)) [1264,1265]. Evidence for a carbocation intermediate was reported for palladium- and platinum-catalyzed cycloisomerizations of dien- and trienols [1266]:



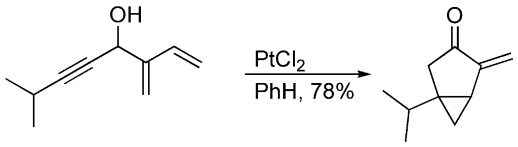
(304)



(305)

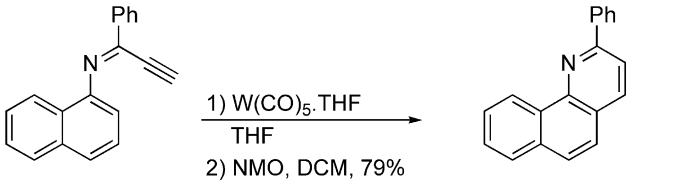


(306)

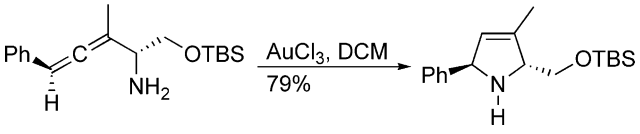


(307)

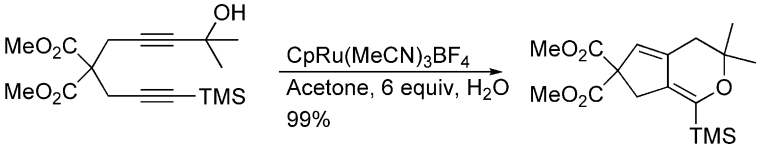
A tungsten-catalyzed cycloisomerization of alkynylimines to give 2-substituted quinolines was reported (Eq. (308)) [1267]. Gold catalyzed the cycloisomerization of 4-amino-1,2-dienes to give 3-pyrrolines (Eq. (309)) [1268] and of propargylic amides to give oxazoles [1269]. Platinum catalyzed cycloisomerizations of 1,6-, 1,7-, and 1,8-enyneamides to give nitrogen containing compounds [1270]. The regioselectivity of ruthenium-catalyzed cycloisomerizations was controlled by the choice of solvent system (Eqs. (310) and (311)) [1271]:



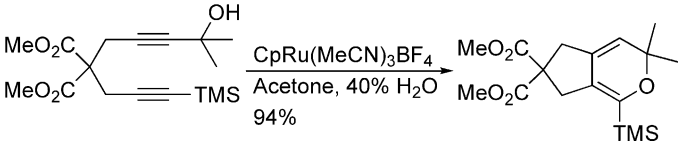
(308)



(309)

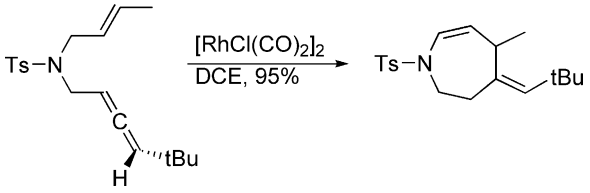


(310)

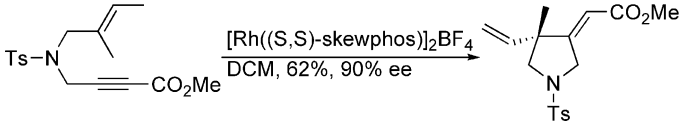


(311)

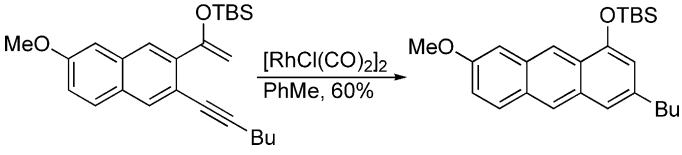
A number of transition metals catalyzed cycloisomerizations of 1,6- and 1,7-enynes to give alkenyl-substituted five- and six-membered rings [1272]. Rhodium catalyzed cycloisomerizations of oxygen and nitrogen containing 1,2-dien-7-ynes to give azepines and oxepines (Eq. (312)) [1273]. Asymmetric rhodium-catalyzed cycloisomerizations of 1,6-enynes were also reported [1274,1275]. Enantioselective cycloisomerizations was observed using 1,6-enynes and a rhodium catalyst (Eq. (313)) [1276]. Rhodium-catalyzed cycloisomerization of 2-(1-silyloxy-1-ethenyl)-1-alkynylarenes to give benzannulated products (Eq. (314)) [1277]. Both 1,6- and 1,7-enyne cyclizations of allylic silyl ether catalyzed by ruthenium to give either five- or six-membered rings having a silyl enol ether were described [1278]:



(312)

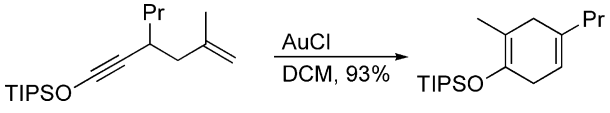


(313)

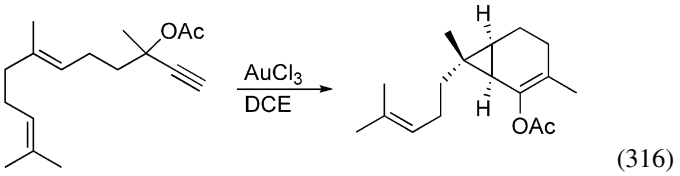


(314)

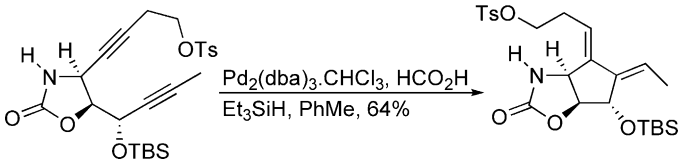
Gold catalyzed the cycloisomerization of 1-silyloxy-5-en-1-ynes to give cyclohexadienes (Eq. (315)) [1279] and of 1,6-enynes to give 1-alkenyl-1-cyclopentenes [1280]. A gold-catalyzed cycloisomerization was used in a synthesis of carene terpenoids (Eq. (316)) [1281]. Gold-, platinum-, gallium-, and indium-catalyzed cycloisomerizations of 2-alkynyl-substituted biaryls (Eq. (317)) [1282]. Gold catalyzed the cycloisomerization of 1,5-enynes to give bicyclo[3.1.0]hexenes (Eq. (318)) [1283]. Iridium catalyzed cycloisomerizations of 1,6-enynes to give alkenyl–alkylidenecyclopentanes [1284]:



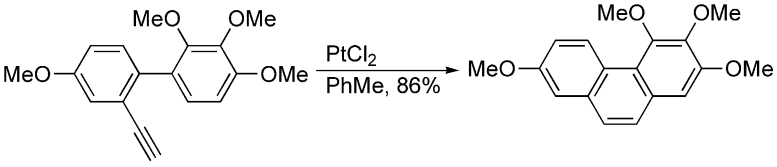
(315)



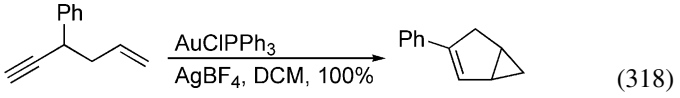
(316)



(321)

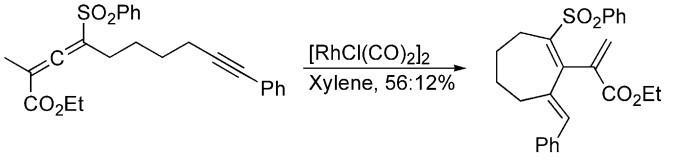


(317)

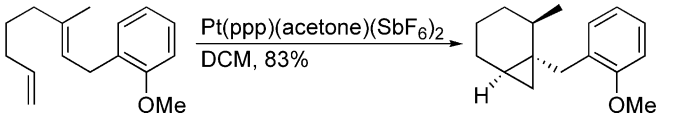


(318)

Rhodium catalyzed cycloisomerizations of 1,2,7-trienes to give methylene-alkenyl-substituted five-membered rings. Seven-membered rings were obtained under a carbon monoxide atmosphere [983]. Rhodium catalyzed cycloisomerizations of 1,2-diene-8-ynes to give cycloheptene trienes and/or bicyclo[5.2.0] derivatives (Eq. (319)) [984]. Rhodium catalyzed a cycloisomerization of 1-chloro-3-ene-7-ynes with a halogen shift (Eq. (320)) [1285]:

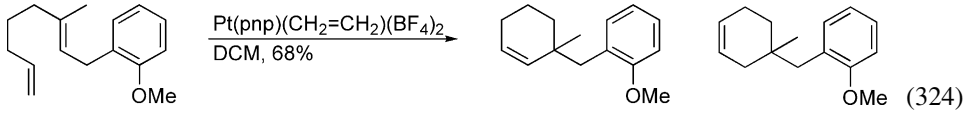


(322)

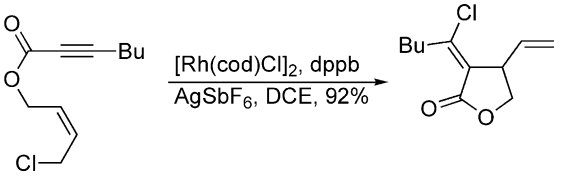


(323)

(319)

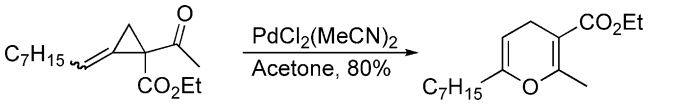


(324)



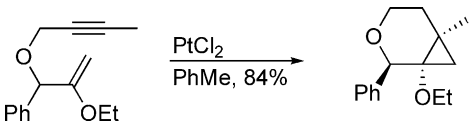
(320)

Palladium was used to catalyze a 1,6-diyne cycloisomerization in a synthesis of (+)-strepazoline (Eq. (321)) [1286]. Palladium-catalyzed cycloisomerizations of 2-en-7-yne-1-ols to give five-membered rings having an exocyclic alkene and an ethanal substituent [1287]. The aldehyde can undergo a subsequent reductive amination with secondary amines and formic acid [1288]. Palladium-catalyzed cycloisomerizations of electron-deficient 1,6-enynes and related enediynes to give six-membered rings (Eq. (322)) [1289]. Depending on the ligand, two different cycloisomerizations of 1,6-dienes were obtained using platinum catalysts (Eqs. (323) and (324)) [1290]. Palladium-catalyzed the cycloisomerization of alkylidenecyclopropyl ketones (Eq. (325)) [1291]:

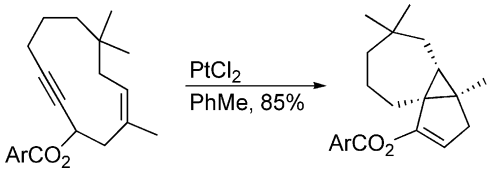


(325)

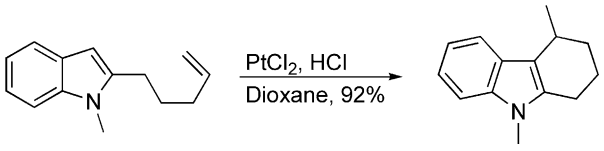
A platinum-catalyzed cycloisomerization of enol ether tethered alkynes was described (Eq. (326)) [1292]. Platinum catalyzed a cycloisomerization of cyclic 1,5-enynes to give tricyclic compounds (Eq. (327)) [1293]. Platinum and silver catalyzed 1,6-enyne cycloisomerizations of yne-tethered cyclic enamides [1294]. Platinum catalyzed 1,2-dien-7-yne and 1,6-diyne cycloisomerizations were reported [1295]. Platinum catalyzed cycloisomerizations of 2-(4-alken-1-yl)-substituted indoles to give substituted carbazoles (Eq. (328)) [1296]. Platinum catalyzed a cycloisomerization of 3-hydroxy-4-amido-1,5-diynes to give a substituted cyclopentenone (Eq. (329)) [1297]. Palladium- and platinum-catalyzed cycloisomerizations of alkyne tethered acetals and thioacetals (Eq. (330)) [1298] were described:



(326)



(327)



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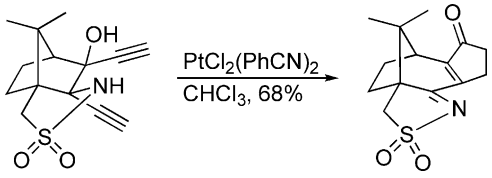
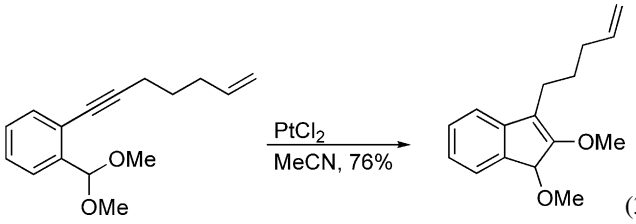
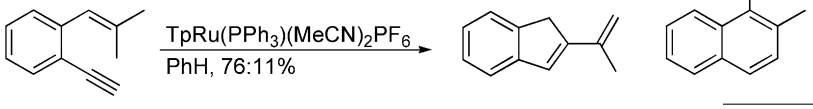


(329)



(330)

Ruthenium catalyzed cycloisomerization followed by skeletal rearrangements of 2-(ethynyl)phenylalkenes (Eq. (331)) [1299]. A copper-mediated cycloisomerization of a 4-(1-alkynyl)-substituted pyrimidine was used in a synthesis of tetraponerin T6 (Eq. (332)) [322]. Related copper-catalyzed reactions were used to prepare analogs of cytidine and 2′-deoxycytidine (Eq. (333)) [303]:



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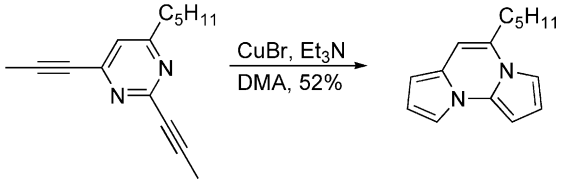

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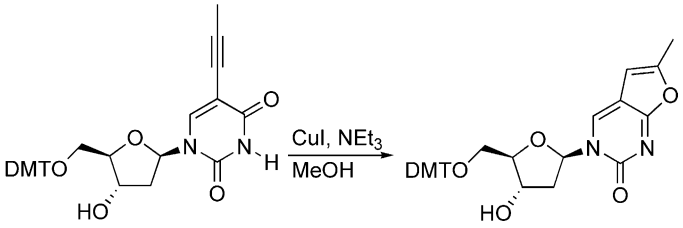
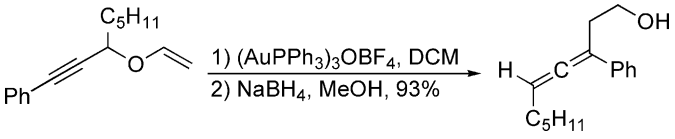


(333)

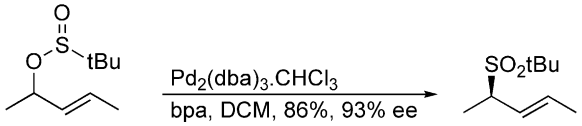
10. Miscellaneous isomerizations

Palladium catalyzed an enantioselective [3,3]sigmatropic rearrangement of allylic trichloroacetimidates [1300]. A related rearrangement of an allylic acetate with chirality transfer was used in a synthesis of (−)-methylenolactocin [1301]. Palladium catalyzed a [3,3]sigmatropic rearrangement of (allyloxy)iminodiazaphospholidines [1302]. Palladium catalyzed an asymmetric Claisen rearrangement [1303]. Gold-catalyzed propargyl Claisen rearrangements (Eq. (334)) [1304]:



(334)

Palladium catalyzed an enantioselective rearrangement of allylic sulfinates to allylic sulfones (Eqs. (335) and (336)) [1305]. Palladium catalyzed a ring-expansion of 1-hydroxy-1-propenoylindanes to 2-hydroxy-2-thenyl-tetralones [1306]. Palladium catalyzed the racemization of optically active allenes [1307] and ruthenium catalyzed racemization of optically active allylic alcohols [1308]:



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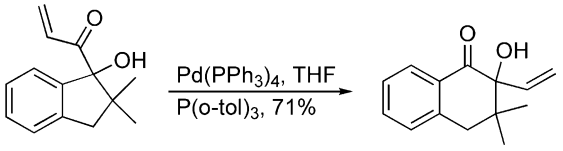
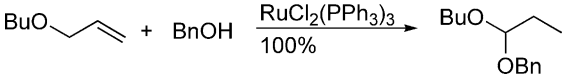


(336)

Titanium mediated a rearrangement of oxiranes to allylic alcohols [1309]. Rhodium and ruthenium catalyzed isomerization of allylic ethers to alkenyl ether [1310]. In the presence of an alcohol, acetals were formed (Eq. (337)). Ruthenium was used as the catalyst to isomerize an allylic ether to an alkenyl ethers in a synthesis toward ciguatoxin (Eq. (338)) [639]. Rhodium was used in a similar reaction toward α-glucofuranosides and β-rhamnopyranosides [1311]. A related palladium-catalyzed 1,6-diene to 1,5-diene isomerization-Claisen rearrangement was reported [1312]. Gold and iridium can also be used as catalysts. A ruthenium catalyst was used to isomerize oxiranes to aldehydes [1313]. Diallyl- and homo-allylethers do not undergo RCM in the presence of ethyl ethenyl ether but instead undergo isomerization followed by a Claisen rearrangement (Eq. (339)) [1314]:



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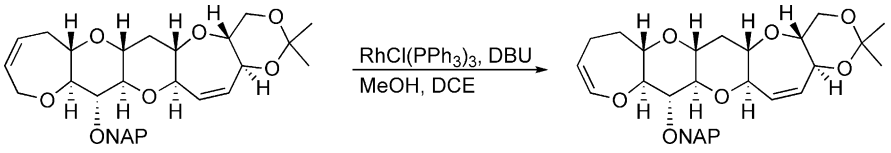

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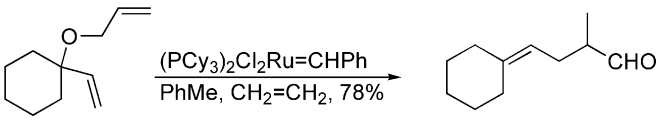
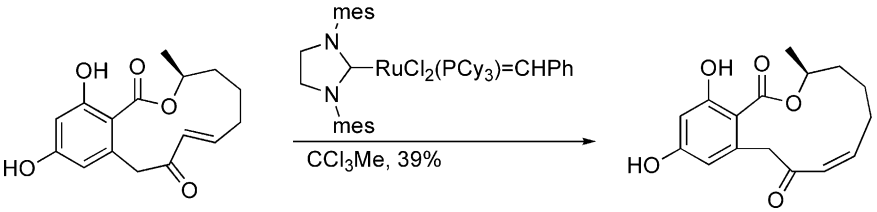
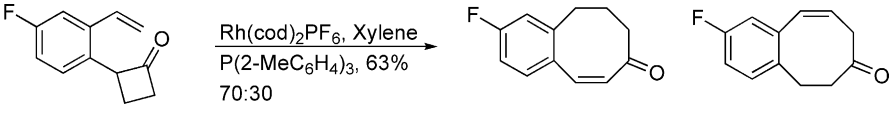


(339)

Ruthenium catalyzed the isomerization of trans to cis resorcylide (Eq. (340)) [497]. Ruthenium catalyzed the isomerization of allylic alcohols to ketones or aldehydes in organic and aqueous media [1315]. Rhodium catalyzed the isomerization of 2-(2-alkenylphenyl)cyclobutanones to give benzene-fused eight-membered rings (Eq. (341)) [1316]. Rhodium catalyzed the isomerization of 2-alkynyl-substituted cyclic ethers to dienones (Eq. (342)) [1317]. A rhodium-catalyzed alkene isomerization to the more substituted alkene was used in a synthesis of cacospongionolides B and E [1087]. Palladium catalyzed the isomerization of terminal to internal alkenes [1201]. Palladium catalyzed the isomerization of 2-alkenylidenefurans to 1,3-dienes (Eq. (343)) [1318]:



(340)



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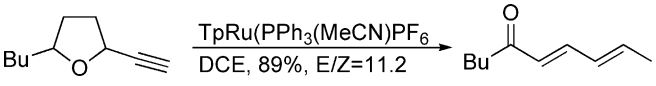

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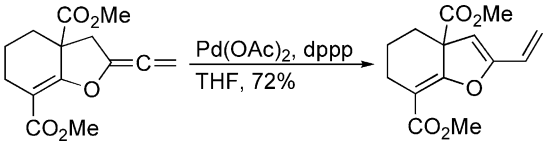
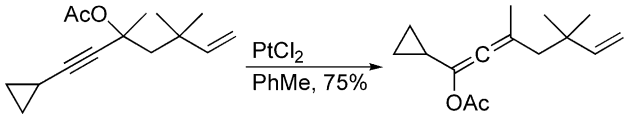


(343)

Iridium, rhodium, and ruthenium catalyzed the isomerization of allylic amines or amides to enamines or enamides [1319,1320]. Platinum catalyzed intramolecular [3,3]rearrangements of enyne acetates (Eq. (344)) [1321]. Alkene isomerization to form the more substituted double bond, catalyzed by rhodium, was used in a total synthesis of irofulvene (Eq. (345)) [727]. Nickel catalyzed an isomerization of alkenylcyclopropanes to give cyclopentenes (Eq. (346)) [1322]. Nickel catalyzed a coupling of enones, alkynes, and alkenes (Eq. (347)) [1323]. Copper-catalyzed enantioselective carbonyl-ene reactions [1324]:



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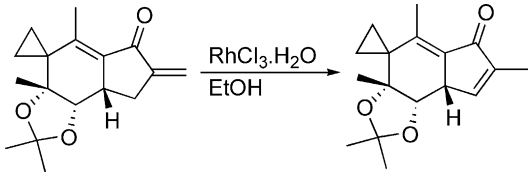

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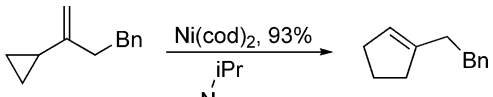

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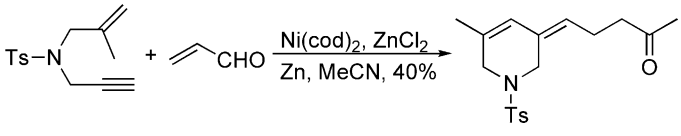
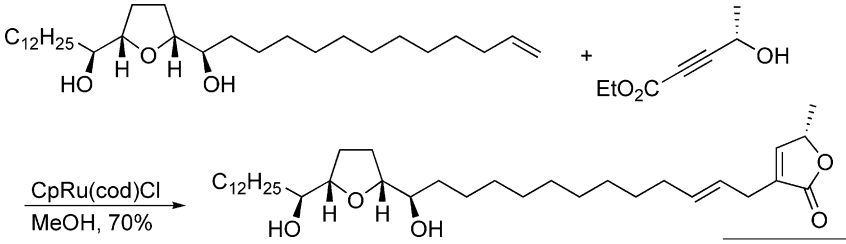


(347)

Intermolecular alkene–alkyne couplings (Alder-ene reactions) to give 1,4-dienes, catalyzed by ruthenium were used

in synthesis of *cis*-solamin isomers (Eq. (348)) [1325], (−)-mycalamide A [585], amphindinolide P [1326], membranacin [1327], and 2-alkenyl-4-methylene tetrahydropyranes [643]. Both inter- and intramolecular alkene–alkyne couplings were used to prepare (+)-amphidinolide A (Eq. (349)) [1328]. A silyloxy tethered 1,*n*-enyne cycloisomerization using ruthenium was reported to give siloxacycles [1329]. The presence or absence of *O*-protecting groups and the nature of the protecting group significantly changed the yield of Alder-ene reactions of hydroxy-group containing alkenes and alkynes [1330]:



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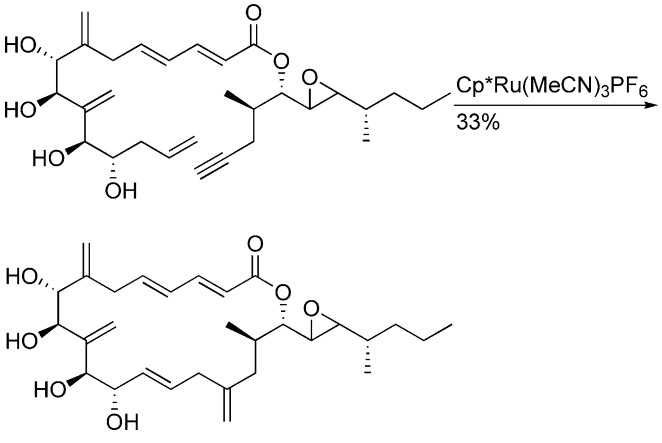
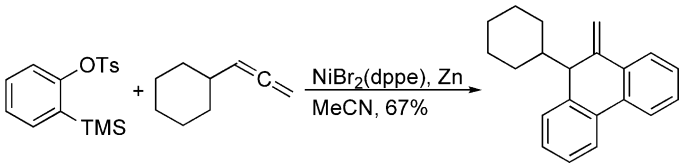


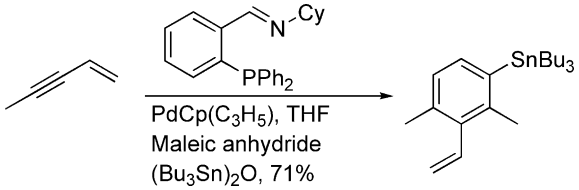
(349)

synthesis of taiwanins C and E [287]. Nickel catalyzed a cyclization of arynes and allenes (Eq. (350)) [1332]:



(350)

Palladium catalyzed the cyclobenzannulations of conjugated enynes in the presence of hexabutyldistannoxane to give tin-substituted aromatic rings. This reaction was employed in a synthesis of alcyopterosin N (Eq. (351)) [1333]. Palladium catalyzed cyclizations of 2-diyn-1-ol carbonates with organoboronic acids to give polycyclic compounds (Eq. (352)) [1334]. Palladium catalyzed a reductive cyclization of 1,2-diene-6-ynes to give 1,2-dialkylidenecyclopentanes (Eq. (353)) [1335]. A titanium-mediated reductive cyclization of 4-oxa-5-sila-1,6-enynes was used in a synthesis of the C9–C19 subunit of dictyostatin-1 (Eq. (354)) [1158]:



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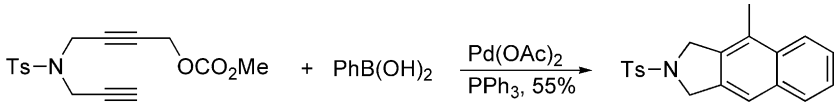
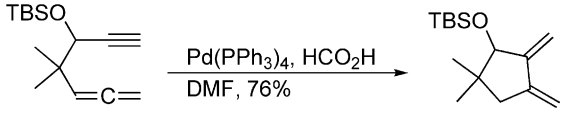


(352)



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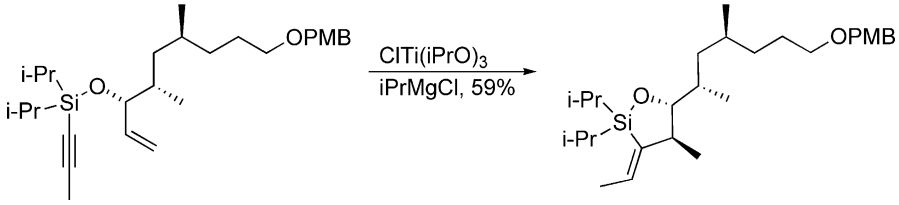


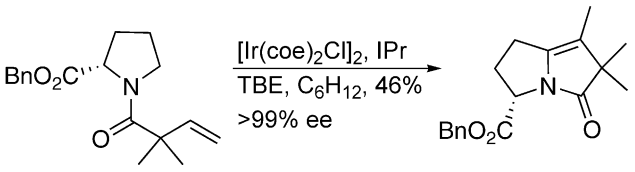
(354)

11. Miscellaneous carbocyclizations

Palladium catalyzed a [2 + 2 + 2]cycloaddition of intermediately formed benzyne with bicyclic alkenes to give polyaromatic compounds [1331]. A related [2 + 2 + 2]cycloisomerization catalyzed by palladium of arynes and tethered diynes was used in

Iridium catalyzed an interesting cyclization of alkene-tethered amides (Eqs. (355) and (356)) [1336]. Ruthenium catalyzed a cyclization of oxiranes tethered to alkynes (Eq. (357)) [1337]. Platinum catalyzed a cyclization of 2-alkynylbenzoates or benzothioates with alkenyl ethers to give substituted naphthalene derivatives (Eq. (358)) [1338]. Gold

catalyzed the cyclization of 2-(1-alkynyl)-2-alken-1-ones to give furans (Eq. (359)) [1339]:



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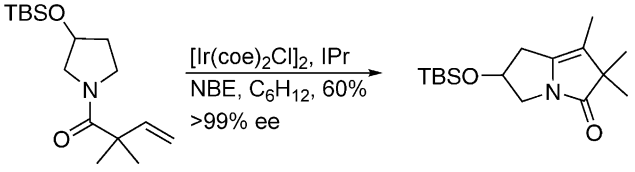

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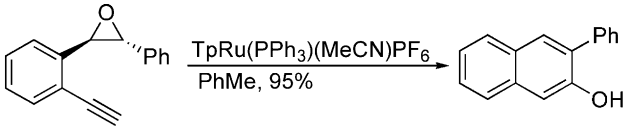

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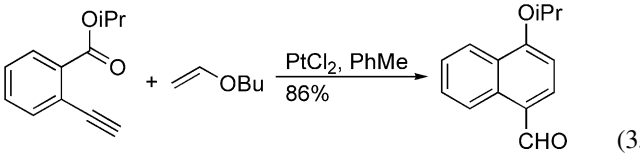

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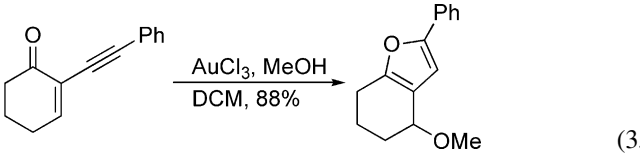
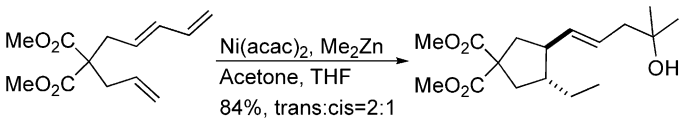


(359)

Nickel catalyzed reactions of 1,3,8-nonatrienes or 1,3,9-decatrienes with dimethylzinc and acetone to give functionalized five- or six-membered rings (Eq. (360)) [1340]. Rhodium catalyzed reductive cyclizations of 1,6-diynes and 1,6-enynes in the presence of hydrogen gas (Eq. (361)) [1341]. Ruthenium catalyzed cyclizations of 8-acetoxy-6-enals and -enones and of 7-acetoxy-5-enals and -enones (Eq. (362)) [1342]. A zirconium mediated reductive cyclization of 1,7-dienes was used to diastereoselectively prepare 2,5-disubstituted decahydroquinolines (Eq. (363)) [1257]:



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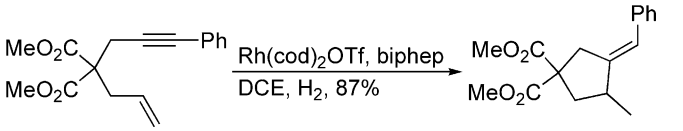
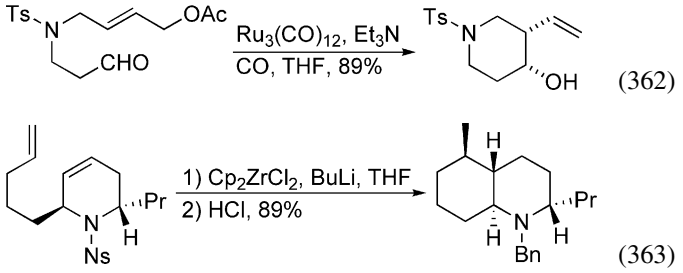
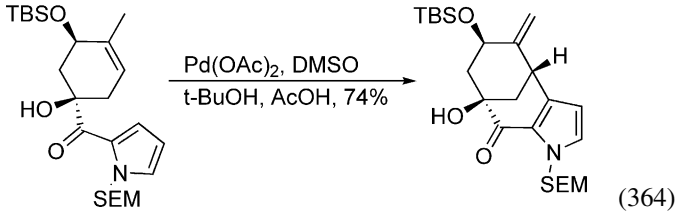


(361)



(362) (363)

A palladium-catalyzed intramolecular dehydrocarbocyclization was used to prepare (+)-dragmacidin F (Eq. (364)) [226]. Palladium catalyzed ring-cleavage cyclizations of cyclobutanone *O*-benzyloximes (Eqs. (365)–(367)) [1343]. Platinum and ruthenium catalyzed the formation of polycyclic cyclopropanes from alkene tethered propargylic alcohols (Eq. (368)) [1344]. A palladium-catalyzed [3 + 2]cycloaddition of trimethylenemethane to an α,β-unsaturated lactone was used in a synthesis of (+)-brefeldin [651]. Palladium-catalyzed cyclizations via alkyl to aryl migration of palladium (Eq. (369)) [1345]. Palladium catalyzed the cyclization of bis-allyl alcohol or ether with aldehydes to give five-membered rings (Eq. (370)) [1346]:



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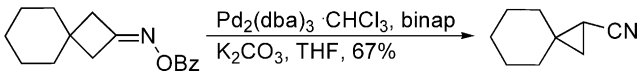

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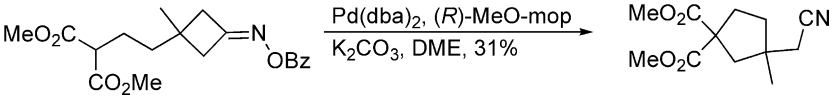
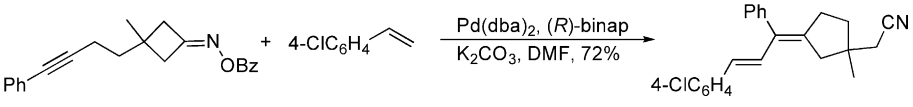


(366)



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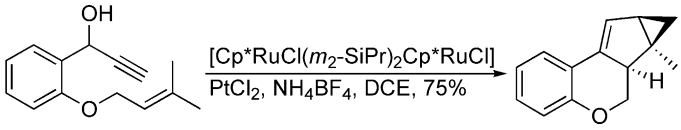

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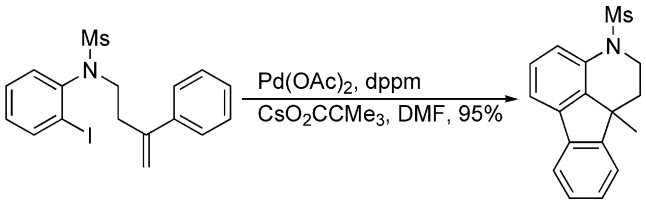

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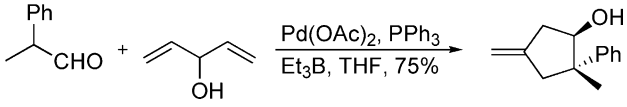
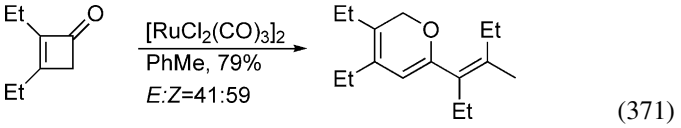


(370)

Rhodium and ruthenium catalyzed the formation of 2-pyranones, cyclopentenes, and cyclohexenones from cyclobutenones (Eqs. (371) and (372)) [1347]. Platinum catalyzed asymmetric alkoxy- and hydroxy-carbocyclizations of 1,6-enynes to give alkoxy- or hydroxymethyl substituted cyclopentanes having an exocyclic double bond [1348]:



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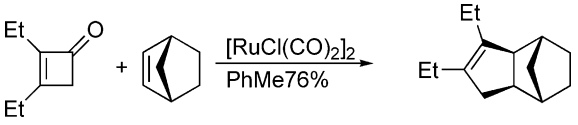


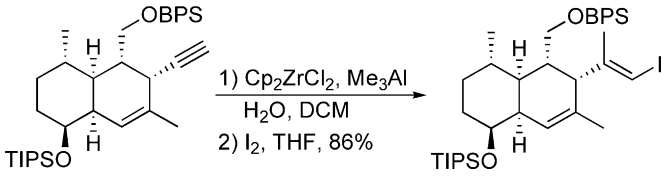
(372)

12. Formation of carbon–halogen bonds

E-Alkenyl iodides were prepared by the chromium-mediated Takai alkenylation of aldehydes using iodoform in the synthesis of tartrolone B [30], cochleamycin A [41], (−)-apicularene [563], (−)-gigantecin [327], zincophorin [141], (−)-dictyostatin [83], gambierol analogs [59], and epothilone [1116]. In a related fashion, chloroform was used in a synthesis of (−)-(*E*)-15,16-dihydrominquartynoic acid [321]. Tributyltindiiodomethane was used in a related reaction introducing an alkenyl stannane in a synthesis of (−)-callystatin A [131].

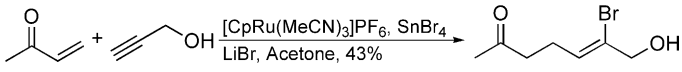
Hydrozirconation of a terminal alkynes followed by addition of iodine to produce alkenyl iodides were used in synthetic applications toward 6,7-dehydrostipiamide [133], octalactin B [868], RK-397 [49], and octalactin B [1349]. Regioselective hydrozirconation–iodinations of internal alkynes were used in syntheses of (−)-callystatin A [131], amphidinolide X [186], scyphostatin side chain [134,135], and (+)-scyphostatin [136].

Methylalumination of a terminal alkyne using trimethylaluminum and $ZrCp_2Cl_2$, followed by treatment with iodine to give a trisubstituted alkenyl iodide was used in synthesis of (+)-phomopsidin (Eq. (373)) [127] and the C1–C11 fragment of bafilomycin A_1 [1350]:



(373)

A three-component ruthenium catalyzed reaction of 1-propyn-3-ol, 3-buten-2-one, and tin tetrabromide furnished functionalized bromo-hydroxy-ketone and this reaction was used in a synthesis of the putative structures of homopumiloto-xins 235C and 233F (Eq. (374)) [439]. Rhodium catalyzed addition of chlorformates to 1,2-dienes (Eq. (375)) [1351]:



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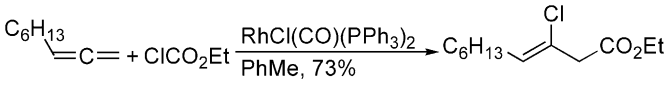
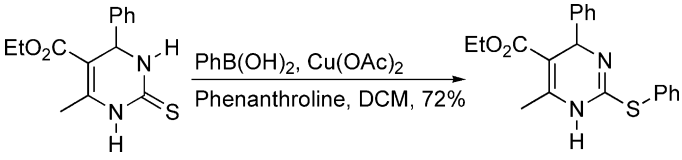


(375)

13. Formation of carbon–nitrogen and –oxygen bonds from boron, bismuth, and tin reagents

Copper-catalyzed *N*-arylations and *O*-arylations of amines and alcohols using triarylbismuth reagents [1352–1355]. An *N*-arylation using triarylbismuth reagents was used to prepare duocarmycin and CC-1065 analogs [542]. Copper catalyzed the formation of diaryl amines from aromatic nitroso compounds and aryl boronic acids [1356]. Copper catalyzed the coupling of aromatic boronic acids with imidazole, amines, amides, imides, and sulfonamides [1357,1358] and of aminopurines and aminopyrimidines [1359]. Copper catalyzed the coupling of dihydropyrimidine-2-thiones with aromatic boronic acids (Eq. (376)) [175] and of sulfinic acid salts with aromatic boronic acids to give aryl sulfones [1360]:

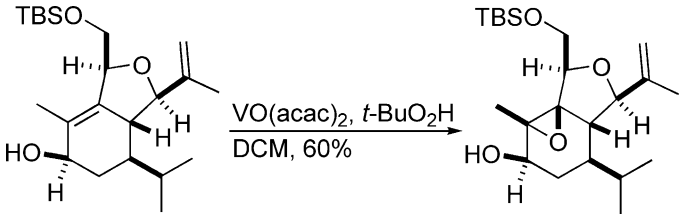


(376)

14. Oxidations

Vanadyl acetylacetonate-catalyzed epoxidations of allylic and in some cases homoallylic alcohols using peroxides con-

tinued to be an invaluable tool in organic total synthesis. Synthetic targets include, massileunicellins (Eq. (377)) [1361] and polyhydroxylated pyrrolidines [1362]. Vanadyl acetylacetonate-catalyzed epoxidation-ring-expansions of furyl alcohols to give pyranones were used in synthesis of (+)-mycoepoxydiene [1212], ocatlactin A [868], and enantiopure oxabicyclo[5.4.0]undecanes [1363]:



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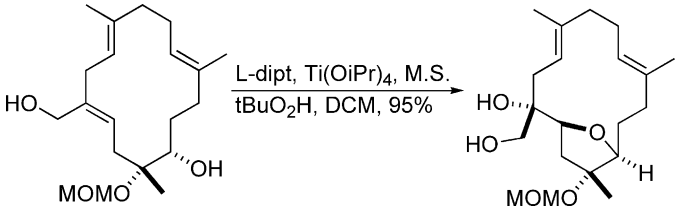
Sharpless enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continued to be extensively used in organic synthesis. For example, this reaction was used in synthetic approaches to laulimalide [856], (−)-SNF4435 C and (+)-SNF4435 D [48], brevetoxin B [1364], zincophorin [141], (+)-pinnatoxinA [898], (−)-nitidon [344], C1–C12 fragment of FD-891 [1365], maduropeptin chromophore [340], methyl isosartortuoate (Eq. (378)) [1366], aminocyclitol moiety of (+)-trehazolin [1367], and EF-ring fragment of ciguatoxin [1145]. A related zirconium-catalyzed enantioselective epoxidation of a homoallylic alcohol using cumene hydroperoxide was used in a synthesis of a tachykinin receptor agonist (Eq. (379)) [1368]:



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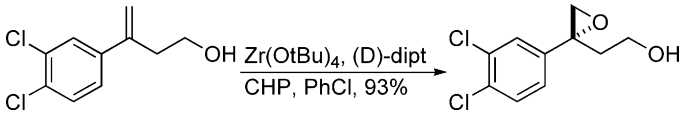
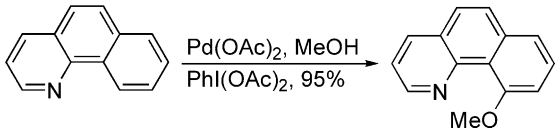


(379)

A number of synthetic applications of the palladium-catalyzed formation of α,β-unsaturated carbonyl compounds from silylenol ethers (Saegusa oxidation) were published. For example, applications toward (−)-cephalotaxine [1369], LY354740 analogs [1370], azaspiracid 1 [625], pentenocin B [1371], (−)-strychnine [67], and the ABCDE ring fragment of ciguatoxins [245] were described.

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. For example, tartrolone B [30], pentenocin B [1372], ferruginine [858], hamigerans [1373], (+)-cassioside and (+)-cassiol [862], brevetoxin B [1364], toward zaragozic acid [1374], hirsutic acid and complicatic acid [1375], α-quarternary pipecolic acid derivatives [1376], a diterpenic lactone [1377], cyclopentanone containing amino acids [1378], and chiloscyphone and isochiloscyphone [1379] were prepared using a Wacker type reaction in one of the steps. A modified Wacker oxidation using mercury acetate was used in a synthesis of ophirin B [1103].

Palladium-catalyzed regioselective allylic oxidation of terminal alkenes forming allylic acetates [1380]. A chelation controlled palladium-catalyzed oxidation of aromatic substrates to give benzylic or aromatic acetates, alkyl ethers, or halides was developed (Eq. (380)) [1381]. Palladium catalyzed related chelation controlled acetoxylations of sp^3–carbon–hydrogen bonds (Eq. (381)) [1382] and this type of reaction was used in a synthesis of rostratone [1383]. Palladium catalyzed the *N*-allylation of aryl amines and benzylic alkylation of 2-arylethanals using 1-substituted-1-propynes (Eq. (382)) [1384]. Rhodium catalyzed the direct formation of alkylboronic acid esters from alkyl-chains containing heteroatoms (Eq. (383)) [1385]. Ruthenium also catalyzed benzylic silylation of 2-(2-methyl)arylpyridines [1386]:



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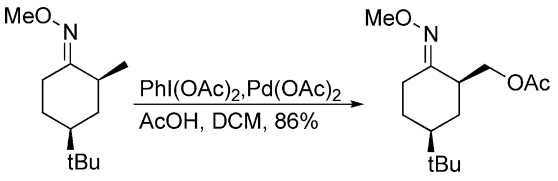

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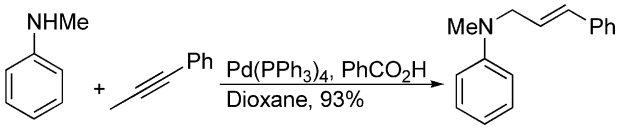

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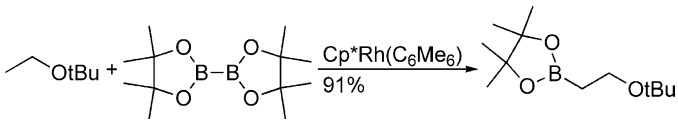


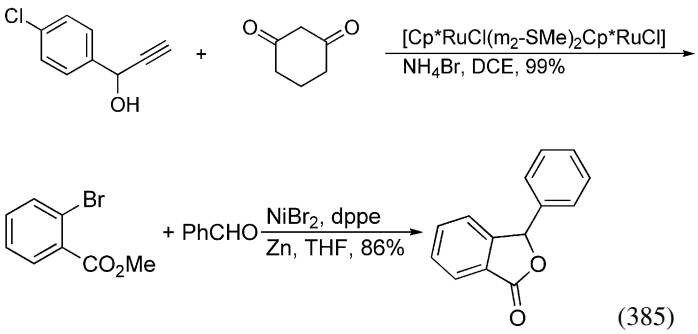
(383)

Iridium catalyzed dehydrogenations of alkanes to form alkenes [1387]. Zirconium mediated the formation of nitriles from amides [1388].

15. Formation of heterocycles

Palladium catalyzed the formation of coumarins from hydroxybenzenes and propynoic acids [1389]. Ruthenium catalyzed cycloadditions of propargylic alcohols and cyclic 1,3-dicarbonyl compounds (Eq. (384)) [1390]. Nickel catalyzed the formation of aromatic phthalides from 2-bromobenzoic acid esters and aldehydes (Eq. (385)) [1391]. A variety of transition

metals catalyzed the formation of (2-furyl)carbenoids from 2-ene-4-yn-1-ones followed by intermolecular cyclopropanation in the presence of alkenes (Eq. (386)) [1392]:



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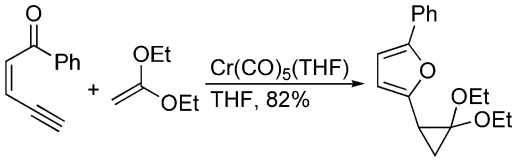
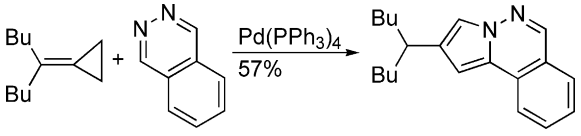


(386)

Palladium catalyzed a cycloaddition of alkylidenecyclopropanes with 1,2-diazines (Eq. (387)) [1393]. Palladium catalyzed the reaction of acetylpyridines with methylene aziridines to give pyridinylpyrroles (Eq. (388)) [1394]. Pyrroles were prepared via palladium-catalyzed coupling of alkynes, imines and acid chlorides (Eq. (389)) [1395]. An interesting cyclization of 2-alkenylpyrrolidines with carbodiimides to give seven-membered cyclic guanidines was developed [1396]. Palladium catalyzed the formation of polyheterocycles via intramolecular *N*-arylation-carbon-hydrogen bond activation-aryl-aryl bond coupling (Eq. (390)) [1397]. Palladium catalyzed the formation of 1,2-dihydroisoquinolines from 2-alkynylbenzaldehyde imines, an allyl stannane, and allyl chloride (Eq. (391)) [1398]. Platinum catalyzed the cyclization of 2-alkynylbenzenamides to give indoles (Eq. (392)) [1399]:



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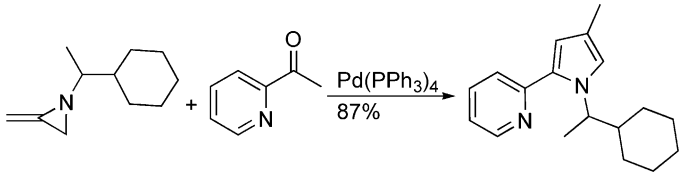

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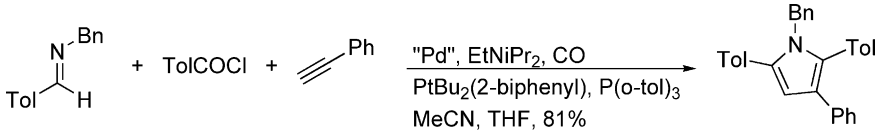

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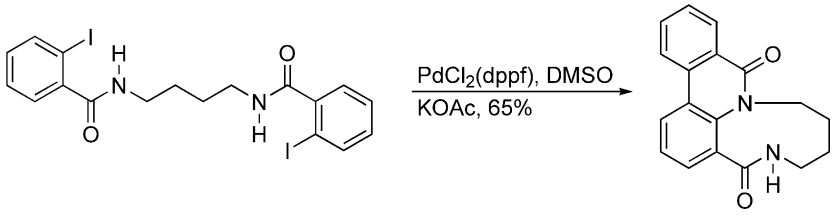

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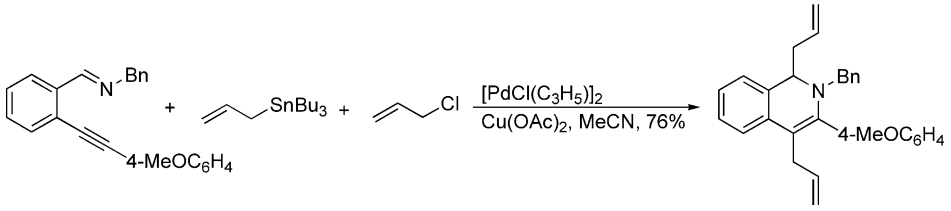

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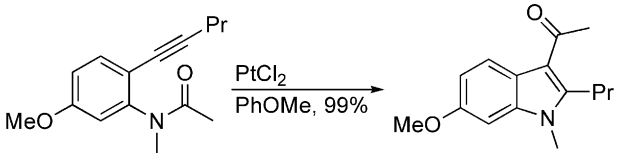
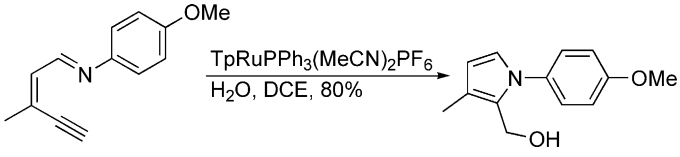


(392)

Ruthenium catalyzed the cyclization of 3-en-ynyl imines in the presence of a nucleophile to give substituted pyrroles (Eq. (393)) [1400]. A rhodium-catalyzed synthesis of five- to seven-membered lactams by oxidative *N*-heteroannulation of aminoalcohols was described (Eq. (394)) [1401]. Rhodium catalyzed aziridinations of allyl-substituted sulfonamides and carbamates (Eq. (395)) [1402]. A copper-catalyzed alkene aziridination using PhI = Nts was employed in a synthesis of kalihinol C (Eq. (396)) [1403]. Zirconium mediated cyclizations of alkenyl-substituted carbamates to give lactams (Eq. (397)) [1404]:



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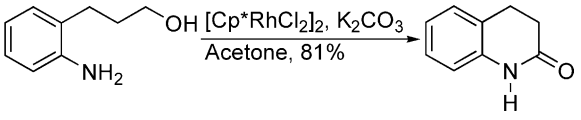

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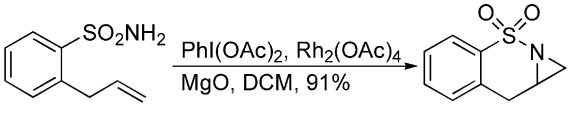

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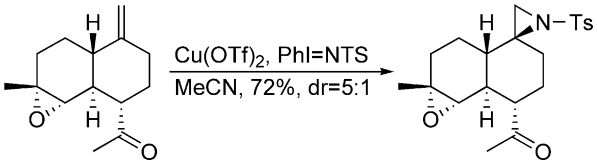

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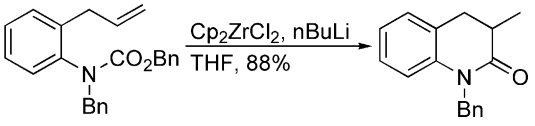
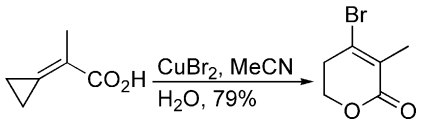


(397)

Copper catalyzed cyclizations of cyclopropylidene acetic acids and esters, and cyclopropylidene acetonitriles to give heterocycles (Eq. (398)) [1405]. A ruthenium–gold catalyst pair was developed to prepare oxazoles from 1-yne-3-ols and primary amides [1406]. Iridium catalyzed an asymmetric *N*-heterocyclization of diols with primary amines (Eq. (399)) [1407]. Zirconium mediated cyclization of a silicon tethered diyne with three molecules of a nitrile to give pyrrolo[3,2]pyridine derivatives (Eq. (400)) [1408]:



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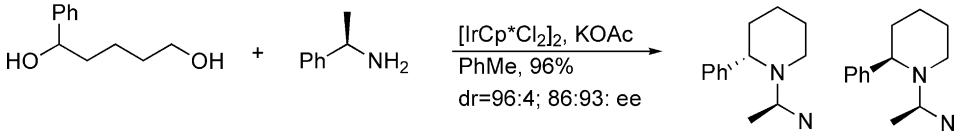

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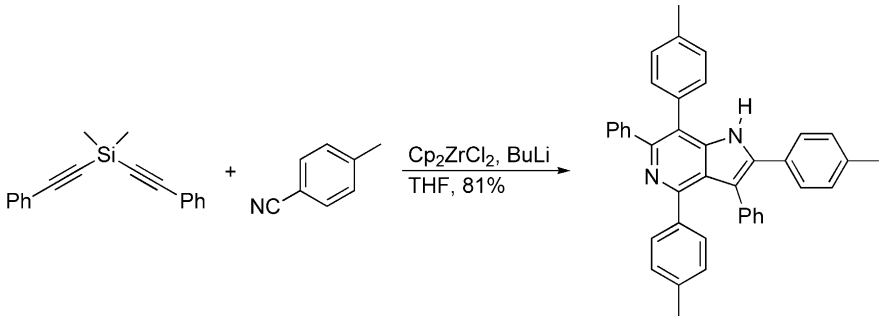
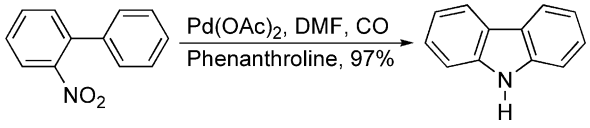


(400)

Palladium catalyzed reductive *N*-heteroannulations of nitrobiphenyl derivatives in the presence of carbon monoxide to afford carbazoles (Eq. (401)) [1409]. Reductive annulation of 2-nitroketoximes using iron as the catalyst gave 1*H*-indazoles (Eq. (402)) [1410] Ruthenium catalyzed the formation of 2,3-disubstituted quinolines from nitroarenes and tetraalkylammonium salts in the presence of tin dichloride (Eq. (403)) [1411]:



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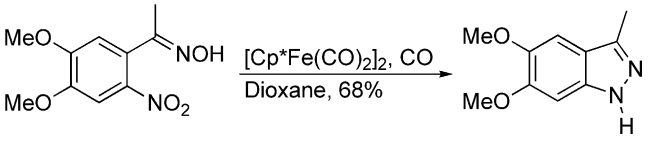

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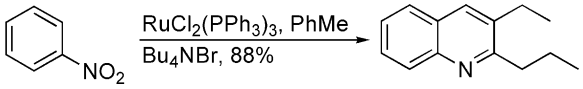


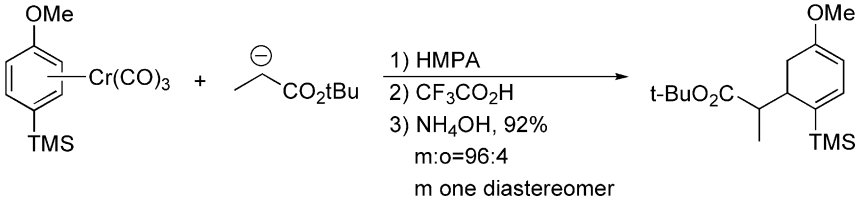
(403)

A palladium–copper-catalyst system was used in a three-component reaction of a terminal alkyne, trimethylsilylazide, and an allylic carbonate affording allyltriazole [1412]. A related four-component reaction of an alkyne two equivalents of an

allylic carbonate, and trimethylsilyl azide to give diallyltriazoles was described [1413].

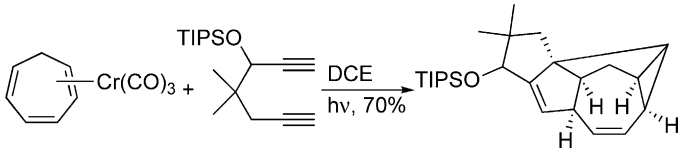
16. Reactions of isolated transitionmetal complexes and titanium- and zirconium-intermediates

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. The metal can then later be removed after serving as a template. Arene tricarbonyl chromium complexes continued to be extensively used as templates for organic reactions. Diastereoselctive SmI_2-mediated coupling of chromium tricarbonyl complexed nitrones with carbonyl compound was described [1414]. A diastereoselective ring-lithiation–bromination was used in a synthesis of korupensamines A and B [220]. A nucleophilic addition to an arene tricarbonylchromium complex with high vicinal stereocontrol was reported and used in a synthesis of erythro juvabione (Eq. (404)) [1415]:



(404)

Cycloadditions of diynes with chromium complexed cycloheptatrienes to give complex polycyclic compounds were developed and used in a synthesis of epi-pentalenic acid (Eq. (405)) [1416]. Rhenium-complexed naphthalenes were diastereo- and enantioselectively dearomatized (Eq. (406)) [1417]. Rhenium complexed anisoles were shown to undergo regioselective *para*-Michael additions (Eq. (407)) [1418]. Osmium, rhenium, and tungsten benzene complexes underwent stereoselective 1,4-addition reactions [1419]:



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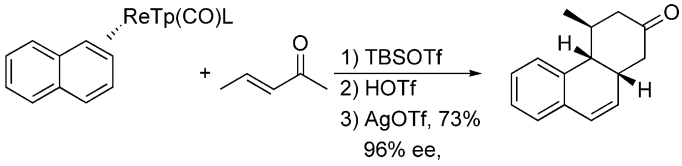

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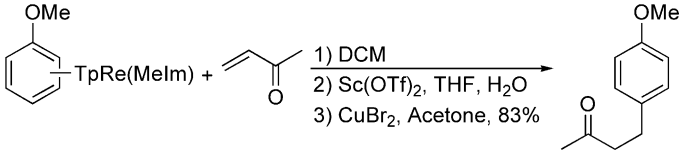
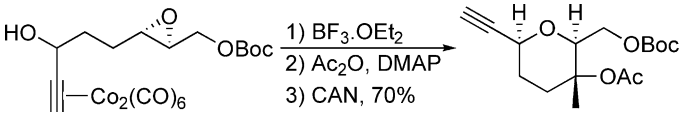


(407)

Oxiranes can be used as the nucleophile in intramolecular Nicholas-type reactions of cobalt alkyne complex stabilized cations (Eq. (408)) [1420]. A Nicholas reaction of a cyclopropyl substituted alkyne cobalt carbonyl complex was reported (Eq. (409)) [1421]. Nicholas-type reactions were used in synthetic applications toward (+)-sorangicine A [1422], cystothiazoles A and B [43], and climacostol (Eq. (410)) [1423]. An intramolecular Nicholas reaction Pauson–Khand sequence was used to prepare oxygen-containing heterocycles [1424]:



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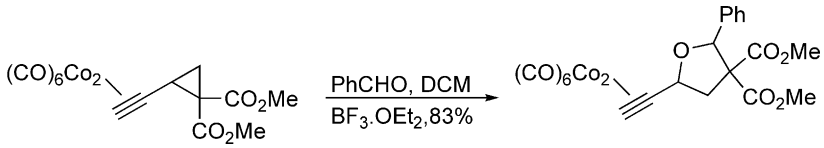

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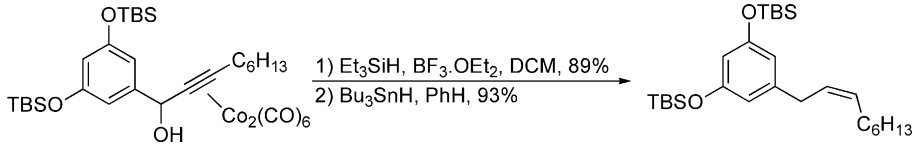
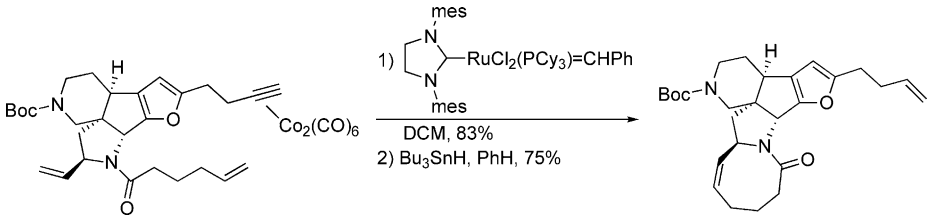
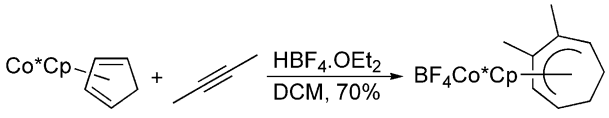


(410)

A cobalt–alkyne complex was utilized as an alkyne protecting group and later reductively decomplexed to an alkene in a synthesis of (−)-nakadoamine (Eq. (411)) [1106]. A two-carbon ring-expansion of cobalt-coordinated cyclopentadiene complexes was described (Eq. (412)) [1425]:

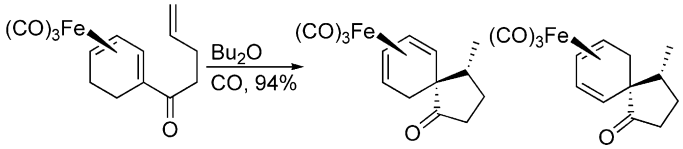


(411)



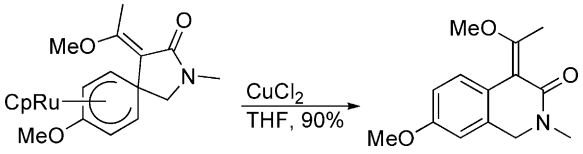
(412)

Iron tricarbonyl complexes continued to be used in organic synthesis. Cyclohexadiene iron tricarbonyl complexes undergo cyclization with a pendant enone unit to give spirocyclic compounds (Eq. (413)) [1426]. Reaction of (1-methoxycarbonyl-5-phenylpentadienyl)iron tricarbonyl complex with carbon and heteroatom nucleophiles occurs generally in the 5-position to afford (2,4-dienoate)iron complexes [1427]. 1-Acetoxy-2,4-hexadiene iron tricarbonyl and 2,4-hexadienal iron tricarbonyl were used as electrophiles in reactions with TMS-enol ethers derived from glycine [1428]. A bicyclo[5.1.0]octadienyliron cation was used to prepare a cis-2-(2′-carboxycyclopropyl)glycine (Eq. (414)) [1429]. An iron tricarbonyl diene complex served as template for polycyclizations affording octahydrophenanthrenes (Eq. (415)) [1430]. 6-Chlorohyellazole was prepared using cationic cyclohexadienyliron tricarbonyl complexes [234]:



(413)

An iron η^3-allyl lactone complex was used as a template in a synthesis of (−)-gloesporone [1431]. The regioselectivity of nucleophilic additions to alkenyl-substituted iron η^3-allyl lactone complexes was examined [1432]. Spirocyclic cyclohexadienyl ruthenium complexes were oxidatively demetallated to give fused heterocycles (Eq. (416)) [1433]. Regio- and diastereoselective insertions of allenes into oxapalladacycles to give 3,4-dihydro-2*H*-1-benzopyranes was described (Eq. (417)) [1434]:



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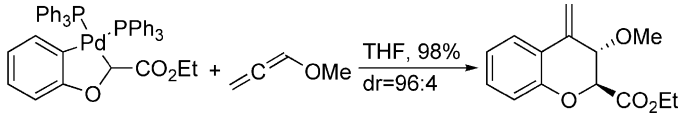
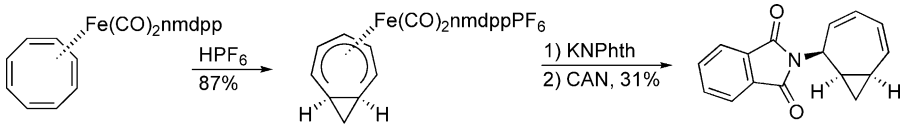
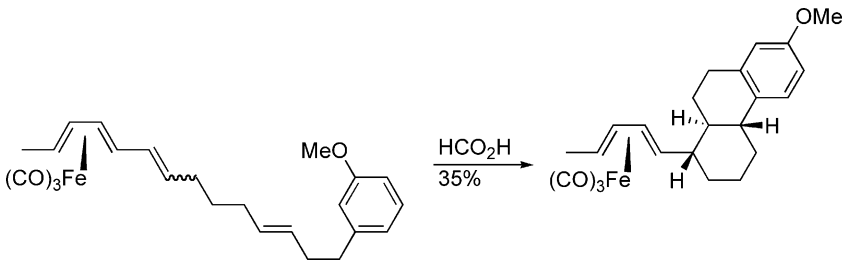


(417)

Zirconacyclopentenes undergo selective oxidation using organoboranes to form 1-alkylidene-2-hydroxymethylcyclopentanes (Eq. (418)) [1435]. Zirconacyclopentanes were expanded to zirconacyclohexanes via carbenoid insertion (Eq. (419)) [1436]. Zirconacyclopentenes were allowed to react with alkynes to form zirconacyclopentadienes (Eq. (420)) [1437]. 5-Hydroxy-1-alkenylboronates were prepared from 1-alkynylboronates and aldehydes via a zirconacyclopentane

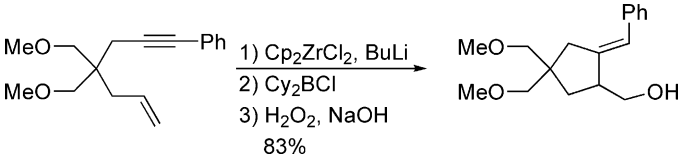


(414)



(415)

intermediate [1438]. Zirconacyclopentenes reacts with two different aldehydes in the presence of copper to give tetrahydrofuran derivatives (Eq. (421)) [1439]. Zirconacyclopentadienes were coupled with aryl-1,2-diiodides to give octa-substituted naphthalenes (Eq. (422)) [1440] and with allylic halides to give 1,3-6-trienes [1441]. Azazirconacyclopentanes were formed by cyclization of ene-imines. The complexes were allowed to react with a variety of reagents to form amino-substituted compounds and cyclic amines (Eq. (423)) [1442,1443]. A zirconium-mediated cyclization of yne-amides followed by electrophilic cleavage of the formed zirconacycle was also described [1444]:



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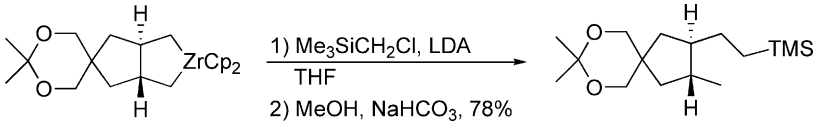

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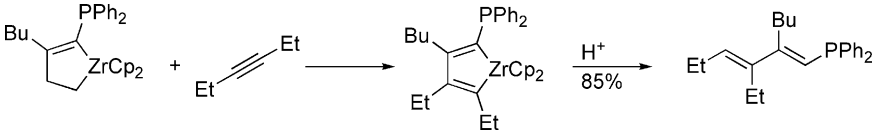
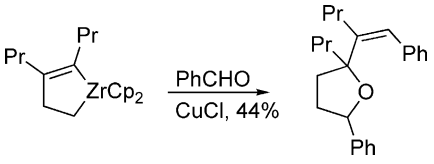
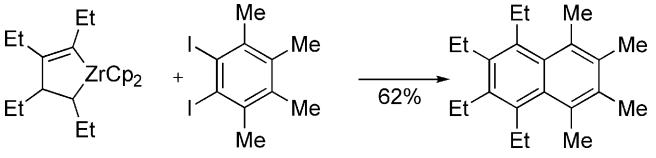


(420)



(421)



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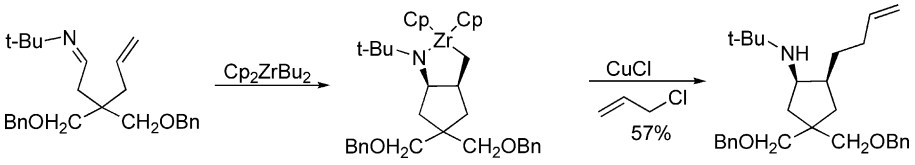
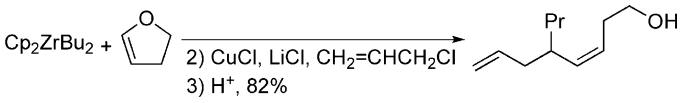


(423)

The formation of alkenylzirconium reagents from alkenyl sulfides, sulfoxide, and sulfones was described [1445]. Zirconocene–alkene complexes prepared in situ were allowed to react with acid chlorides to give homoallylic alcohols [1446]. Zirconocene–alkene complexes react with dihydropyrane and an electrophile to give cyclobutanes or with furans to give alkenol derivatives (Eqs. (424) and (425)) [1447]. Related reactions of zirconocene–alkyne complexes with alkenyl bromides furnished cyclobutenes and 1,3-dienes [1448]. Imine–zirconocene complexes formed in situ were coupled with enol ethers or allyl ethers to give allylic- or homoallylic amines (Eq. (426)) [1449]:



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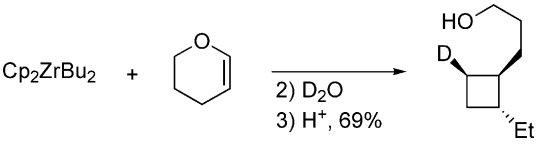
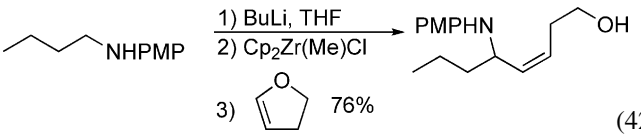
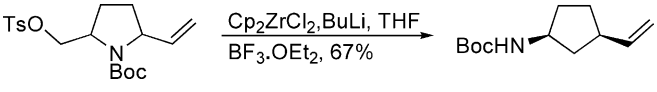


(425)



(426)

Zirconocene mediated transformations of 2-alkenylheterocycles to give alkenylcarbocycles were reported (Eq. (427)) [1450]. Hydrozirconation followed by oxidation of 1,5- and 1,6-dienes furnished cyclic compounds (Eq. (428)) [1451]. Copper catalyzed the coupling of 2-alkenylbenzylzirconocene intermediates with acid chlorides, allylic halides, propargyl bromide, and an allylic phosphonate (Eq. (429)) [1452,1453]. Cyclopropylmethylzirconium compounds were coupled with acid chlorides, allylic chlorides [1454]:



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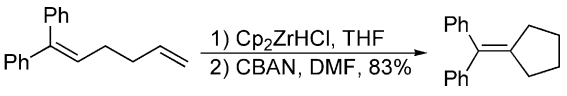
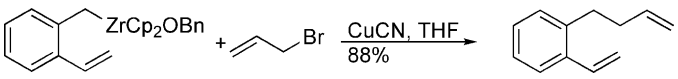
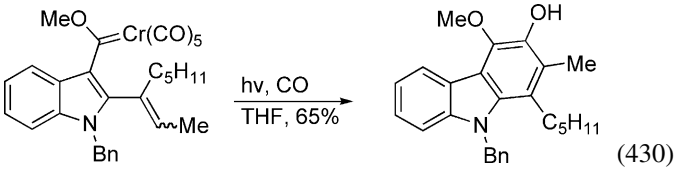


(428)

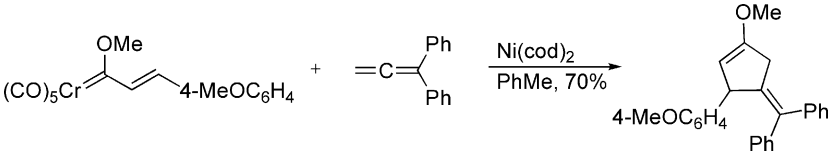


(429)

Fischer carbene complexes were used in organic synthesis of, for example, carbazoquinocine C (Eq. (430)) [1455]. Related benzannulations were used in synthesis of the proposed structure of landomycinone [1456], dibenzofuran-1,4-diones [1457], and shikonin [1458]. Nickel and rhodium catalyzed cycloaddition reactions of unsaturated Fischer carbene complexes with allenes (Eqs. (431) and (432)) [1459,1460]. A formal [5 + 2]cycloaddition of α,β,γ,δ-unsaturated Fischer carbenes and ketone enolates was described (Eq. (433)) [1461]. β-Amino-substituted α,β-unsaturated Fischer chromium carbenes produced aminosubstituted 1,3-cyclopentadienes upon reaction with alkynes [1462]:



(430)



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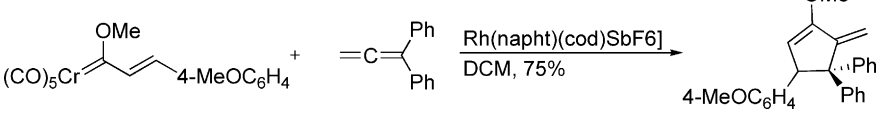

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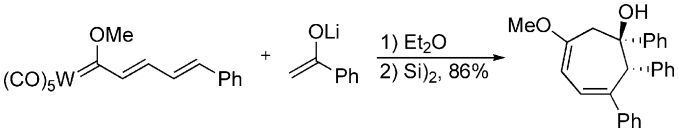
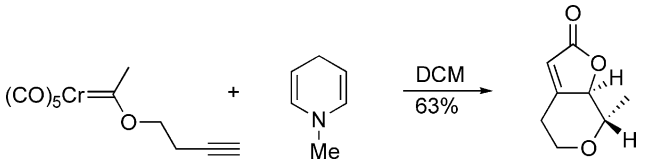
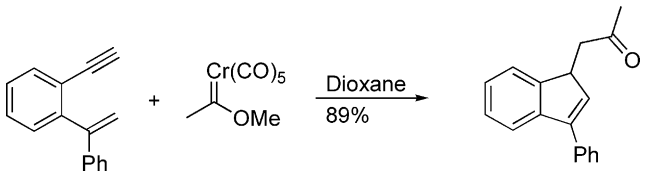


(433)

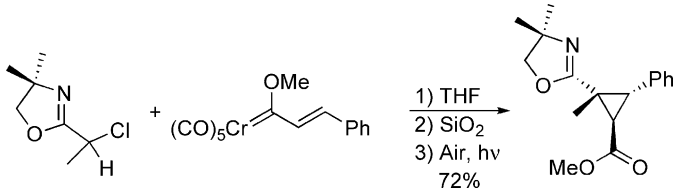
Reactions of alkyne-substituted Fischer chromium carbenes with dihydropyridines affords polycyclic butenolides or lactams in a diastereo- and enantioselective fashion (Eq. (434)) [1463]. Reaction of Fischer chromium carbenes with alkynyl–alkenylsubstituted aromatic compounds gave indenes (Eq. (435)) [1464]. Substituted cyclopropanes were prepared from lithiated chloroalkyloxazolines and α,β-unsaturated Fischer carbene complexes (Eq. (436)) [1465]. Rhodium catalyzed a [3 + 2]cycloaddition using Fischer carbenes and alkynes to give 2-cyclopentenones (Eq. (437)) [1466]. Copper-catalyzed dimerization of Fischer chromium carbenes [1467]. Reaction of propargylsilanes with Fischer chromium carbenes gave conjugated dienol ethers (Eq. (438)) [1468]:



(434)



(435)



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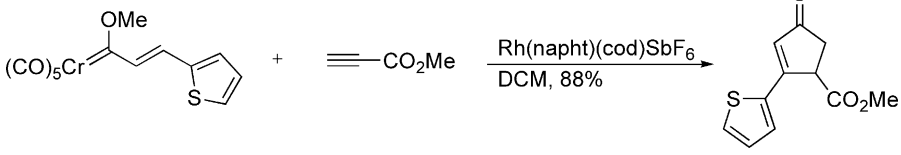
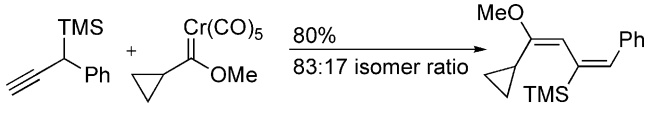
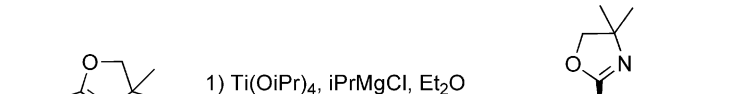


(437)

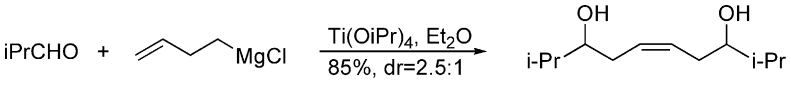


(438)

Asymmetric couplings of alkenyloxazoline–titanium complexes with alkynes and an electrophile were reported (Eq. (439)) [1469]. Titanacyclopentenes couple with carbonyl compounds to give 2-en-1,4-diols (Eq. (440)) [1470]:

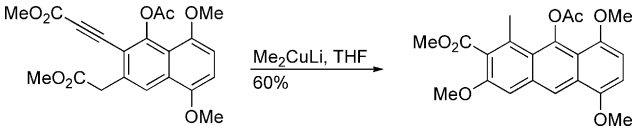


(439)

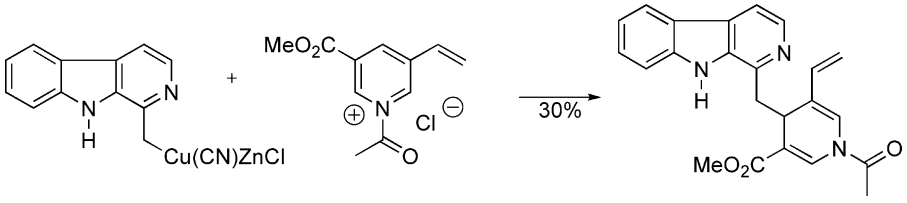


(440)

Stereoselective Michael additions of dialkylcuprates and diarylcuprates were used in synthetic applications toward, MetAP-2 inhibitors [125], tetracenomycin A_2 (Eq. (441)) [1471], (−)-cochleamycin A [323], (+)-tricycloclavulone [914], 3-desmethyl 251F and 251F [859], (−)-terpestacin [788], desmethylpallescensin-A, isopallesencin-A, and isopallesencin-1 [1472], pancratistatins [1080], alkaloids 223A and 205B [491], spiro segment of halichlorine [1473], carboskeleton of isaindigotidione [1474], and the tricyclic core of guanacastepene A [74]. Michael addition of MeCu was used in a synthesis of alcyopterosin N [1333]. Addition of a copper intermediate to a *N*-acetylpyridinium salt was used in a synthesis of the proposed structures of lualine and lyadine (Eq. (442)) [1475]:

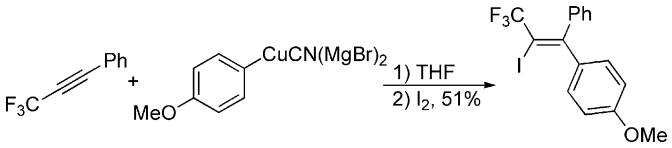


(441)

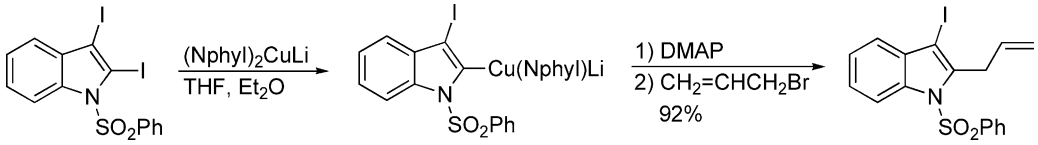


(442)

Regio- and stereoselective oxirane ring opening using Me_2CuLi_2CN or Me_2CuLi were used in a synthesis of pectenotoxin [1476], zincophorin [141], eriolangin [918], and bis-spiroacetal core of spirolide B [1477]. A selective carbocupration of an internal alkyne was used in a synthesis of panomifene (Eq. (443)) [217]. A regioselective iodine-copper exchange followed by reaction with electrophiles was described (Eq. (444)) [1032]:



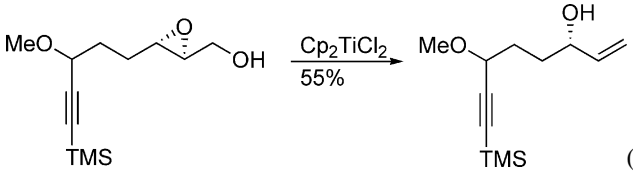
(443)



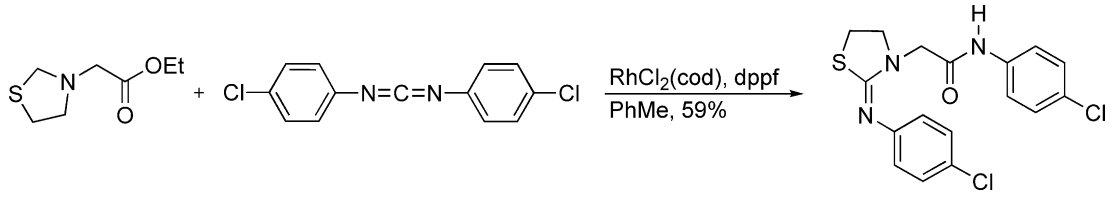
(444)

17. Miscellaneous reactions

Titanium mediated the transformation of a hydroxymethyl-substituted oxirane to an allylic alcohol (Eq. (445)) [1420]. Reduction of a thioester to an aldehyde using Et_3SiH in the presence of a palladium catalyst was used to prepare functionalized 14-β-hydroxysteroids [1478]. Rhodium catalyzed the formation of thiazolidinimines from thiazoles and carbodiimides (Eq. (446)) [1479]. Ruthenium catalyzed the formation of silyl ethers from alcohols and diphenyl-alkynylsilane affording hydrogen gas as the only side product [1329]:



(445)



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