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Review

Transition metals in organic synthesis: Highlights for the year 2005

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Abstract: A review with 1627 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 2005. Comprehensive literature coverage with extensive citations is presented. The field of transition metal chemistry continued to expand in 2005 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. Most copper catalyzed alkylations and Michael type reactions 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. Transition-metal catalyzed reactions used to remove allyl groups from allylic ether and amines have also not been reviewed. Most hydroformylations were omitted apart from some reactions used in total synthesis.

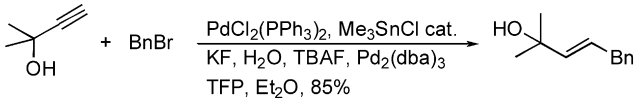
New catalyst systems including ligands, solvents, and reaction conditions for reactions that have ample examples in the literature have not been included unless truly remarkable results were obtained.

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

2.1. Carbon–carbon bond-forming reactions via transmetallation

Palladium-catalyzed cross-coupling reactions of organotin reagents with a large variety of electrophiles [1] (Stille couplings) continued to be developed and extensively used in organic synthesis. Addition of cesium fluoride and copper iodide to the reaction significantly improved the yields of Stille couplings [2]. A Stille coupling catalytic in tin was developed (Eq. (1)) [3].

Both palladium [4] and nickel [5] catalyzed Stille couplings of monoalkyltin derivatives were reported. (*E*)- and (*Z*)-Difluoro-3-stannylpropenoic acid esters were used [6]. Sequential Stille–Suzuki couplings of a 4-tributylstannyl-1,3-butadienylboronic acid ester were reported (Eq. (2)) [7]. 1,2-Dienyl-1-stannyl reagents were used in tandem intermolecular Stille-heterocyclization reactions affording isocoumarins (Eq. (3)) [8], indoles [9], and 2-pyridinones and (2*H*)-isoquinolin-1-ones [10].



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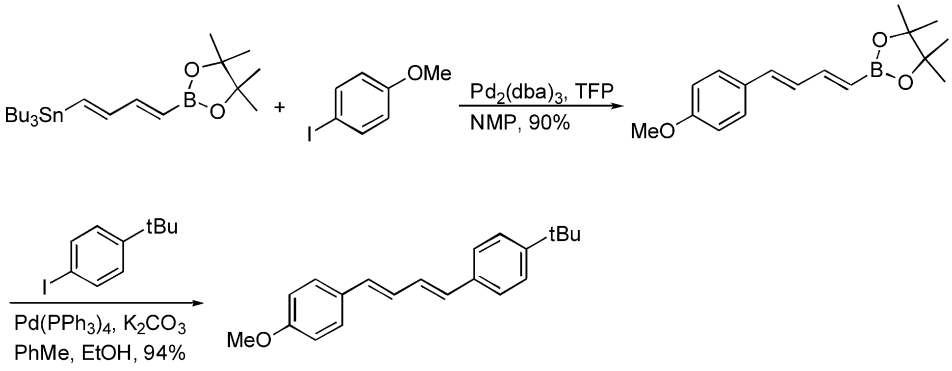

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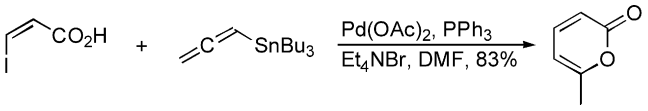


(3)

A variety of heterocyclic tin derivatives, such as 4-fluoro-5-pyrazolyl [11], 2-indolyl [12], 2-pyridyl [13], 6-chloro-2-pyrazinyl [14], 2-oxazolyl [15] and 3-pyrazinyl, 2-pyrimidinyl, and 2-pyridazinyl [16] stannanes were used in Stille-type couplings with aromatic halides. A ferrocenyl tin reagent was coupled with bromopyridine *N*-oxides [17].

Inversion of stereochemistry was observed using (*Z*)- or (*E*)-2-bromo-1,3-enynes [18]. Acid chlorides were used as electrophiles without decarboxylation in Stille-type couplings [19] and this type of reaction was used in a synthesis of methyl sarcoate [20] and of the core of reseophilin [21].

Palladium catalyzed the coupling of acid chlorides and benzylic bromides with trimethylstannylphenylselenide to give phenylselenides [22]. A 3-tosyl-2-eneoate [23], 2-fluorobenzoic acid salts [24], 4-[^{18}F]fluoroiodobenzene [25], thiomethylimidazolin-5-one [26], and a 1-bromo-1,2-diene [27] were used as electrophiles.

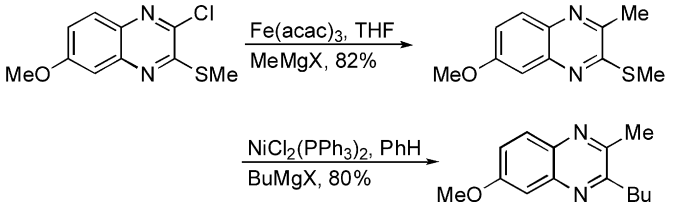
A variety of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and alkenyltin reagents. Examples of ring systems include, bromooxabicyclo[3.2.1]octadiene [28], 5-trifloxyindoles [29], 6-halopurines [30] and 3,5-dichloro-2(*H*)-1,4-oxazin-2-one [31]. A palladium-catalyzed coupling of a 2-thiomethylbenzoxazole with a 2-pyridyltin reagent was used in a synthesis of pyridinyl boxazomycin C analogs [32].

The intermolecular Stille reaction continued to be extensively used in organic synthesis [33]. Synthetic targets include, the heterocyclic core of GE 2270 [34], peridinin [35], pyrones isolated from placobranchus ocellatus [36], terpene-polyketide natural products [37], dihydroxerulic and xerulinic acid [38], bipinnatin J [39], elipticine [40], (+)-7-deoxy-*trans*-dihydronarciclasine [41], gambierol [42], taiwaniaquinol [43], (−)-scabronine G [44], garsubellin A [45], piericidin A1 and B1 [46], lucilactaene [47], fostriecin [48], lupine alkaloids [49], β-C-glycosides [50], (+)-crocacin D [51], lobatamide analogs [52], epoxyquinoid compounds [53,54], (+)-SCH 351448 [55], 3-methyl-2,5-dihydro-1-benzoxepin carboxylic acids [56], (+)-ochromycinone and (+)-rubiginone B_2 [57], EI-1941-1 and EI-1941-2 [58], mycothiazole [59], 6′-epiperidinin [60], aureothin and *N*-acylaureothamine [61], cyercene A and placidenes [62], herbindole B [63], (+)-tubelactomicin A [64], saudin [65], peroxyacarnoates A and D [66], aureothin, *N*-acetylaureothamine, aureonitrile [67], elysiapyrones [68], altromycin B [69], (+)-phorboxazole A and analogues [70,71], (−)-SNF4435 C and (+)-SNF4435 D [72], A2E [73], paracentrone [74], diazonamide A [75], 8,18-$^{13}C_2$-labeled retinal [76], diospyrol [77], (−)-eburnamonine [78], toward herbimycin A [79], lissoclinolide [80], tricyclic core of GKK1032 [81], macrolactin analogs [82], zoanthamine [83], epoxyquinols A and B [84], umbrosone [85], hazimycin [86], xanthocillin X dimethylether [87], (−)-pyricuol [88], (−)- and (+)-sparteine [89], and toward merrilactone [90]. Intramolecular Stille-type macrocyclization were used in synthesis of bis-deoxylophotoxin [91] and rhizoxin D [92].

Nickel and palladium were used as catalysts in coupling reactions of Grignard reagents with organic halides [93]. Nickel and palladium catalyzed the coupling of aryl fluorides with aryl magnesium reagents [94]. Nickel catalyzed coupling of a variety of aromatic chlorides and fluorides with Grignard reagents [95,96]. Neopentyl arenesulfonates were used in place of aryl halides in cross-coupling reaction with alkyl magnesium bromides [97]. Nickel catalyzed couplings of aryl *O*-sulfamates with Grignard reagents [98,99].

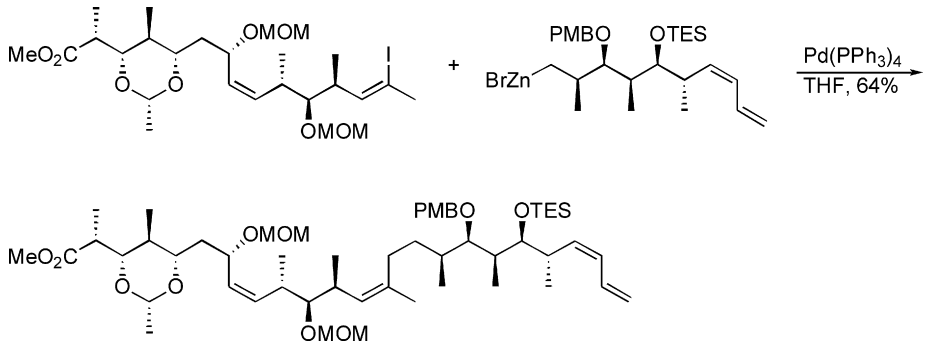
Palladium catalyzed couplings of 2- and 6-haloazulenes with Grignard reagents [100] and of aryl and alkenyl tosylates with aryl, alkenyl, and alkyl Grignard reagents [101]. Palladium-catalyzed coupling of lithium tri(2-oxazolyl)magnesate and tri(2-benzoxazolyl)magnesates with arylhalides [102]. A palladium-catalyzed alkyl Grignard–alkenyl triflate coupling was used in a synthesis of 15-deoxy-16-(*m*-tolyl)-17,18,19,20-tetranorisocarbacyclin [103].

Iron catalyzed cross-coupling of Grignard derived copper reagents and aryl iodides [104]. Iron and nickel catalyzed sequential couplings of 3-chloro-2-thiomethylquinoxalines with Grignard reagents (Eq. (4)) [105]. Chloro-pyrazine, -pyrimidine, -pyridazine, and -pyridine derivatives were coupled with alkyl and aryl Grignard reagents using iron catalysts [106,107]. An iron-catalyzed cross-coupling of a Grignard reagent and an alkenyl triflate was used in a synthesis of latrunculin A [108]. Iron catalyzed cross-couplings of alkyl halides [109] and alkenylsulfides [110] with Grignard reagents. Zirconium catalyzed the coupling of alkyl Grignard reagents with alkyl tosylates, bromides, fluorides and sulfates [111].



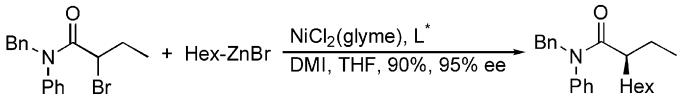
(4)

Palladium- or nickel-catalyzed couplings of organic halides and triflates with organozinc reagents (Negishi coupling) continued to grow [93,112,113]. Nickel catalyzed the coupling of tosylates derived from 4-hydroxy-2(1*H*)-quinolones, 4-hydroxypyrone, and 4-hydroxy-2(5*H*)-furanone with zinc reagents [114]. Selective coupling at the sp^2-carbon was observed using 2,4-diiodo-1-enes. Elimination was observed from the sp^3-bonded iodide [115]. Nickel catalyzed coupling of cyclic anhydrides with organozinc reagents to give ketone-acids [116]. An asymmetric nickel-catalyzed Negishi coupling of α-bromoamides with organozinc reagents was reported (Eq. (5)) [117]. Asymmetric nickel-catalyzed reactions of racemic secondary benzylic halides were also described [118]. The transmetallation from organozinc reagents to a series of diphoshine-phenylplatinum halides indicated that transmetalla-



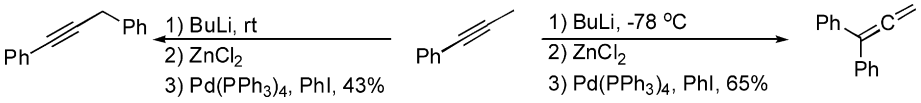
(8)

tion was the fastest for sterically less demanding diphosphines [119]. Iron catalyzed couplings of alkyl halides with aromatic zinc reagents [120].



(5)

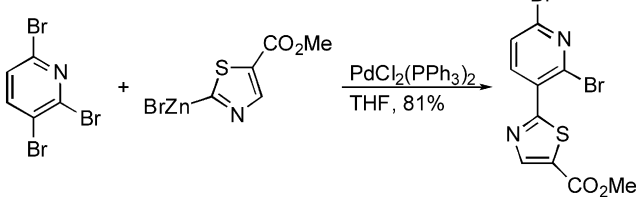
Palladium-catalyzed couplings of zinc α-cyanoalkyls with aryl bromides [121]. Depending on the base and reaction temperature, either 1,1-diaryl-1,2-propadienes or 1,3-diaryl-1-propynes can be obtained by deprotonation of arylpropynes, transmetallation to zinc and coupling with an aryl iodide in the presence of a palladium catalyst (Eq. (6)) [122].



(6)

Negishi couplings of alkyl bromides with alkylzinc reagents were reported [123,124].

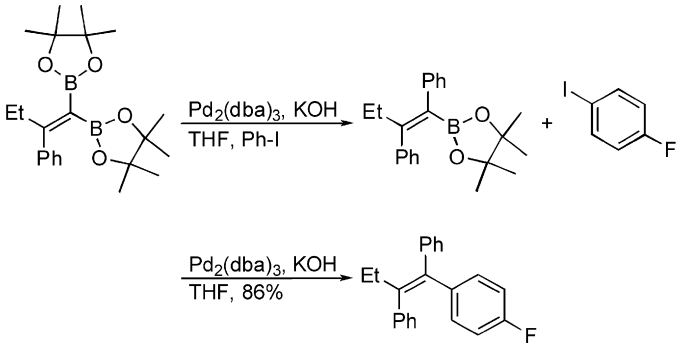
Couplings of 2-fluoro-4-pyrrolylzinc with 2,4- and 3,6-dichloropyrimidine [125] and 2-furyl-, 3-pyridyl-, 5-pyrimid-2,4-dionyl-, and 3-indazolyl-zinc reagents [126] were described. Synthetic targets using palladium- or nickel-catalyzed Negishi couplings include, heterocyclic core of GE 2270 (Eq. (7)) [34], (+)-apiosporamide [127], (+)-allocyathine B_2 [128], (+)-discodermolide (Eq. (8)) [129], brevetoxin B [130], (−)-scabronine G [44], isodityrosine [131], spectinabilin [132], 6(*E*)-plaunotol [133], kidamycin aglycone [134], δ-*trans*-tocotrienoloic acid [135], gizzerosine [136], Ro 67-8867 [137], GKK1032A [138], (+)-diplyne C and E [139], and (+)-curcuphenoil [140]. A regioselective coupling in the 2-position of 2,4-dibromopyridine was used in a synthesis of A2E [73].



(7)

The Suzuki (or Suzuki–Miyaura) 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 [93]. The mechanism of the coupling anhydrides with aryl boronic acids was examined in a DFT study [141]. Asymmetric biaryl-coupling was observed using aryliodides having a chiral chelating functionality adjacent to the iodide [142]. Enantioselective Suzuki couplings of optically active aryliodides were described [143]. Oxidative addition to halo-diaryl was found to be faster compared to dihaloaryls [144].

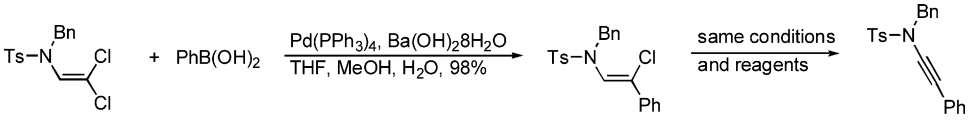
1,4-Cyclohexadienylboronic acids [145], 2-ethenyl-[1,3,2]-dioxaborinane [146,147] and 2-(1-ethoxybuta-1,3-dienyl)-[1,3,2]dioxaborinane [148] were used in Suzuki couplings. Arylboronic acid esters were coupled with carbamoyl chlorides [149]. Stereoselective coupling of 1,1-diboryl-1-alkenes with aryl iodides was reported (Eq. (9)) [150]. A variety of heteroaryl boronic acid derivatives were used such as, haloimidazolyl [151], 2- and 3-thiophenyl [152] in a synthesis of rebeccamycin analogs [153], 3-pyridyl [154], 5-pyrimidyl [155], 1,4,5,6-tetrahydropyridyl [156], 2-indolyl [157] and 3-furyl [158]. A ferrocenyl boronic acid reagent was coupled with a 1-iodonaphthalene [17].



(9)

2-Iodo-2-ene-1-ones [159], 3-tosyl-2-eneoates [23,160], benzylic carbonates and acetates [161,162], 2-fluorobenzoic acid salts [24], benzylic phosphates [163], 4-tosylphosphacoumarin [164], chloroformates and carbamoyl

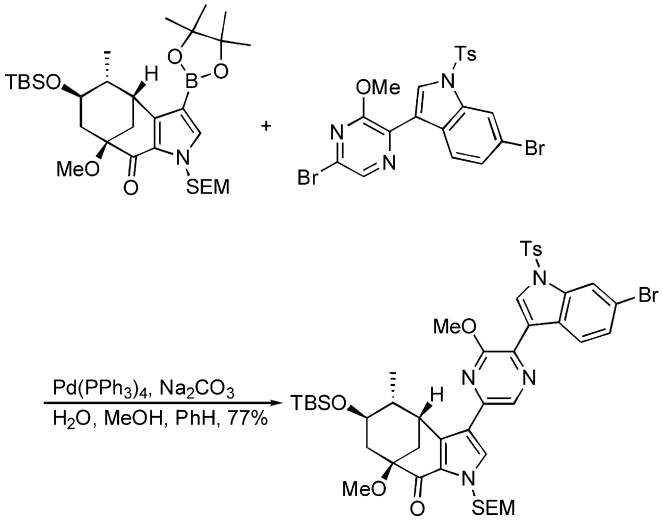
chlorides [165], bis-alkenylphosphates [166], enol phosphates [167], halo-1-azaazulenes [168] and thioenol ethers [169] were coupled with organoboron reagents. β,β-Dichloroenamides were coupled with organoboron reagents to give β-substituted-β-chloroenamides. Attempted second coupling gave elimination products (Eq. (10)) [170]. Selective Suzuki-coupling at the sp^2-carbon was observed using 2,4-diiod-1-enes. Elimination was observed from the sp^3-bonded iodide [115]. Palladium-catalyzed couplings of *in situ* formed aryl dibromoborane with aryl halides [171]. Nitrogen heterocycles can also be used in place of halides and triflates. Palladium catalyzed Suzuki reactions of a triazolopyrazinyl bromide [172] were described employing boronic acid derivatives. Palladium catalyzed a tandem cyclization Suzuki couplings of 1-chloro-2,7-enynes [173].



(10)

A large variety of functionalized heterocyclic halides and sulfonates were employed in the palladium-catalyzed Suzuki reaction. Heterocyclic ring-systems such as, bromo-oxabicyclo[3.2.1]octadienes [28], 6-bromoimidazo[1,2-*a*]pyridine [174], 3,5-dichloropyrimidine and 3,5-dichloropyridine [175], 4,7-dibromo-2,1,3-benzothiazole with a 3-indolylboronic acid [176], halo-amino-pyridines, pyrazines and pyrimidines [154], chloropyrazines in a synthesis of botryllazines [14], 4-chloroquinoline-2-one [177], 3-trifloxypyrazole [178], 8-bromo-2′-deoxyguanosine [179], 2-chloro-2′-deoxyinosine [180], 6-halopurines [30], 6-fluoro-, 6-(3-methylbutylsulfanyl)-, and 6-(3-methylsulfonyl)purine [181], 5-trifloxyindoles [29], 3-iodo-5-bromoindole [182], 4-bromobenzofuran [183], 5,7-dichloro-1,6-naphthyridin-2-(1*H*)-one [184], 7-bromo-pyrido[2,3-*b*]pyrazine [185], 5-iodo-1,2,3-triazoles [186], 3-bromo-2,5-dihydrofuran-2-one [187], 3-halo-indazoles [188,189], 3-chloro-2-pyrazolines and 3-chloro-1-phenyl-1,4,5,6-tetrahydropyridazine [190] and 3-iodo-4*H*-chromen-4-one [191] were used in Suzuki type cross-couplings. Regioselective coupling in the 4-position of 3-bromo-4-trifloxyquinolin-2(1*H*)-one [192], the 2-position of di- and tribrominated pyrroles [193] and the 3-position of 3,5-dichloropyrazin-2-ones [194] were reported. A regioselective coupling in the 2-position of 2,4-dibromopyridine was used in a synthesis of A2E [73].

A number of syntheses wherein aryl or alkenyl boronic acids or esters were coupled with aryl- and alkenyl-halides, -triflates, or -phosphonates were reported [195–197]. Synthetic targets included, prodigiosines [198], chartelline alkaloids [199], eupomatilone-3 [200], 9,10-deoxytridachione [201], hamigeran B [202], leprapinic acid and calycin [203], dehydrocoelenterazine analogs [204], gymnocin A (Eq. (11)) [205], murrastifoline A [206], styelsamine C and norsegoline [207], trispheridine [208], dragmacin F [209], ningalin D [210], eupomatilones [211], lucilactaene [47], epoxyquinols A–C and epoxytwinol A [212], mukonine [213], cepharanone [214], $\alpha_v\beta_3$ antagonist [215], alternariol [216], core of roseophilin [21], (−)-callystatin A [217], dibenzo[*c*,*p*]chrysene [218], GABA agonist [219,220], diazonamide A [221], dityrosine, trityrosine, pulcherosine [222], lamellarin D [223], integramycin [224], (+)-discodermolide and analogues [225,226], AB-ring system of hexacyclinic acid [227], δ-*trans*-tocotrienoloic acid [135], spirofungins [228], kwakhurin [229], toward (+)- and (−)-deplanchein [230], combretastatin analogs [231], diazonamide A [75], cadiolide B [232], pukeleimide A [233], toward apoptolidin [234], polycitone A and B [235], pulvinic acids [236], CB92834 [237], 2*E*,4*E*,6*E*,11*Z*-octadecatetraenoic acid [238], lamellarin analogs [239], dragmacidin B, *trans*-dragmacidin C, and *cis*- and *trans*-dihydrohamacanthins A [240], arcyriaflavin analogs [241], SCH-56036 [242] and eupomatilones 2 and 5 [243]. Intramolecular couplings were used in synthesis of isocomplestatin [244] and biphenomycin B [245].



(11)

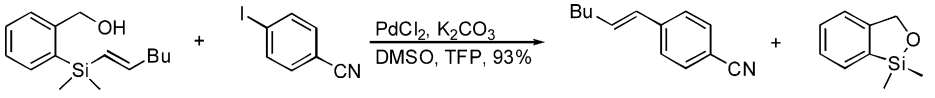
Alkyl boron reagents were used in synthesis of for example minfiensine [246], zoapatanol [247], dibenzocyclooctadiene lignans [248,249], the C1–C25 fragment of amphidinol 3 [250], (+)-SCH-351448 [251], the C10–C24 fragment of inostamycins [252], (+)-brefeldin C [253], (+)-wortmannin [254], cordiaquinone J and K [255] and cannabinoid antagonists [256].

Suzuki couplings of potassium alkyl-, aryl-, alkynyl-, and alkenyltrifluoroborates [23,257–262], potassium triethyl 2-indolylborate [263], and lithium alkynyltriisopropoxyborates [264] were described. Alkenyl tellurides were coupled with alkynyltrifluoroborates [265]. Coupling of alkyl borates were used in synthesis of EI-1941-1 and EI-1941-2 [58], of alkenylborate in synthesis of gustastatin [266], toward (+)-sorangicin [267], serofendic acids A and B [268] and C6–C21 segment of

amphidinolide E [269] and of an alkynylborate in a synthesis of latrunculin A [108].

Copper catalyzed the coupling of alkenyl boron compounds with 1-trimethylsilyl-2-bromoethyne [270]. Nickel catalyzed selective coupling in the 4,6-position of 5-chloro-2,4,6-trifluoropyrimidine with aryl boronic acids [271]. Nickel-catalyzed Suzuki couplings were used to prepare syn-7-substituted 2-azanorbornanes [272]. Rhodium catalyzed the coupling of arylboron compounds and arylhalides [273].

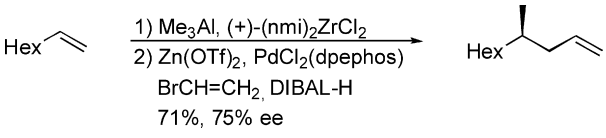
Silicon reagents continue to be used in coupling reactions using palladium catalysts. Palladium catalyzed cross-couplings of an alkenyldimethylbenzylsilane with iodobenzene [274], of alkenyl and aryl(2-hydroxymethyl)dimethylsilanes with aryl and alkenyl iodides (Eq. (12)) [275], of (*Z*)-3-trimethylsilyl-2-alkenoates [276] and of 1,4-bissilyl-1,3-butadienes with aryl iodides [277] and this reaction was utilized in a synthesis of RK-397 [278], of α-silyl nitriles with aryl bromides [121], of dimethyl(4-isoxazolyl)silanes with aryl iodides [279], of carbamoylsilanes with imidoyl chlorides [280] and benzyl bromides and chlorides [281], of (*E*)-*N*-(2-silylethenyl)carbazole with iodobenzene [282], of trimethyl-2-pyridylsilanes and aryl halides [283], and of dimethylfluorosilylalkenes with aryl iodides [284]. A palladium-catalyzed couplings of 5-bromotropolone and arylsiloxanes was used in an approach to colchicine [285].



(12)

Transmetallation and cross-coupling reactions of some more unusual organometallic reagents were described. Alkynyl indium and manganese reagents were coupled with alkenyl iodides using a novel palladium catalyst [286]. Triorganoindium reagents were coupled with aryl halides [287]. Alkyl indium reagents were coupled with thiol esters using a palladium catalyst forming unsymmetrical ketones [288]. Palladium catalyzed the coupling of diphenylalkynylstibanes with bromo- and iodo-alkenes [289]. Cobalt catalyzed the coupling of arylcopper reagents with aryl bromides and chlorides [290].

Nickel catalyzed couplings of allylzirconium reagents with aromatic halides [291]. Palladium catalyzed the coupling of alkenyl zirconium reagents prepared *in situ* by hydrozirconation of a terminal alkynes [292] and this reaction was used in a synthesis of (+)-pseudodistomin D [293], (+)-leucascandrolide A [294], and of mycolactones A and B [295]. An asymmetric zirconium catalyzed methylalumination followed by a palladium-catalyzed alkenylation was described (Eq. (13)) [296].

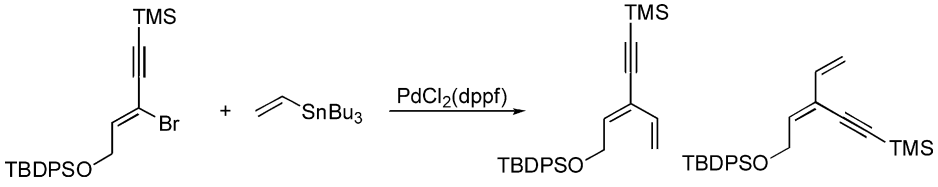


(13)

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 [297–299]. Silver-acetylides were formed *in situ* and are indicated as intermediates in palladium–silver catalyzed Sonogashira couplings [300].

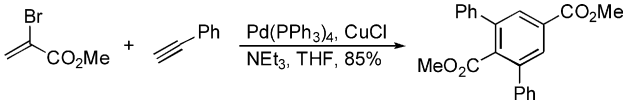
Palladium-catalyzed couplings of acid chlorides [301], a 3-tosyl-2-eneoate [23], 4-phenyltelluro-alkenes replacing the phenyltelluro group [302–304] and 4-tosylphosphacoumarins [164]. Selective Sonogashira-coupling at the sp^2-carbon was observed using 2,4-diiod-1-enes. Elimination was observed from the sp^3-bonded iodide [115,305]. Inversion of stereochemistry was observed using 2-bromo-1,3-enynes (Eq. (14)) [18].



(14)

Heterocyclic ring systems were shown to readily participate in Sonogashira-type couplings [175,306]. Some more complex examples include reaction of 5-iodoarabinosyluridine [307], 3-iodofuran [308], 6-chloro-9*H*-purine [309], 8-bromoadenosine [310], 2,6-bis(4-iodopyrazol-1-yl)pyridine [311], 6-chloro-3*H*-pyrimidin-4-one [312], bromo-oxabicyclo[3.2.1]octadienes [28], a triazolopyrazinyl bromide [172], 5-trifloxyindoles [29], 5-bromobenzofuran [183], 3-iodo-5-bromoindole and 3-iodo-5-bromo-3-indazole [182], 7-bromo-pyrido[2,3-*b*]pyrazine [185], 5-iodo-1,2,3-triazoles [186,313], 4,5-diiodoimidazole [314] and 4-iodo-2-bromoquinoline [315] derivatives. Sonogashira coupling of 2-iodophenols followed by cyclization to give benzofurans was reported [183].

A sequential Sonogashira coupling–cyclization–Suzuki coupling was described [316]. Sonogashira coupling of 2-iodobenzoic acids with terminal alkynes produced isocoumarins [317]. Sonogashira reaction of 2-iodo-1-benzenamine with propargylic alcohols gave quinolines [318]. Palladium catalyzed a Sonogashira coupling-benzannulation sequence using terminal alkynes and 2-bromopropenoates (Eq. (15)) [319]. An unusual intermolecular alkyne insertion alkyne termination was observed using 3-iodothioflavones under copper free conditions (Eq. (16)) [320].



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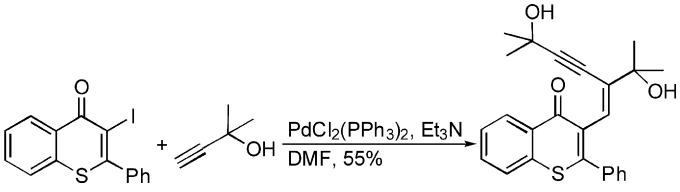
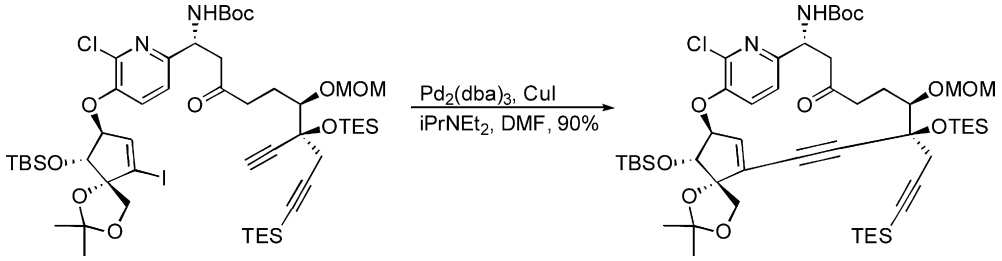


(16)

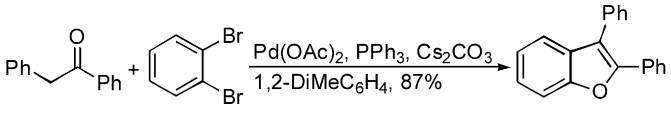
The Sonogashira reaction continued to be extensively used in organic synthesis [321]. Examples of synthetic targets include, 10-hydroxyasimicin [322], cylindramine [323], murisolins [324], norzoanthamine [325], salicylihalamide core [326], (+)-laudanosine and (−)-xylopine [327], 5,5-difluoroleukotrienes B_3 [328], dehydrocoelenterazine analogs [204], (−)-epiindolizidine 167B and 5*E*,9*Z*-indolizidine 223AB [329], (+)-allocyathine B_2 [128], amphidinolides T1 and T4 [330], (4*E*,6*Z*,10*Z*)-4,6,10-hexadecatrien-1-ol [331], diacetylenic spiroacetal enol ethers [332], epoxyquinoid compounds [53], heliophenanthrone [333], bulgaramine [334], (−)-siphonodiol [335], the ergoline skeleton [336], (−)-isoprelaurefucin [337], elipticine [338], pyranicin [339], peroxyacarnoates A and D [66], allocolchicinoid [340], (−)-217 A [341], DE ring system of upenamide [342], A2E [73], α-glycosylindole [343], mycolactones A and B [296], (+)-obtusenyne [344], tetradec-6-en-1,3-diyne-13-one [345], virol C and 1-dehydroxyvirol A [346], terreinol [347], (+)-trifluoromethyl monomorine [348], 2*E*,4*E*,6*E*,11*Z*-octadecatetraenoic acid [239], 7(*S*),17(*S*)-resolvin D5 [349], erogorgiaene [350], solamine analogs [351], longimicin C [352], furanopyrimidine nucleoside analogs [353] and polyacetylenic acids [354]. Intramolecular Sonogashira couplings were used in synthesis of cyclic peptides [355] and of the kedarcidin chromophore (Eq. (17)) [356].



(17)

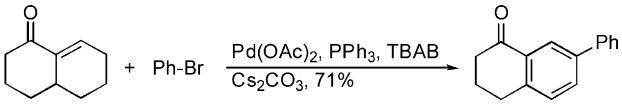
Related coupling reactions using copper catalysts have also been reported. Copper catalyzed the coupling of terminal alkynes with alkenyl and aryl halides [357]. Copper-catalyzed couplings of alkynyl halides with terminal alkynes, Cadiot–Chodkiewicz-type reaction, were used in synthesis of diacetylenic spiroacetal enol ethers [333], (−)-siphonodiol [336], panaxytriol [358], strongylodiols [359], (+)-gymnasterkoreayne [360], (+)-diplyne C and E [139] and polyacetylenic acids [355]. A copper-mediated cross-coupling of a terminal alkyne and an aryl iodide was used in a synthesis of renieramycin G and lemonomycinone analog [361].

Other carbon nucleophiles were employed in palladium catalyzed carbon–carbon bond forming reactions employing organohalides and triflates. Palladium catalyzed the arylation of sulfonamide enolates with aryl halides [362]. A sequential enolate *C*- and *O*-alkylation of 1,2-dibromobenzenes leading to substituted furans was described (Eq. (18)) [363]. A related sequential *C*- and *N*-arylation of 2-amino-acetophenone with 1,2-dibromobenzene was used in a synthesis of trileptal [364]. Palladium catalyzed α-arylations of ketones with aryl chlorides [365] and of sulfoximines with aryl bromides [366].



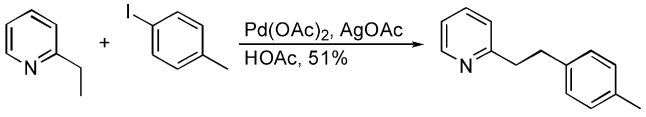
(18)

Palladium catalyzed an interesting γ-alkylation–aromatization of bicyclic enones to give 7-aryltetralones (Eq. (19)) [367]. Both *C*- and *O*-alkylation was observed upon arylation of 6,8-dimethoxybenzofuran-3-one [368]. Intramolecular palladium-catalyzed α-alkenylations or -arylations of ketones were used to polycyclic indole alkaloids [369], *N*-methylwelwitindolinone skeleton [370,371], ABC ring system of calyciphylline A [372], gardnerine and gardnutine [373], and lennoxamine, 13-deoxychilenine, and chilenine [374]. Palladium catalyzed an intramolecular α-arylation of an imine (Eq. (20)) [375].



(19)

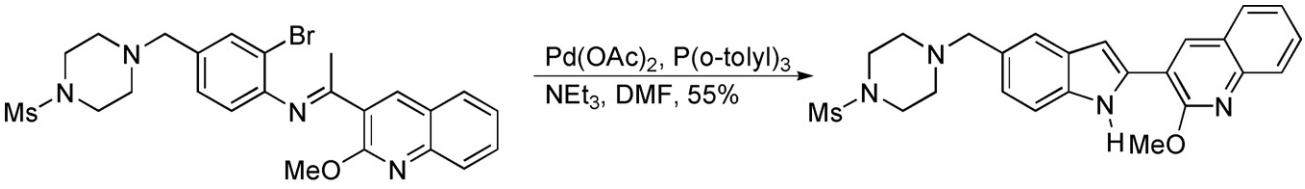
Palladium catalyzed *ortho*-arylations of anilides using aryl iodides, diaryl iodonium salts, or bromoalkenes [376,377]. Ruthenium catalyzed chelation assisted *ortho*-arylations of (2-pyridyl)arenes and acetophenone imines with aryl chlorides [378]. Related ruthenium-catalyzed *ortho*-arylations and -alkenylation of 2-aryloxazolines and 2-arylimidazolines were described [379]. Regioselective chelation-directed alkylation of sp^3-carbon-hydrogen bonds of 2-alkylpyridines with aryl iodides was reported (Eq. (21)) [380]. Palladium catalyzed a related reaction [381].



(21)

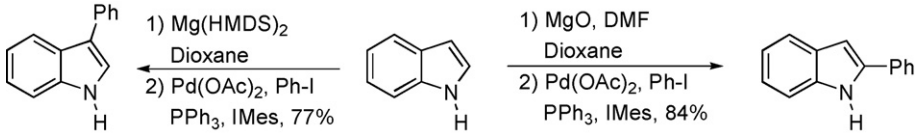
Ruthenium catalyzed couplings in the 2-position of pyridine with iodobenzene [382], of *N*-unprotected indoles and pyrrole using aryliodides [383] and of *N*-phenylpyrrolidine [384].

A mechanism for the related palladium-catalyzed arylation of indoles was proposed and based on this mechanism a selective arylation in the 2- or 3-position was developed (Eq. (22)) [385]. Palladium catalyzed cross-couplings of pyridine *N*-oxides in the 2-position using aromatic bromides [386].



(20)

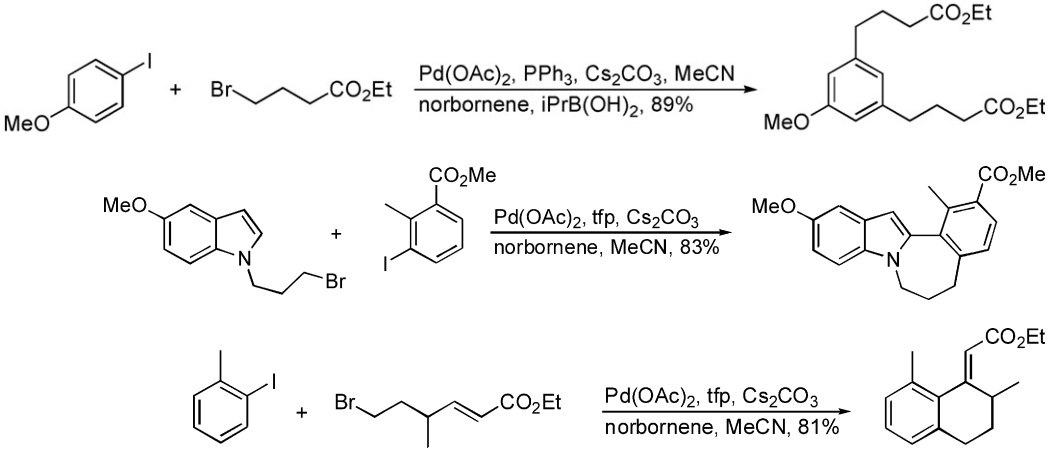
Benzothiophens and benzothiazoles were coupled in the 2-position with aryl bromides [387,388]. 1-Aryl-1*H*-imidazoles were coupled in the 5-position with aryl bromides, iodides and triflates [389] and ethyl 4-oxazolecarboxylate was coupled in the 2-position using iodobenzene [390]. Arylation in the 2-position of an indole using 3-bromoquinolin-2(1*H*)-one was used in a synthesis of an antiangiogenic tyrosine kinase inhibitor [391].



(22)

The palladium-catalyzed intramolecular variation ("aromatic-Heck") using aromatic rings in place of alkenes in couplings with organic and halides and triflates [392–394] was used to prepare benanomicin B [395], cavicularin and riccardin C [396], 5,6-dihydro-4*H*,8*H*-pyrido[3,2,1-*de*]phenanthridin-8-one [397], GABA agonist [219,220], lamellarin D and lamellarin analogs [223,240], allocolchicinoid [341], rhazinilam [398], givocarcin M [399], isoneocryptolepine [400], graphislactones A–D [401] and fused benzazepinones [402].

Palladium catalyzed a norbornene-mediated reductive dialkylation of iodobenzenes (Eq. (23)) [403]. An arylation–direct arylation sequence was used to prepare annulated indoles (Eq. (24)) [404]. Palladium catalyzed an alkylation-Heck-type reaction of iodobenzenes in the presence of norbornene to form fused aromatic rings (Eq. (25)) [405].



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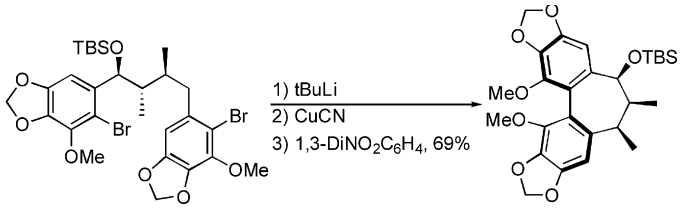
Copper catalyzed coupling of aryl halides with activated methylene compounds [406]. Copper mediated an intramolecular coupling of two aryl bromides in a synthesis of dibenzocyclooctadiene lignans (Eq. (26)) [249,250].



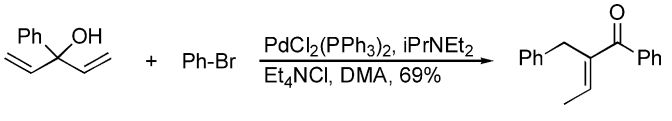
(26)

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

The palladium-catalyzed Heck (or Mizoroki-Heck) reaction remained one of the most versatile methods for the alkylation of alkenes [93,407]. The mechanism and kinetics of the Heck reaction was examined [408].

A palladium(II) intermediate in decarboxylative Heck reactions of carboxylic acids was isolated and further mechanistic steps were examined [409]. Aryl sulfonyl chlorides were used in place of halides via an initial desulfination. Both palladium and rhodium were used as catalysts [410]. Tetraarylphosphonium halides [411] and aryl diazonium tetrafluoroborates [412], in a synthesis of rolipram [413] and tosylates and mesylates derived from 1,3-dicarbonyl compounds [414] were used in place of halides and triflates. Halides and triflates of heterocyclic ring systems, such as 3-bromoquinoline-2-ones [177] and 7-bromo-pyrido[2,3-*b*]pyrazine [185] were used in Heck reactions. Regioselective reaction in the 3-position of 3,5-dichloropyrazin-2-ones was reported [194].

An interesting rearrangement was observed upon Heck reactions of 1,4-dien-3-ols and 1,4-enyn-3-ols (Eq. (27)) [415]. Migration of the palladium was observed in some intermolecular Heck reactions (Eq. (28)) [416]. A Heck reaction–intramolecular alkylation was reported using trifloxybenzaldehydes, ethylene glycol ethenyl ether, and a secondary amine (Eq. (29)) [417]. A double Heck reaction of dimethyl(2-pyridyl or 2-pyrimidyl)ethenylsilane with two different aryl halides was reported [418]. A related reaction of 2-pyrimidyl ethenyl sulfide was described [169].



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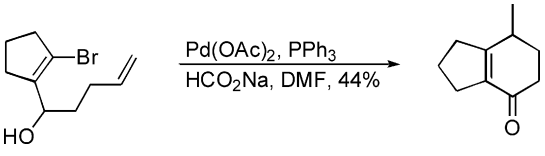

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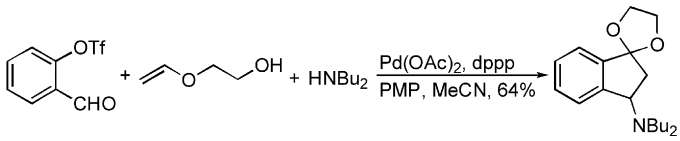
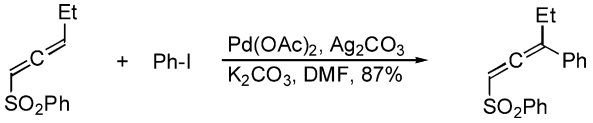


(29)

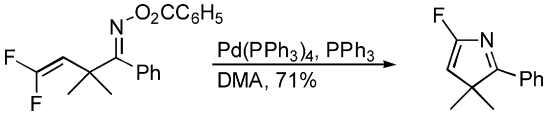
1-Cyclopropyl-1,2-dienes were used in Heck reactions to form 1,3,5-trienes [419]. Related reactions of 3,4-dienoates with aryl halides forming 4-aryl-2,4-dienoates were reported [420]. Heck reactions of 1,2-allenylsulfones with aryl halides affording a new allene were reported (Eq. (30)) [421]. Selective arylation of the more substituted position of *N*-acyl-*N*-ethenylamine derivatives using aryl triflates was reported [422]. Alkenylsilanes [423] and allylic acetates [424] were used as the alkene in Heck reactions. Cyclopropenes [425] were also used as the alkene in Heck-like reactions. The mechanism is not known.



(30)

The intermolecular Heck reaction was used in organic synthesis of, for example, glycocinnasperimicin D [426], cribrostatin IV [427], novel pyrimidine nucleosides [428], $\alpha_v\beta_3$ antagonist [215], KDR kinase inhibitors [376], selaginoidine [429], pancracine [430], pyrrolidino pseudoisocytidine [431], (+)- and (−)-deplanchein [230], rhizoxin D [432], 2-acetonylinosine [433] and 10-deoxyartemisinins [434]. A combination of an intermolecular and intramolecular Heck reaction of iodoarylalkenes was used to prepare macrocycles [435].

Intramolecular Heck-type reactions of 1,1-difluoroalkenes with *O*-pentafluorobenzoyloximes (Eq. (31)) [436] and of alkenes with *O*-pentafluorobenzoylamidoximes [437] were reported. Intramolecular Heck reactions [394,438–442] were used as the key step toward an array of synthetic targets for example, stephacidin A [443], 1-epi aglycon of cripowellins A and B [444], hamigeran B [202], mensacarcin [445], (−)-galanthamine and (−)-morphine [446], gelsemine [447], 5-substituted azepino[3,4-*b*]indoles [448], komaroviquinone and faveline methyl ether [449], pseudopteroxazole [450], diazonamide A [75], 4,7-azaindoles [451], substituted tryptophans (Eq. (32)) [452], 3,5,7-substituted indoles [453], morphine fragments [454], (+)-wortmannin [255], umbrosone [85] and the tricyclic core of cyathin diterpenoids [455]. An intramolecular reaction of an oxime ester was used synthesis toward prodigiosines (Eq. (33)) [198].



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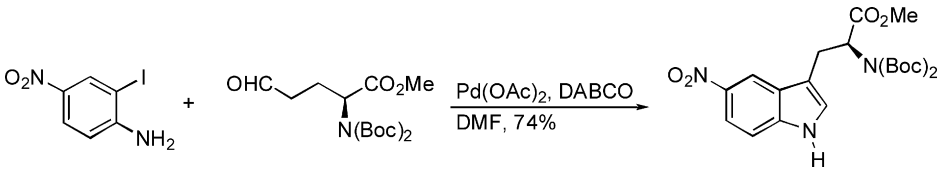

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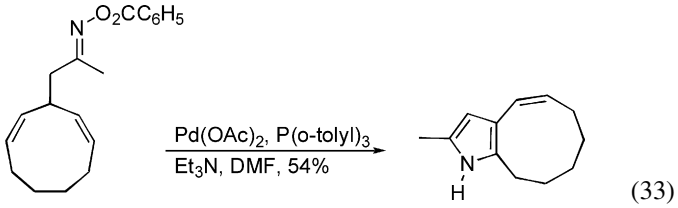
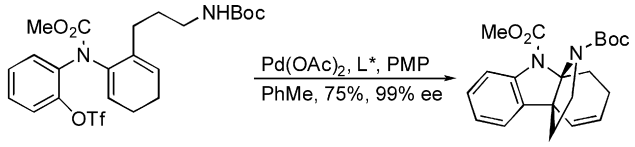


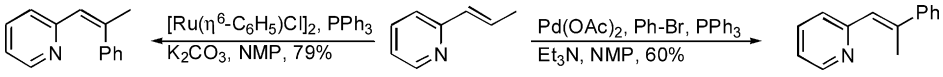
(33)

An intramolecular reductive Heck reaction was used in a synthesis of (−)-eudistomine [456]. Desymmetrization of meso compounds was achieved using an intramolecular Heck reaction [457]. A sequential asymmetric intramolecular Heck reaction–iminium ion cyclization was used in a synthesis of minfiensine (Eq. (34)) [247].



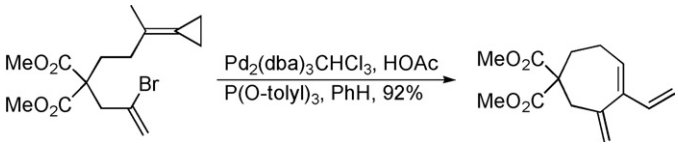
(34)

A copper-catalyzed intermolecular Heck reaction in ionic liquids was developed [458]. Nickel also catalyzed intermolecular Heck reactions [459]. Ruthenium catalyzed a chelation-assisted 2-alkenylpyridine arylation with opposite stereochemistry compared to the palladium-catalyzed Heck reaction (Eq. (35)) [460].



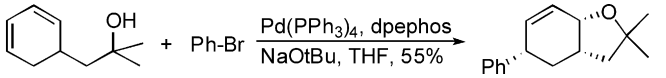
(35)

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. Bromoalkenes and alkyne-substituted methylenecyclopropanes participated in oxidative addition–insertion reactions to give polyfunctionalized compounds (Eq. (36)) [461]. An oxidative addition–intermolecular alkene insertion–intramolecular alkene insertion–elimination sequence was used to prepare the batrachotoxin ring system [462].



(36)

An "oxidative addition–intermolecular alkene insertion–intermolecular alkoxylation" to give 2,1′-substituted tetrahydrofurans was described [463]. Evidence for initial alkoxy-palladation instead of alkene insertion was reported [464]. A related reaction using both cyclic and acyclic 1,3-diene-7-ols was described. Cyclic substrates gave 1,4-addition and acyclic substrates afforded 1,2-addition products (Eqs. (37) and (38)) [465]. Control of alkene regioselectivity of sequential intermolecular Heck reaction followed by intramolecular amination was observed in some cases (Eq. (39)) [466,467].



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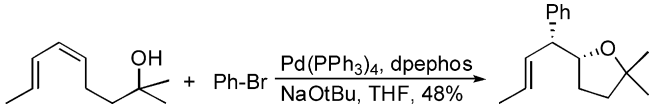

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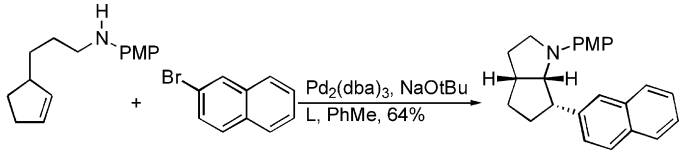


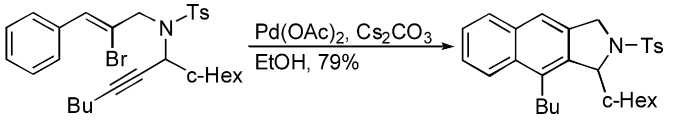
(39)

2.4. Carbon–carbon bond formation via insertion of alkynes

Reactions that can be formalized as the formation of σ-palladium complex intermediates by oxidative addition of palladium(0) to aryl halides and triflates followed by sequential insertion of an alkyne and a termination step for example nucleophilic addition or carbonylation are discussed in this section.

Palladium catalyzed an oxidative addition–alkyne insertion–arene insertion to form polycyclic aromatic systems (Eq. (40)) [468]. Palladium catalyzed an oxidative

addition–alkyne insertion–bicyclopropylidene insertion to give cyclopropanated bi-, tri-, and tetracyclic frameworks [469]. Palladium catalyzed the cyclization of aryl halides with tertiary propargylic alcohols to give 3-benzylidene-2,3-dihydrofurans (Eq. (41)) [470]. Reactions of aryl iodides with internal alkynes resulted in products derived from multiple migrations of palladium (Eq. (42)) [471]. A three component reaction was used in a synthesis of 13-hydroxy- and 13-acetoxy-14-nordehydrocacalohastine (Eq. (43)) [472]. Aryl iodides were coupled with diarylalkynes and activated alkenes (Eq. (44)) [473]. An oxidative addition–intermolecular alkyne insertion–intramolecular Heck reaction was used in a synthesis of 24,24-ethanovitamin D_3 lactones [474].



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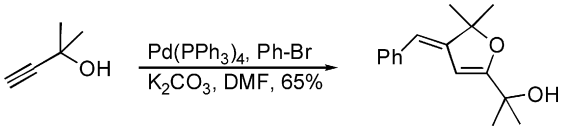

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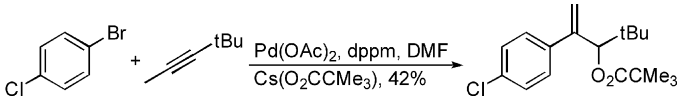

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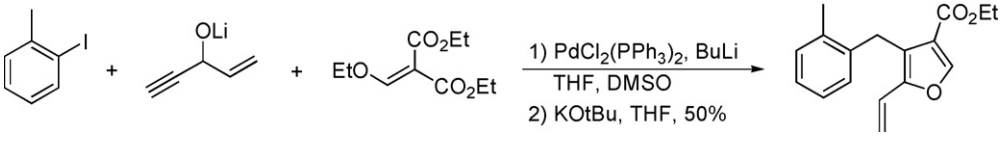
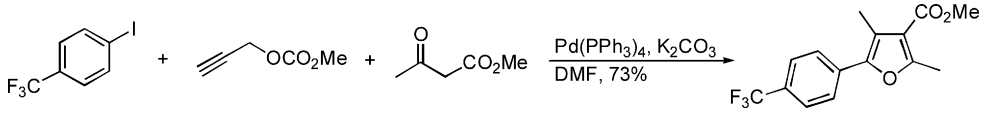


(43)

(44)

Palladium catalyzed a three-component reaction of an activated methylene, a propargylic bromide or carbonate, and an aryl iodide to give substituted furans (Eq. (45)) [475]. Palladium catalyzed a cyclization of activated methylene compounds having a tethered alkyne and in the presence of an aryl iodide (Eq. (46)) [476]. Indenes were formed by a palladium-catalyzed reaction of internal alkynes and (2-halophenyl)-substituted activated methylene compounds [477]. Oxidative addition to 2-iodo-4-(phenylchalcogenyl)-1-butenes followed alkyne insertion and Sonogashira termination was reported (Eq. (47)) [478].



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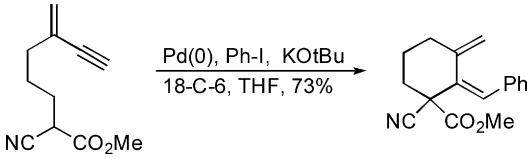

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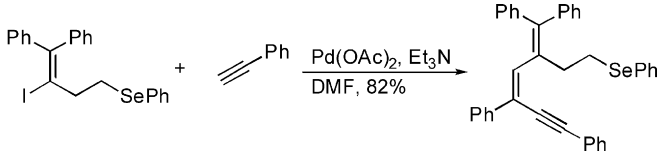
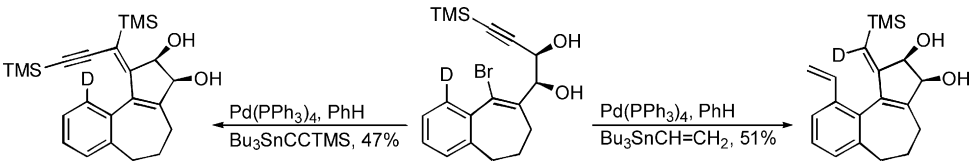


(47)

Palladium catalyzed an oxidative addition–intramolecular alkyne insertion–hydride termination sequence to give chromenes [479] and tricyclic furans [394], or arylboronic acid termination to give 3-(diarylmethylidene)indolin-2-ones [480]. The palladium can migrate to an adjacent ring or undergo alkynylstannane termination to give polycyclic compounds (Eq. (48)) [481] and carbazoles (Eq. (49)) [482]. Palladium catalyzed an oxidative addition–intermolecular alkyne insertion–organoboronic acid termination to give tetrasubstituted alkenes [483]. An oxidative addition–intramolecular

alkyne insertion–organozinc termination sequence was used in a synthesis of 1α,25-dihydroxyvitamin D_3 [484]. The σ-palladium complex formed from an oxidative addition–intramolecular alkyne insertion sequence was terminated with an alkene, an organoboronic acid, hydride reduction or a carbonylation-boronic acid to give substituted 3-alkyliden-indolin-2-ones or -benzofuran-2-ones [485].



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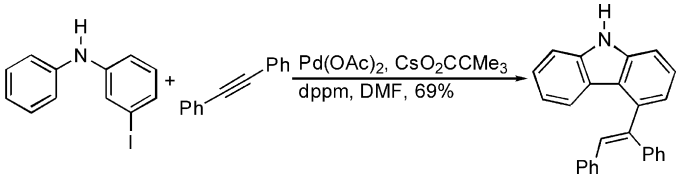
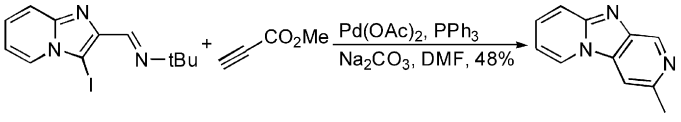


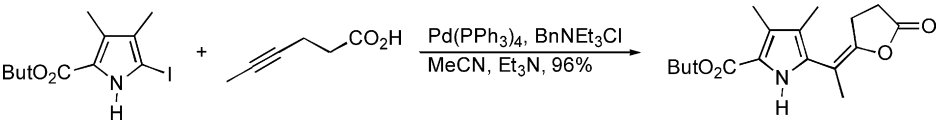
(49)

Palladium catalyzed the formation of isoquinolines from 2-iodobenzaldehyde imines and fluorinated alkynes [486]. A related nickel-catalyzed reaction was reported [487]. Palladium catalyzed the formation of benzothiazines and benzoisothiazoles from *S*-2-bromophenyl-*S*-methylsulfoxime and terminal alkynes [488]. Palladium catalyzed the reaction of 3-haloimidazo[1,2-*a*]pyridine-2-carbaldehyde imine with alkynes to give dipyrido[1,2-*a*;3′,4′-*d*]imidazoles (Eq. (50)) [489].



(50)

Palladium-catalyzed oxidative addition to 2-iodobenzenamine derivatives followed by alkyne insertion and intramolecular amino-palladation producing indoles were used to prepare a variety of alkaloids [370,490,491,373]. Palladium catalyzed the reaction of 2-iodopyrroles with 4-ynoic acids (Eq. (51)) [492]. Rhodium catalyzed an alkyne arylation-1,3-acyl migration sequence using alkyne tethered β-ketoesters (Eq. (52)) [493].



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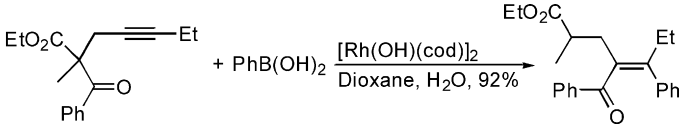
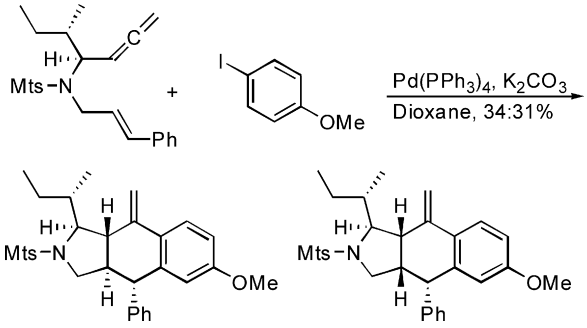


(52)

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, intermolecular allene insertion, and intramolecular "aromatic-Heck" reaction gave tricyclic heterocycles (Eq. (53)) [494]. A four component reaction using a 2-halobenzaldehyde, 1,2-propadiene, an amino acid, and *N*-methylmaleiimide to give polycyclic isoquinolines was described (Eq. (54)) [495]. Palladium–indium catalyzed the formation of fused benzazepines from isoquinolines, 1,2-butadiene, and 2-iodobenzylbromide (Eq. (55)) [496]. Palladium catalyzed an oxidative addition–intermolecular allene insertion–nucleophilic addition sequence using malonitriles as nucleophiles [497]. A related reaction using amines as the nucleophile was also presented [498].



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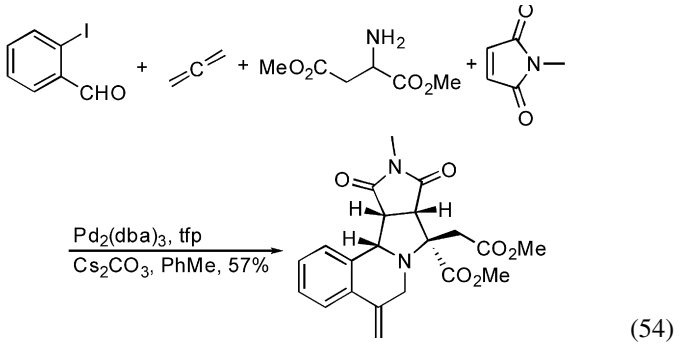

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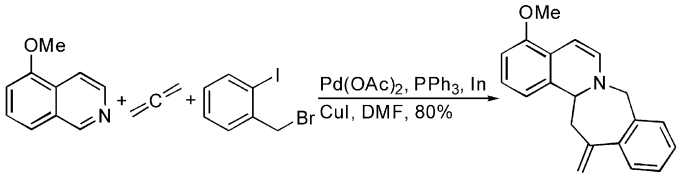
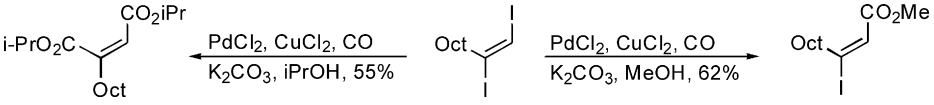


(55)

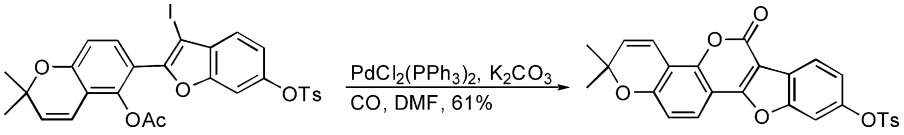
2.6. Carbon–carbon bond formation via insertion of carbon monoxide

A variety of palladium complexes and catalyst systems catalyzed the carbonylation of alkenyl and aryl halides and triflates to afford carboxylic acids and derivatives [394,499]. Palladium catalyzed selective mono-carbomethoxylation of (*E*)-1,2-diiodides. Interestingly, dicarbonylation of the same substrates gave malic acid diesters (Eq. (56)) [500]. Formation of medium-sized lactones, lactams, and thiolactones using a palladium catalyst was reported [501]. Mixtures of 1-bromo-1-fluoro-1-alkenes (*Z*/*E* ≈ 1:1) were transformed into (*Z*)-2-fluoro-2-enoic acid esters and amides having a *Z*/*E* ratio > 98:2 [502].



(56)

The palladium-catalyzed alkoxy-carbonylation of organic halides and triflates was used in synthetic applications toward fumagalone analogs [503], Uhle's ketone and the ergoline skeleton [337], pyrrolizidines [504], pilcadin (Eq. (57)) [505], 1,2-anhydro methyl rocaglate [506], (+)-8-epi-xanthin [507], carbasugars [508] and 261C [509].

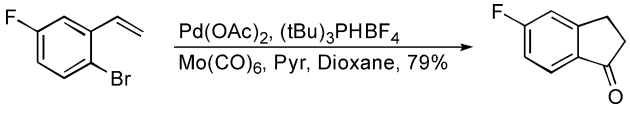


(57)

Cobalt mediated a microwave accelerated formation of ureas from amines and carbon monoxide [510]. A palladium catalyzed amide formation was used in a synthesis of gelsemine [448]. A palladium-catalyzed synthesis of acylsulfonamides from aryl halides and sulfonamides using $Mo(CO)_6$ as the CO source under microwave activation was reported. This reaction was used to prepare a hepatitis C virus NS3 protease inhibitor [511].

Palladium catalyzed the carbonylative coupling of terminal alkynes with aryl halides, in the absence of copper, to give aryl alkynyl ketones [512]. A palladium-catalyzed carbonylative coupling of phenyl bromomethyl sulfoxide with aryl boronic acids to give β-keto sulfoxides was reported [513]. Palladium-catalyzed carbonylative couplings of alkenyl triflates with alkenyl tin reagents were used in synthesis of C(1–4)-linked disaccharides [514]. ^{11}C-Labelled aryl alkyl ketones were prepared from organic iodides, ^{11}C-labeled carbon monoxide and organotin reagents [515]. Copper catalyzed carbonylative cross-couplings of alkynyl iodonium salts with organostannanes in the presence of carbon monoxide [516]. A palladium-catalyzed carbonylative coupling of 3-iodoindoles with a terminal alkyne was used in a synthesis of meridianines [517].

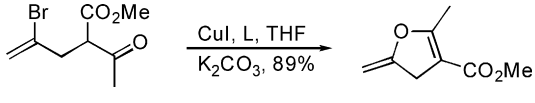
Palladium catalyzed an oxidative addition–carbon monoxide insertion–intramolecular alkene insertion sequence of 2-halo-1-alkenylarenes (Eq. (58)) [518]. Palladium also catalyzed an oxidative addition–carbon monoxide insertion–intermolecular allene insertion–amine termination sequence [519].



(58)

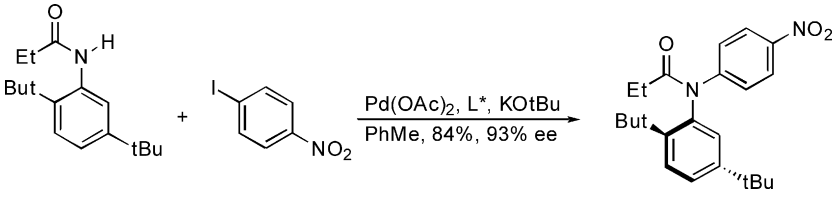
2.7. Carbon–oxygen, –nitrogen, and –sulfur bond-forming reactions

Intramolecular palladium-catalyzed *O*-alkylation using organic halides to give 2-methylchromans was reported [520]. A copper-catalyzed intramolecular *O*-arylation of an enolate with a pendant aryl halide was used to prepare benzofurans [521]. Related reactions of alkenylbromides were reported (Eq. (59)) [522]. Copper catalyzed intermolecular *O*-arylations of aryl iodides [523] and a copper-catalyzed intermolecular reaction was used in synthesis of aspercyclide C [524].



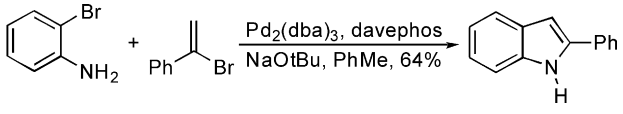
(59)

A variety of amines and amides were coupled with aryl and alkenyl halides using palladium or copper catalysts [1,440,525–527]. Microwave accelerated intermolecular palladium-catalyzed *N*-arylations of amides forming oxindoles were reported [528]. The addition of a small amount of water was found beneficial in some amidation reactions [529]. Palladium catalyzed the intermolecular *N*-arylation or *N*-alkenylation of primary and secondary amines [530,531], polyamines [532], imidazoles [533], benzophenone hydrazine [376], *N*,*N*-dialkylhydrazines [534], sulfoximines [535,536] in a synthesis of erogorgiaene [350], sulfamides [537], pyrroles and indoles [538], aziridines [539], zinc trimethylsilylamide, as an ammonia equivalent [540], of ^{15}N-labelled benzamide [541], ureas [542] and linear polyamines [543] using aryl halides or alkenyl triflates. This type of reaction using a 3,5-dibromopyridine and aza-substituted 1,*n*-diamines was used to prepare polyazamacrocycles [544]. An asymmetric intermolecular reaction leading to optically active anilides was reported (Eq. (60)) [545].



(60)

Palladium catalyzed the *N*-arylation using 6-bromoazulenes [546], 2-chloro-2′-deoxyinosine [180], heteroaryl halides [547] and 7-bromo-pyrido[2,3-*b*]pyrazine [185] as the electrophile. Palladium catalyzed the *N*-alkenylation of amides using alkenyl tosylates [548]. An intermolecular amidation-intramolecular Heck reaction of 2-halobenzenamines and alkenylhalides to give indoles was described (Eq. (61)) [549]. Palladium catalyzed a sequential *N*-arylation-carboamination of 1-amino-4-enes (Eq. (62)) [550].



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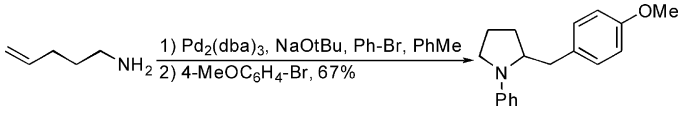
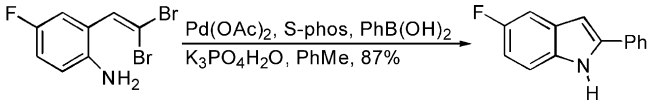


(62)

Intermolecular carbon-nitrogen couplings were used in synthesis of (−)-2-fluoronorapomorphine [551], myrmicarin alkaloids [552] and trichostatin D [553]. A double *N*-arylation of 2,2′-biphenylene triflates with primary amines to give carbazoles was reported and this reaction was used in a synthesis of mukonine [213]. Related reactions were used to prepare hetero[7]helicenes [554], indoles [555], murrastifoline A [206] and anhydrolycorine, hippadine, oxoassonine, and pratosine [556].

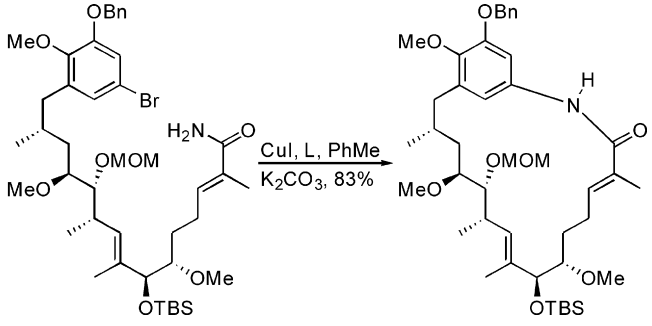
Intramolecular palladium-catalyzed carbon-nitrogen coupling reactions were used in synthesis of 1-aryl-1*H*-indazoles [557], pyrrolo[1,2-*a*]quinoxalines and indolo[1,2-*a*]quinoxalines [558] and tarpane [559]. A combination of copper- and palladium-catalyzed reactions was used to prepare of *N*,2-disubstituted indoles from 2-(2,2-dihaloethenyl)aminobenzenes and boronic acids (Eq. (63)) [560]. Indoles were prepared by palladium-catalyzed reaction of 1-(1-alkynyl)-2-chloroarenes with primary amines [561].



(63)

Transition metals other than palladium, mainly copper, also catalyzed carbon–nitrogen bond forming reactions of amides and amines. A new iridium catalyst was reported [562]. Nickel catalyzed *N*-arylation of aryl diamines using aryl chlorides [563]. Copper catalyzed *N*-arylations of amines and nitrogen heterocycles in the presence of an amino acid [564], an amino-phosphonate [565] or an amine [566]. Copper catalyzed *N*-arylations of sulfonamides [567]. Copper catalyzed intermolecular *N*-arylations of *N*-heterocycles using aromatic chlorides and fluorides [568], for example, N^6-arylation of 2-deoxyadenosine [569]. Copper-catalyzed intermolecular *N*-alkenylation of an amine was used in synthesis of (+)-crocacin D [51].

Copper-catalyzed intramolecular *N*-arylations leading to medium- and large-sized nitrogen heterocycles were described [570]. Copper catalyzed intermolecular *N*-alkenylations of amides with alkenyl iodides [571] used in a synthesis of lobatamide analogs [52] and *N*-allenylation of amides, carbamates, and ureas using allenyl halides [572,573]. Copper catalyzed intramolecular couplings of amides with aryl halides [574] and this type of reaction was used in a synthesis of reblastatin (Eq. (64)) [575].



(64)

Palladium catalyzed *S*-arylation and *S*-alkenylation of thiols derived from cystein to form thioethers [576]. Palladium catalyzed the coupling of benzyl carbonates with sulfinic acid salts [577]. Nickel catalyzed the coupling of aryl halides with thiols [578]. Alkyl bromides were coupled with disulfides or diselenides in the presence of a rhodium catalyst and hydrogen gas to give unsymmetrical sulfides or selenides [579]. Copper catalyzed *S*-arylations of sulfinic acid salts to give aryl sulfones [580].

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

Palladium-catalyzed reactions of hexamethylditin with aryl halides and alkenyl triflates to give aryl and alkenyl tin reagents [155,581] were employed in the synthesis of heterocyclic core of GE 2270 [34], core of roseophilin [21], frondosin C ring system [582], phorboxazole A analogs [71] and functionalized azaindoles [583].

Palladium catalyzed the cross-coupling of aryl iodides and alkenyl triflates with dipinacolyldiborane, pinacolborane, and bis(neopentyl glycato)diboron producing aryl- and alkenylpinacolyl boronic esters [156,168,581,584,585] and this type of reaction was used in synthesis of isocomplestatin [244], diazonamide A [221], dityrosine, trityrosine, pulcherosine [222], biphenomycin B [246], spirofungins [228], toward apoptolidin [234] and CB92834 [237]. *N*,*N*-Diisopropylaminoboranes can also be used as the boron source [586].

Palladium mediated the formation of a 3,4-diphosphonate from a 3,4-diiodothiophene and triethylphosphite [587]. A palladium catalyzed transformation of an aryl bromide to an aryl phosphonate was used in a synthesis of (*S*)-APICA [588]. Copper catalyzed the coupling of alkenyliodonium salts with phosphonates [589].

Palladium catalyzed the formation of aryl silanes using aryl halides and trisubstituted silanes [590]. Palladium catalyzed arsination of aryl triflates with triphenylarsine [591].

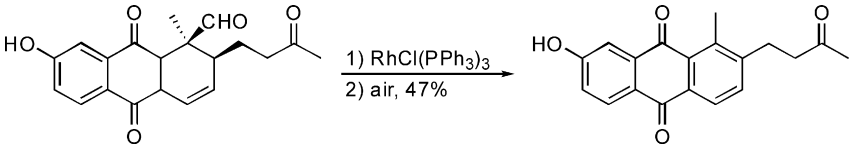
2.9. Carbon–hydrogen bond-forming reactions (including decarbonylation of aldehydes)

A variety of functional groups were replaced by a hydrogen atom using palladium-catalyzed methodologies. Palladium complexes, together with a hydride source, such as tributyltin hydride, triethylsilane, and triethylamine–formic acid catalyzed the reduction organic bromides, iodides, and triflates. Synthetic applications include, 1,8-naphthyrine C-nucleosides [592], cylindramine [324], azadirachtin [593], dihydroxerulic and xerulinic acid [38], bergapten [594], 3-methoxyelipticine and elipticine [595], (−)-scabronine G [44], (−)-galanthamine and (−)-morphine [447], toward (+)-sorangicin [268], 2-fluoro-11-hydroxy-*N*-propylnoraporphine [596], hetsine alkaloids [597], pancracine [430] and the DEF-rings of FR182877 [598].

Monodebromination of 1,1-dibromo-1-alkenes was achieved using tributyltin hydride in the presence of a palladium catalyst forming *cis*-bromoalkenes and this type of reaction was used in the synthesis of combretastatin analogs [231] and toward herbimycin A [79].

A palladium-catalyzed reductive ring-opening of an alkynyloxirane was used in a synthesis of the EF-ring segment of ciguatoxin CTX1B [599].

Wilkinson's catalyst was used in decarbonylations of aldehydes in synthesis of antimalarial naphthoquinones (Eq. (65)) [600] and (−)-physovenine and (−)-3a-hydroxyfuroindoline [601].

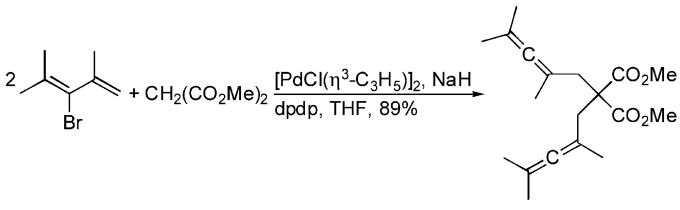


(65)

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. Allylic alkylations of stabilized carbon nucleophiles, and amides using 2-bromo- or 2-trifloxy-1,3-dienes to give allenic products were described (Eq. (66)) [602–604]. This reaction was used in a synthesis of the pheromone (*E*)-tetradeca-2,4,5-trienoate [605]. The ratio of S_N2- to S_N2'-substitution of bicyclic dienylacetates could be affected by the choice of phosphine ligand for the palladium-catalyst [606]. Di- and sesterpenes were prepared using an allylation of a trimethylsilylenol ether [607]. Palladium catalyzed allylic alkylation using allylic alcohols and meldrum's acid [608]. C-3 selective allylation of indoles using allyl alcohols in the presence of triethylborane was reported (Eq. (67)) [609].



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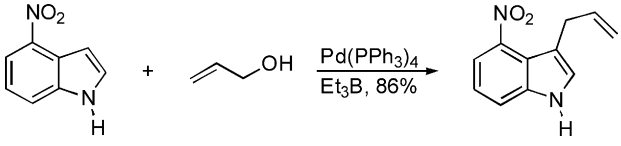

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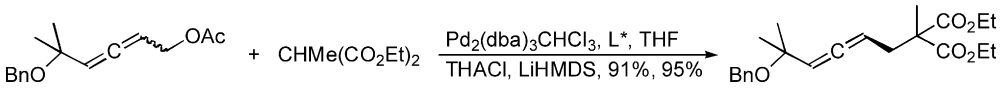

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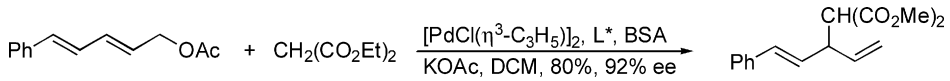
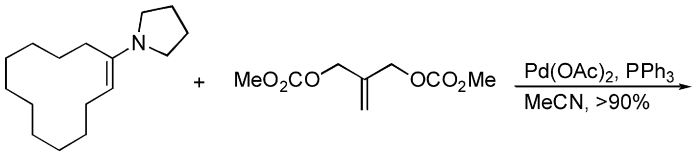


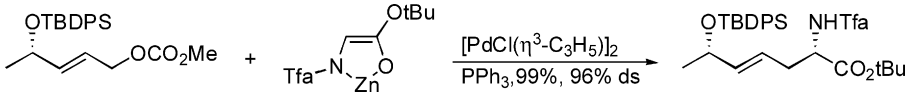
(73)

Palladium-catalyzed allylic alkylations were used in synthetic applications toward tremulenediol A and tremulenolide A [610]. A double allylic alkylation was used in a synthesis of roseophilin (Eq. (68)) [611].



(68)

Palladium catalyzed enantioselective allylic alkylation of allyl-enol carbonates derived from ketones [612,613]. A palladium-catalyzed asymmetric alkylation of 3-aryloxindoles with allyl acetate was developed [614]. 1,5-Chirality transfer was observed in palladium-catalyzed allylic alkylations using chelated amino ester enolates (Eq. (69)) [615]. Palladium-catalyzed asymmetric decarboxylative allylation of β-keto esters (Eq. (70)) [616] and β-fluoro-β-ketoesters [617]. Palladium catalyzed a decarboxylative reaction of allyl alkynoates to give 1,4-enynes (Eq. (71)) [618]. Ruthenium also catalyzed a stereospecific decarboxylative allylation of allyl β-keto esters [619].



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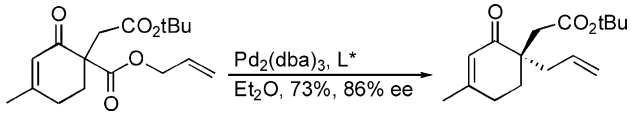

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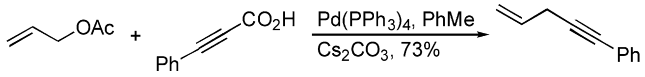
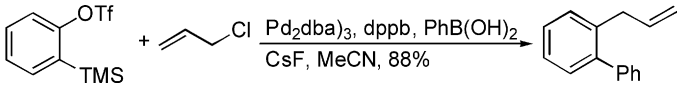


(71)

Palladium-catalyzed asymmetric α-allylation of ketone enolates [620,621] and this type of reaction was used in synthesis of hamigeran B [202] and (+)-allocyathine B_2 [128]. Palladium catalyzed dynamic kinetic asymmetric allylic alkylations of allenyl acetates with carbon nucleophiles (Eq. (72)) [622]. A regioselective asymmetric allylation of dimethyl malonate or benzylamine with 1-acetoxy-2,4-dienes, adding the nucleophile to the 3-position, was reported (Eq. (73)) [623].

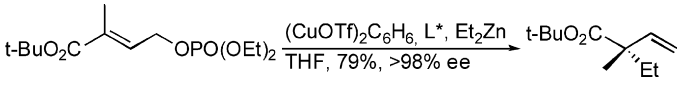
A palladium-catalyzed coupling of an allylic bromide with an alkenyltin reagent was used in a synthesis of (+)-dactylolide [624]. Palladium catalyzed the coupling of Baylis–Hillman acetates with organosilanes [625]. Palladium-catalyzed couplings of alkynyl zinc reagents with allylic chlorides, bromides, and acetates [626]. Palladium catalyzed a three-component coupling of benzynes, allylic halides and organo-boron, -silicon, and -tin reagents (Eq. (74)) [627,628].



(74)

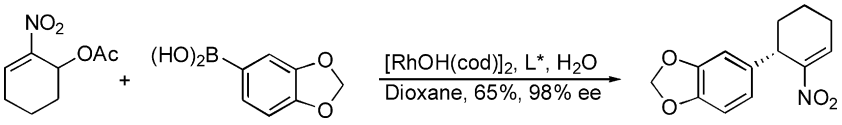
Palladium catalyzed an arylation of acetylated glycals using arylboronic acids. Ring-opened arylated products were observed as side-products [629]. Palladium catalyzed the reaction of allylic oxiranes and aziridines with aromatic boron reagents to give allylic alcohols and amines, respectively [630]. Related reactions of alkynyl oxiranes with arylboronic acids gave 1-aryl-1,2-dien-4-ols [631].

Copper catalyzed desymmetrizations of cyclic meso allylic bisphosphonates using alkylzinc reagents [632]. Copper catalyzed asymmetric alkylations of γ-phosphate-α,β-unsaturated esters in the α-position using dialkylzinc reagents (Eq. (75)) [633]. Iridium catalyzed asymmetric inter- and intramolecular allylic alkylations using allylic carbonates [634]. Iridium catalyzed regio- and enantioselective allylic alkylations of ketone enolates was also reported [635].



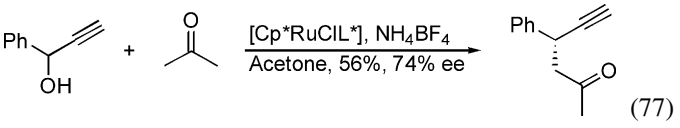
(75)

An asymmetric rhodium-catalyzed allylation of an arylboronic acid was used in a synthesis of (+)-γ-lycorane (Eq. (76)) [636]. A rhodium-catalyzed allylic alkylation was used in a synthesis of tremulenediol A and tremulenolide A [610].



(76)

Ruthenium catalyzed an asymmetric direct propargylation of acetone with propargylic alcohols (Eq. (77)) [637]. Either a 4-yne-1-one or a 2,4-diene-1-one can be formed depending on the ruthenium catalyst [638]. Ruthenium catalyzed a decarboxylative Michael addition–allylation of allyl β-ketoesters (Eq. (78)) [639]. Gold catalyzed propargylic substitution reactions of propargyl alcohols employing carbon, oxygen, or nitrogen nucleophiles (Eq. (79)) [640].



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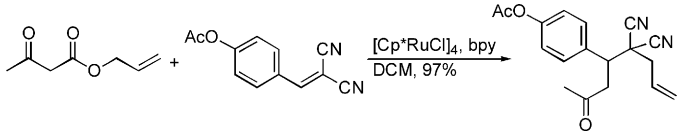

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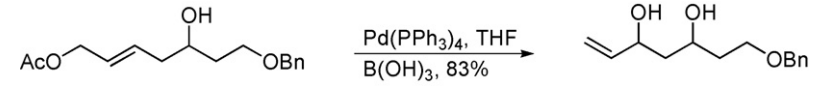

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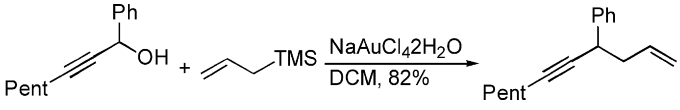
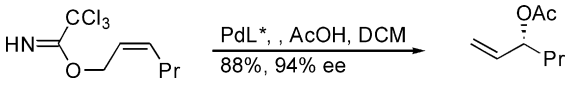


(79)

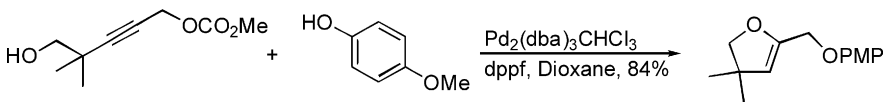
3.2. Carbon–heteroatom bond-forming reactions using heteroatom nucleophiles

Allylation of sulfur, oxygen, nitrogen, and carbon nucleophiles in water using allylic acetates in the presence of palladium on carbon was reported [641]. A palladium-catalyzed asymmetric synthesis of allylic esters from (*Z*)-allylic trichloroacetimidates was developed (Eq. (80)) [642]. Palladium catalyzed a dynamic kinetic resolution of racemic allylic carbonates to give allylic alcohols [643]. Regioselective hydroxylation and alkoxylation of hydroxy-functionalized allylic acetates and carbonates using boric acid or esters was described (Eq. (81)) [644].



(80)

Palladium-catalyzed asymmetric cyclizations of benzene-1,2-diol and secondary propargylic carbonates to give 2-alkylidene-2,3-dihydro-1,4-benzodioxanes [645]. Monocarbonates from 2-yne-1,4-diol were reacted with alkoxides and stabilized methylene compounds to form 2,3-dihydrofurans and benzofurans (Eq. (82)) [646]. A palladium-catalyzed reaction of alkynyl oxiranes with phenols to give phenoxy-substituted alkenyl oxiranes was reported (Eq. (83)) [647]. An intermolecular allylation of an alcohol using an allylic phosphonate was employed in a synthesis of pyranicin [340]. Palladium catalyzed *O*-allylation and *N*-allylation of oximes using allylic acetates and carbonates. The product formed depended on the catalyst used (Eqs. (84) and (85)) [648].



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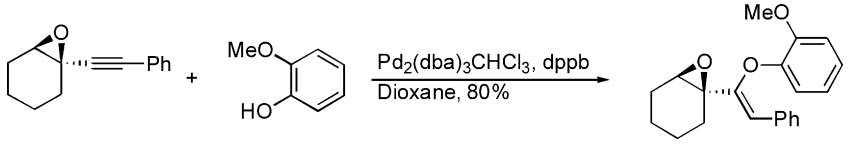

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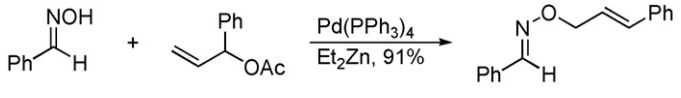

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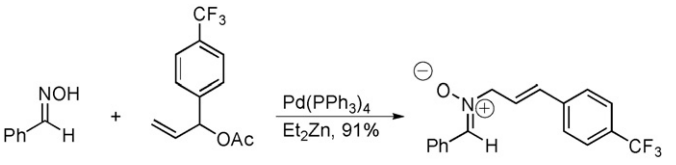
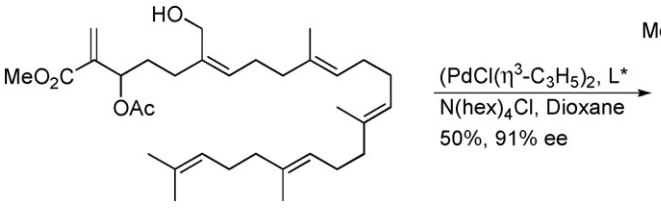


(85)

Palladium-catalyzed asymmetric allylation of alkoxides or alcohols were used in the synthesis of (−)-galanthamine and (−)-morphine [447], ustiloxins A and B [649] and orbicuside A analogues [650]. A dynamic kinetic asymmetric transformation of an ethenyloxirane with 4-methoxybenzylacohol was used in a synthesis toward amphidinolide B1 [651].

Palladium-catalyzed allylation of an alcohol was used in the synthesis of heliannuols G and H [652], (+)-hippospongic acid A (Eq. (86)) [653], C22–C36 subunit of halichondrin B [654] and anamarine [655], (−)-laulimalide (Eq. (87)) [656] and daumone [657].



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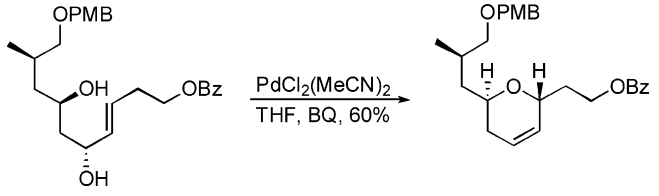
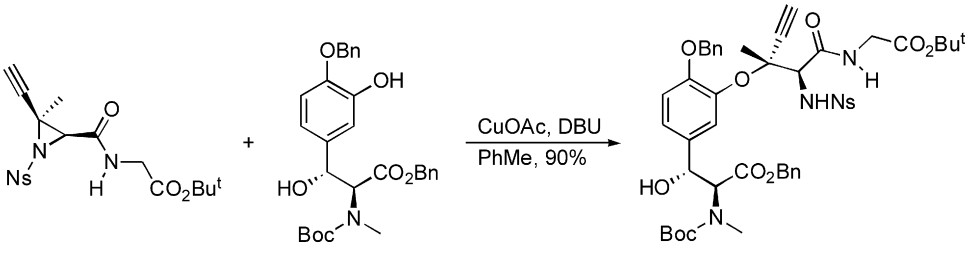


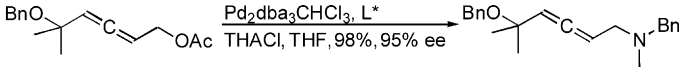
(87)

Iridium catalyzed inter- and intramolecular *O*-allylations of alcohols with allylic carbonates [658]. Iridium catalyzed enantioselective *O*-allylations of hydroxylamines using allylic carbonates [659]. Iridium catalyzed asymmetric allylations of C-, N-, and O-nucleophiles affording selectively the more substituted product [660–662]. Ruthenium catalyzed propargylic alkoxylation, amination, and phosphorylation of 1-yne-3-ols using alcohols, amines, amides and diarylphosphineoxides, respectively [663]. Ruthenium catalyzed the direct formation of allylic ethers from an allylic alcohol and a second alcohol [664]. A copper-catalyzed alkynyl aziridine ring opening with phenols was used in a synthesis of ustiloxin D (Eq. (88)) [665].



(88)

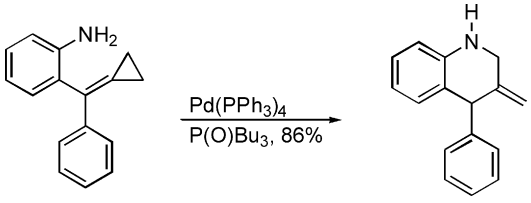
A number of reports dealing with allylation of nitrogen nucleophiles appeared in the literature. Palladium catalyzed dynamic kinetic asymmetric *N*-allylations of allenyl acetates with nitrogen nucleophiles (Eq. (89)) [622]. Palladium catalyzed *N*-allylations of aziridines and the mechanism was examined [666]. Palladium catalyzed *N*-allylation using allylic alcohols [667]. A palladium-catalyzed decarboxylative amination of allyl carbamates was described [668].



(89)

Palladium-catalyzed *N*-allylations were used to prepare novel carbocyclic nucleoside analogs [669], a polycyclic pyrazolo[3,4-*d*]pyrimidine [670], deoxymannojirimycin [671], (−)-norsecurine [672], gizzerosine [136] and tetrahydroisoquinolines [673].

A palladium-catalyzed asymmetric reaction of 2,3-dienyl-1-phosphates with nitrogen and oxygen nucleophiles was reported [674]. Palladium catalyzed both inter- and intramolecular *N*-allylations of amines using alkylidene cyclopropanes (Eq. (90)) [675]. Intramolecular palladium-catalyzed *N*-allylations of amino-tethered allylic alcohols to give 3-methylene-piperidines were reported (Eq. (91)) [676]. An interesting palladium-catalyzed aziridine ring-opening intramolecular amination reaction was examined as an approach toward nufar alkaloids [677].



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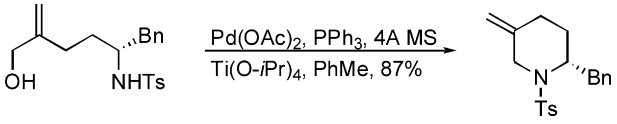
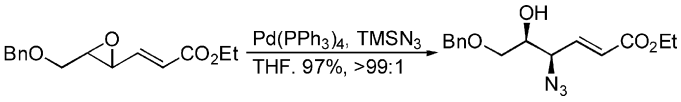


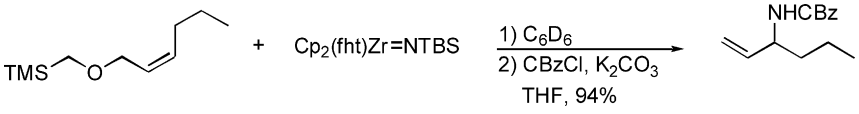
(91)

Palladium catalyzed the azidation of electron deficient alkenyloxiranes (Eq. (92)) [678]. Sodium azide was used as the nucleophile in the preparation of azidosugars [679]. Iridium catalyzed *N*-allylations of hydrazines [680]. An iridium catalyzed *N*-allylation was used in a synthesis of (+)- and (−)-nicotine [681]. Rhenium catalyzed *N*-propargylation of amines with propargylic alcohols [682].



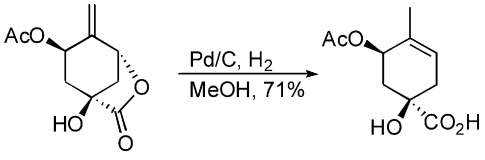
(92)

Palladium catalyzed *S*-allylations of arenethiols [683] and of sodium phenylsulfinate [684] with allylic alcohols. An S_N2' reaction of allylic trimethylsilylmethyl ethers using zirconocene imido complexes as the nucleophile was reported (Eq. (93)) [685].



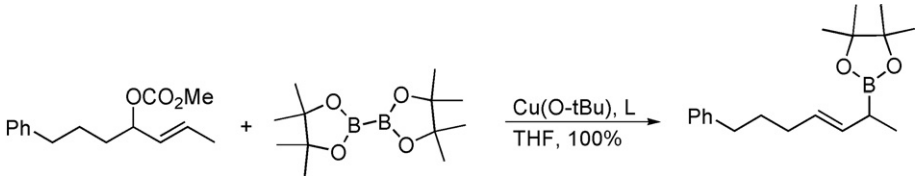
(93)

A palladium-catalyzed regioselective reduction of an allylic epoxide using formic acid and triethyl amine was used to prepare stereoidal taxoid mimics [686]. Palladium-catalyzed reductive isomerizations of allylic carbonates (Eq. (94)) [687]. A palladium-catalyzed formation of a 1,3-diene from an allylic benzoate was used in a synthesis of tuberostemonines [688]. Palladium catalyzed the transformation of 1-aryl-3-*N*,*N*-dialkyl-1-propynes to 1-aryl-1,2-propadienes [306,689].



(94)

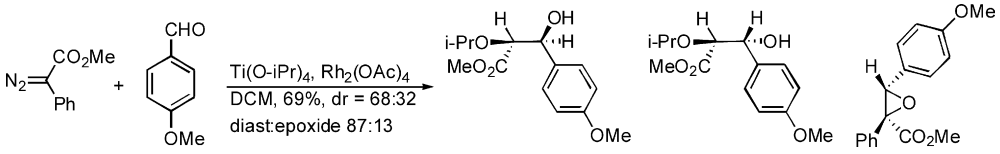
Palladium-catalyzed coupling of allylic bromides with trimethylstannylphenylselenide to give allylic phenylselenides [22]. A related formation of propagylic phenylselenides from propargylic bromides and trimethylstannylphenylselenide was described [22]. Palladium catalyzed the formation of propargyl and allenyl stannanes and silicon compounds from propargylic chlorides and epoxides and hexamethylditin or trimethylsilyl–trialkyltin [690]. Palladium catalyzed the reaction of disilanes and digermanes with carbomethoxy substituted allylic actetates, derived from a Baylis–Hillman reaction, affording carbomethoxy-substituted allylic silanes and germanes [691]. Palladium catalyzed the formation of an allylic boronate from an allylic acetate and bis(pinacolato)diboron [243]. Copper catalyzed a γ-selective and stereospecific allylic borination of allylic carbonates (Eq. (95)) [692].



(95)

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

Rhodium catalyzed the reaction of diazoacetates with aldehydes in the presence of alkoxides to give α-alkoxy-β-hydroxy acid derivatives (Eq. (96)) [693]. Related reactions between aryl diazoacetates, alcohols, and aldehydes to give aldol-like products were reported [694]. Rhodium catalyzed the methylenation of aldehydes using trimethylsilyldiazomethane [695,696]. Tertiary formamides were alkenylated with silylated diazoesters (Eq. (97)) [697]. Rhodium catalyzed diazo-decomposition alkenylation was used in a synthesis toward rubromycins (Eq. (98)) [698]. Palladium catalyzed the reaction of benzylic bromides with ethyl diazoacetate to give 3-aryl-propenoates [699].



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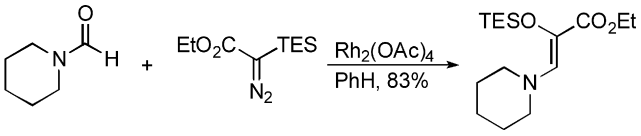

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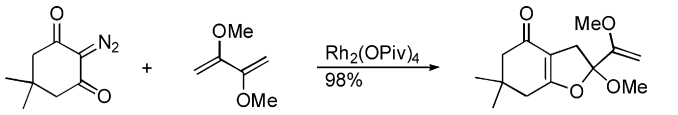

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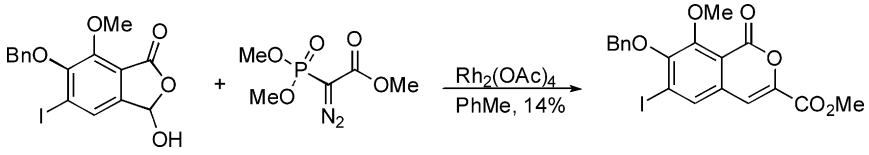
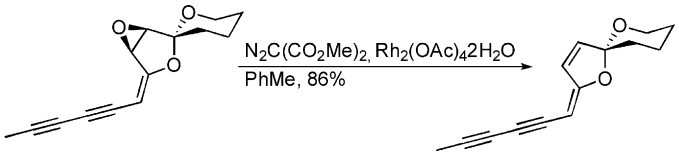


(98)

A rhodium-catalyzed oxirane deoxygenation using dimethyl diazomalonate was used in a synthesis of diacetylenic spiroacetal enol ethers (Eq. (99)) [333]. Rhodium catalyzed the reaction of trimethylsilyldiazomethane with alkynylboronates to give 1,4-silyl-2-boryl-1,3-dienes [700]. Rhodium catalyzed the formation of aryl α-imino esters from aryl diazoacetates and an amine (Eq. (100)) [701]. Rhodium catalyzed *O*-alkylation of sulfonimidates with diazocompounds (Eq. (101)) [702].



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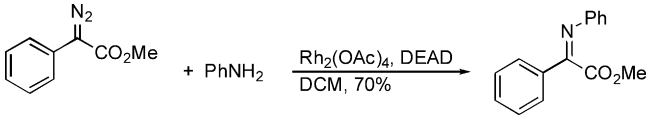

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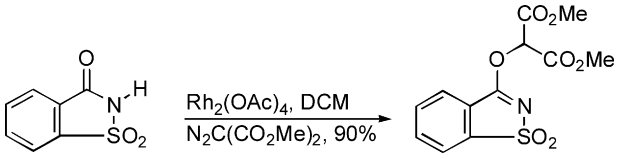
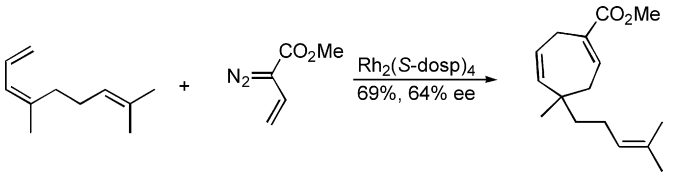


(101)

Rhodium catalyzed [3 + 2] and [3 + 4] cycloadditions of diazodicarbonyl compounds and conjugated dienes (Eq. (102)) [703]. This type of reaction was used in a synthesis of 5-epi-10-epi-vibsanin E (Eq. (103)) [704]. Rhodium catalyzed a reaction of 1,6- and 1,7-enynes with trimethylsilyldiazomethane to give bicyclic compounds having a ethenylsilane substituent (Eq. (104)) [705].



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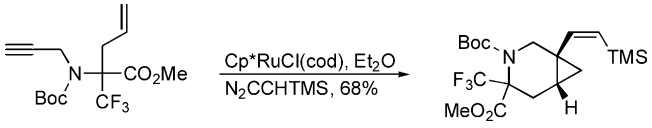
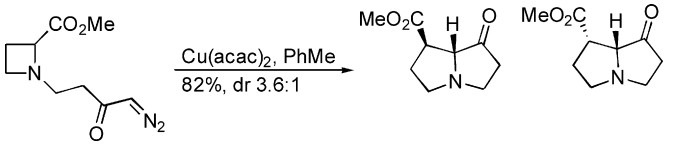


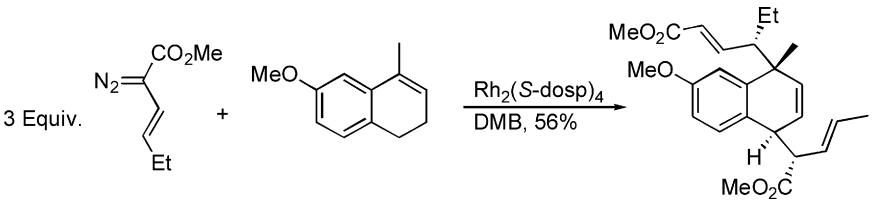
(104)

A 1,2-thio-group migration of β-thioether α-diazoesters was observed upon decomposition of diazo compounds [706,707]. Related 1,2-migrations of alkenyl- and alkynyl-groups were described [708]. A copper catalyzed diazo decomposition—1,2-shift was used in synthesis of turneforcidine and platynecine (Eq. (105)) [709].



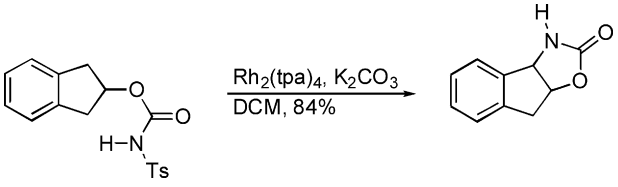
(105)

Rhodium-catalyzed intramolecular carbon–hydrogen bond insertions using water as the solvent [710]. Asymmetric intermolecular carbon–hydrogen bond insertions of oxygen-bonded carbons were described [711,712]. Rhodium catalyzed asymmetric intramolecular carbon–hydrogen insertions [713]. Rhodium catalyzed an intermolecular enantioselective double carbon–hydrogen bond insertion of dihydronaphthalenes (Eq. (106)) [714] and this type of reaction was used in a synthesis of (+)-erogorgiaene [715]. Rhodium-catalyzed decomposition of 4*Z*-β-alkenyl-α-diazo-β-ketoesters to give five- or six-membered rings [716].



(106)

Rhodium-catalyzed intramolecular carbon–hydrogen bond insertions of carbamates to form five-membered heterocycles, in the presence of $PhI(OAc)_2$ as the oxidant, and this type of reaction was used in synthesis of L-daunosamine, D-saccharosamine, and L-rhistosamine [717]. Related intramolecular reactions of sulfamates to give oxathiazines [99] and carbon–hydrogen bond insertions and aziridinations of *N*-tosyloxycarbamates (Eq. (107)) [718] were reported. Copper catalyzed the carbon-hydrogen bond insertion using chloramine T hydrate [719].



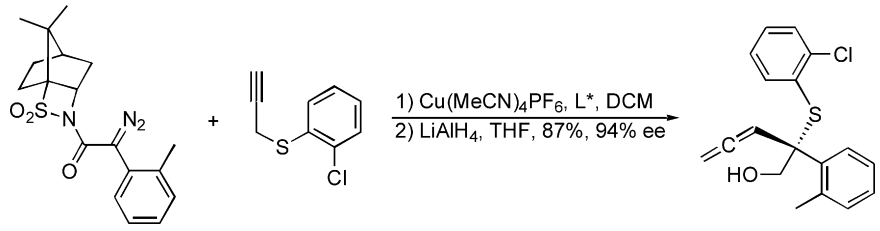
(107)

Intramolecular rhodium-catalyzed carbon-hydrogen bond insertions via decomposition of diazo compounds forming four to six membered rings were used in a number of synthetic applications, for example, toward (+)-sulcatine G [720], β- and γ-lactams [721], baclophen and GABOB [722], (+)-β-herbertenol [723], 9-isocyanoneopupukeanane [724], (−)-rolipram [725], toward cyperanes [726], chiral γ-lactams [727], viridenomycin [728] and 3,6-epoxy-4,6,8-triethyl-2,4,9-dodecatrienoate [729]. An intermolecular rhodium-catalyzed aromatic carbon-hydrogen bond insertion was used in a synthesis of derrusnin [730].

The carbenoids derived from rhodium-catalyzed decomposition of diazo compounds insert into nitrogen–hydrogen bonds. Intermolecular amide nitrogen–hydrogen bond insertions were used in approaches to martefragine [731] and diazonamide A [75,221]. Intramolecular nitrogen–hydrogen bond insertions to give *cis*-2,4-disubstituted azetidin-3-ones [732] and (−)-*cis*-carboxyazetidine-3-acetic acid [733] were reported.

A chiral tethered controlled an intramolecular asymmetric rhodium-catalyzed oxygen–hydrogen bond insertion [734]. Rhodium-catalyzed oxygen–hydrogen bond insertions were used in synthesis of (+)-zoapatanol [247], (−)-dihydroxanthatin [735] and (−)-xeniolide F [736].

A highly stereoselective copper-catalyzed sulfonium ylide formation–[2,3]-sigmatropic rearrangement was described (Eq. (108)) [737]. [2,3]-Sigmatropic rearrangements of spirocyclic ammonium ylides were described [738]. Rhodium catalyzed a diastereoselective sulfur ylide formation–[3,3]-sigmatropic rearrangement (Eq. (109)) [739].



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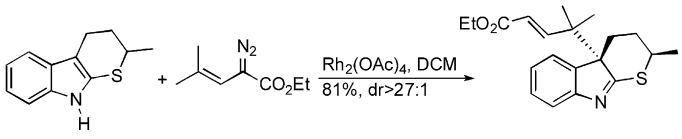
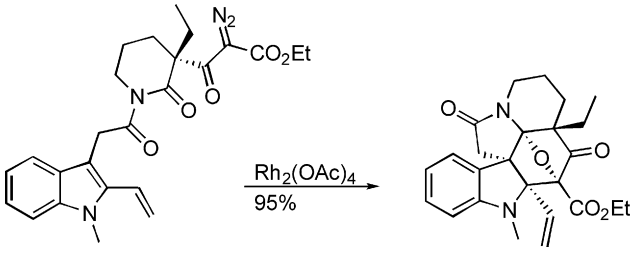


(109)

A variety of cycloaddition reactions of carbonyl ylides formed by rhodium-catalyzed decomposition of α-diazocarbonyl compounds were described [440], for example, enantioselective [3 + 2]cycloadditions with alkenes [740], reactions with imines [741] and methylenecyclopropanes [742]. Intramolecular carbonyl ylide-alkene cycloadditions were used to prepare polycyclic indoles [743], the oxatricyclo[6.3.1.$0^{o,o}$]dodecane core of komaroviquinone [744] and (−)-colchicine [745]. Intermolecular carbonyl ylide-alkene [3 + 2]cycloadditions were used to prepare polycyclic compounds (Eq. (110)) [12].

A diastereoselective addition of oxygen-, nitrogen- and sulfur-nucleophiles to carbonyl ylides derived from diazo compounds was reported [746]. Rhodium catalyzed an asymmetric synthesis of pyrrolines from a diazoketone, an imine, and an alkyne (Eq. (111)) [747].



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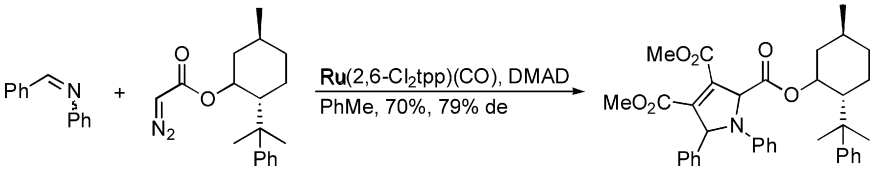
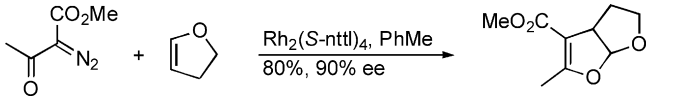


(111)

Cyclopropanations of alkenes via transition metal-catalyzed decomposition of diazo compounds continued to be developed. Rhodium-catalyzed intermolecular enantioselective cyclopropenations of alkynes [748] and of 3-ethenylindoles [749]. A highly *cis*- and enantioselective asymmetric intermolecular cyclopropanation catalyzed by ruthenium was reported [750].

Rhodium catalyzed an asymmetric intermolecular cyclopropanation of enol ethers followed by rearrangement to give dihydrofurans (Eq. (112)) [751]. Rhodium-catalyzed enantioselective formation of substituted cycloheptadienes from alkenyl diazoacetates and 3-substituted-1-alkoxy-1,3-dienes via a cyclopropanation-Cope rearrangement (Eq. (113)) [752]. Silver catalyzed the reaction of ethyldiazoethanoate with aromatic compounds forming cycloheptatrienes [753]. A related copper-catalyzed ring-expansion of benzopyrylium triflates was reported affording 2,3-benzooxepins [754].



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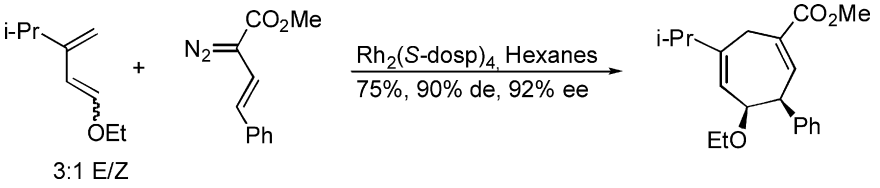
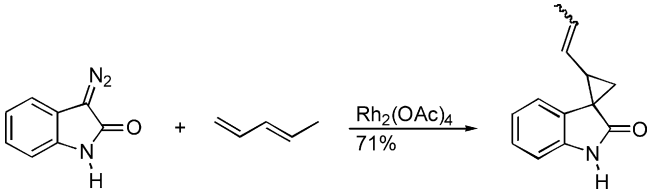


(113)

A rhodium-catalyzed intramolecular cyclopropanation was used in a synthesis of tremulenediol A and tremulenolide A [610] and an intermolecular variation was used in a synthesis of (−)-spirotryprostatin B (Eq. (114)) [755].

A copper-catalyzed intermolecular cyclopropanation was used in a synthesis of (−)-malyngolide (Eq. (115)) [756]. Copper catalyzed intermolecular cyclopropanation of arylalkenes and alkenylethers using tributylstannyl diazoacetate esters [757]. Copper also catalyzed cyclopropanations of furans [758].

Copper catalyzed intramolecular cyclopropanations were used in synthesis of (−)-microbiotol and (+)-β-microbiotene [759].



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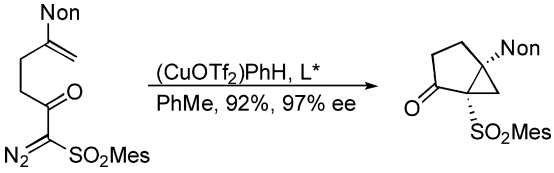
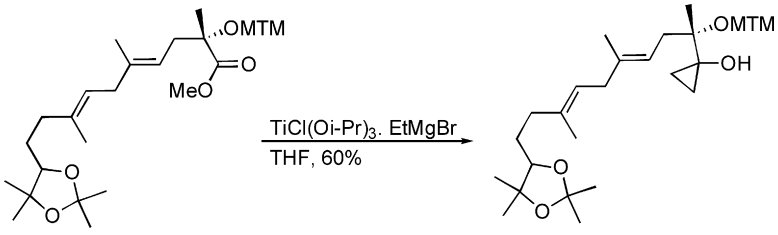


(115)

The Kulinkovich–de Meijere cyclopropanation of alkenes using esters and amides mediated by titanium and in the presence of a Grignard reagent was further developed [760]. This type of reaction was used in a synthesis of isoedunol and β-arosene (Eq. (116)) [761]. Titanium mediated intramolecular cyclopropanations of alkene tethered amides [762]. A titanium-mediated cyclopropanation of nitriles with Grignard reagents to give amino-substituted cyclopropanes was described [763]. Cyclopropanation of alkenes was reported using 2-(1-alkyne-1-yl)-2-(trialkylsilyl)-1,3-dithianes and a titanium catalyst (Eq. (117)) [764]. Titanium mediated the formation of spirocyclopropanated amides from nitrile esters (Eq. (118)) [765]. Titanium mediated the formation of cyclopropanes from thioacetals and alkenes [766].



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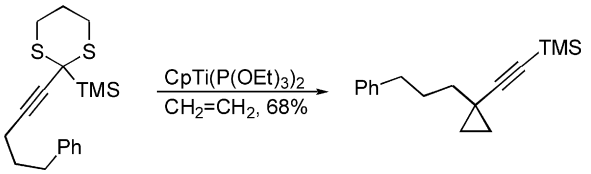

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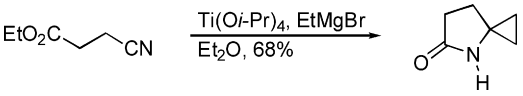
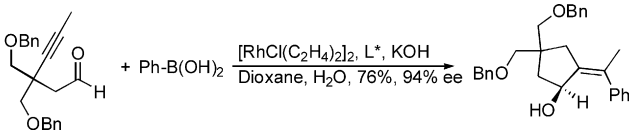


(118)

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

5.1. Formation of carbon–carbon bonds from alkene and alkynes

A rhodium-catalyzed oxidative addition of arylboronic acids to 2-propen-1-yl ethers to give 3-aryl-2-propenyl ethers was described [767] and used in a synthesis of rosavin [768]. Rhodium catalyzed an asymmetric arylation-cyclization of 5- and 6-yne-als using arylboronic acids to give five- and six-membered rings (Eq. (119)) [769]. Rhodium catalyzed the formation of indenones and indanones from 2-cyanophenylboronic acids and alkynes or alkenes (Eq. (120)) [770].



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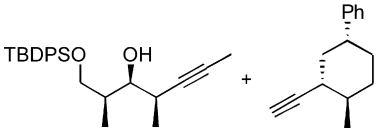

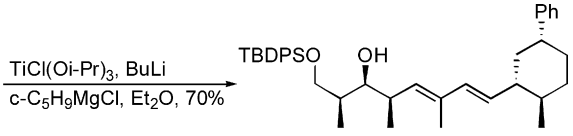

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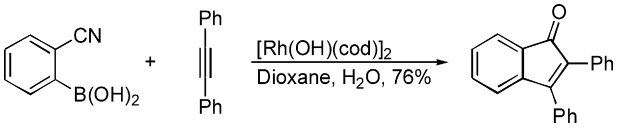
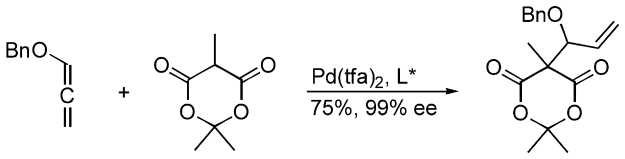


(120)

Palladium catalyzed 1,2-diarylations of internal alkynes using arylboronic acids to give *cis*-1,2-diarylated alkenes [771]. Palladium-catalyzed intermolecular hydroarylations of internal alkynes using aryl- and alkenyl-boron reagents [772] and asymmetric hydroarylations of activated alkenes using triarylbismuth, arylbismuth, and arylsilicon compounds [773].

Rhodium catalyzed a cyclization of 2-(carbonylfunctionalized)arylboronic acid with alkynes to give indenols [774,775]. Platinum-catalyzed intermolecular hydroarylations of 2-ynoates [776].

Palladium-catalyzed asymmetric intermolecular hydroalkylations of 1-benzyloxy-1,2-propadiene using 1,3-dicarbonyl compounds (Eq. (121)) [777]. Silver catalyzed selective Markovikov hydroalkylations of phenylethene using activated methylene compounds [778]. Gold catalyzed intermolecular hydroalkylations using activated methylene compounds and dienes, trienens, or cyclic enol, alkenes [779].



(121)

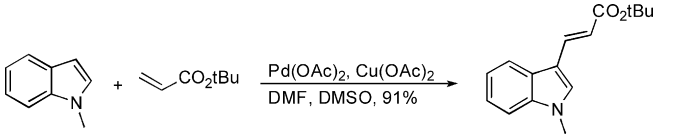
An asymmetric palladium-catalyzed hydroalkenylation of styrenes was described [780]. An iridium-catalyzed hydroalkenylation of ethene was reported and experimental and theoretical evidence supports an alkene carbon–hydrogen bond activation–insertion into a iridium-alkenyl intermediate [781].

Ruthenium catalyzed hydroalkynylation of arylalkynes with silylalkynes forming 5-silyl-substituted-1-aryl-1,3-enynes [782]. Ruthenium catalyzed a hydroalkynylation-dimerization of terminal alkynes in the presence of methyl iodide to give *E*-1,4-disubstituted 1,3-enynes as the major isomer. In contrast, using methanol as the solvent gave selectively the corresponding *Z*-isomer [783]. Related reactions in the absence of methyl iodide gave (*Z*)-1,4-disubstituted-1,3-enynes from terminal alkynes [784]. Titanium mediated a reductive hydroalkynylation of internal alkynes with terminal alkynes to give 1,3-dienes (Eq. (122)) [785].

Palladium-catalyzed intermolecular oxidative hydroarylations of alkenes using indoles (Eq. (123)) [786]. Palladium-catalyzed oxidative hydroarylations of activated aldehydes with benzenes (Eq. (124)) [787]. *N*-2-Pyridylmethylindoles were exclusively substituted in the 2-position [788]. Palladium-catalyzed intermolecular hydroarylations alkynes using pyrroles and thiophens [789]. A platinum–silver catalyst system was used in anti-hydroarylations of 2-alkynoic acids [790]. An iridium catalyst system for hydroarylations of alkenes was reported [791]. Rhodium catalyzed chelation-controlled hydroarylations of alkenes using acetophenone imines [792]. Rhodium-catalyzed hydroarylations of alkenes using aromatic azines was also described [793].

Nickel catalyzed regio- and stereoselective reductive hydroacylations of alkynes with α-oxo-aldehydes to give allylic alcohols [794]. Rhodium catalyzed chelation-assisted intermolecular hydroacylations of alkynes to give *E*-2-alken-1-ones [795]. A rhodium-catalyzed asymmetric reductive hydroacyla-

tion of 2-enoates with aldehydes affording 3-hydroxyalkanoates was used in a synthesis toward C10–C24 fragment of inostamycins [253]. A nickel-catalyzed reductive hydroacylations using ynals was used to prepare macrocycles (Eq. (125)) [796].



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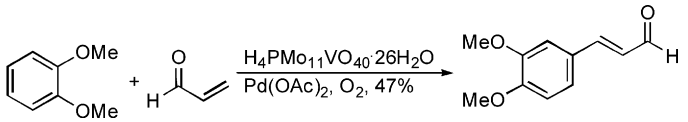

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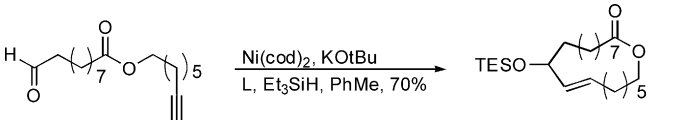
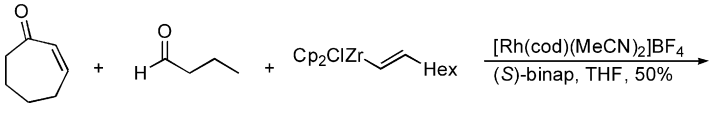
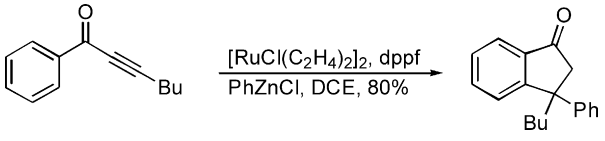


(125)

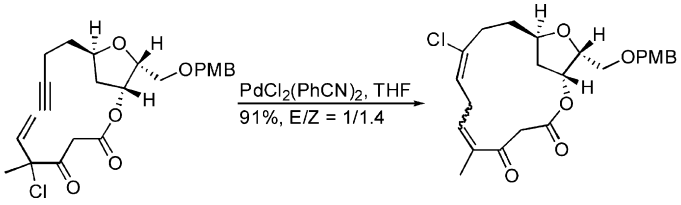
An enantioselective rhodium-catalyzed Michael addition using an alkenylzirconium reagent followed by trapping of the enolate with an aldehyde was developed and used in a synthesis toward vannusal A (Eq. (126)) [797]. Rhodium catalyzed asymmetric 1,6-additions of aryl zinc reagents to 2,4-dienones [798]. Rhodium catalyzed asymmetric 1,4-addition of alkenylboronic acid to β,β-disubstituted alkenyl pyridyl sulfones [799]. Ruthenium catalyzed the formation of 3,3-disubstituted-1-indanones from aryl alkynyl ketones and arylzinc reagents (Eq. (127)) [800].





(127)

A zirconium-catalyzed asymmetric methylalumination–oxidation of terminal alkenes was used in synthesis of (+)-bistramide C [801]. Two palladium catalyzed arylations of 2,6-dichloro-benzoquinone using arylmercury reagents were used in the synthesis of demethylasterriquinone B4 [802]. Palladium catalyzed haloallylations of alkynes and this reaction was used in synthesis of (−)-haterumalide NA/(−)-oocydin A (Eq. (128)) [803]. Rhodium catalyzed the addition of ethoxallylchloride (Eq. (129)) [804] and of perfluorinated acid chlorides [805] to alkynes. Nickel catalyzed an alkylative carboxylation of alkynes using organozinc reagents and carbon dioxide (Eq. (130)) [806]. A copper–iron catalyzed arylmagnesation of alkynes was developed [807].



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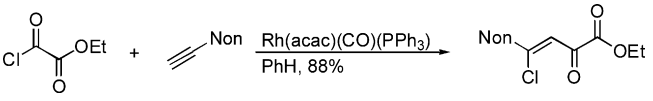

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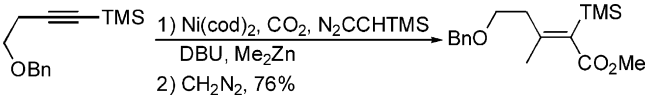
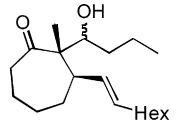


(130)

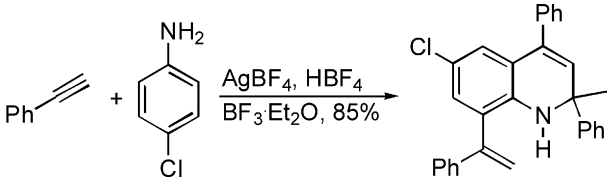
5.2. Formation of carbon–nitrogen bonds from alkenes, alkynes, and allenes

Palladium-catalyzed intermolecular hydroaminations of alkynes with anilines to give ketones after hydrolytic workup of the intermediately formed imine [808]. Palladium-catalyzed intermolecular hydroaminations of 1-benzyloxy-1,2-propadiene to give 3-amino-substituted 3-benzyloxy-1-propenes [809].



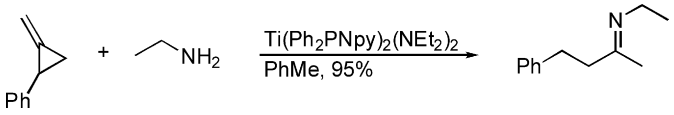
(126)

Cobalt catalyzed intermolecular hydrohydrazinations of 1,3-dienes or 1,3-enynes to give allylic or propargylic hydrazides [810]. Silver catalyzed intermolecular hydroaminations of alkynes using cyclic secondary vinylogous carbamates to give enamines [811]. Silver catalyzed a sequential hydroamination–cyclization of aryl alkynes with aryl amines (Eq. (131)) [812]. Rhodium and iridium catalyzed intermolecular hydroaminations of alkynes [813].



(131)

Platinum-catalyzed intermolecular hydroamidations of alkenes using sulfonamides [814] and carboxamides [815]. Highly Markovnikov selective platinum-catalyzed hydroaminations of alkenes was reported [816]. Titanium catalyzed intermolecular hydroaminations of methylidenecyclopropanes to give acyclic imines (Eq. (132)) [817]. Titanium catalyzed a three component coupling of an alkyne, a hydrazine, and an isonitrile (Eq. (133)) [818]. Titanium catalyzed the formation of arylhydrazones from alkynes and arylhydrazines [819]. Palladium catalyzed an intramolecular diamination of alkenes (Eq. (134)) [820].



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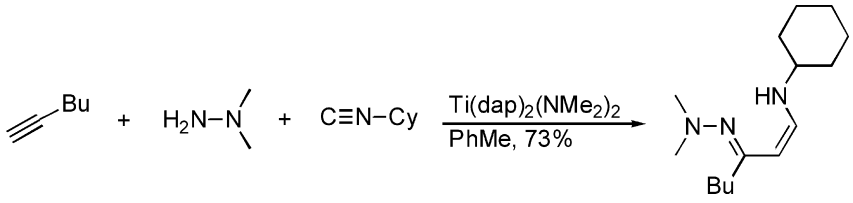

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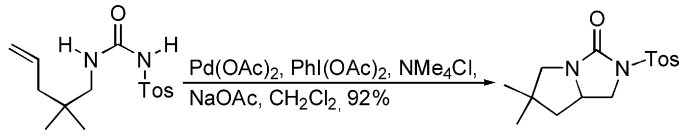
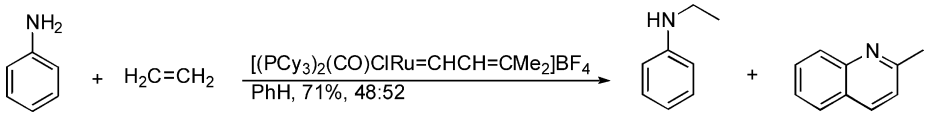


(134)

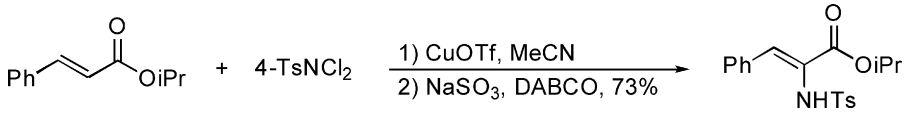
Titanium catalyzed intermolecular hydroaminations of 1-aryl-2-alkylalkynes affording 2-aryl-ethylamines as the major regioisomer after sodium cyanoborohydride reduction [821]. Titanium catalyzed intermolecular hydroaminations of terminal alkynes to form both ald- and ketimines [822]. Ruthenium catalyzed hydroaminations of arylamines with alkenes or 1,3-dienes to give *N*-alkylated products and heterocycles (Eq. (135)) [823]. Cobalt catalyzed hydroazination of alkenes using tosylazide in the presence of a silane and *t*-butylhydrogen peroxide [824].



(135)

Copper catalyzed 3-chloro-amidation of α,β-unsaturated ketones and esters that upon base treatment gave α,β-unsaturated α-amidoketones and esters (Eq. (136)) [825]. Copper catalyzed the addition of 2-oxazolidinones to arylidenecyclopropanes (Eq. (137)) [826]. Copper catalyzed the formation of *N*-sulfonylamidines from a three component reaction of a sulfonyl azide, an alkyne, and an amine [827]. Copper catalyzed the formation of amides from reaction of terminal alkynes, water and sulfonylazide (Eq. (138)) [828].

A nitrosarene–copper complex was isolated and suggested as an intermediate in copper catalyzed amination of alkenes using hydroxylamines to form allylic amines [829]. Copper catalyzed the formation of allylic hydroxylamines and amines from alkenes and BOC-hydroxylamine [830]. The kinetics of the iron-catalyzed amination of alkenes using nitroarenes forming allylic amines was examined. An iron–nitrosarene–alkene complex was proposed as an intermediate [831].



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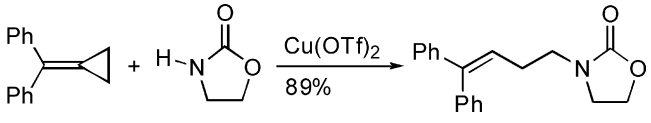

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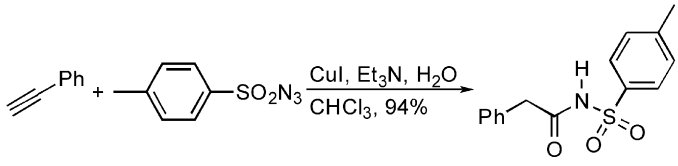
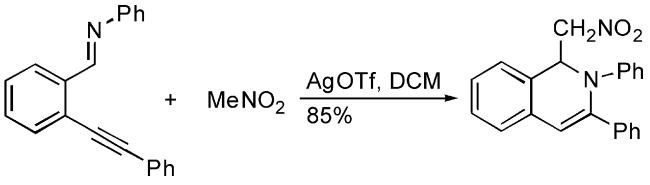


(138)

Silver and copper catalyzed the addition of pronucleophiles to 2-alkynylbenzaldehyde imines to give 1-substituted 1,2-dihydroisoquinolines (Eq. (139)) [832]. Ruthenium catalyzed an interesting hydroamination–cyclization of benzofused cyclic amines to form tricyclic quinolines (Eq. (140)) [833].

Ruthenium catalyzed anti-Markovnikov hydroamidations of alkynes to form enamides [834].



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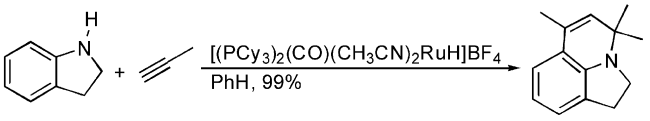

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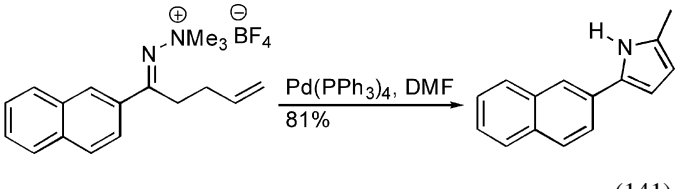
Palladium-catalyzed an oxidative amination of alkenes to give enamines, imines, or allylic amines depending on substrate [835]. The regioselectivity of the amination could be modulated by the addition of a base [836]. Palladium catalyzed an intramolecular amination of 4-alken-1-one-derived *N*,*N*,*N*-trimethylhydrazonium salts to give pyrroles (Eq. (141)) [837]. A palladium-catalyzed intramolecular amino-acetoxylation was reported (Eq. (142)) [838]. Palladium catalyzed a 1,2-diamination of conjugated dienes (Eq. (143)) [839]. A copper-mediated intramolecular diamination of a pendant alkene was reported (Eq. (144)) [840]. Amino-palladation of 2-(1-alkyne-1-yl)trifluoroacetanilides followed by intermolecular reaction with an alkynyl halides was used to prepare 3-alkynylindoles [841]. Gold catalyzed a sequential intramolecular amination–Michael addition of 2-alkynyl-1-aminobenzenes with α,β-unsaturated ketones (Eq. (145)) [842].



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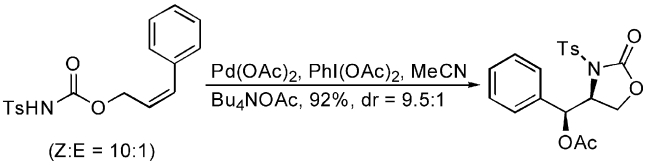

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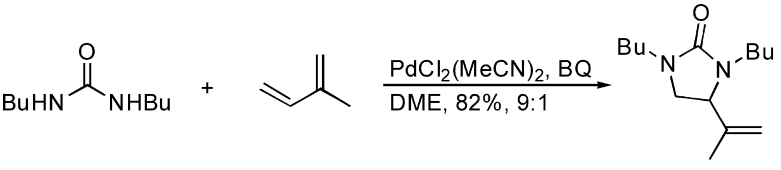

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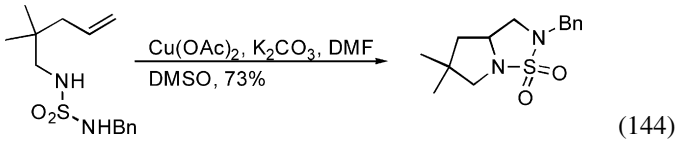

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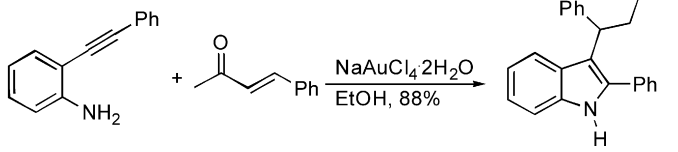
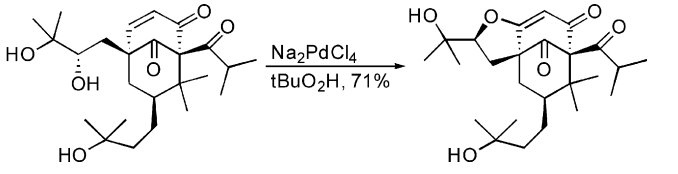


(145)

5.3. Formation of carbon–oxygen, –selenium and –sulfur bond from alkenes, alkynes, and allenes

The stereochemistry of oxidative intramolecular alkoxy-palladations was studied [843]. Oxidative intramolecular alkoxy-palladation (formally alkoxy-palladation-β-elimination) of a pendant alkene was used to form functionalized furans and benzofurans [440,844], dihydropyranones and furanones [845], (−)-15-oxopuupehenol [846], garsubellin A (Eq. (146)) [45] and alstonerine, 6-oxoalstophylline, alstophylline, and macralstonine [847]. A palladium catalyzed alkoxypalladation of pendant alcohols to allylic alcohols furnished unsaturated six-membered rings via elimination of hydroxide (Eq. (147)) [848]. A related oxidative intramolecular acyloxy-palladation of a pendent alkene was used in a synthesis of EI-1941-1 and EI-1941-2 [58]. Palladium catalyzed the formation of γ-methylene-α,β-unsaturated γ-lactones from 3,4-dienoates [849].



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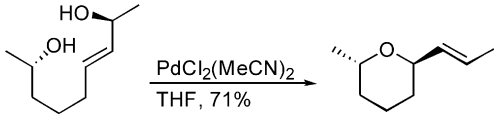

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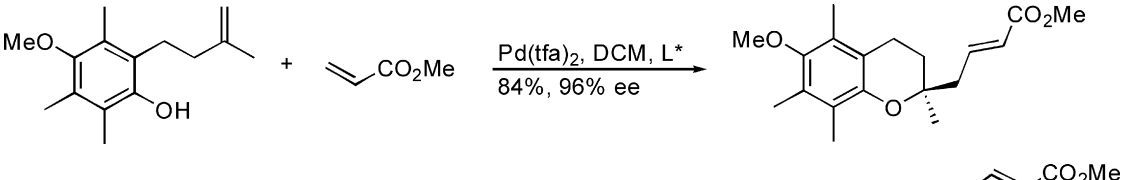
Intramolecular alkoxy–palladation–carbonylation of 2-(1-alkyn-1-yl)phenols to give benzo[*b*]furan-3-carboxylic acids was reported [850]. Intramolecular alkoxy-palladation–carbonylation–lactone formations of 1-ene-3,4-diols were used in synthesis of (+)-goniothalesdiol

and (+)-7-epi-goniothalesdiol [851]. Vitamin E (Eq. (148)) [852] and mycalamide A [853] were prepared via intramolecular alkoxypalladation followed by a Heck-type reaction of the σ-palladium intermediate. An intramolecular alkoxy-palladation of 3-hydroxy-1,2-dienes to give a σ-palladium complex followed by either a Heck reaction, a Suzuki coupling, or a Sonogashira coupling was described (Eq. (149)) [854]. A related reaction was used in a synthesis toward merrilactone A (Eq. (150)) [855]. Copper catalyzed an intramolecular alkoxy–cupration–allylation sequence (Eq. (151)) [856].



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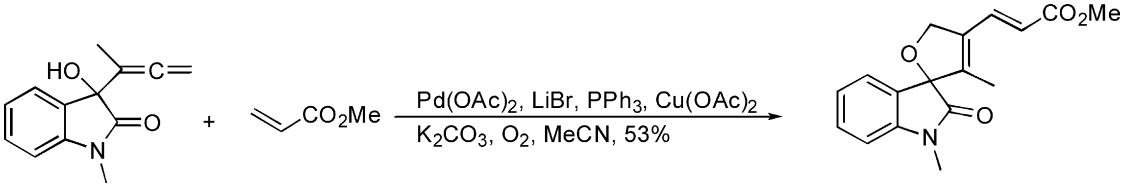

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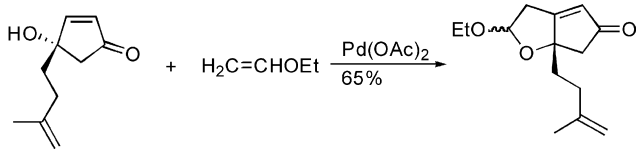

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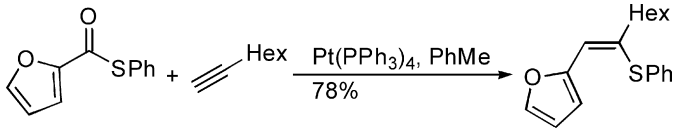

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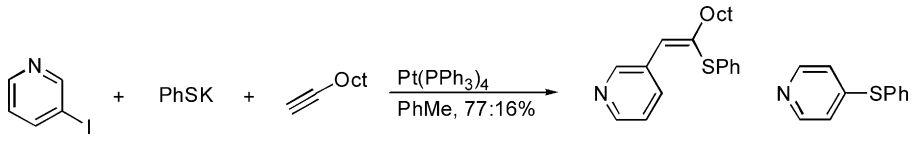

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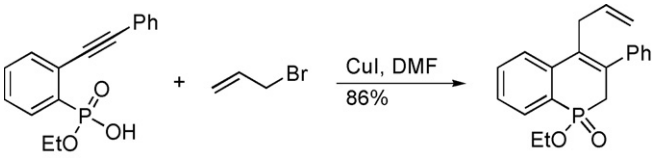


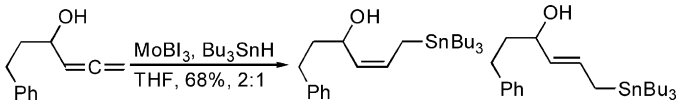
(151)

Gold catalyzed intermolecular hydroalkoxylation and hydroacyloxylations of alkenes to give saturated ethers and esters [857]. Ruthenium catalyzed related intermolecular hydroalkoxylations of alkenes affording saturated ethers [858].

Rhodium-catalyzed hydrothiolations of terminal alkynes [859] and platinum catalyzed furylthiolation (Eq. (152)) [860] and pyridylthiolation (Eq. (153)) [861] of alkynes. Palladium catalyzed the addition of diaryldisulfides and diaryldiselenides to alkynes forming *Z*-1,2-addition products [862,863]. Palladium catalyzed the formation of benzo[*b*]naphth[2,3-*d*]thiophen-6,11-diones via oxidative hydrothioarylation of benzoquinones with arylthiol [864]. Rhodium catalyzed the formation of alkynylthioethers from terminal alkynes and disulfides [865]. Palladium catalyzed 1,2-diselenations of allenes using diphenyldiselenide [866] and regioselective hydroselenations of terminal alkynes affording 2-seleno-1-alkenes as the only product [867].

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 synthesis toward peridinin [35], 6′-epi-peridinin [60], dihydroxerulic and xerulinic acid [38], (+)-tubelactomicin A [64,868] and macrolactin analogs [82]. Regioselective palladium-catalyzed hydrostannations of internal alkynes were used in synthesis toward mycolactones A and B [296], tricyclic core of GKK1032 [81], zoanthamine [83] and xanthocillin X dimethylether [87]. Palladium-catalyzed hydrostannation of bromoalkynes to afford a *trans*-alkenylstannanes were used in synthesis of (+)-phorboxazole A [70] and rhizoxin D [92]. Stryker's reagent catalyzed hydrostannations of 2-alkynoates and 2-alkyn-1-ones to give 2-stannylated 2-alkenoates and 2-alken-1-ones [869]. Molybdenum catalyzed hydrostannation of 1,3-dien-4-ols to give allylic stannanes as the major product (Eq. (154)) [870].



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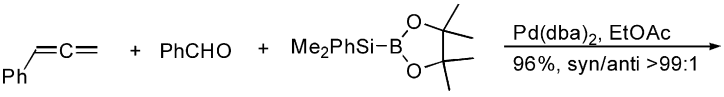
Palladium catalyzed a dimerization–distannation of benzynes using hexabutylditin to give 2,2′-distannylated biphenyl [871]. Palladium catalyzed a *syn*-alkynylstannylation of alkynes using tributyl(3,3,3-trifluoropropynyl)stannane [872].

A stereoselective palladium-catalyzed *cis*-addition of cyanoboranes to alkynes was reported [873]. The intramolecular variation was also described [874]. Palladium catalyzed a 1,2-silaboration of allenes using an organic iodide as the initiator. Alkylation occurs in the presence of an aldehyde (Eq. (155)) [875]. Nickel catalyzed an alkynylboration of an boron-tethered alkyne (Eq. (156)) [876]. Rhodium catalyzed an diboration of alkenes [877] including asymmetric reactions [878]. Palladium catalyzed an asymmetric allene 1,2-diboration followed by aldehyde alkylation and oxidative work-up (Eq. (157)) [879].



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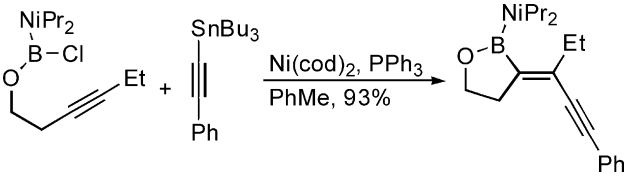

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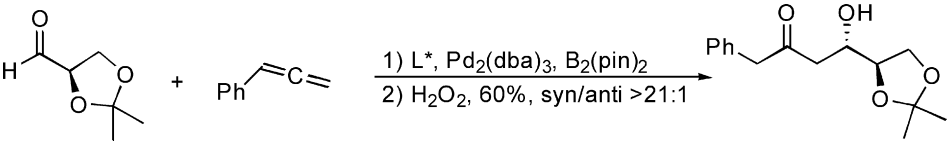
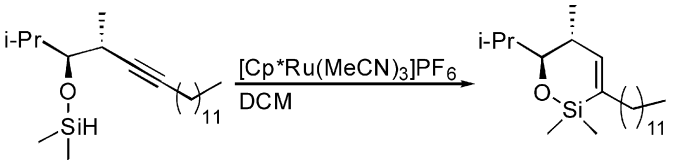


(157)

An intramolecular ruthenium-catalyzed hydrosilylation of propargylic and homopropargylic alkynes was described and this reaction was utilized in a synthesis of spectaline (Eq. (158)) [880]. Ruthenium carbene complexes catalyzed Markovnikov hydrosilylations of alkynes [881]. A *trans*-selective hydrosilylation of alkynes catalyzed by ruthenium was reported [882]. Platinum [883] and gold [884] catalyzed *cis*-hydrosilylations of terminal alkynes adding the silicon to the terminal carbon. Palladium catalyzed an intramolecular bis-silylation of disilanyl allylic ethers (Eq. (159)) [885].



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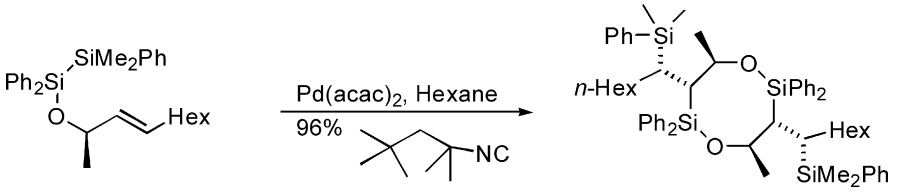
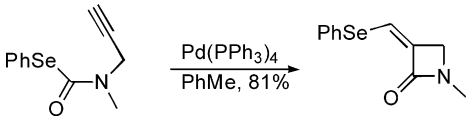


(159)

Palladium-catalyzed intramolecular selenocarbamoylations of alkynes (Eq. (160)) [886]. Nickel catalyzed hydrophosphination of alkynes using alkylphosphinates [887]. Nickel catalyzed reactions of propargylic alcohols with diphenylphosphine oxide and related phosphines to give phosphinoly-1,3-dienes [888]. Nickel catalyzed the formation of 1,3-dien-2-yl zinc intermediates from 3-trimethylsilyl-1-benzyloxy-2-propyne and bis(iodozincio)methane. The intermediates were coupled with aryl iodides (Eq. (161)) [285].



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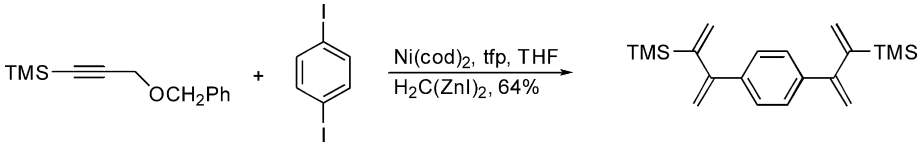
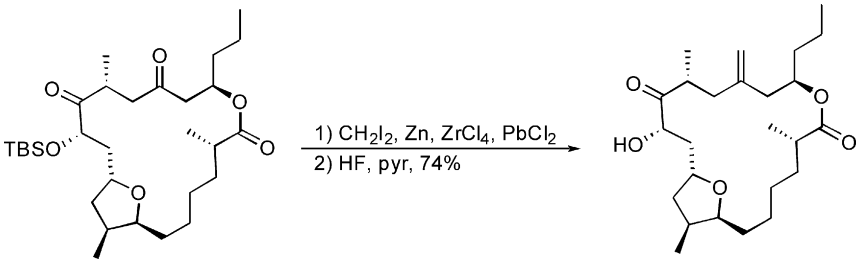


(161)

5.5. Formation of carbon–carbon and –nitrogen bonds from carbonyl compounds, imines, and azocompounds

A number of carbonyl to alkene transformations was 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 α-kainic acid [889] and toward spongistatin 1 [890]. $Zn(CH_2ZnBr)_2$–titanium tetrachloride was used to methylenate a ketone in a synthesis toward viridenomycin [891]. Zirconium tetrachloride was used in place of titanium tetrachloride in a synthesis of amphidinolides T4 (Eq. (162)) [331]. This type of alkenylation was also used to prepare alkenyl ethers from esters in a synthesis of β-*C*-glycosides [50]. An alkenylation-metathesis sequence using titanium tetrachloride, zinc, lead dichloride, and 1,2-dibromoethane [892] was used in a synthesis of gambierol (Eq. (163)) [42].



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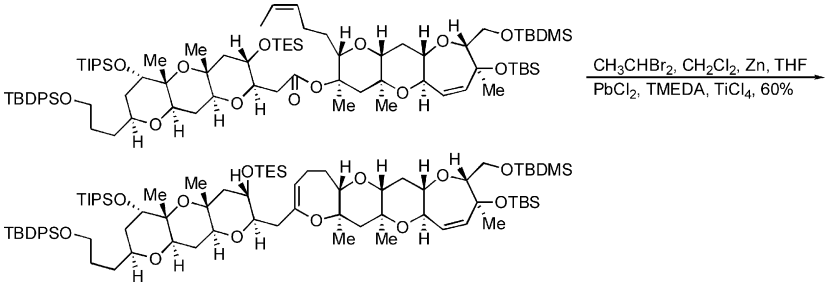

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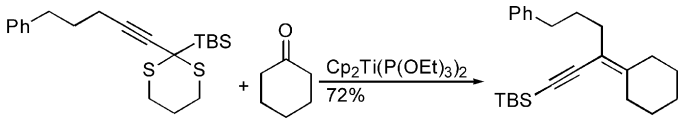
Tebbe's reagent was used to transform esters to alkenyl ethers in synthetic approaches to integramycin [224], α-(1–6)-*C*-disaccharides [893] and *C*-glycolyl amino acids [894]. Petasis reagent, Cp_2TiMe_2, was used to methylenate esters, lactones, or ketones in synthesis of mycalamide A [853], dihydropyrrins [492], toward vinigrol [895], (+)-phorboxazole A [70] and spongistatin 1 [896]. Petasis reagent was also used to methylenate unsymmetrical oxalates [897] and acetamides [898].

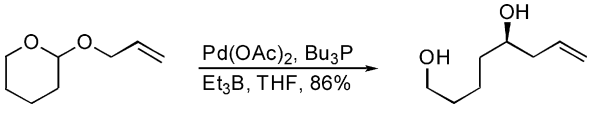
A titanium carbene complex formed *in situ* from 2-(1-alkyne-1-yl)-2-(trialkylsilyl)-1,3-dithianes was used to form trialkylsilyl-substituted 1,3-enynes (Eq. (164)) [764]. Titanium promoted the formation of allenes from 1,1-dichloro-1-alkenes and ketones [899].



(164)

Umpolung of intermediately formed η^3-allyl palladium complexes by transmetallation to indium or zinc was used in a number of cases. Palladium catalyzed the allylation of allyl 2-tetrahydropyranyl and 2-tetrahydrofuranyl ethers (Eq. (165)) [900]. Palladium catalyzed *in situ* formation of allylic boronates from allylic acetates followed by alkylation of aldehydes and imines [901]. A related reaction of allylic alcohols was described [902,903] in addition to an asymmetric reaction using allylic alcohols [904].

Palladium-catalyzed diasteroselective alkylation of an aldehyde by a propargylic mesylate in the presence of indium tribromide was used in a synthesis of the C20–C26 fragment of superstolide A [905], C6–C21 segment of amphidinolide E [270] and 2-*C*- and 4-*C*-branched sugars [906]. A three-component palladium-catalyzed reaction of arylboronic acids, an allene, and an aldehyde affording pyrones was described (Eq. (166)) [907].



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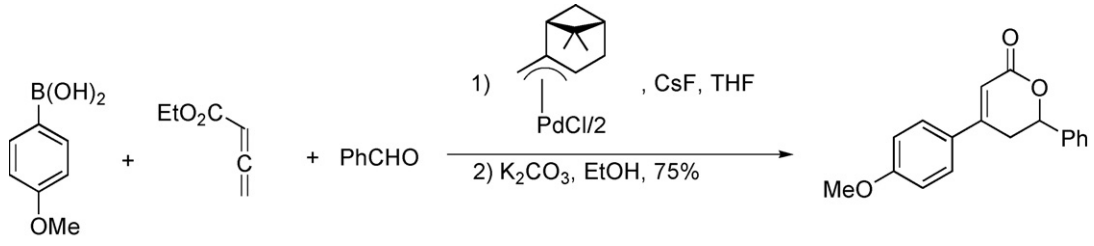
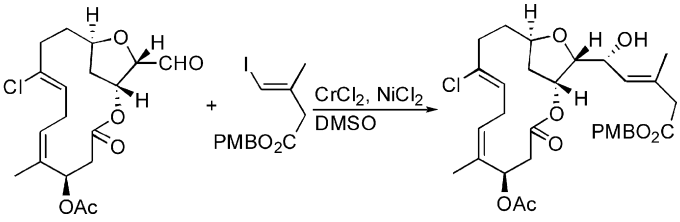


(166)

Nickel catalyzed a reductive coupling of an aldehyde and an alkyne in the presence of triethylboron was used in a synthesis of amphidinolides T1 and T4 (Eq. (167)) [330]. A related reductive coupling of an alkyne and an epoxide was used in a synthesis toward amphidinolide T2 [908]. Nickel catalyzed asymmetric reductive couplings of 1,3-enynes with ketones to give 5-hydroxy-1,3-dienes [909]. Nickel catalyzed an asymmetric coupling of an allene, an aldehyde, and a hydrosilane to give an allyl silylether (Eq. (168)) [910]. A nickel-catalyzed reductive coupling of allenes with aldehydes and an organozinc reagent was described [911]. Nickel catalyzed the coupling of mono-substituted alkenes with aldehydes in the presence of TMS-triflate to give allylic TMS-ethers (Eq. (169)) [912]. Rhodium catalyzed a reductive coupling of conjugated alkynes with ethyl (*N*-sulfinyl)iminoacetates in the presence of hydrogen gas (Eq. (170)) [913]. Rhodium catalyzed a reductive coupling of oxiranes with aldehydes (Eq. (171)) [914].



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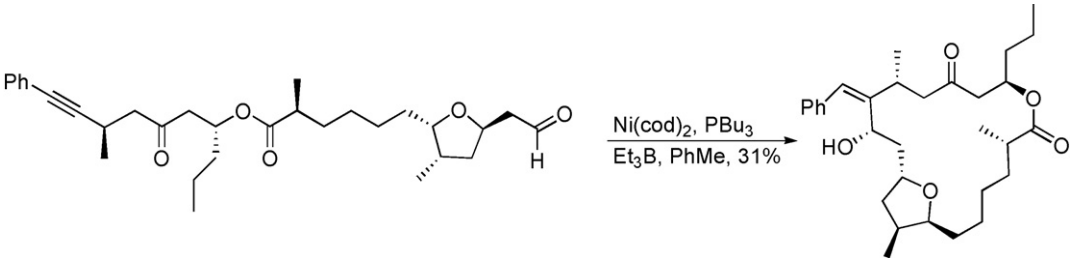
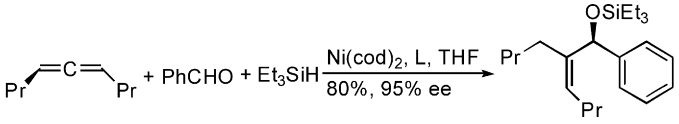


(167)

Titanium catalyzed an asymmetric alkenylation of ketones using alkenylzirconium reagents (Eq. (173)) [927]. A regioselective hydrozirconantion of an internal alkyne followed by transmetallation to zinc and aldehyde alkylation was used in



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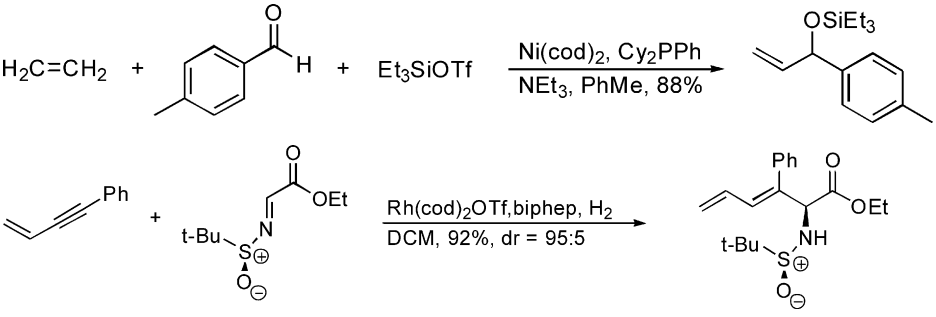

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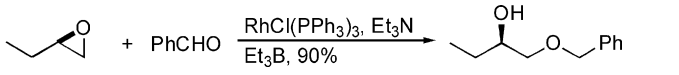
synthesis of reblastatin [575] and sphingadienine derivatives [928] or imine alkylation in synthesis of α,β-cyclopropyl-γ-amino acids [929], macrolactin analogs [87] and methyl sarcoate [20]. Hydrozirconation of allenes followed by transmetallation to zinc and imine alkylation furnished homoallylic amines [930].

A zirconium-catalyzed methylalumination of a terminal alkyne followed by imine alkylation was used in a synthesis of α,β-cyclopropyl-γ-amino acids [929] and sphingadienine derivatives [928].

Zirconium mediated a diastereoselective alkene-carbonyl coupling of *N*-alkenylcarbamates (Eq. (174)) [931].



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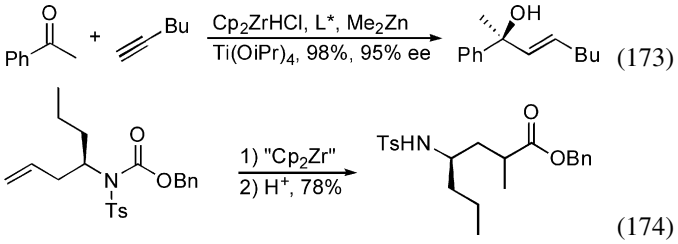
An asymmetric rhodium-catalyzed reductive aldol reaction of α,β-unsaturated esters with aldehydes in the presence of a hydrosilane was reported [915]. Rhodium catalyzed an enantioselective arylation of chiral aldimines using arylboronic acids [916]. Rhodium catalyzed an *ortho*-arylation of a benzophenone imine using $NaBPh_4$ [273].

Chromium–nickel mediated Nozaki–Hiyama–Kishi-type reactions were used in synthesis of clavosolide [917], norrisolide [918], (−)-haterumalide NA/(−)-oocydin A (Eq. (172)) [803], narbonolide [919], polycavernoside A [920], laulimalide analog [921] and (−)-gabosine [922]. A chromium–nickel mediated Nozaki–Hiyama–Kishi reaction was used to prepare macrocycles without the need for high-dilution techniques [923]. A Nozaki–Hiyama–Kishi reaction of 1-bromo-1-fluoro-1-alkenes substituting the bromide was reported [924]. An asymmetric nickel–chromium catalyzed Nozaki–Hiyama–Kishi reaction of alkenyl iodides with aldehydes was developed [925,926].



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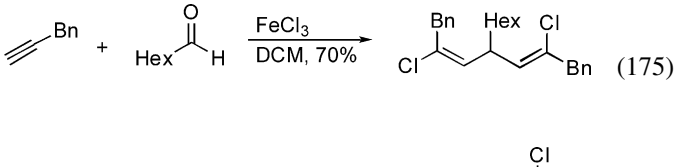
An iron-catalyzed asymmetric addition of alcohols to ketenes forming esters was developed [932]. Iron catalyzed the formation of 1,5-dihalo-1,4-dienes, α,β-unsaturated ketones, or cyclic ethers from aldehydes and alkynes (Eqs. (175) and (176)) [933]. Copper catalyzed a coupling of imines, acid chlorides and organotin reagents (Eq. (177)) [934]. Copper catalyzed the addition of arylboronic acids to azodicarboxylates forming aryl-substituted hydrazines [935]. Copper catalyzed a decarboxylative intermolecular aldol condensation of methyl malonic acid half thioester (Eq. (178)) [936].



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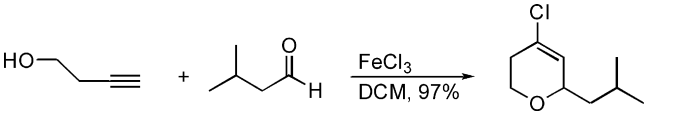

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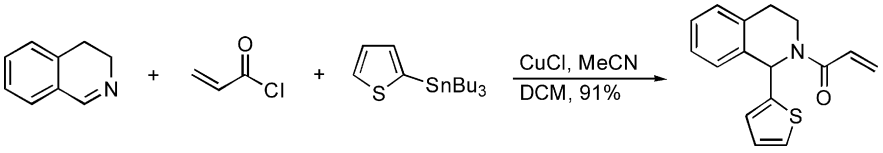

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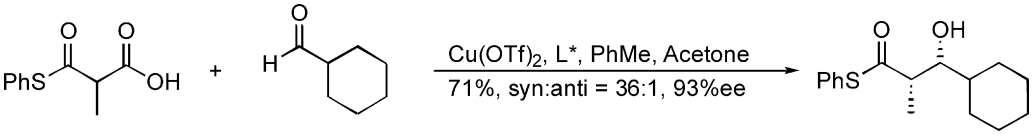

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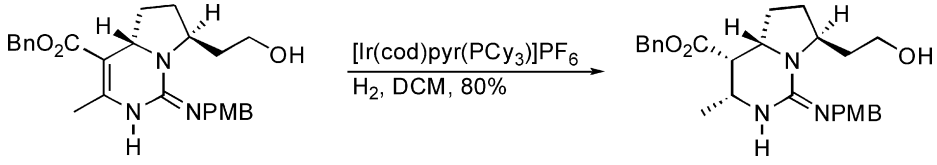
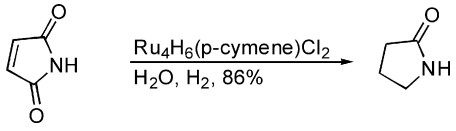


(181)

5.6. Formation of carbon–hydrogen bonds from alkenes, alkynes, carbonyl compounds, and imines

Ruthenium catalyzed mono-reductions of cyclic imides to lactams and hydrogenation of aromatic rings (Eq. (179)) [937]. Selective conjugate reductions of electron-deficient alkenes using Stryker's reagent, $[CuHPPh_3]_6$, was used in synthesis toward vannusal A [797], (−)-salicylhalamides A and B [938], *trans*-kumausyne [939], BCDE fragment of brevetoxin A [940], C26–C40 subunit of spirastrellolide [941] and a laulimalide analog [921]. A copper-catalyzed dynamic kinetic resolution of α,β-unsaturated lactones via asymmetric conjugate reduction was used in a synthesis of eupomatilone-3 (Eq. (180)) [200].



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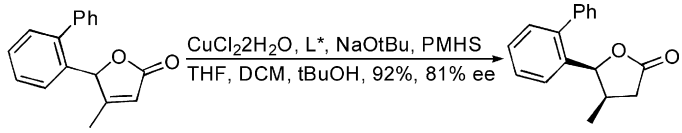

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Carbon–carbon double bond or triple bond reduction using Wilkinson's catalyst were used in synthesis of 10-hydroxyasimicin [323], murisolins [324], tuberostemonines [688], eupomatilones [211], guanacastepene A [942], pyragonicin [943], mucocin [944], 3-hydroxy-4-methyl-2-tetradecyl-4-butenolide [945], gleenol and axeenol [946], (−)-jimenezin [947], epi-(+)-SCH-642305 [948], methionine aminopeptidase-2-inhibitor [949] and the tricyclic core of cyathin diterpenoids [455]. Crabtree's iridium catalyst was used in synthesis of prodigiosines [198], batzelladine alkaloids (Eq. (181)) [950] and BCDE fragment of brevetoxin A [940].

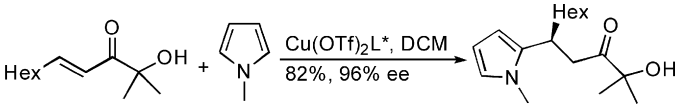
Rhodium catalyzed asymmetric reductions of (*Z*)-3-arylidene-4-acyl-3,4-dihydro-2*H*-benzoxazines [951]. Ruthenium catalyzed related reductions of *N*-sulfonated-α-dehydroamino acids in synthesis of anthrax lethal factor inhibitor [952]. Asymmetric reductions of 2-amido-2-alkeneoates using rhodium as the catalyst were used in synthetic applications toward (−)-aphanorphine [953] and cribrostatin IV [428]. Asymmetric ruthenium-catalyzed Noyori-type reductions of ketones [954] were used to prepare fasicularin [955], cribrostatin IV [428], daumone [657], (−)-colchicine [745] and strongylodiols [360]. Noyori's ruthenium

catalyst was used to reduce an imine in a synthesis of (+)-laudanosine and (−)-xylopine [327]. A Noyori-type reduction of a *cis*-alkene in the presence of *trans*-alkenes was used in a synthesis of C1–C25 fragment of amphidinol 3 [250].

A rhodium-catalyzed hydrosilylation of an enone to give a silylenol ether was used in a synthesis of 1,2-anhydro methyl rocaglate [506] and toward solanoeclepin A [956].

6. Miscellaneous carbon–carbon bond forming reactions

Palladium catalyzed direct carboxylation of arenes forming aromatic acids [957]. A number of transition metals catalyzed Friedel–Crafts type alkylations of arenes with benzylic acetates, alcohols, and carbonates [958,959]. Copper catalyzed enantioselective alkylations of pyrroles and indoles using α′-hydroxyenones (Eq. (182)) [960]. Copper catalyzed an oxidative coupling of tetrahydroisoquinolines, *N*,*N*-dimethylbenzeneamine, or 1-phenylpyrrolidine with activated methylene compounds, nitromethane, or indoles (Eq. (183)) [961–963].



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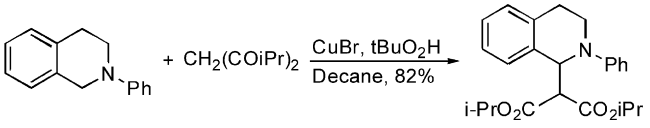
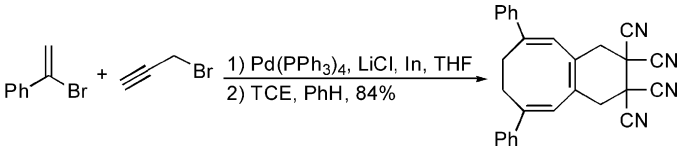


(183)

Palladium catalyzed tandem cross-coupling-[4 + 4] and [4 + 2]cycloadditions (Eq. (184)) [964]. Palladium catalyzed hydrosilylative couplings of alkynes with alkenes (Eq. (185)) [965]. Rhodium catalyzed homocouplings of tertiary propargyl alcohols with the loss of a ketone (Eq. (186)) [966]. A rhodium-catalyzed oxidative aryl–aryl coupling was used in synthesis of indolo[2,3-*a*]pyrrolo[3,4-*c*]carbazoles (Eq. (187)) [967].



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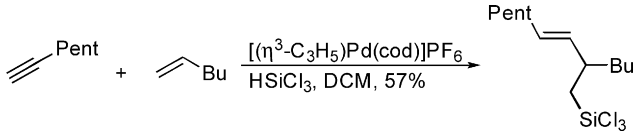

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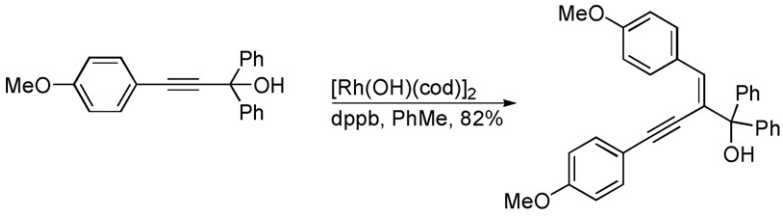

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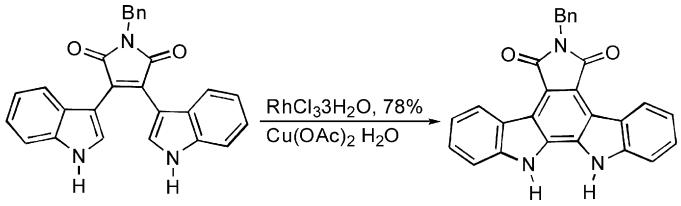
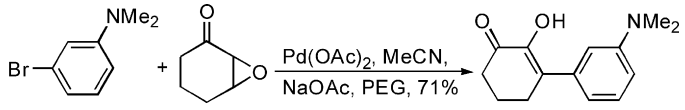


(187)

Palladium catalyzed the reaction of 2,3-epoxycyclohexanone with aryl bromides under microwave activation to give 3-aryl-2-hydroxy-2-cyclohexen-1-ones (Eq. (188)) [968]. A palladium–ruthenium catalyst system was used to couple chelating aldehydes with aryl iodides to give unsymmetrical ketones (Eq. (189)) [969]. Ruthenium catalyzed a regioselective chelation controlled *ortho*-arylation of aryl ketones using arylboronic esters (Eq. (190)) [970]. A related palladium catalyzed *ortho*-arylation of amido- or pyridine-substituted arenes using Ph_2IBF_4 as the source of the aryl group was described [971].



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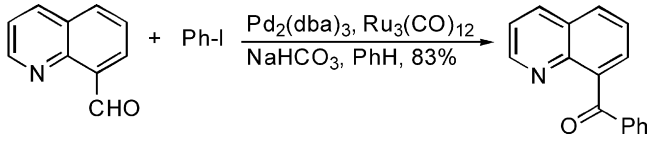

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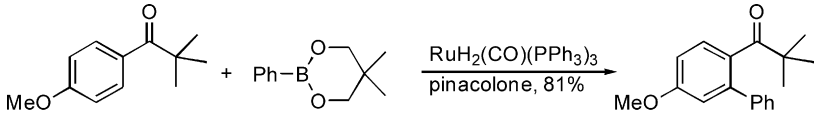
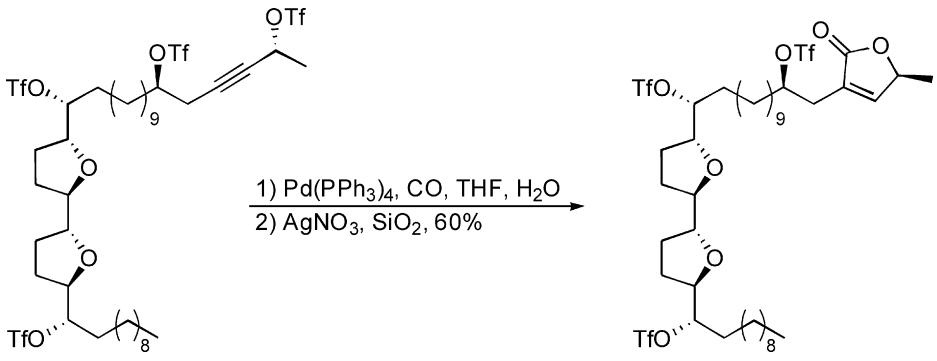


(190)

Copper catalyzed coupling of aryl iodides with perfluoroalkyliodides [972]. A palladium-catalyzed copper-mediated coupling of 1-iodo-2-nitrobenzene with a 2-iodo-2-cyclohexen-1-one was used in a synthesis of aspidospermidine [973]. Palladium-catalyzed coupling of pyrrolothimethylimidates with aryl halides [974] and of allylic acetates and carbonates with a pendant aryl iodide (Eq. (191)) [975]. Cobalt-catalyzed coupling of aryl chlorides and bromides with alkenyl acetates to give arylalkenes (Eq. (192)) [976]. Nickel catalyzed enantioselective orthoester alkylations of *N*-acylthiazolidinones (Eq. (193))



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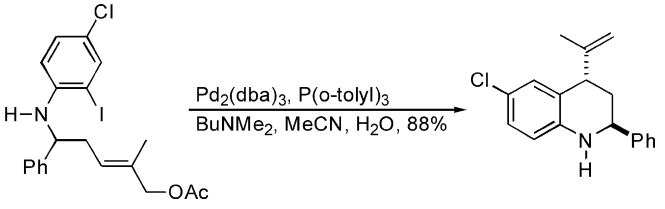
[977]. Ruthenium catalyzed an interesting reaction between propargylic alcohols and allyl ethyl ether to give cyclic acetals (Eq. (194)) [954].



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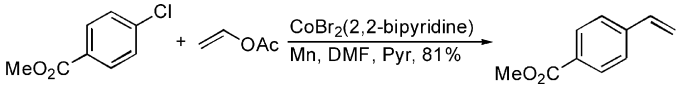

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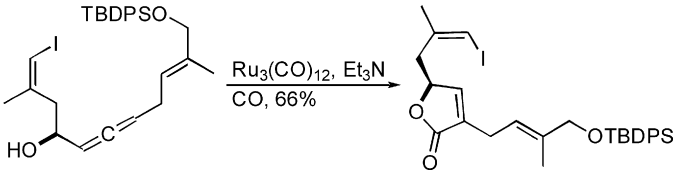
7. Carbonylations

7.1. Carbonylations of alkenes, allenes, and arenes

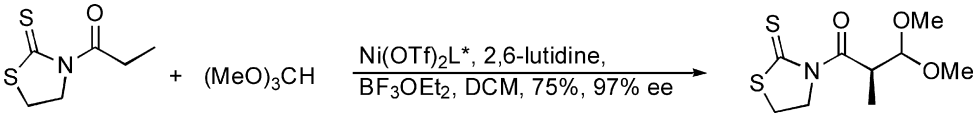
Palladium-catalyzed hydrocarbonylation of ethenyl acetate and styrene in the presence of an alcohol to give esters [978,979]. A palladium-catalyzed hydrocarbonylation was used in a synthesis of (+)-bullatacin (Eq. (195)) [980]. A ruthenium-catalyzed hydrocarbonylation of an allene was used in a synthesis of bipinnatin J (Eq. (196)) [39].



(196)

Rhodium catalyzed a desymmetrizing hydroformylation of diallylcarbinols [981]. A rhodium-catalyzed hydroformylation–Fischer indole synthesis sequence was described (Eq. (197)) [982]. A silylformylation-allylation was used in a synthesis of (+)-SCH-351448 (Eq. (198)) [983].

Rhodium-catalyzed hydroformylations were used in synthesis of polyketide-like macrolides [984], toward tashironin [985] and the C1–C13 segment of dolabelide B [986]. A rhodium catalyzed alkene hydroformylation–imine formation–reduction sequence was used to prepare ibutilide and aripiprazole [987].



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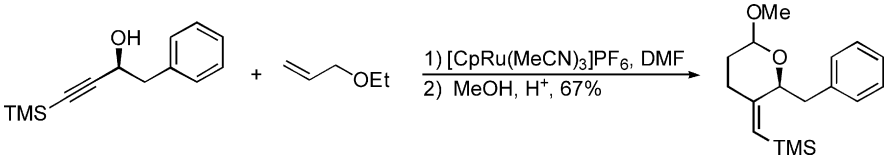

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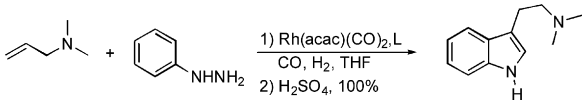

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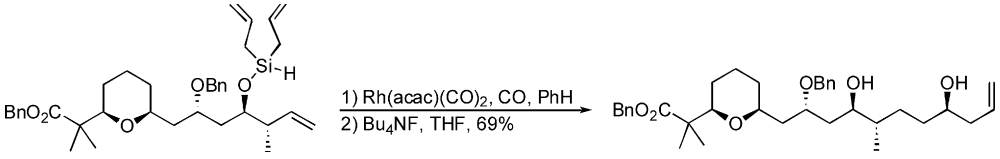
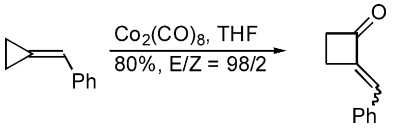


(198)

Cobalt catalyzed a [3 + 1]cycloaddition of methylene cyclopropanes and carbon monoxide (Eq. (199)) [988]. Nickel catalyzed a double carbonylation of 1-trimethylsilyl-1,2-dienes using carbon dioxide and this reaction was utilized in a synthesis of chaetomellic acid A anhydride (Eq. (200)) [989].



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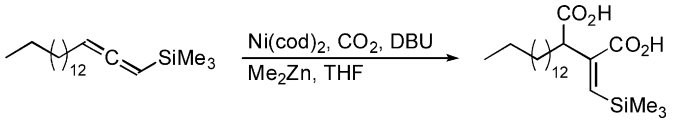

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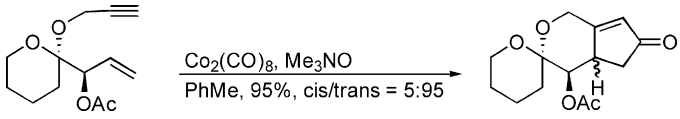
7.2. Carbonylations of alkynes (including the Pauson–Khand reaction)

Formation of cyclo-2-penten-1-ones via [2 + 2 + 1]ene-yne-carbon monoxide cycloaddition continued to be extensively studied, in particular the cobalt-mediated or -catalyzed, Pauson–Khand reaction. TEMPO [990] and dodecyl methyl sulfide [991] were shown to promote Pauson–Khand reactions. A catalyst system of dicobalt octacarbonyl-tetramethylthiourea and carbon monoxide was reported [992] and used in synthesis toward micrandilactone [993]. Palladium catalyzed Pauson–Khand reactions using tetramethylthiourea and carbon monoxide [994]. Molybdenum mediated Pauson–Khand reactions at room temperature [995].

Diastereoselective intermolecular Pauson-Khand reactions of chiral cyclopropenes were described [996]. Asymmetric cobalt-catalyzed intermolecular Pauson–Khand reactions were reported [997]. An asymmetric intermolecular Pauson–Khand reaction of cobalt–alkyne complexes having a chiral bridging ligand [998] and a rhodium-catalyzed asymmetric Pauson–Khand reaction in water [999] was described. Rhodium catalyzed an allylic alkylation-Pauson–Khand sequence [1000].

Intermolecular Pauson–Khand reactions of 2-ethynyl-1-aminobenzene [1001] and allylphosphonates [1002] and reactions of substrates having an arene chromium tricarbonyl complex as part of the molecule [1003] were reported.

Intramolecular Pauson–Khand reactions of alkynes tethered to methylencyclopropanes were described [1004]. A number of intramolecular Pauson–Khand reactions of 1,6- and 1,7-enynes [1005–1010] were reported. Anomeric control was observed in intramolecular Pauson–Khand reactions (Eq. (201)) [1011]. Intramolecular Pauson–Khand reactions were used in synthetic applications toward carbocyclic nucleoside analogs [669], ABC ring core of hexacyclinic acid [1012], ingenol (Eq. (202) [1013], Japanese hop ether [1014], toward pactamycin [1015], core structure of asperparaline [1016] and magellanine [1017]. Iridium catalyzed asymmetric intramolecular Pauson–Khand reactions of 1,6-enynes [1018].



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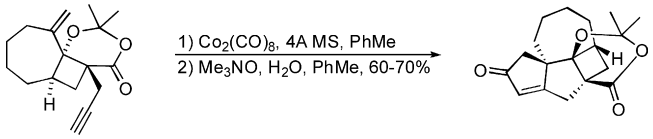
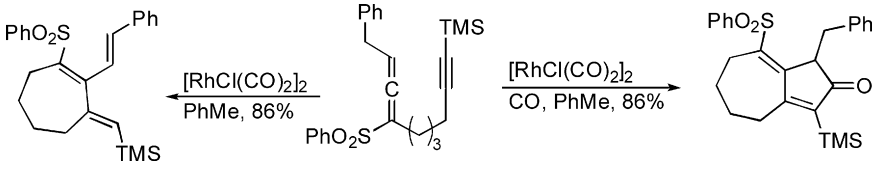


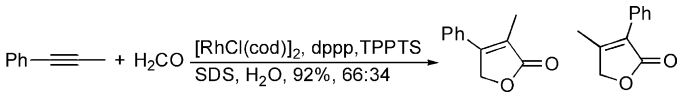
(202)

Intramolecular molybdenum-mediated allenic Pauson–Khand reactions using 1,2-diene-6-ynes and -7-ynes were described [1019–1022]. Rhodium-catalyzed intramolecular allenic Pauson–Khand reactions of 1,2-diene-7-ynes, -8-ynes, and -9-ynes in the presence of carbon monoxide to give bicyclic cyclopentenones and cycloisomerized products [1023,1024]. Only cycloisomerization is observed in the absence of carbon monoxide (Eq. (203)). A molybdenum-mediated intramolecular double allenic Pauson–Khand reaction was used to prepare dicyclopenta[*a*,*e*]pentalenes [1025].



(203)

Palladium catalyzed a hydrocarbonylation of phenylethyne using carbon monoxide to form methyl 2-phenylpropenoate with high selectivity [1026]. Rhodium catalyzed a cyclocarbonylation of alkynes forming butenolides using methanal as the CO source (Eq. (204)) [1027]. Rhodium catalyzed a [2 + 2 + 2 + 1]cycloaddition of enediynes in the presence of a silane and carbon monoxide (Eq. (205)) [1028]. Cobalt and rhodium catalyzed a [5 + 1]cycloaddition of ethenylcyclopropanes with carbon monoxide to give 3-cyclohexen-1-ones (Eq. (206)) [1029]. Cobalt–rhodium catalyzed a reductive cyclocarbonylation to give butenolides from conjugated ynones (Eq. (207)) [1030]. Palladium catalyzed the formation of 4-dialkylamino-1,5-dihydropyrrol-2-ones by reaction of 3-amino-1-alkynes with a secondary amine and carbon monoxide [1031].



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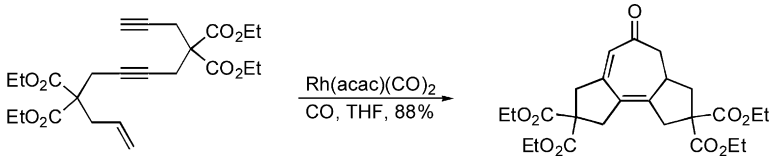

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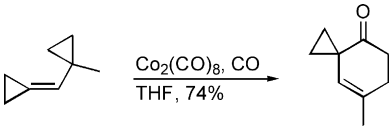

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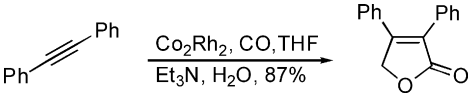
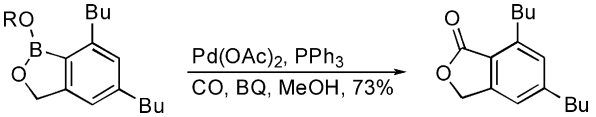


(207)

7.3. Miscellaneous carbonylations

Palladium catalyzed a carbonylation of arylboronates to form phthalides (Eq. (208)) [1032]. A palladium catalyzed alkoxycarbonylation of an allylic acetate was used in a synthesis of (+)-kalafungin and (−)-nanaomycin D [1033].

Palladium catalyzed carbonylation of terminal alkynes in the presence of halides to give (*Z*)-3-halo-2-alkenoate esters (Eq. (209)) [1034]. Palladium catalyzed a stereoselective dithiocarbonylation of propargylic mesylates with thiols (Eq. (210)) [1035]. Palladium catalyzed the formation of thiocarbamates from disulfides and an amine [1036]. Palladium catalyzed a four-component reaction of carbon monoxide, a terminal alkyne, diphenyldiselenide and a sulfenamide to give (*Z*)-3-phenylselenyl-2-alkenylcarboxamides (Eq. (211)) [1037].



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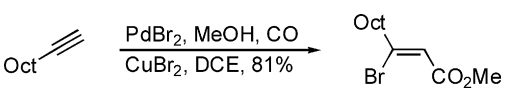

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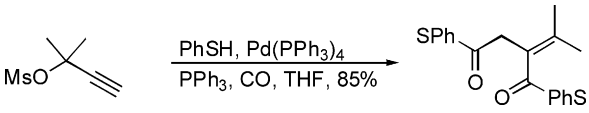

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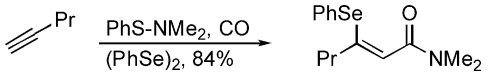
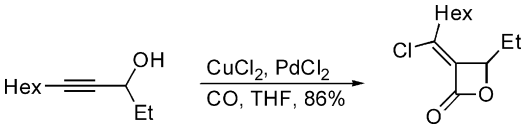


(211)

Palladium catalyzed a carbonylative cyclization of propargylic alcohols in the presence of copper dihalide to give (*Z*)-α-haloalkylidene-β-lactones (Eq. (212)) [1038]. Related reactions of propargylic amines gave (*E*)-α-haloalkylidene-β-lactones [1039]. An intramolecular palladium-catalyzed alkoxy-palladation carbonylations-lactonization of an 1-ene-1,6-diol was used to prepare micrandilactone [993]. Palladium catalyzed a carbonylative cyclization of 5-ynoates to give γ-alkylidene-α,β-unsaturated γ-lactones (Eq. (213)) [1040].



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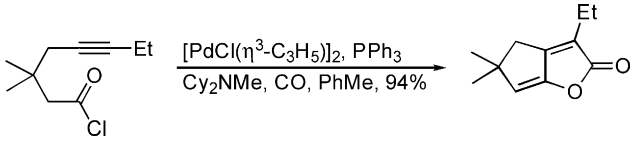
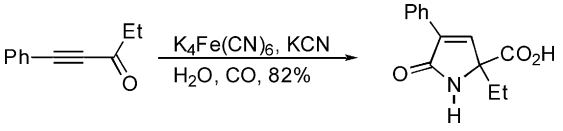


(213)

Iron catalyzed a cyclocarbonylation of 1-yne-3-ones in the presence of potassium cyanide to give 3,5-disubstituted-3-pyrrolin-2-one-5-carboxylic acids (Eq. (214)) [1041]. Nickel catalyzed a carbonylative cyclization of alkynes with allylic halides forming cyclopentenones (Eq. (215)) [1042]. Chromium catalyzed carbonylations of epoxides to form lactones (Eq. (216)) [1043].



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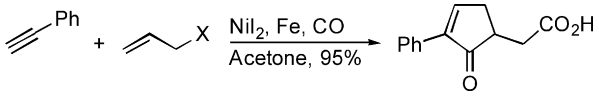

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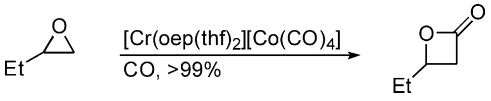
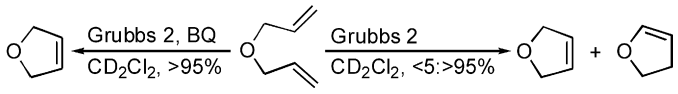


(216)

8. Metathesis reactions

A large number of reports on alkene–alkene ring-closing metathesis (RCM) and cross-metathesis (CM) reactions using Grubbs' type ruthenium catalysts were published. A number of new ruthenium catalysts were developed for both RCM and CM [1044–1049] including recyclable catalysts [1050]. A Lewis acid assisted RCM was described [1051]. An osmium catalyst was developed for RCM and CM [1052]. Addition of phenol to Grubbs first generation catalyst gave better yield of CM products [1053].

The selectivity and mechanism of a double RCM was affected by the choice of catalyst [1054]. Undesirable alkene isomerization during ring-closing and cross-metathesis was not observed upon addition of benzoquinone (Eq. (217)) [1055]. A tandem ring-closing metathesis-Kharasch reaction was described (Eq. (218)) [1056]. A diastereoselective RCM of prochiral phosphinic acid derivatives was reported [1057]. RCM of 3-oxo-1,4,7-trienes or cross-metathesis of 3-oxo-1,4-diene with 1,4-dienes afforded phenols (Eq. (219)) [1058]. Double bond isomerization RCM sequences using two different ruthenium catalysts were described [1059,1060]. A domino-RCM was reported (Eq. (220)) [1061].



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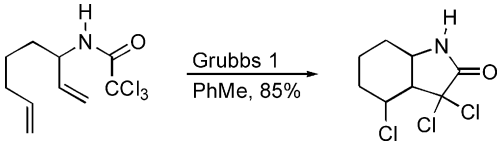

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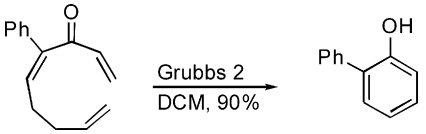

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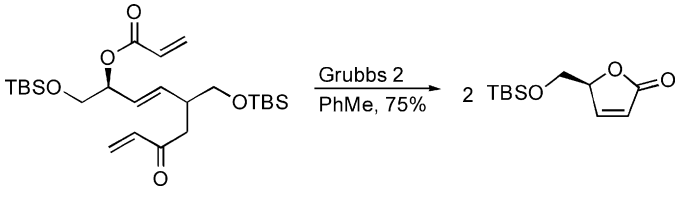
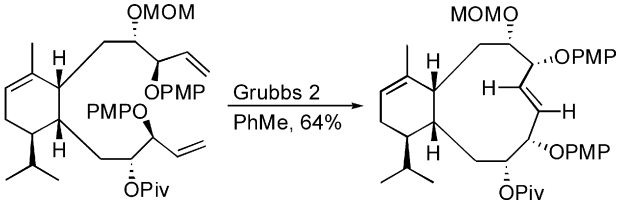


(220)

A large variety of ring systems were prepared via alkene-alkene RCM using ruthenium catalysts [441] including, furans and pyrroles [1062], macrocycles [1063–1071], five- to nine-membered unsaturated cyclic amines [498,519,898,1072–1077], cyclooctenes [1078], chiral azepin-3-ols and azocin-3-ols [1079], oxepin- and oxocin-annulated 2-quinolones [1080], benzazepines [1081,1082], 1-azaspiro[5.5]undacanes [1083], *N*-sulfonyl-2-quinolinones [1084], bis(silyl)-[3]-ferrocenophanes [1085], ferroceno-quinolines and -isoquinolines [1086], polycyclic hydrocarbons [1087], cyclic α-arylthiophosphonate esters [1088], carbohydrate-based macrolides [1089], fused carbazoles [157], 4-methyl-5-alkyl-2(5*H*)-furanones [1090], bibicyclo[8.8.8]hexacosanes [1091], bicyclic imidazoles [1092], bicyclic phosphates [1093], bicyclic lactams [1094], hexofuranose-like imino sugars [1095], C8-glycomimetics [1096], *cis*-perhydroisoquinolines [1097], restricted cyclic pentapeptide [1098], cyclic peptides [1099], cycloalkylalanines [1100], furanyl-glycin, -alanine and homo-furanyl glycine derivatives [1101], cyclic sulfoximines [1102], cyclic sulfamoyl carbamates [1103], 1,5-diaza-2,4-disilacycloheptanes [1104], silacyclopentenes [1105], bicyclic diketopiperazines [1106], unsaturated cyclic ethers [1006,1107–1109], indenones [1110], azabicyclophosphinic acids [1111], benzo[*f*,1,2]oxasilepines [1112], 2*H*- and 4*H*-chromenes [1113], the C2–C16 fragment of phorboxazole A [1114], aza-heteroannulated sugars [1115] and azepinoindoles [1116].

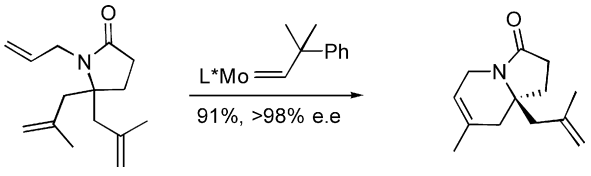
Synthetic targets using alkene–alkene ring-closing metathesis include 10-hydroxyasimicin [323], eleutherobin and analogs (Eq. (221)) [1117,1118], hybrids of D-galactose with 1-deoxynojirimycin analogs [1119], the tetracyclic core of tetrapentalones [1120], 1-epi aglycon of cripowellins A and B [445], gambieric acid [1121], aspercyclide C [524], radicicol and pochonin C [1122], trachelanthamidine [1123], heliannuols G and H [652], (−)-Geismann–Waiss lactone [1124], tuberostemonines [688], (−)-kendomycin [1125], pochonin D [1126], (−)-223A [1127], coleophomones B–D [1128], brevetoxin B [130], garsubellin A [45], fostriecin [48], 11-acetoxy-4-deoxyasbestin D [1129], (+)-cyclophellitol [1130], β-*C*-glycosides [50], zoapatanol [247], deoxymannojirimycin and swainsonine [1131], valienamine [1132], A_3 adenosine recetor agonists [1133], vincantril [810], (−)-perhydrohistrionicotoxin [1134], annonaceous acetogenins [1135], deoxycombrestatin A-4 analogs [1136], (+)-lentiginosine and analogs [1137], *N*-malayamycin A [1138], (−)-salicylhalamides A and B [938], oxytocin analogs [1139], steroid-like compounds [440], (−)-isoprelaurefucin [338], halichondrin B [1140], (−)-microcarpalide and (+)-lethaloxin [1141,1142], anamarine [655], (+)-cyclophellitol [1143], ovalicin [1144], OSW sapinines [1145], (+)-discodermolide analogues [226,1146], (+)-14-normethyldiscodermolide analogues [1147], mycothiazole [59], dictyostatin [1148], tonantzitlolone [1149], (−)-agelastatin A [1150], sarain B [1151], 10-epi-anamarine [1152], the proposed structure of feigrisolide [1153], rollicosin [1154], epothilones [1155], the C12–C19 fragment off (+)-peloruside A [1156], the C11–C23 fragment of spirastrellolide A [1157], (−)-dactylolide and (−)-zampanolide

[1158], gamberic acid [1159], carbocylic core of tricycloclavulone [1160], buflavine analogs [1161], (−)-epibatidine [1162], (+)-SCH-351448 [983], (+)-longicin [1163], (+)-epiquinamide [1164], the BCDE fragment of brevetoxin A [940], α-isospertein [1165], (−)-oleocanthal [1166], (+)-phomopsolide C [1167], polyketide-like macrolides [984], pochonin A [1168], (−)-cladospolide B [1169], (−)-lentigiosine [1170], (+)- and (−)-nicotine [681], conduritols [1171], tricyclic core of stemoamide [1172], untenone A and plakevulin A [1173], herbertene-1,13-diol and α-herbertenol [1174], 1,4-dideoxy-1,4-imino-D-talitol [1175], the ABCD ring system of manzamine [1176], 1-doexygulonojirimycin [1177], eleutheside analogs [1178], (−)-microcarpalide [1179], the EF-ring segment of ciguatoxin CTX1B [599], the B-ring of eleutherobin [1180], (+)-angustureine [1181], toward viridenomycin [891], di- and sesterpenes [607], methyl sarcoate [20], the tricyclic framework of eunicin [1182], (−)-microcarpalide [1183], (−)-centrolobin [1184], merrilactone A [1185], (+)-boronolide [1186], a cystein protease cathepsin K inhibitor [1187], halichlorin and pinnaic acid-like products [1188], (−)-gabosine [922], herbertane sesquiterpenes [1189], (−)-deoxynupharidine [1190], core of otteliones A and B [1191], HM-1 and HM-2 [1192], valienamine [1193], SCH-56036 [242], tarchonanthuslactone [1194], epi-(+)-SCH-642305 [948], methionine aminopeptidase-2 inhibitor [949], (+)-laurencin [1195], a spirobenzofuran metabolite [1196], the octahydroisobenzofuran skeleton of eunicellins [1197], the core of streptorubin B [1198], the floresolide B hydroquinone lactone core [1199], agardhilactone [1200], the IJKLM-segment of ciguatoxin CTX3C [1201] and *E*- and *Z*-germacrenes [1202].



(221)

A diastereoselective ruthenium-catalyzed RCM was used in a synthesis of (−)-eburnamonine [78]. Chiral cyclic amides and amines were prepared by a molybdenum-catalyzed asymmetric RCM (Eq. (222)) [1203]. The mechanism of the molybdenum-catalyzed asymmetric RCM was examined by computational methods [1204]. Molybdenum catalyzed intermolecular alkyne–alkyne ring closing metatheses were used in synthesis of latrunculin A (Eq. (223)) [108] and nisin Z mimics [1205].



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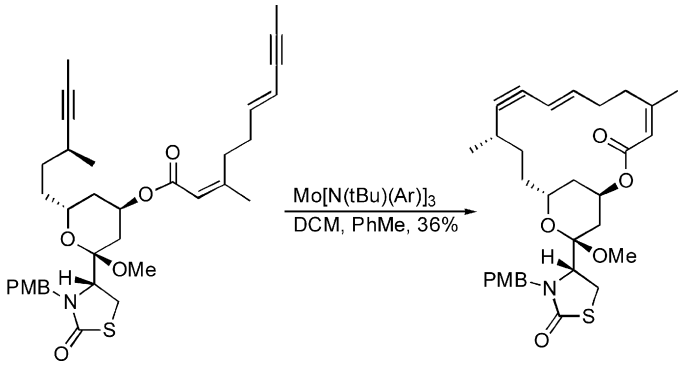
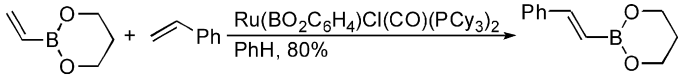


(223)

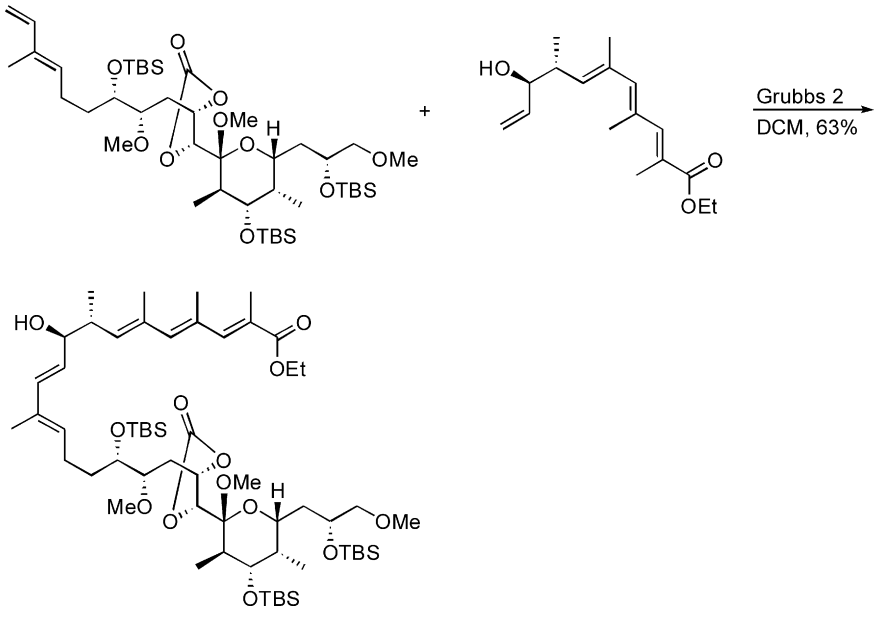
Alkene–alkene CM and alkene isomerizations were observed using urea- and amide-substituted alkenes [1206]. Microwave accelerated alkene–alkene CM was reported [1207]. Desymmetrizations of di- and trialkenyl phosphine oxides via CM were reported [1208]. An unusual *cis*-selective CM was reported [1209]. A non-carbenoid ruthenium complex catalyzed a CM of ethenylboronates with terminal alkenes to afford alkenylboronates (Eq. (224)) [1210]. Homo-alkene–alkene CM of was described [1211,1212].



(224)

Ruthenium catalyzed alkene–alkene CM of styrenes [1213], 1-allyl-tetrahydro-β-carboline and -tetrahydroquinoline [1214], propenoic esters [1215,1216], alkenylsilanes [283,1217], allyl trimethylsilane [1218], conjugated dienes [1219,1220], allyl cyanide [1221], acrylonitrile [1222], ethenylazulene [1223], *N*-allylpyrimidines [1224], terminal 1-en-4-ols [644] and β-hydroxyenones [1225] with terminal alkenes were described.

Alkene–alkene CM were used in synthesis toward viridiofungin A [1226], 2,7-diaminosuberic acid derivatives [1227], radicicol and pochonin C [1122], (+)-cyanthiwigin U [1228], apoptolidinone (Eq. (225)) [1229], the C9–C16 segment of ambruticin [1230], (−)-lemonomycin [1231], aureothin and *N*-acylaureothamine [61], the C22–C36 subunit of halichondrin B [654], the A ring of bryostatin analogs [1232], misakinolide A [1233], α-*C*-glucosyl serine and alanine [1234], the C26–C40 subunit of spirastrellolide [941], glutamic acid side chain homologues [1235], discodermolide analogues [1236], the C6–C21 segment of amphidinolide E [270], fraxinellone limonoids [1237], (+)-phomopsolide C [1167], pinnaic acid and halichlorine [1238], sphingosines [1239], mollugin and microphyllaquinone [1240], mucocin [645], (+)-anatoxin-a [1241], colletodiol [1242], toward paulitin [1243], tashiromine [1244], (−)-nupharamine [1245], octalactin lactone [1246], 3-hydroxy-4-methyl-2-tetradecyl-4-butenolide [945], 5,5-dimethylproline [1247], xenovenine and indolizine 209D [1248] and bengamides [1249]. An alkene–alkene scrambling cross-metathesis was used to prepare labeled sphingolipids (Eq. (226)) [1250]. A CM of an propenyl group to give an ethenyl group was used in synthesis of tuberostemonines (Eq. (227)) [688].



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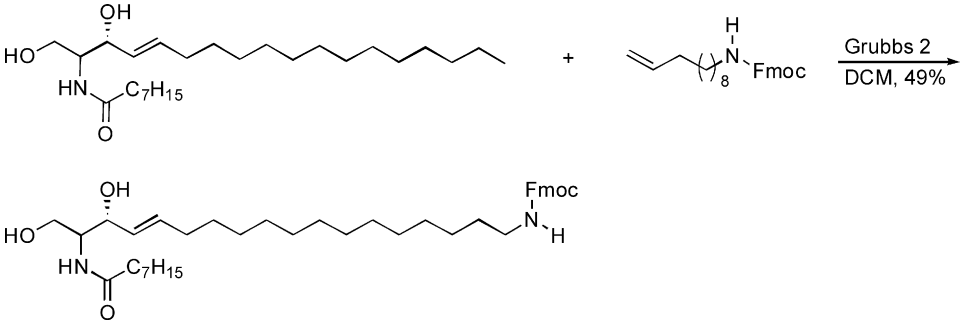

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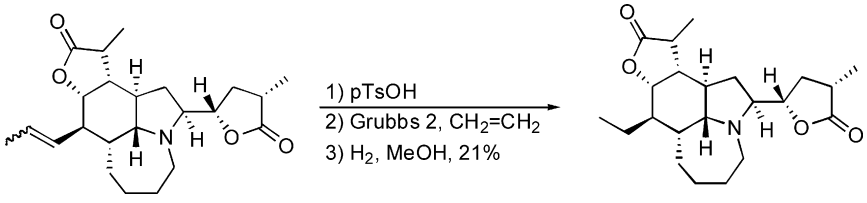
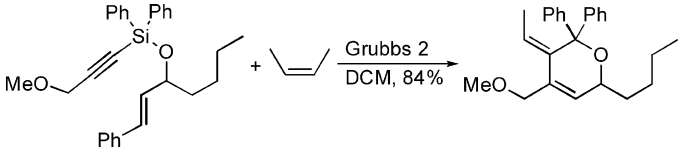


(227)

A tandem CM-RCM was described (Eq. (228)) [1251]. A ring-opening cross-metathesis [1252] and a regioselective intermolecular ring-opening-cross-metathesis of norbornene derivatives was reported [1253]. A related intramolecular variation was also described [1254]. Ring-rearrangement metatheses were used in syntheses of (+)-cyanthiwigin U (Eq. (229)) [1228], (+)-*trans*-195A [1255], *trans*-kumausyne [939] and (+)-castoanospermine [1256].



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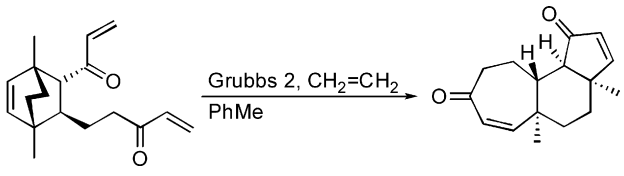
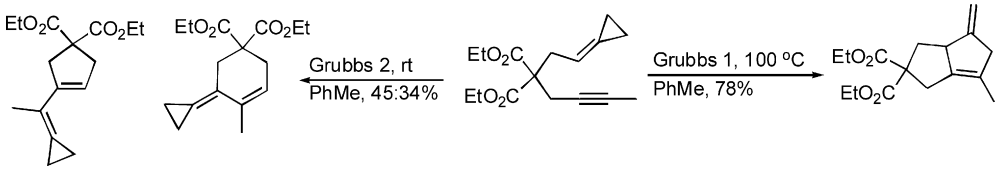


(229)

Ruthenium-catalyzed RCM of 1,7-, 1,8-, and 1,9-enynes were described [1257–1260]. A mechanism involving an ene-then-yne metathesis of tethered enynes was supported by labeling experiments [1261]. Ruthenium-catalyzed ene-yne RCM reactions of cyclopropane substituted 1,6-enynes forming either bicyclic or monocyclic compounds depending on the catalyst

(Eq. (230)) [1262]. A related cyclization of 1,2-diene-7-ynes was reported (Eq. (231)) [1263]. Ruthenium catalyzed enyne RCM were used in syntheses of (−)-dihydroxanthatin [735] and toward paulitin [1243]. A ruthenium-catalyzed ene-diyne metathesis followed by a metallotropic [1,3]-shift was described [1264]. A ruthenium-catalyzed enyne RCM followed by cross-metathesis was used in a synthesis of (+)-8-epi-xanthin (Eq. (232)) [507]. An interesting polycyclization of polyenynes was reported [1265]. Ruthenium catalyzed ene-yne-ene metatheses were used in approaches to silicon containing bicyclic compounds (Eq. (233)) [1266], thapsigargin skeleton [1267] and paulitin [1243].



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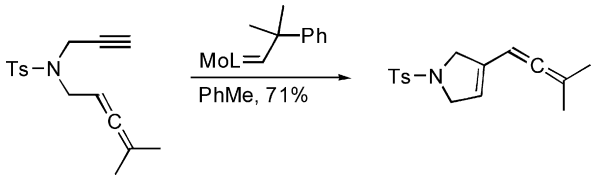

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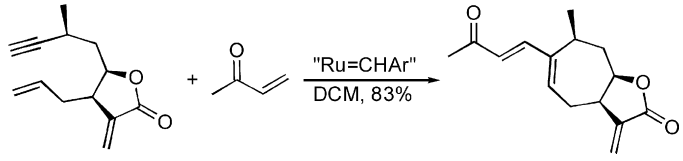

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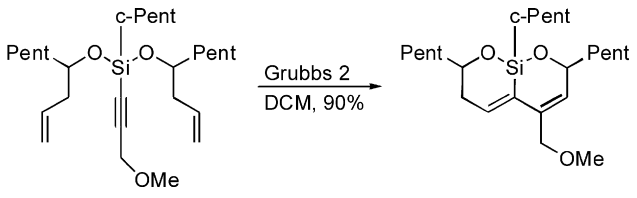
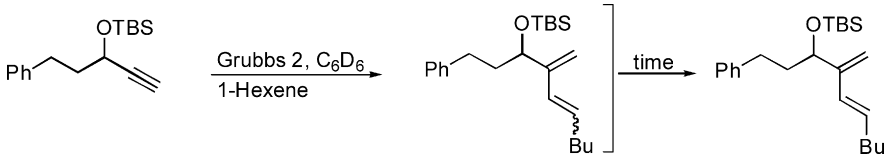


(233)

Ruthenium catalyzed a number of ene-yne CM reactions forming conjugated dienes [1223,1268,1269]. The mechanism of the ruthenium-catalyzed cross-coupling coupling reaction to give 1,3-dienes was examined and a ruthenacyclobut-2-ene intermediate is probably not part of the catalytic cycle [1270,1271]. Ene-yne CM between 1-hexene and an alkyne gave *E*/*Z*-mixtures of 1,3-dienes. The mixture equilibrates over time with an excess of 1-hexene to give the thermodynamically more stable *E*-isomer via an ene-ene cross metathesis (Eq. (234)) [1272]. The kinetic of the ruthenium catalyzed ene-yne CM was examined [1273]. A boron-directed ene-yne cross metathesis was described (Eq. (235)) [1274]. Ruthenium catalyzed ene-yne CM was used in a synthesis of 24,24-ethanovitamin D_3 lactones [474].



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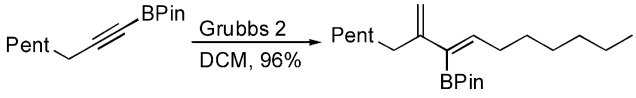
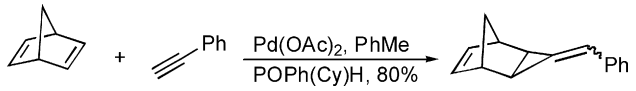


(235)

9. Cyclizations

9.1. Cycloadditions

Reactions wherein a cyclic product is formed from two (or more) compounds without loss of atoms are grouped in this section including all [*n* + *m* + ···]cycloadditions. A palladium-catalyzed intermolecular alkylidenecyclopropanations of bridged bicyclic alkenes and terminal alkynes was reported (Eq. (236)) [1275]. An interesting rhodium-catalyzed dimerization of bisallenes to give steroid ring system was described (Eq. (237)) [1276]. Rhodium catalyzed a cycloaddition of 4-ynals with *N*,*N*-dialkyl propenamides (Eq. (238)) [1277]. Iron catalyzed a cyclization of 1,3-dienes with oxiranes forming tetrahydrofurans [1278].



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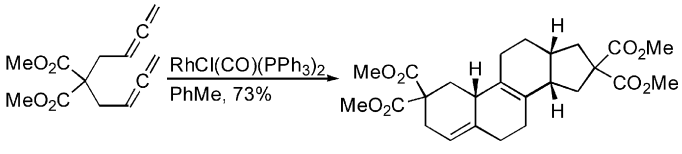

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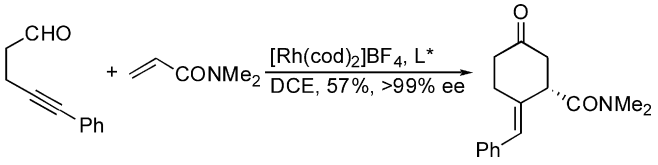
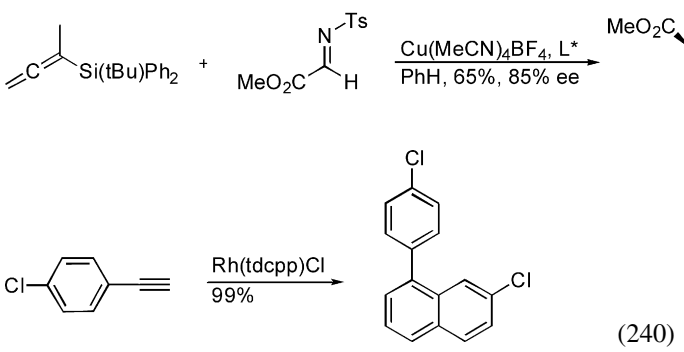


(238)

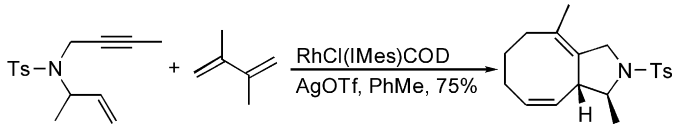
Ruthenium catalyzed a [2 + 2]cycloaddition of allenylboronates [1279]. Iron catalyzed asymmetric Staudinger reactions to form β-lactams [1280]. Copper catalyzed an asymmetric [3 + 2]cycloaddition of (1-alkyl-1,2-propadienyl)silanes with α-imino esters (Eq. (239)) [1281]. A related asymmetric copper-catalyzed [3 + 2]cycloaddition of azomethine ylides with 2-alkenoates was reported [1282,1283]. Rhodium-catalyzed intermolecular [4 + 2]cycloisomerizations of arylalkynes to give 1-arylnaphthalenes (Eq. (240)) [1284]. Rhodium-catalyzed intramolecular [4 + 2]cycloadditions of 9-halo-1,3-dien-8-ynes and 10-halo-1,3-dien-9-ynes [1285].



(240)

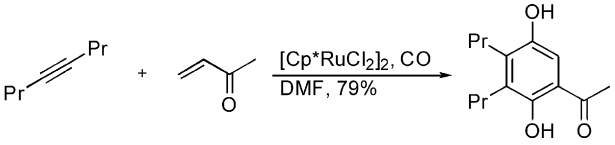
A cobalt-catalyzed [4 + 2 + 2]cycloaddition of a norbornadiene derivative with 1,3-butadiene was used to prepare the core of portulal [1286]. Rhodium catalyzed a diastereoselective [4 + 2 + 2]cycloaddition of 1,6-enynes with 1,3-butadiene to give fused bicyclic compounds (Eq. (241)) [1287]. Cobalt catalyzed a [6 + 2]cycloaddition of cycloheptatrienes with alkynes [1288].

Cobalt mediated an intramolecular [2 + 2 + 2]cycloaddition of methylenecyclopropyldiynes to give cyclopropanated polycycles [1289]. Cobalt mediated [2 + 2 + 2]cycloadditions were used to prepare aryl-containing macrocycles [1290], [7]helicene-like molecules [1291] and the deoxygenated pancrastistatin core [1292]. Benzolactones and lactams were prepared via a cobalt-catalyzed [2 + 2 + 2]cycloadditions of (2–4)-yne-1-ols and 2-yne-1-amines with propynoic acid esters [1293].



(241)

Ruthenium catalyzed [2 + 2 + 2]cycloadditions of 1,6- and 1,7-diynes with an ethynylboronate [1294], a *C*-alkynylglycoside [1295] and electron-deficient nitriles and heterocumulenes [1296]. Ruthenium catalyzed a cycloaddition of alkynes, carbon monoxide, and α,β-unsaturated carbonyl compounds to give hydroquinones (Eq. (242)) [1297]. Nickel catalyzed a related reaction of 1,6-, 1,7- and 1,8-diynes and nitriles to give fused bicyclic pyridines [1298]. Nickel catalyzed a cycloaddition of two alkynes and an isocyanate to give pyridones [1299]. Nickel catalyzed cycloadditions of 1,6- and 1,7-diynes with aldehydes to give five- and sex-membered rings having a tethered carbonyl (Eq. (243)) [1300]. Iridium catalyzed [2 + 2 + 2]cycloadditions of 1,6-enynes with alkynes [1301].



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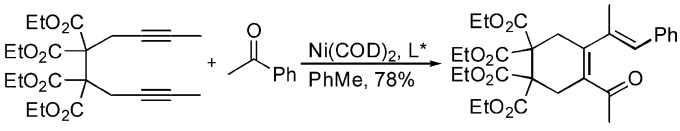
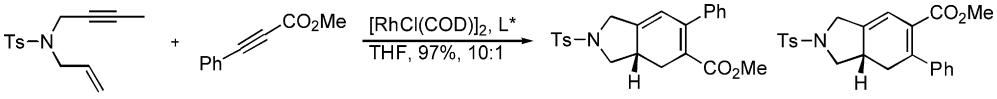


(243)

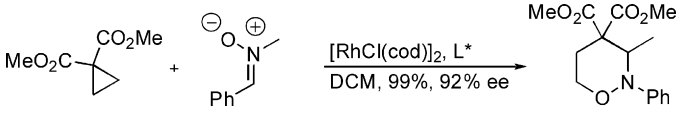
Rhodium catalyzed an intramolecular [2 + 2 + 2]cycloaddition of cyclic triynes and endiynes [1302]. Axially chiral biaryls were prepared by asymmetric intermolecular [2 + 2 + 2]cyclization [1303]. Rhodium catalyzed regioselective eyne-diyne[2 + 2 + 2]cycloadditions to give paracyclophanes [1304]. Rhodium-catalyzed intermolecular [2 + 2 + 2]cycloaddition of 1,6-enynes with alkynes to give bicyclic cyclohexadienes [1305]. Rhodium catalyzed [2 + 2 + 2]cycloadditions of 1,6-, 1,7-, and 1,8-diynes with isocyanates to give fused 2-pyridones [1306]. Enantioselective reactions were reported (Eq. (244)) [1307]. Enantioselective rhodium-catalyzed [2 + 2 + 2]cycloaddition of 1,6-enynes with alkynes was described [1308].



(244)

Nickel catalyzed an asymmetric cycloaddition of nitrones with activated cyclopropanes (Eq. (245)) [1309]. Nickel catalyzed cycloadditions of cyclobutanones with alkynes forming cyclohexenones (Eq. (246)) [1310]. Rhodium catalyzed [5 + 2] and [5 + 2 + 1]cycloadditions of allenes with alkenylcyclopropanes (Eqs. (247) and (248)) [1311]. Rhodium catalyzed an allylic alkylation-[5 + 2]cycloaddition or a cycloisomerization sequence (Eqs. (249) and (250)) [1000]. Rhenium catalyzed the

formation of indenes from aromatic aldimines and alkynes (Eq. (251)) [1312]. Gold catalyzed an interesting asymmetric alkene cyclopropanation using propargylic esters (Eq. (252)) [1313].



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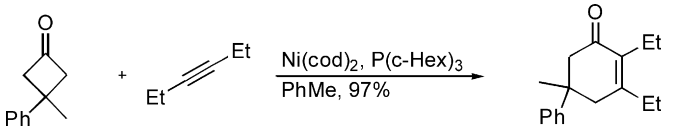

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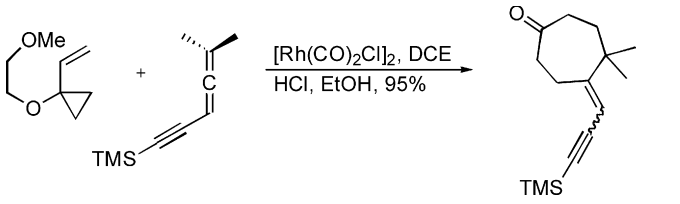

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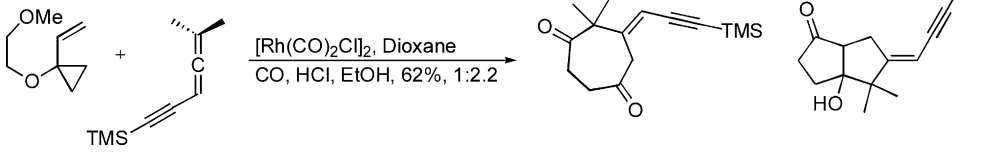

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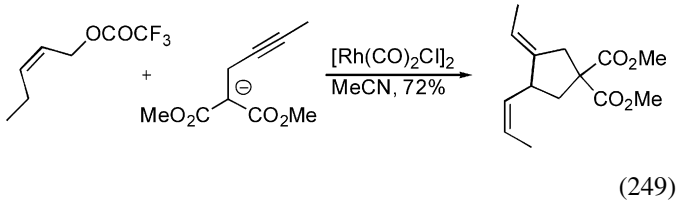

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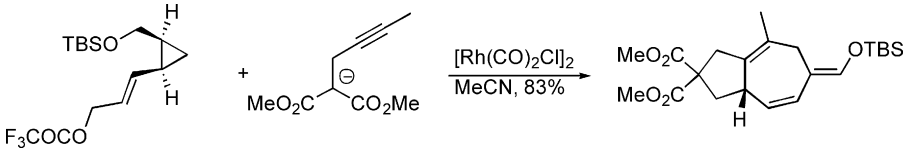

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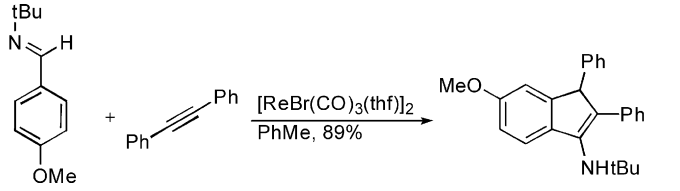

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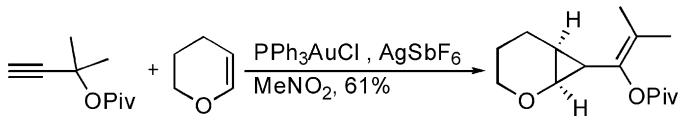
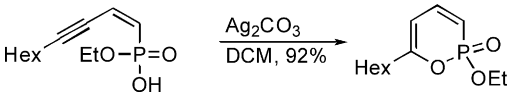


(252)

9.2. Cycloisomerizations

Intramolecular reactions wherein all atoms from the starting material are found in the product are included in this section. The mechanism of the palladium-catalyzed intramolecular hydroalkylation of 6-ene-1,3-diones was examined [1314]. An efficient catalyst system was reported for the intramolecular hydroalkylation of 6- and 7-ene-1,3-dicarbonyl compounds to give cyclohexanones [1315]. A related oxidative hydroalkylation of 7-, 8-, and 9-ene-1,3-dicarbonyl compounds to give heterocycloalkenones was reported using a palladium–ytterbium [1316] or a platinum–europium [1317] catalyst system. A nickel–ytterbium catalyst system was reported for the intramolecular hydroalkylation of alkyne-tethered 1,3-carbonyl compounds [1318].

A palladium-catalyzed intramolecular hydroalkoxylation of a pendant alkyne was used in a synthesis of terreinol [347]. Silver catalyzed intramolecular hydroalkoxylations and hydroacyloxylations of 1-ene-5-ols and 4-enoic acids [1319]. Silver catalyzed intramolecular hydroalkoxylations of (*Z*)-2-en-4-yne phosphonic monoesters to give 2*H*-1,2-oxaphosphorin 2-oxides (Eq. (253)) [1320]. Iridium catalyzed a tandem Claisen rearrangement–intramolecular hydroalkoxylation of aryl allyl ethers to give dihydrobenzofurans [1321].

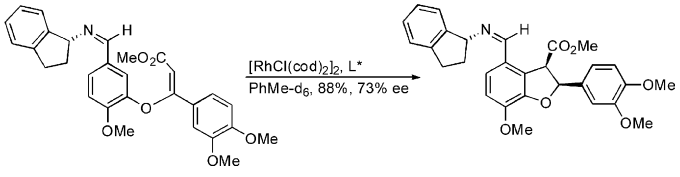


(253)

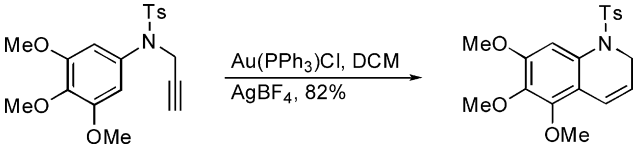
Rhodium catalyzed asymmetric intramolecular hydroacylations of 2-alkenylbenzaldehydes to give indanones [1322]. A titanium–rhodium-catalyzed intramolecular hydroacylation of 4-ynals forming cyclopentanones was reported [1323]. A

related rhodium-catalyzed intramolecular hydroacylation of formamides having a pendant alkyne leading to α-alkylidene-γ-lactams was described [1324].

Evidence for an aromatic electrophilic substitution not a carbon–hydrogen bond activation mechanism for the intramolecular hydroarylation of phenol esters of propargylic acids was reported [1325]. A rhodium-catalyzed hydroarylation of an alkenylphenol was used in a synthesis of (+)-lithospermic acid (Eq. (254)) [1326]. Gold and platinum catalyzed intramolecular hydroarylations of alkynes (Eq. (255)) [1327].



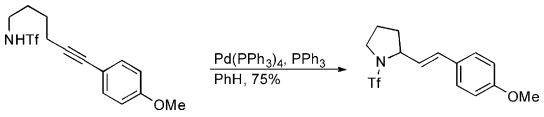
(254)



(255)

Palladium catalyzed the formation of five- and six-membered *N*-heterocycles from 1-amino-5- or -6-ynes (Eq. (256)) [1328]. Platinum-catalyzed intramolecular hydroaminations of pendant alkenes to give five- or six-membered rings [1329]. Silver and ruthenium catalyzed intramolecular hydroaminations of 1-amino-3,4-dienes to give 2,5-dihydropyrroles [1330]. A silver-catalyzed intramolecular alkyne hydroamination was used in a synthesis of (+)-pseudodistomin D [294].

Copper-catalyzed intramolecular hydroaminations of pendant alkynes were used to prepare (*Z*)-3-arylidene-4-acyl-3,4-dihydro-2*H*-benzoxazines [951] and 5-amino-7-azaindoles [1331]. A number of transition metals including silver, gold, copper, and palladium-catalyzed intramolecular hydroaminations of pendant alkynes to give 5-substituted proline derivatives [321]. A palladium-catalyzed intramolecular alkyne hydroamination was used in a synthesis of indolizine (−)-209D [322]. A titanium catalyzed intramolecular hydroamination of an alkyne was used in a synthesis of (+)-laudanosine and (−)-xylopine (Eq. (257)) [327]. Iridium catalyzed intramolecular hydroaminations, hydroacyloxylations, and hydroalkoxylations of 2-(1-alkyn-1-yl)-benzenamines, -benzylalcohols, -phenols, and -benzenamines [1332].



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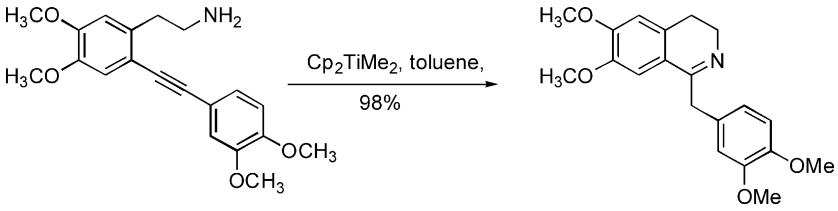
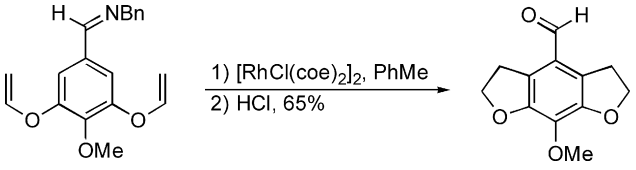


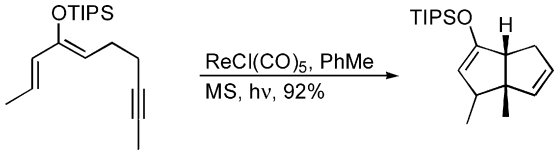
(257)

Rhodium catalyzed directed intramolecular hydroarylations of aromatic imines. This reaction was used in a synthesis of a mescaline analog (Eq. (258)) [1333]. Palladium catalyzed an intramolecular hydroarylation of 2-alkynoic acid esters of phenols forming coumarins [1334]. Palladium catalyzed the formation of benzofurans and chromenes from allyl phenyl ethers or homoallyl phenyl ethers [1335].



(258)

Gold, platinum, and tungsten but in particular rhenium catalyzed cycloisomerizations of 3-silyloxy-1,3-diene-7-ynes to give bicyclic compounds (Eq. (259)) [1336]. Platinum catalyzed an cycloisomerization-rearrangement of 2-(1,5-dihydro-3*H*-2,4-dioxepinyl)arylalkynes (Eq. (260)) [1337]. Platinum catalyzed cycloisomerizations of acetals derived from 2-alkynylphenols (Eq. (261)) [1338,1339]. A platinum catalyzed cycloisomerization of a 1,5-diene was used in a synthesis of *cis*-thujane (Eq. (262)) [1340].



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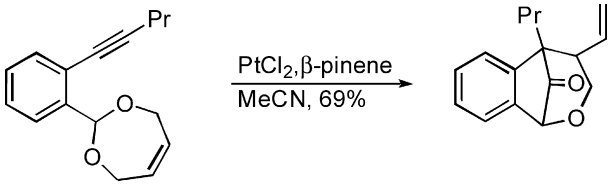

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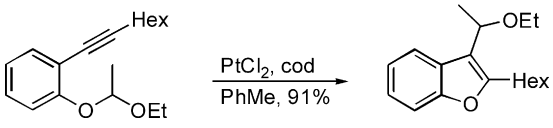

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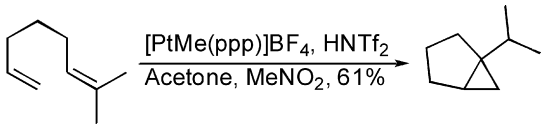
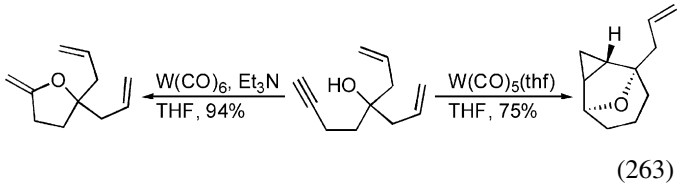


(262)

Tungsten catalyzed cycloisomerizations of 5-hydroxy-1-alkynes. Depending on the conditions, cyclopropanation of a pendant alkene may occur (Eq. (263)) [1341]. Tungsten catalyzed cycloisomerizations of a 1-yne-5-ols were used in syntheses toward altromycin B (Eq. (264)) [69] and erythro-4-deoxyglycals [1342]. Silver catalyzed cycloisomerizations of 6- and 7-ynals to give ketone-substituted five- and six-membered rings (Eq. (265)) [795].



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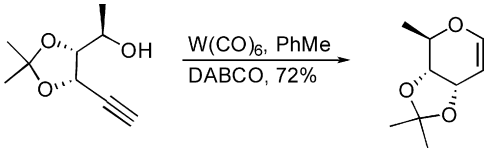

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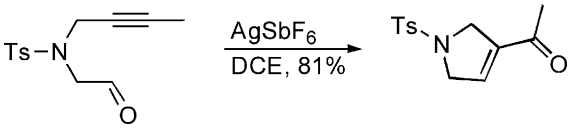
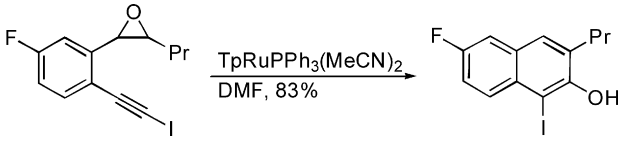


(265)

Ruthenium catalyzed a solvent dependent cycloisomerization of 2-(2-iodoethenyl)phenyloxiranes to give six- or seven-membered rings depending on the solvent (Eqs. (266) and (267)) [1343]. Ruthenium catalyzed a cycloisomerization of 1,3-diene-5-ynes having an adjacent methylene group to form substituted benzenes (Eq. (268)) [1344]. Ruthenium catalyzed *cis*-3-en-1-yne cycloisomerizations (Eq. (269)) [1345].



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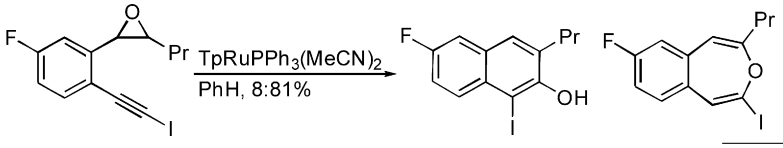

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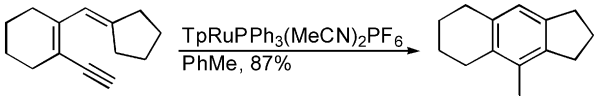

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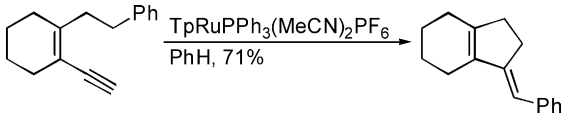
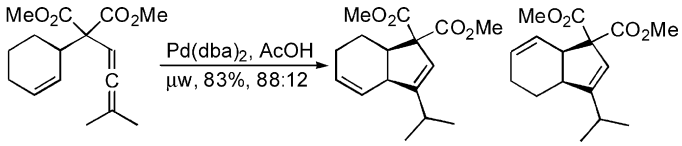


(269)

Palladium catalyzed cycloisomerizations of 1,2,6-trienes (Eq. (270)) [1346]. Palladium catalyzed a cycloisomerization of 6-ynylidenecyclopropanes to give bicyclo[3.3.0]ring-systems [1347]. Rhodium catalyzed cycloisomerizations of 1,6- and 1,7-enynes to give six- or seven-membered rings (Eq. (271)) [1348]. A rhodium-catalyzed cycloisomerization of an 1-cyclopropyl-1,6-enyne was used in synthesis of tremulenediol A and tremulenolide A (Eq. (272)) [610].



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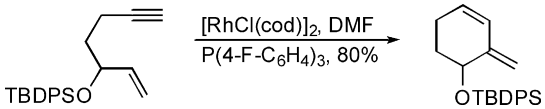

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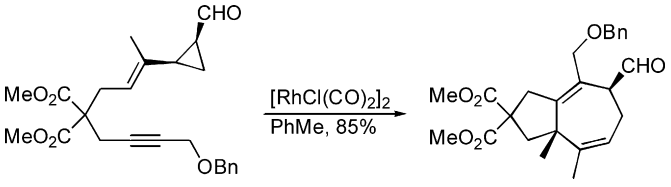
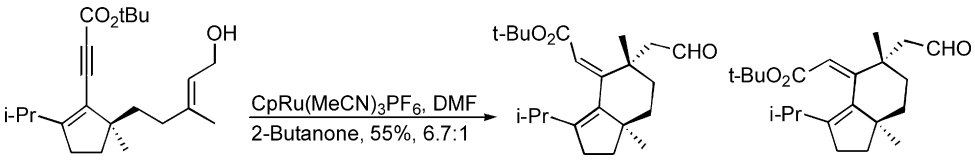


(272)

A ruthenium-catalyzed cycloisomerization of a 2-ene-8-yne-1-ol was used in a synthesis of (+)-allocyathine B_2 (Eq. (273)) [128,1349] and a related rhodium-catalyzed reaction of a 2-ene-7-yne-1-ol was used in a synthesis of (−)-blastmycinolactol [1350]. A related ruthenium catalyzed cyclization of 1-hydroxy-2,7 (or 8)-diynes to give substituted five- or six-membered rings was developed and used in a synthesis of α-kainic acid (Eq. (274)) [1351].



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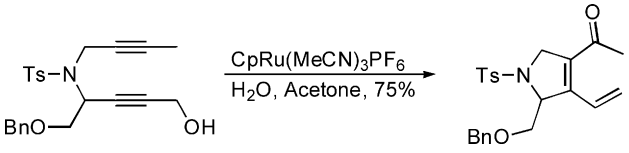
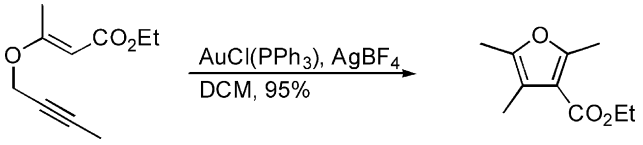


(274)

The gold-catalyzed cycloisomerization of 2-(4-alkyn-1-yl)furans to give substituted arenes was shown to proceed via an arene oxide intermediate [1352]. Gold catalyzed an intramolecular [4 + 2]cycloisomerization of 1-aryl-1,6-diynes [1353]. Gold catalyzed the cycloisomerization of propargyl enol ethers (Eq. (275)) [1354] and of (*Z*)-2,4-enyne-ols [1355] to give furan derivatives. Gold catalyzed cycloisomerizations of 3-hydroxy- or -alkoxy-1,5-enynes (Eq. (276)) [1356] and of 4-(1,2-dienyl)-2-azetidinones (Eq. (277)) [1357]. Gold catalyzed a [4 + 2]cycloisomerization of 1,3-enynes or arylalkynes with alkenes (Eq. (278)) [1358].



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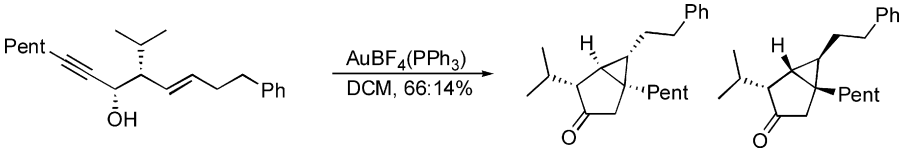

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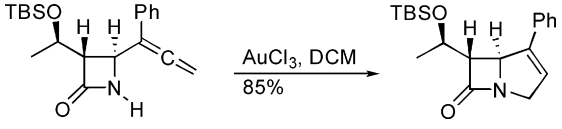

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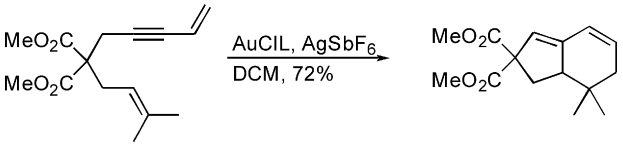
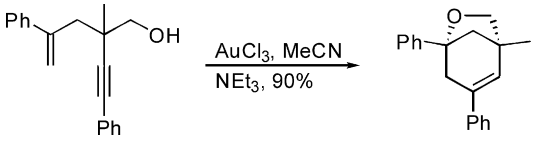


(278)

Gold-catalyzed cycloisomerizations of 1,6-enynes to give a variety of cyclic products depending on the substrate [1359]. Gold-catalyzed cycloisomerizations of amide- or hydroxy-tethered 1,5-enynes to give heterocyclic bicycles(Eq. (279)) [1360]. Gold also catalyzed cycloisomerizations of bis-homopropargylic diols to give strained bicyclic ketals (Eq. (280)) [1361]. Gold catalyzed a tandem [3,3]rearrangement-[2 + 2]cycloaddition of propargylic esters Eq. (281)) [1362]. Gold, copper, and platinum catalyzed a [4 + 2]cycloisomerization of 1,9-diyne-6-ones, 1,10-diyne-7-ones, 1,6-diyne-10-als, 1,9-enyne-6-ones, 1,10-enyne-7-ones, and 1,6-enyne-10-als to give polycyclic compounds [1363,1364]. This reaction was used in a synthesis of (+)-ochromycinone and (+)-rubiginone B_2 (Eq. (282)) [57]. A related cyclization was used in a synthesis of heliophenanthrone [334]. Gold catalyzed the cycloisomerization of 1-halo-4-oxo-1,2-dienes to give halogenated furans. Depending on the catalyst, the halogen may migrate (Eq. (283)) [1365].



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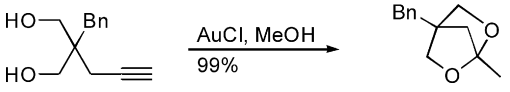

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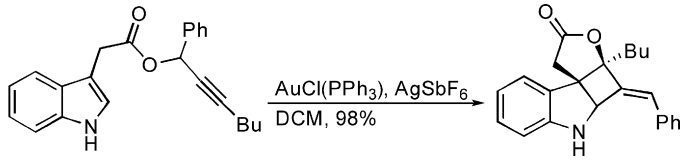

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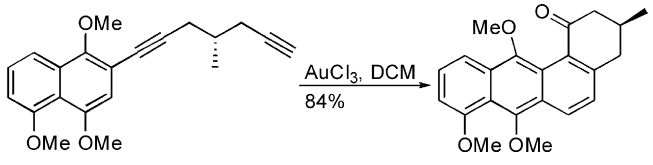

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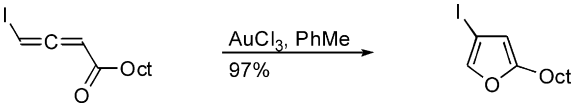
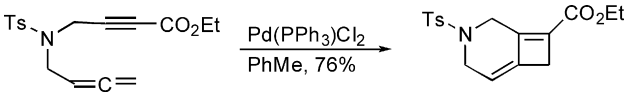


(283)

Ruthenium, molybdenum, and palladium catalyzed [2 + 2]cycloisomerizations of 5,6-, 6,7-, and 7,8-diene-1-ynes (Eq. (284)) [1366,1367]. Seven-membered rings were prepared by ruthenium-catalyzed [5 + 2]cycloisomerization of 1-cyclopropyl-6-yne-1-enes (Eq. (285)) [1368]. In contrast nickel catalyzed the formation of five-membered rings from 1-cyclopropyl-6-yne-1-enes (Eq. (286)) [1369]. Nickel catalyzed an asymmetric cycloisomerization of a 1,6-diene to give a methylidenecyclopentane [1370]. Rhodium catalyzed a cycloisomerization of 1-aryl-1-hydroxy-3-ynes to give indanones (Eq. (287)) [1371,1372]. Trimethylaluminum mediated a nickel-catalyzed cycloisomerization of 1,6-enones (Eq. (288)) [1373].



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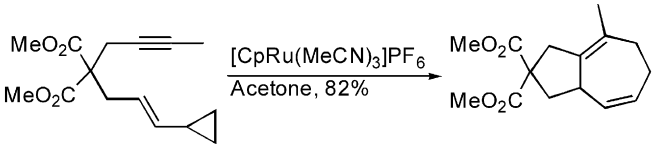

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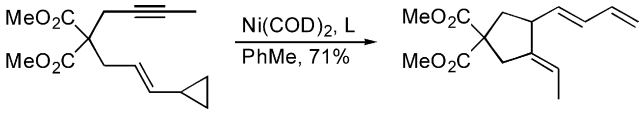

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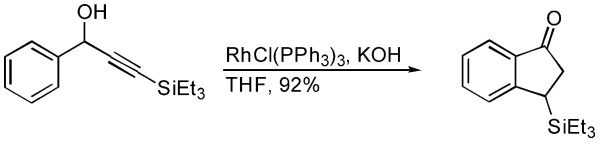
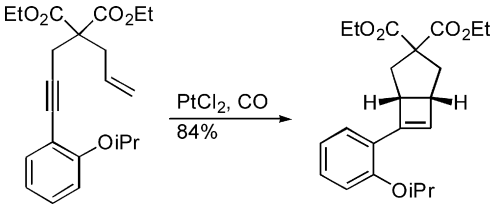


(287)

(288)

The mechanism of platinum-catalyzed enyne metathesis was examined computationally [1374]. Platinum catalyzed cycloisomerizations of 1,6-enynes to give cyclobutene containing compounds (Eq. (289)) [1375]. Platinum catalyzed cycloisomerizations of enyne propargyl esters to form five-membered rings (Eq. (290)) [1376]. A related reaction of esters derived from 5-hydroxy-1,6-enynes to give bicyclo[4.1.0]-ring systems was reported (Eq. (291)) [1377]. Platinum catalyzed cycloisomerizations of 2-amino-1,5-enynes to give amino-substituted aromatic compounds (Eq. (292)) [1378].



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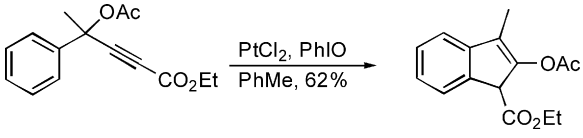

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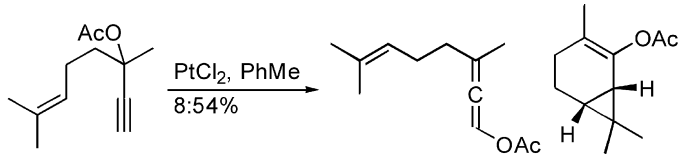
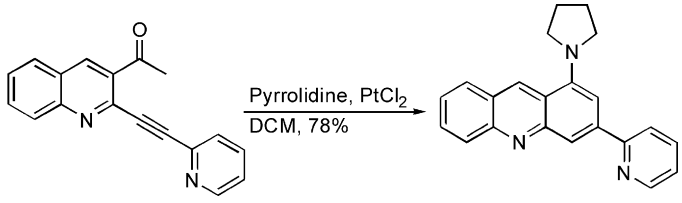
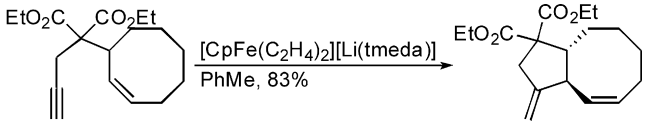


(291)



(292)

Cycloisomerizations of 1,6-enynes catalyzed by iron were reported (Eq. (293)) [1379]. Iridium catalyzed a 1,6-enyne cycloisomerizations–Diels–Alder reaction (Eq. (294)) [1380]. Iridium catalyzed *Z*-selective cycloisomerizations of 1,6-enynes [1301]. Iridium catalyzed cycloisomerizations of 4-aza-1,6-enynes to give 3-azabicyclo[4.1.0]heptenes [1381]. Rhodium catalyzed asymmetric cycloisomerizations of 1,6-enynes [1382]. Ruthenium catalyzed cycloisomerizations of 1,6-enynes to give alkenyl-substituted five-membered unsaturated rings [1383].



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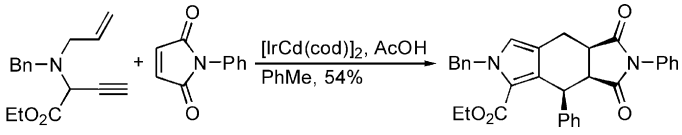
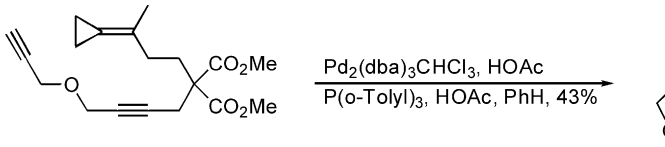


(294)

Palladium catalyzed cycloisomerizations of alkyne- and diyne-substituted methylenecyclopropanes was reported affording polyfunctionalized compounds (Eq. (295)) [461]. Palladium catalyzed a cycloisomerization of alkynyl-substituted benzaldehyde acetals wherein both alkoxy groups moved (Eq. (296)) [1384].



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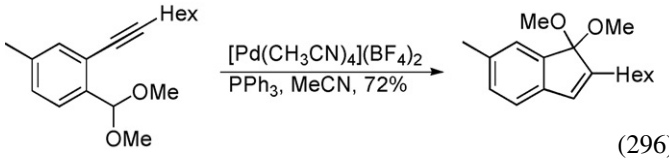
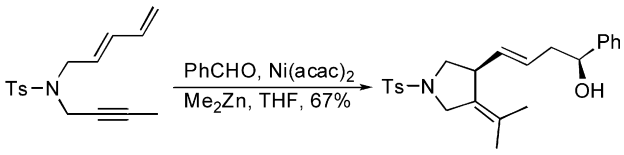


(296)

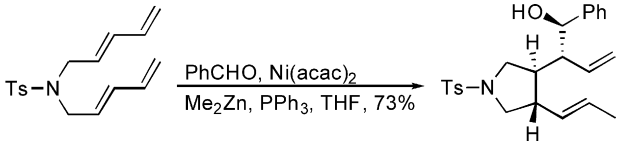
Palladium catalyzed cycloisomerizations of 1-acetoxy-2,7-enyne to give five-membered rings having an 1′-acetoxyalkylidene substituent. One example of an asymmetric reaction was reported [1385]. A related reaction of 1-chloro (or bromo)-2,7-enynes gave the corresponding 1′-chloro (or bromo) derivative [1386]. Palladium catalyzed the cycloisomerization of 1-hydroxy-5-2,7-enynes to give ethanal-substituted five-membered rings [1387,1388].

9.3. Other carbocyclizations

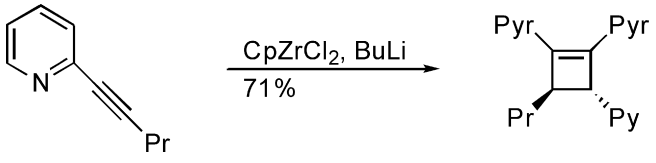
Rhodium catalyzed a [2 + 2 + 2]cyclization of alkynylboronates to give aromatic products [1032,1389]. Nickel catalyzed the [2 + 2 + 2]cycloaddition of intermediately formed benzyne and diynes to give naphthalene derivatives [1390]. Nickel catalyzed four-component couplings of dimethylzinc, an alkyne, a 1,3-diene, and an aldehyde. When the alkyne and diene is part of the same molecule, five- or six-membered rings are formed (Eq. (297)) [1391]. Nickel catalyzed a related enantioselective reaction of dimethylzinc, an aldehyde and a 1,3,8,10-tetraene (Eq. (298)) [1392]. Nickel catalyzed a regio- and stereoselective formation of cyclobutenes from alkynes (Eq. (299)) [1393]. Nickel catalyzed the cyclization of 5,7- and 6,8-dienals with an aldehyde, and a diboryl reagent (Eq. (300)) [1394]. Nickel catalyzed a diastereoselective cyclization of chiral alkenyl sulfoxides having a tethered alkenyl ester (Eq. (301)) [1395]. Nickel catalyzed a carboxylative cyclization of 1,6- and 1,7-enynes in the presence of carbon dioxide (Eq. (302)) [1396].



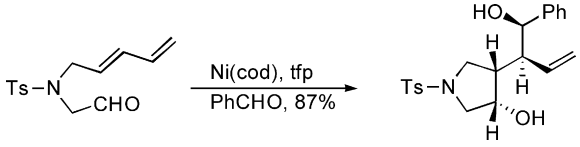
(297)



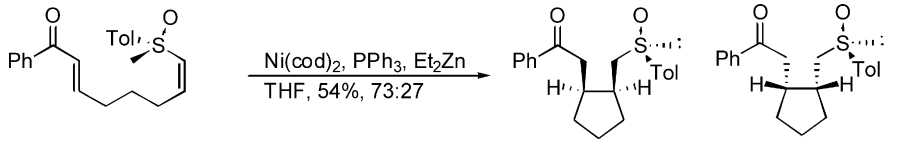
(298)



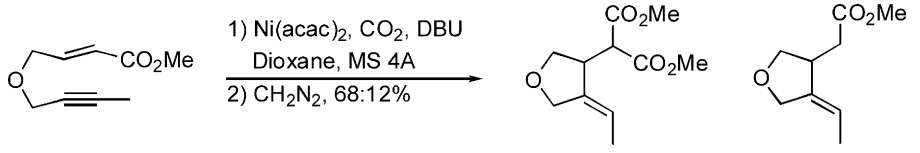
(299)



(300)



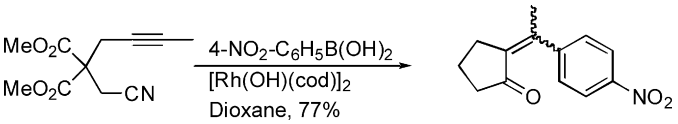
(301)



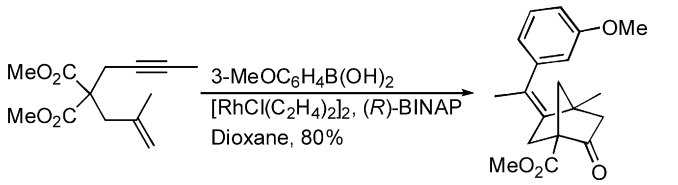
(302)

Ruthenium, palladium, iridium, and gold catalyzed Markovnikov hydroiodinations of 1,2-bis(ethynyl)benzene [1397]. In contrast, platinum catalyzed a hydroiodination cyclization. Rhodium catalyzed an alkyne arylation–cyclization sequence using arylboronic acids and yne-nitriles (Eq. (303)) [1398] or of yne-ones [1399]. Rhodium catalyzed a related reaction of 1-alkoxy-2,7-enynes with aryl boronic acids [1400]. Alkylation of a pendant ester functionality was reported (Eq. (304)) [1401]. A rhodium catalyzed cyclization–cycloaddition sequence using ene-yne-benzaldehydes was reported (Eq. (305)) [1402]. Rhodium catalyzed a four-component cyclization of a alkenylcyclopropane, an alkyne and two carbon

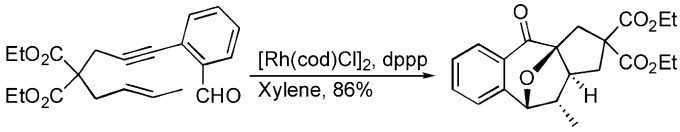
monoxides (Eq. (306)) [1403]. Rhodium catalyzed an enantioselective reductive cyclization of 1,6-enynes (Eq. (307)) [1404]. Rhodium catalyzed a enantioselective arylation–cyclization of alkynes having an electron deficient alkene (Eq. (308)) [1405]. A rhodium-catalyzed alkyne–arylation intramolecular cyclobutanone alkylation ring expansion was described (Eq. (309)) [1406].



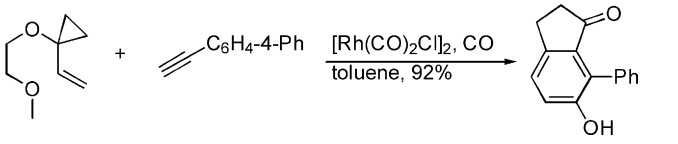
(303)



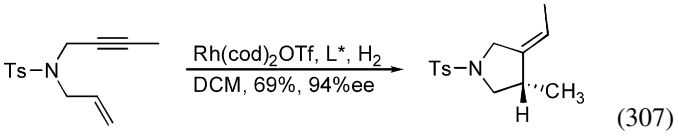
(304)



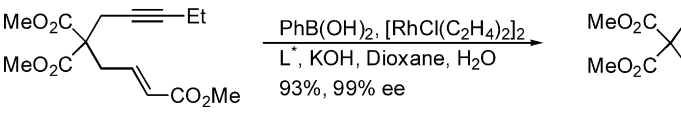
(305)



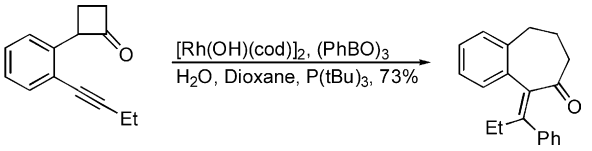
(306)



(307)

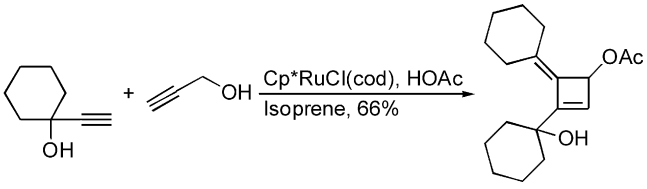


(308)



(309)

Ruthenium catalyzed cyclization of 1-yne-ols in the presence of an acid to give alkylidenecyclobutenes (Eq. (310)) [1407]. Ruthenium catalyzed cycloisomerizations of 1,5-enynes in the presence of water to form cyclopentanones (Eq. (311)) [1408]. Ruthenium catalyzed the cyclization of 1,1-diaryl-1-penten-4-yn-3-ols with diphenylphosphine oxide to give polyaromatic products (Eq. (312)) [1409]. Palladium catalyzed silylative cyclizations of alkadiynes or enynes [1410]. Ruthenium catalyzed a hydrative 1,6-diyne cyclization forming enones (Eq. (313)) [1411]. Ruthenium catalyzed cyclization of 3-ene-1,5-diynes in the presence of a nucleophile (Eq. (314)) [1412].



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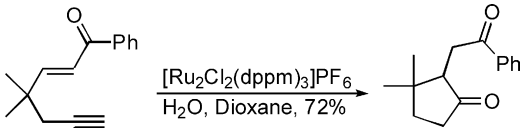

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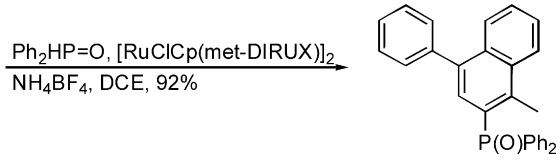

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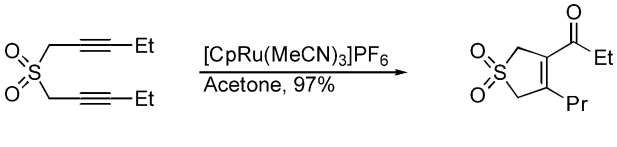

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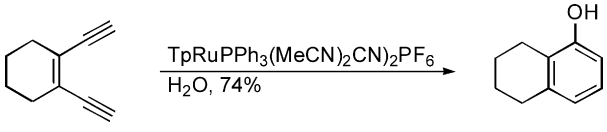
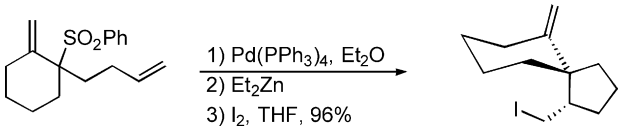


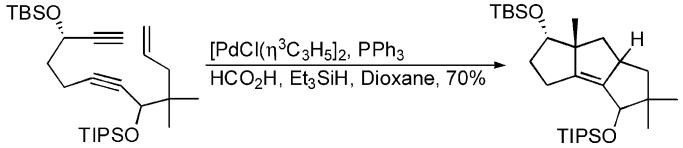
(314)

Palladium-catalyzed cyclization–acetoxylation of 2,7-enyne-1-ones to give 1′-acetoxyalkylidene substituted five-membered rings [1413]. A palladium-catalyzed, zinc-mediated reaction of phenylsulfonyl-substitute 2,7-dienes followed by addition of iodide was used in a synthesis of (−)-erythrodiene (Eq. (315)) [1414]. Palladium-catalyzed reductive and arylative cyclizations of 1,2-diene-6/7-ynes using triethylsilane and aromatic boronic acids, respectively [1367,1415]. Palladium-catalyzed cyclizations of 1,3,7,9- and 1,3,8,10-tetraenes in the presence of phenol or a sulfonamide to give five- or six-membered rings [1416].



(315)

A palladium-catalyzed reductive cyclization of 1-ene-6,11 diynes was used in a synthesis of ceratopicanol (Eq. (316)) [1417]. A palladium-mediated oxidative coupling of a diaryl amine was used in a synthesis of murrastifoline A (Eq. (317)) [206]. An unusual palladium-mediated cyclization was used in a synthesis of dragmacin F (Eq. (318)) [209]. The stereoselectivity of the palladium-catalyzed cyclization of 1,2-diene-7-als with hexamethylditin was controlled by addition of Lewis acid (Eq. (319)) [1418].



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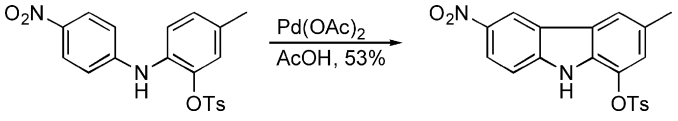

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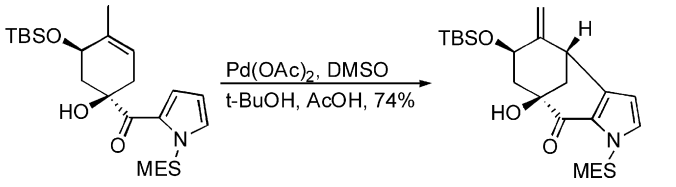

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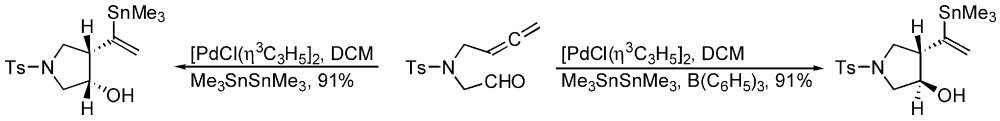
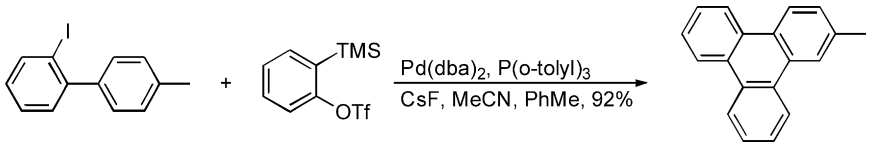


(319)

Palladium catalyzed reactions between 2-halobiphenyls and intermediately formed arynes to give polycyclic aromatic compounds (Eq. (320)) [1419]. Palladium catalyzed the cyclization of 2-halobenzaldehydes with arynes to give fluoren-9-ones (Eq. (321)) [1420]. Palladium catalyzed a cyclization of activated methylene compounds having a tethered alkyne and in the presence of an aryl iodide (Eq. (322)) [476]. Palladium-catalyzed cyclizations of cyclic non-conjugated dienolsilyl ethers (Eq. (323)) [1421]. A palladium catalyzed [3 + 2]cycloaddition of trimethylenemethane to an aldehyde forming a 3-methylenecyclopentanone was used in a synthesis of aureothin and *N*-acylaureothamine [61].



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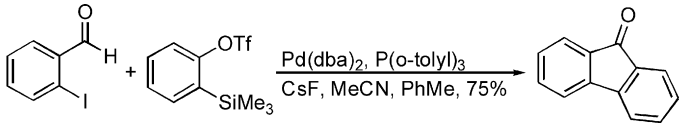

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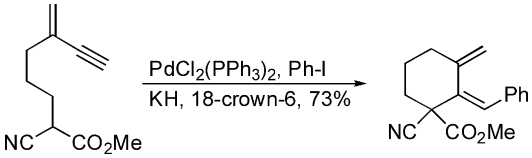

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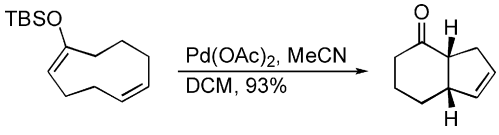
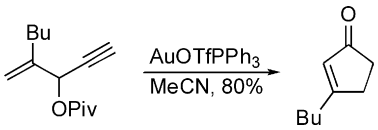


(323)

Gold catalyzed the cyclization of pivalic esters of 3-hydroxy-1-en-4-ynes to give 2-cyclopentenones (Eq. (324)) [1422]. A platinum and gold catalyzed asymmetric cyclization of 1,6-enynes in the presence of an alcohol to give methylidene cyclopentanes having the alcohol incorporated was described (Eq. (325)) [1423]. Platinum and palladium catalyzed enyne metathesis-aromatization of 1-(1-methoxy-3-buten-1-yl)-2-(1-

alkynyl)benzenes to give alkenyl-substituted naphthalenes (Eq. (326)) [1424].



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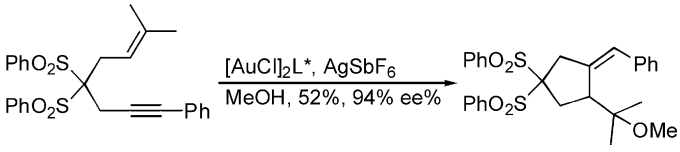

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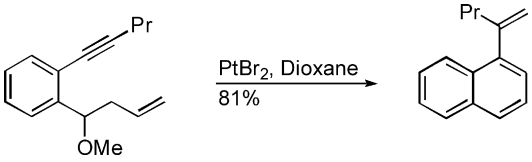
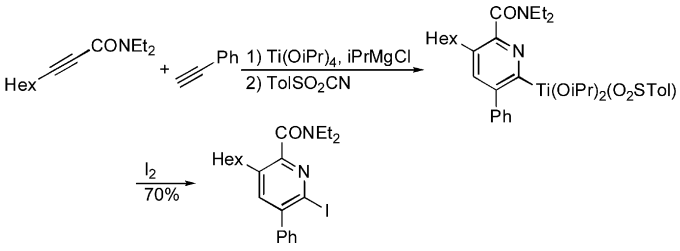


(326)

Titanium-mediated cyclization of two alkynes and a nitrile to give titanium-substituted pyridines followed by electrophilic cleavage (Eq. (327)) [1425]. Zirconium mediated a cyclization–elimination of 2-heterosubstituted 1,6-dienes and 1,6-enynes (Eq. (328)) [1426]. Iron catalyzed a cyclization of 3-ene-1,5-diynes to give aromatic compounds [1427]. Chromium mediated the formation of polycyclic aromatic compounds from dihalobiaryls and alkynes (Eq. (329)) [1428]. Tungsten catalyzed [3 + 2]cycloaddition reactions of intermediate carbonyl ylids (Eq. (330)) [1429].



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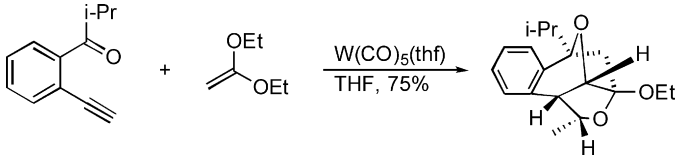
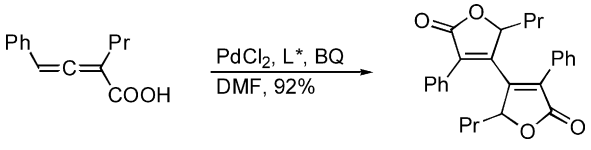


(330)

9.4. Other heterocyclizations

Reactions wherein a carbon–heteroatom bond was formed in the cyclization and with gain/loss of atoms are discussed herein. Palladium catalyzed an oxidative cyclization dimerization of 2,3-dienoic acids (Eq. (331)) [1430]. A related homodimerization of 1,2-dien-3-ones with 2,3-dienoic acid amides was also described [1431]. Palladium catalyzed a two component coupling of 2,3-dienoic acids with 2,3-diene-1-ols to give substituted furanones (Eq. (332)) [1432]. Palladium catalyzed the formation of 2-alkoxy-substituted tetrahydrofurans from allylic alcohols and alkenyl ethers [1433,1434]. This reaction was used to prepare (−)-dihydroxanthatin (Eq. (333)) [735] and fraxinellone limonoids [1237]. Palladium catalyzed the reaction of 2-alkynylbenzaldehydes with allyl silanes to give isochromene derivatives [1435]. Palladium catalyzed a cyclization–alkoxycarbonylation sequence of 5-oxo-1-ynes to give heterocyclic compounds (Eq. (334)) [1436]. This type of reaction was used in syntheses of diacetylenic spiroacetal enol ethers (Eq. (335)) [333].



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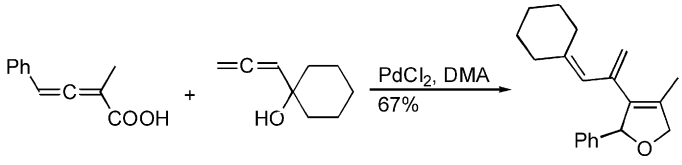

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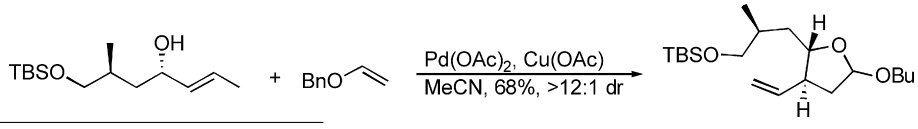

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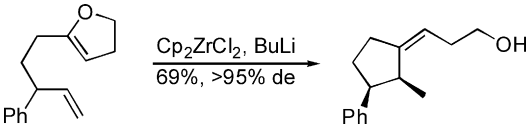

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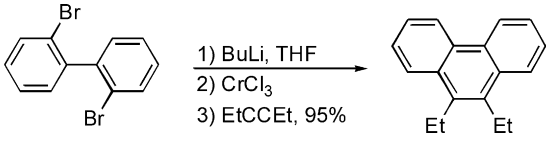

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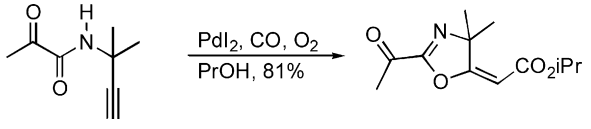

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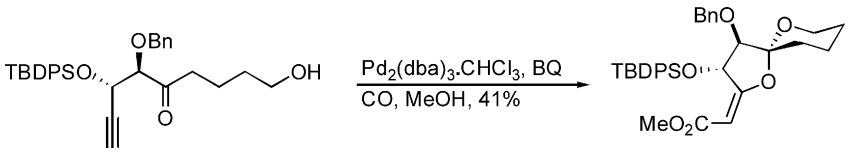
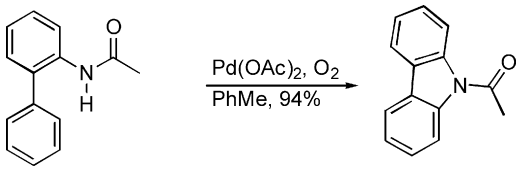


(335)

Palladium catalyzed the formation of oxidoles from 2-alkynylbenzenisocyanates and terminal alkynes (Eq. (336)) [1437]. Palladium catalyzed the formation of indole derivatives from 3-aryl-2-aminopropenoic acid esters (Eq. (337)) [1438]. Palladium catalyzed an intramolecular carbon–nitrogen bond formation via carbon–hydrogen bond activation (Eq. (338)) [1439]. Palladium catalyzed a coupling of terminal alkynes, hydrazine or hydroxylamine, aryl iodides, and carbon monoxide to give pyrazoles or isoxazoles (Eq. (339)) [1440]. Palladium catalyzed the formation of 2-aryl-4-aminoquinolines from 2-(1-alkyn-1-yl)benzenamines, carbon monoxide, and aryl iodides and an amine (Eq. (340)) [1441]. Palladium catalyzed the formation of pyridone derivatives from isoxazolidine-5-



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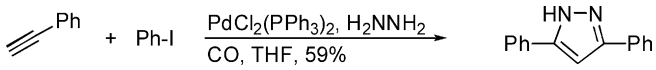

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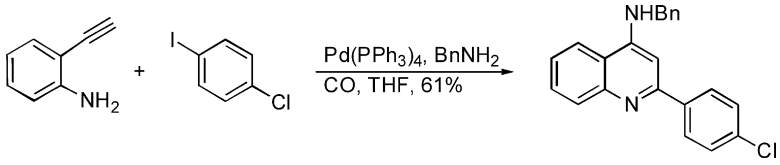

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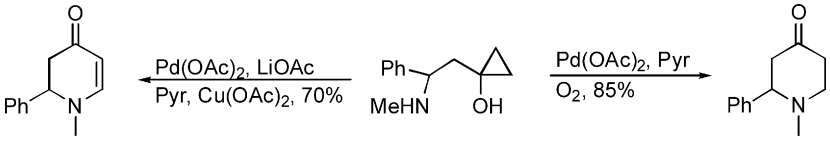
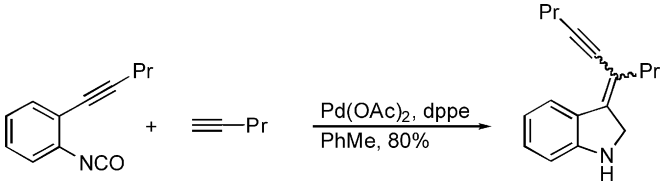


(341)

spirocyclopropanes. Different oxidation states of the product can be obtained depending on the catalyst used (Eq. (341)) [1442]. Palladium-catalyzed reductive *N*-heteroannulations of aromatic 2-nitro-1-alkene derivatives in the presence of carbon monoxide to afford indoles [1443,1444] was used in a synthesis of KDR kinase inhibitors [376].



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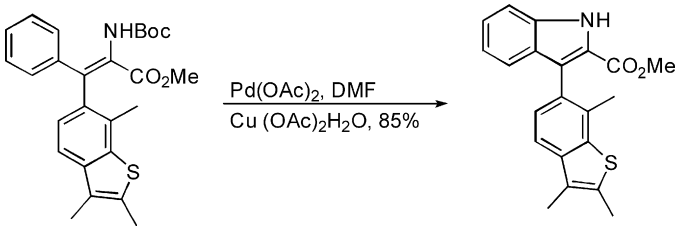
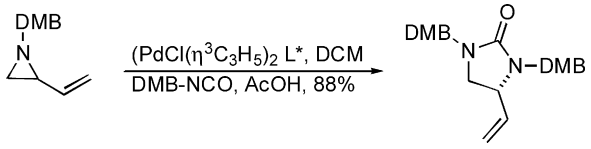


(337)

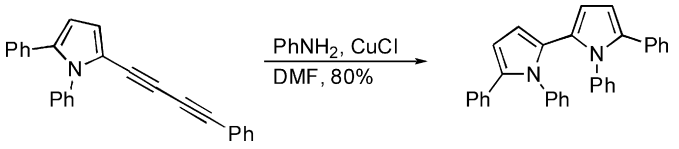
Palladium-catalyzed intramolecular hydroaminations and hydroarylations of alkene tethered pyrrolo-2-carboxamindes [1445]. Palladium catalyzed a decarboxylative [2 + 3]cycloaddition of 5,5-dithenylkoxazolidin-2-ones with alkylidene malonate derivatives to give substituted pyrolidines [1446]. A palladium-catalyzed dynamic kinetic asymmetric transformation of an ethenylaziridine with benzylisocyanate was used in a synthesis of (+)-pseudodistomin D (Eq. (342)) [294].



(342)

Copper mediated the formation of 2,2′-bipyrroles from 1,7-diene-3,4-ynes and an aromatic amine (Eq. (343)) [1447]. Copper catalyzed the formation of pyrroles from an alkyne and an isocyanide [1448]. Copper catalyzed an asymmetric 1,3-dipolar cycloaddition of azomethine ylides and alkenes to give substituted pyrrolidines [1449]. Copper and gold catalyzed the formation of alkoxy-substituted furan derivatives from 2-(1-alkyn-1-yl)-2-alkene-1-ones (Eq. (344)) [394,1450].

Copper catalyzed the formation of 5-methylene-1,3-oxazolidin-2-ones from propargyl alcohols, amines, and carbon dioxide [1451].



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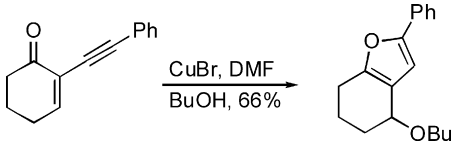
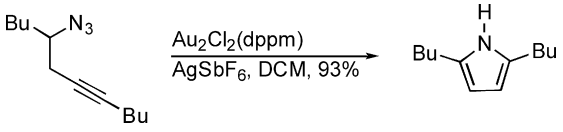


(344)

Gold catalyzed the formation of pyrroles from 1-azido-3-ynes (Eq. (345)) [1452]. A gold-catalyzed cyclization of *t*-butyl 2,3-dienoates to give 2,4-functionalized butenolides was reported [1453]. Silver catalyzed the formation of isochromenes from an alcohol and 1,6-diyne-3-ones (Eq. (346)) [1454]. Platinum catalyzed the annulation of 1-trimethylsilyl-1-alken-5/6-ols with aldehydes to give tetrahydropyranes and tetrahydrofurans (Eq. (347)) [1455]. Iridium catalyzed a sequential cycloisomerization–hydroalkoxylation of bis-homopropagylic alcohols (Eq. (348)) [1456].



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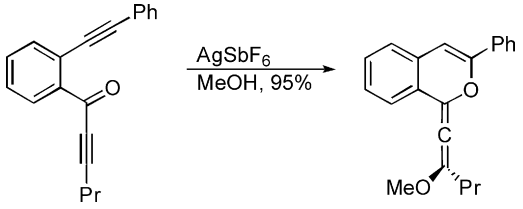

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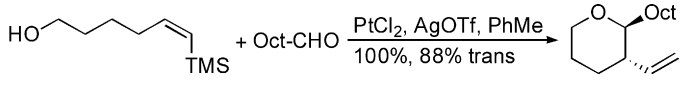

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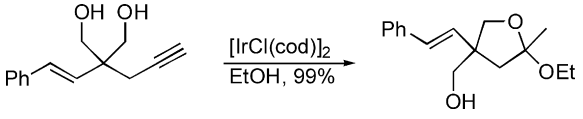
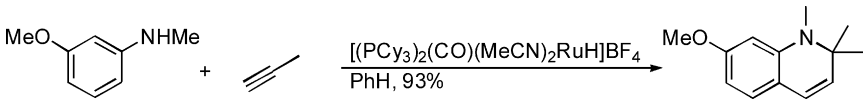


(348)

Rhodium catalyzed the formation of quinoline derivatives from benzenamines and two terminal alkynes (Eq. (349)) [1457]. Rhodium catalyzed the formation of *E*-alkylidene derivatives of 3*H*-furan-2-ones from 4-pentynoic acid and aldehydes (Eq. (350)) [1458]. Ruthenium catalyzed an oxidative cyclization of 1,5-dienes to give functionalized tetrahydrofurans (Eq. (351)) [1459]. Ruthenium catalyzed an asymmetric intramolecular [4 + 2]cycloaddition forming oxazinolactams [1460]. Nickel catalyzed the formation of phthalimides and maleimides from 2-iodobenzoic acid esters and 3-iodo-propenoic acid esters and isocyanates [1461].



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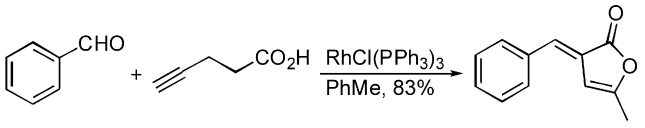

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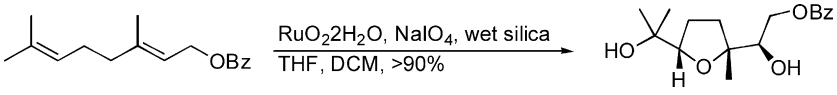
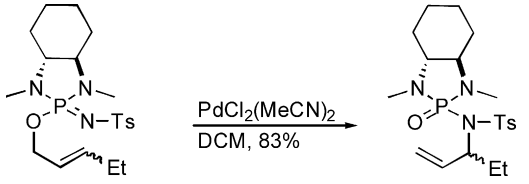


(351)

10. Miscellaneous isomerizations (including alkene isomerizations)

Palladium catalyzed an asymmetric Carroll rearrangement of allylic α-acetamido-β-ketoesters [1462]. Palladium catalyzed a Cope rearrangement [1463] and an [3,3]-aza-phospha-Cope rearrangement (Eq. (352)) [1464]. Tungsten catalyzed a Cope rearrangement of allenyl silyl enol ethers (Eq. (353)) [1465]. Palladium-catalyzed asymmetric rearrangements of trifluoroacetimidates to allylic amides [1466–1468].



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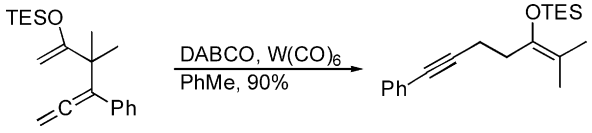
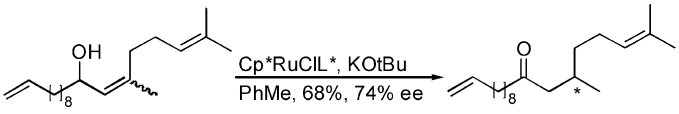


(353)

Ruthenium catalyzed the isomerization of allylic alcohols to aldehydes or ketones [1469] and this reaction was used in an asymmetric synthesis of muscone (Eq. (354)) [1470]. Mechanistic insights were presented [1471]. Rhodium catalyzed the isomerization of an allylic ether to an enol ether [1472]. Palladium catalyzed an alkene isomerization to the more substituted alkene in a synthesis of (−)-physovenine and

(−)-3*a*-hydroxyfuroindoline [601]. Rhodium catalyzed alkene isomerizations to the more substituted alkene was used in synthesis of the C7–C15 decalin system of tetrodomycin [1473] and pandamarilactonine A [1474]. Rhodium catalyzed the isomerization of propargylic sulfinic esters to allenyl sulfones [1024]. Rhodium and copper catalyzed a rearrangement of 5-alkynals to give 6-yn-2-ones (Eq. (355)) [1475]. A rhodium catalyzed kinetic resolution isomerization of propargylic alcohols to 2-enones was reported [1476].



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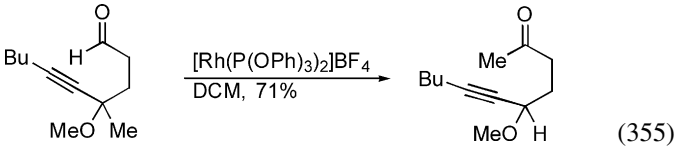

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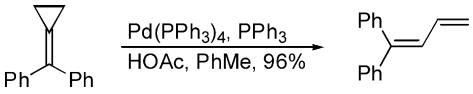
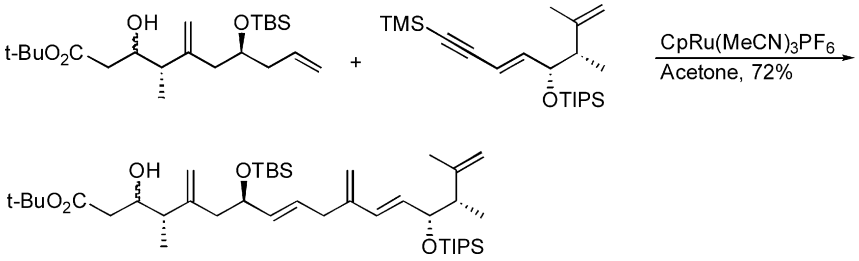
A palladium catalyzed yne-one to dienone isomerization was used in a synthesis of bisorbicillinolide, bisorbicillinol, and bisorbibutenolide [1477]. Palladium catalyzed the isomerization of alkylidenecyclopropanes to 1,3-dienes in the presence of acetic acid (Eq. (356)) [1478].



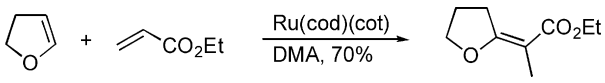
(356)

Platinum catalyzed enantioselective carbonyl-ene reactions [1479]. A copper catalyzed asymmetric carbonyl-ene reaction was used in a synthesis toward amphidinolide B1 [651]. Intermolecular alkene–alkyne couplings (Alder-ene reactions) to give 1,4-dienes, catalyzed by ruthenium were used in a synthesis of 21,22-diepimembrarollin [1480], of 1,4-dien-1-ylboronic esters [1481], (+)-amphidinolide A [1482,1483] and amphidinolide P (Eq. (357)) [1484]. A rhodium-catalyzed Alder-ene reaction–intramolecular Michael addition of propargyl alcohols with 3-enones was reported to give 4-methylene-2,6-dihydropyrans [499].



(357)

Ruthenium catalyzed a coupling of dihydrofurans with enoates (Eq. (358)) [1485]. A ruthenium-catalyzed allylic peroxide to bisepoxide was used in a synthesis of elysiapyrones (Eq. (359)) [68]. A gold-catalyzed ring-expansion of cyclopropanols and cyclobutanols was reported (Eq. (360)) [1486].



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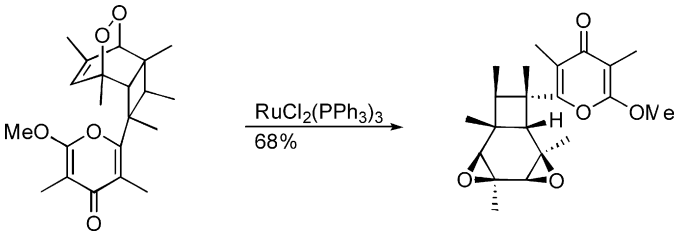

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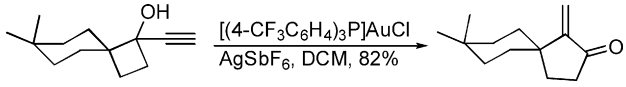

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11. Formation of carbon–halogen bonds

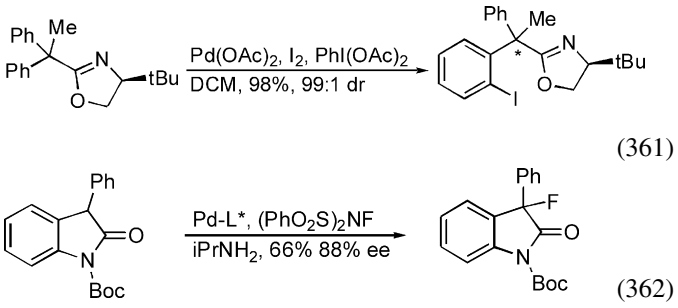
E-Alkenyl iodides were prepared by the chromium-mediated Takai alkenylation of aldehydes using iodoform in synthesis of 10-hydroxyasimicin (*E*/*Z* = 3.7:1) [323], C6–C21 segment of amphidinolide E [270] and spirofungins [228]. In a related fashion, 1,1-diiodoethane was used in a synthesis of musacins [1487].

Hydrozirconation of terminal alkynes followed by addition of iodine or NBS to produce alkenyl iodides or bromides were used in synthetic applications toward latrunculin A [108], (+)-apiosporamide [127], octalactin A [1488], (4*E*,6*Z*,10*Z*)-4,6,10-hexadecatrien-1-ol [331], lobatamide analogs [52], pyranicin [340], toward (+)-sorangicin [268] and the C10–C24 fragment of inostamycins [253]. Regioselective hydrozirconation of an internal alkyne followed by iodine quench was used in a synthesis of spirofungins [228].

Zirconium-catalyzed methylalumination–iodine quench sequences to form trisubstituted alkenyl iodides were used in syntheses of δ-*trans*-tocotrienoloic acid [135], bis-deoxylophotoxin [91], toward herbimycin A [79] and methyl

sarcoate [20]. Methylalumination of a terminal alkyne using trimethylaluminum and $ZrCp_2Cl_2$ was reported [1489].

Palladium catalyzed an asymmetric monoiodination of unactivated carbon–hydrogen bonds having an adjacent oxazoline group (Eq. (361)) [1490]. Palladium-catalyzed asymmetric fluorination of oxindoles (Eq. (362)) [1491], α-cyanoacetates [1492] and β-ketophosphonates [1493] were described.



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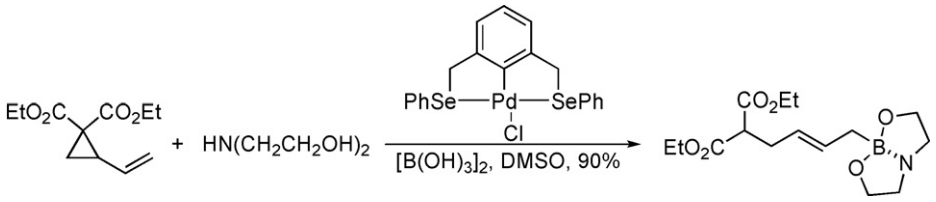
12. Formation of carbon–heteroatom bonds from boron, bismuth, silicon, zinc and tin reagents

Copper catalyzed the *N*-arylation cyocine and adenine [1494], cyclic imides [1495], sulfoximines [1496], 3-trimethylsilylindazole [1497] and purine nucleosides [1498] with arylboronic acids. Copper catalyzed *N*-alkenylation of aziridines using alkenylboronic acids [539]. Copper catalyzed amination of organozinc reagents using *O*-acyl hydroxylamines as the electrophile [1499]. Nickel catalyzed *N*-arylations of bromomagnesium diaryl amides with aryl halides [1500].

Palladium catalyzed the formation of allyl boronic acids from alkenyl cyclopropanes, alkenyl aziridines, or allyl acetates using tetrahydroxydiboron (Eq. (363)) [1501]. Iridium catalyzed a direct borylation of aromatic hydrocarbons using bis(dipinacolylborane) [1502]. The mechanism of this reaction was studied [1503]. Selective *ortho*-borylation of aromatic nitriles was observed [1504]. Iridium catalyzed the formation of 2-silicon-substituted heteroarenes using 1,2-di-*t*-butyl-1,1,2,-tetrafluorodisilane [1505]. Platinum catalyzed both intra- and intermolecular dehydrogenative couplings of trialkylsilanes with aromatic rings and aliphatic methyl groups (Eq. (364)) [1506]. Titanium catalyzed the formation of allylic silanes from terminal alkenes (Eq. (365)) [1507]. Copper catalyzed the formation of unsymmetrical diaryl selenides and tellurides from diphenyl diselenide or ditelluride and aromatic boronic acids [1508].



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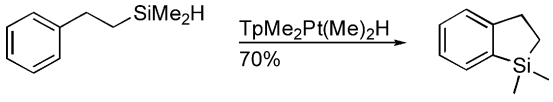

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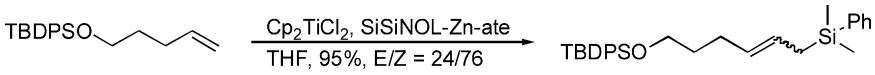

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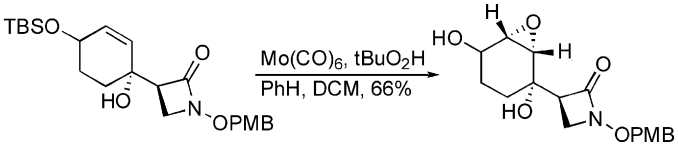
13. Oxidations

Vanadyl acetylacetonate-catalyzed epoxidations of allylic and in some cases homoallylic alcohols using peroxides continued to be an invaluable tool in organic total synthesis. Synthetic targets include, tetracyclic core of ingenol [1509], epoxyquinols A–C and epoxytwinol A [212], 1-deoxypaclitaxel precursor [1510], ovalicin [1144], polar core of scyphostatin [1511], abyssomicin C [1512] and ABCD ring of gambierol [1513]. A molybdenum catalyzed epoxidation was used in a synthesis of (+)-apiosporamide (Eq. (366)) [127].



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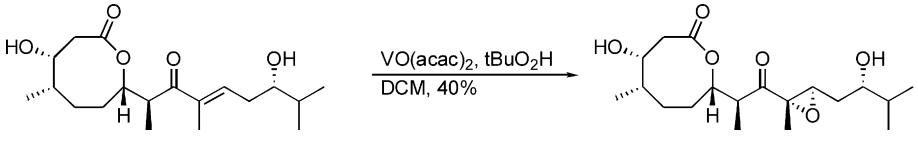
Sharpless enantioselective epoxidations of allylic (and in some cases homoallylic) alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continued to be extensively used in organic synthesis. For example, this reaction was used in synthetic approaches to octalactin A (Eq. (367)) [1488], the functionalized taxoid ABC-ring system [1514], (−)-malyngolide [756], the C22–C36 subunit of halichondrin B [654], the EF-ring segment of ciguatoxin CTX1B [599] and a laulimalide analog [921].



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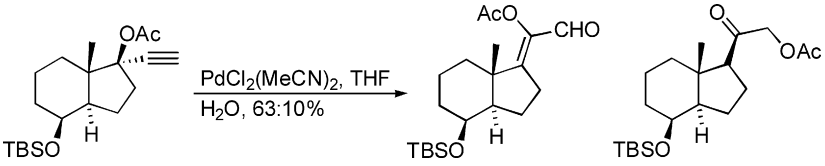
A number of synthetic applications of the palladium-catalyzed formation of α,β-unsaturated carbonyl compounds from silylenol ethers (Saegusa oxidation) were published. For example, applications toward kainoids [1515], gymnocin A [205], fumagalone analogs [503], guanacastepenes [1516], GKK1032s [138,1517], the ABCD ring system of manzamine [1176], xestobergsterol A [1518] and (+)-solanascone, (+)-dehydrosolanascone, and (+)-anhydro-β-rotunol [1519] were described. Palladium hydroxide on carbon using *t*-butylhydrogen peroxide as the reoxidant was used in Saegusa-type oxidations [1520].

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 [1521]. For example, spongistatin 1 [1522], (+)-anatoxin-a [1241], *N*-oxido-3-aza-1,3,5(10)-trieno steroids [1523], 11-thiasteroid derivatives [1524], CJ-13,015, CJ-13,102, CJ-13,103, CJ-13,104, CJ-13,108 [1525], epoxyquinols A and B [84], (−)-blastmycinolactol [1350], (+)-abresoline [1526], (−)-tarchonanthuslactone [1527] and (+)-solanascone, (+)-dehydrosolanascone, and (+)-anhydro-β-rotunol [1519] were prepared using a Wacker type reaction in one of the steps.

Support for a hydride shift mechanism for Wacker-type oxidations of styrenes using hydrogen peroxide was reported [1528]. A regioselective Wacker-type reaction of an internal alkene was used in a synthesis of annuionone [1529].

Palladium catalyzed regioselective allylic oxidation (acetoxylation) of terminal alkenes without alkene isomerization probably via a π-allyl complex [1530]. Similar reactions of terminal alkenes in DMSO-acetic acid and in the presence of benzoquinone gave allylic acetates with migration of the double bond [1531]. A palladium-catalyzed oxidative rearrangement of an propargyl acetate was used in a synthesis of OCT analogs (Eq. (368)) [1532].



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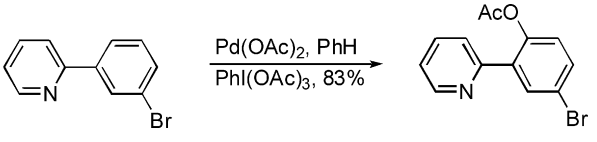
A chelation controlled regioselective palladium-catalyzed oxidation of 2-arylpyridines was described (Eq. (369)) [1533]. Ruthenium catalyzed oxidation of amines to give imines [1534]. Ruthenium catalyzed an interesting ketohydroxylation of alkenes (Eq. (370)) [1535]. Ruthenium catalyzed *cis*-dihydroxylations of alkenes [1536]. Copper catalyzed asymmetric allylic aminations and oxidations of alkenes [1537].



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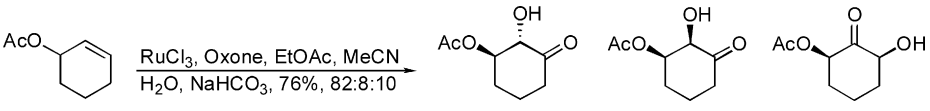

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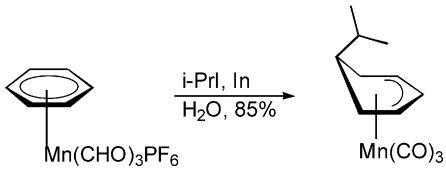
14. Reactions of isolated transitionmetal complexes and copper-, titanium-, and zirconium-intermediates

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. Enantioselective lithiation and alkylation of chromium tricarbonyl complexes of benzaldehyde derived chiral aminals was reported [1538]. Diastereoselective enolate and enamine addition to chiral arene chromium tricarbonyl propargyl cations was described [1539]. A nucleophilic substitution of a fluoroarene tricarbonylchromium complex using a chiral alcohol as the nucleophile was used in a synthesis of (+)-reboxetine [1540].

Tungsten pyridine complexes underwent Diels–Alder reactions [1541]. Indium mediated alkyl radical additions to (η^6-arene)-manganese complexes in aqueous media (Eq. (371)) [1542]. Nucleophilic substitution of a 1,4-dichlorobenzene η^6-iron complex with phenols was used to prepare thyroid hormone analogs [1543]. A related reaction of a chlorobenzene η^6-ruthenium complex was used in a synthesis toward ristocetin A aglycon [1544].



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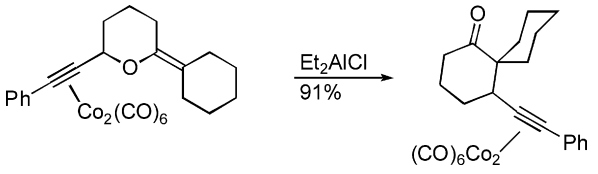
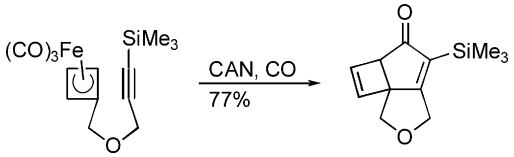
Nicholas-type reactions were used in synthetic applications toward macrocyclic diolides [1545] and cobalt complexed benzocycloheptynes [1546]. Diastereoselective allylations of cobalt coordinated ethynals using allylic stannanes were reported [1547]. Formation of a cobalt–alkyne complex made intramolecular [4 + 2]cycloadditions possible [1548]. Oxygen to carbon rearrangement of alkyne–cobalt complexes were reported (Eq. (372)) [1549]. A cobalt-alkyne complex was utilized as an alkyne protecting group and later oxidatively decomplexed in a synthesis of *trans*-kumausyne [939]. A regioselective hydrosilylative-decomplexation of cobalt–alkyne complexes was reported [1550]. The role of the base used for isomerization of alkyne-manganese to allene-manganese complexes was examined [1551].



(372)

Iron tricarbonyl complexes continued to be used in organic synthesis. An intramolecular [2 + 2 + 1]cycloaddition of alkyne-tethered cyclobutadiene iron tricarbonyl complexes in the presence of carbon monoxide was described (Eq. (373)) [1552]. Reaction of (1-methoxycarbonylpentadienyl)iron tricarbonyl complex with methyl lithium followed by oxidative decomplexation was used in a synthesis of the C9–C16 segment of ambuticin (Eq. (374)) [1230]. Related reactions using a variety of alkenyl Grignard reagents as the nucleophile furnished dialkenylcyclopropanes [1553].



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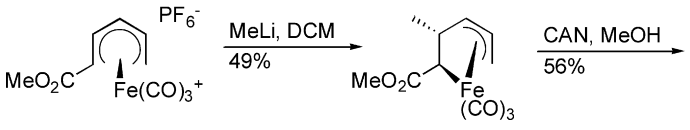

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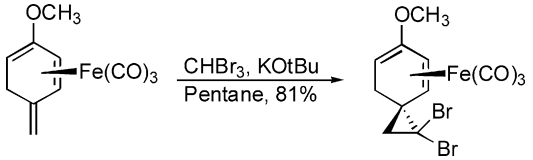
Reactions of polymer-bound cationic cyclohexadienyliron tricarbonyl complexes with nucleophiles were described [1554]. Nucleophilic addition of arylamines to cationic 2-methoxy-cyclohexadienyliron tricarbonyl complex followed by oxidative cyclization was used in synthesis of 7-methoxy-*O*-methylmukonal, clausine H, K, and O [1555]. Cyclopropanation of tricarbonyl(5-alkylidene-1,3-cyclohexadiene)iron complexes with carbenes was reported (Eq. (375)) [1556]. Dynamic kinetic resolution was observed upon [6 + 2]ene-type reaction of cyclohexadiene-iron complexes [1557]. Spirocyclic cyclohexadienyl ruthenium complexes were demetallated using halide ions or Lewis acids to give spirocyclic heterocycles (Eq. (376)) [1558].



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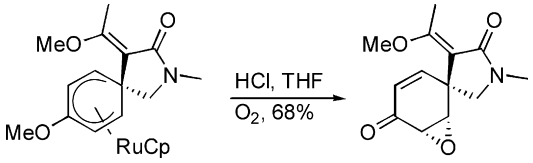
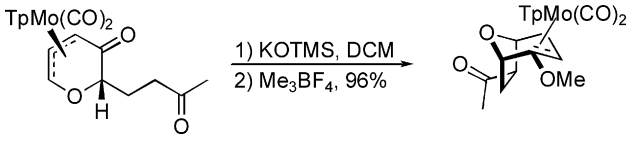


(376)

Cyclic molybdenum η^3-allyl complexes underwent intramolecular 1,5-Michael additions to give bicyclic products (Eq. (377)) [1559]. Allylation of α-metallated carbamates with η^3-allylmolybdenum complexes was described [1560]. A molybdenum η^3-allyl complex was used in a synthesis of the C14–C18 fragment of tetronasin [1561]. The *cis-trans* isomerization of cyclic η^3-allylpalladium complexes derived from allylic carbonates was slowed down upon addition of chloride anion [1562].



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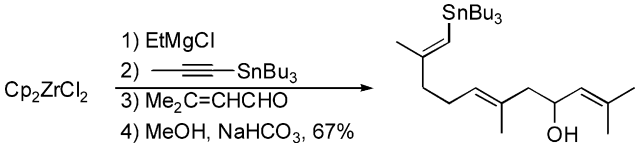
Zirconacyclopentenes served as templates in the synthesis of a number of natural products via reactions with 3-chloro-2-methylpropene and an aldehyde (Eq. (378)) [37]. Formation of a zirconacyclopentane followed by hydrolysis was used in a synthesis of (+)-*trans*-195A (Eq. (379)) [1255]. Reaction of zirconcyclopentenes with methyl 2-bromoethanoate to give cyclopropanated products was described (Eq. (380)) [1563]. Zirconacylopentene underwent a number of demetallation reactions including reactions with iodine forming monoiodides, with an excess iodine to give diiodides, with allylbromide in the presence of copper to give allylated products, and with propargyl bromide in the presence of palladium to give allene-ynes or allenes depending on the amount of propargylic bromide (Eq. (381)) [1564]. Zirconacyclopentenes react with acyl cyanides to give either 1-ene-5-ones or cyclopentadienes depending on the reaction temperature (Eqs. (382) and (383)) [1565]. Titanacyclopentenes were coupled with nitriles to form 3-cyclopentenylamines (Eq. (384)) [1566].



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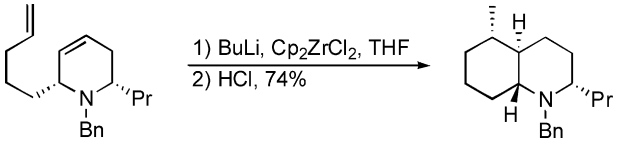

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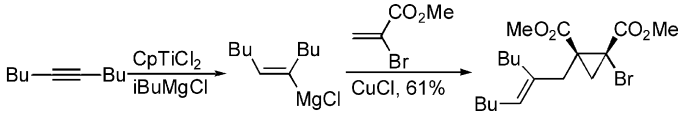

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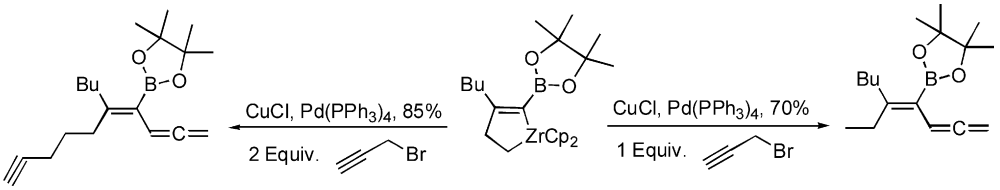

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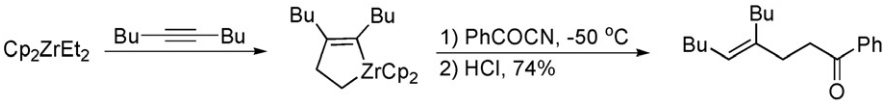

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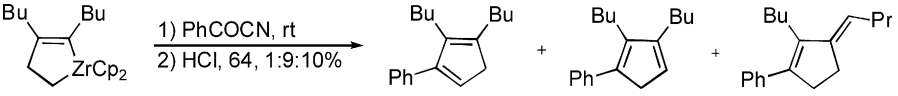

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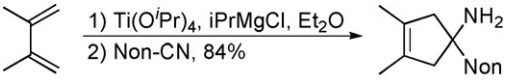
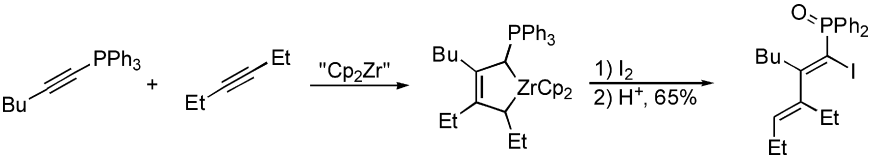


(384)

Zirconacyclopentadienes were coupled with oxalyl chloride in the presence of a copper catalyst to give cyclopentadienones [1567]. α-Phosphinozirconacyclopentadienes were cleaved with protons or iodine to give phosphino-dienes or phosphinoiododienes (Eq. (385)) [1568]. Five-membered zirconacycles derived from carbon dioxide and an alkene or an alkyne were allowed to react, in the presence of a copper or nickel promoter, with allylic and propargylic bromides, ethenyloxirane, iodobenzene, or 2-cyclohexenone to give functionalized carboxylic acids (Eq. (386)) [1569].



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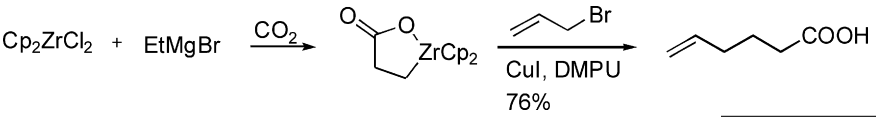
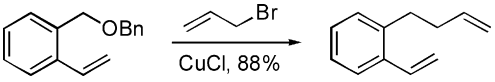


(386)

Benzylzirconocene intermediates were allylated using a copper catalyst (Eq. (387)) [1570]. Aryne-zirconocene complexes were transformed into functionalized arenes upon reaction with enol ethers and an electrophile (Eq. (388)) [1571].

Hydrozirconation of terminal alkynes followed by silver-catalyzed epoxide ring opening was reported [1572]. A hydrozirconation cyclization sequence was used to prepare pyrrolidines from *N*-allyl oxazolidines (Eq. (389)) [1573]. The formation of alkenylzirconium reagents from alkenyl carbamates was described [1574].



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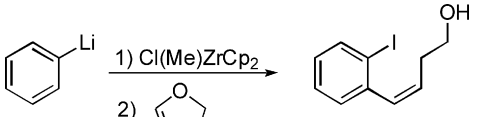

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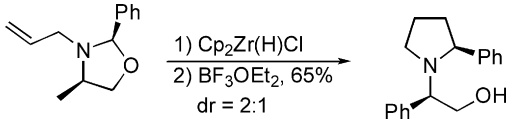
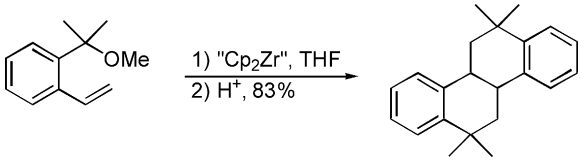


(389)

Zirconocene mediated a coupling of alkoxymethyl-substituted alkenylbenzenes to give polycyclic compounds (Eq. (390)) [1575]. Zirconocene mediated a coupling of an alkyne, a ketone, and an alkynylbromide to give 2-ene-4-yne-1-ols (Eq. (391)) [1576]. γ,γ-Dialkoxyallylic zirconium reagents reacts with carbonyl compounds to form cyclopropanated products (Eq. (392)) [1577]. A double carbonylation of zirconocene-alkyne complexes forming 4-hydroxy-2-cyclobuten-1-ones was described (Eq. (393)) [1578]. A chromium–zirconium mediated tetramerization of diarylalkynes was reported [1579].



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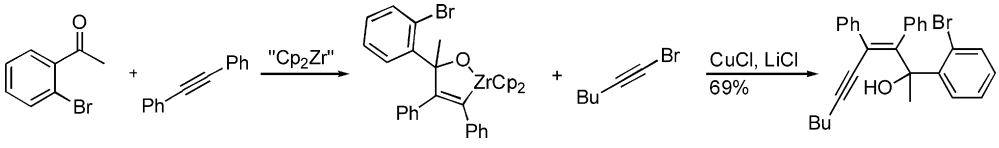

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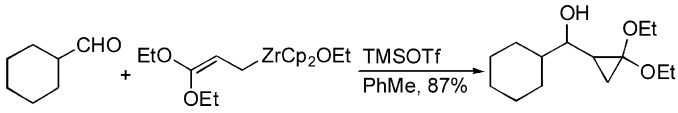

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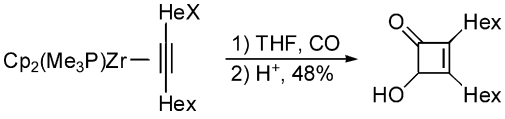
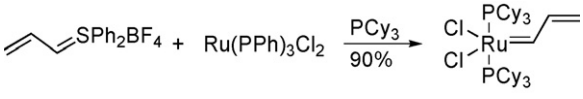


(393)

A new and selective synthesis of metal carbene complexes from sulfur ylides was developed (Eq. (394)) [1580]. Reactions of Fischer carbenes bound to a polymer formed by replacement of a carbon monoxide ligand with a polymer bound isonitrile were described [1581]. Fragmentation patterns of Fischer carbene complexes by electrospray ionization Fourier transform ion cyclotron mass spectrometry were reported [1582]. The mechanism of electrospray ionization was also examined [1583]. Cyclopropanation of ketene acetals with Fischer carbenes at elevated CO pressures was reported [1584]. Chiral cyclopropyl Fischer tungsten carbenes were prepared from alkenylcarbene and lithiated aryloxiranes (Eq. (395)) [1585]. Reaction of Fischer carbenes with alkoxyacetylides afforde new alkyne substituted carbenes (Eq. (396)) [1586].



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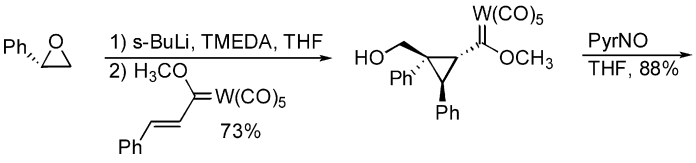
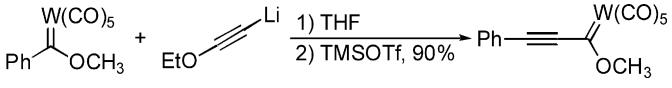
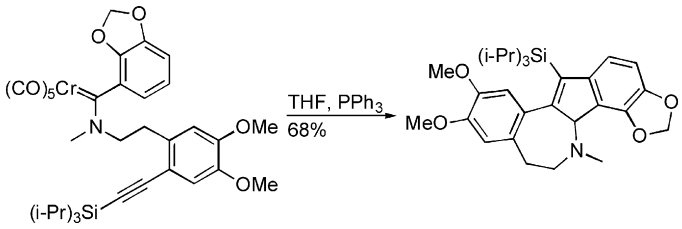




(396)

Dötz benzannulation of Fischer chromium carbene complexes [1587] were used in organic synthesis of, for example [6]- an [7]-helicene quinones [1588], (−)-curcuquinone [1589], isodityrosine [131], (−)-kendomycin [1590] and arylglycines [1591]. An interesting annulation of an amino carbene complex was used in a synthesis of bulgaramine (Eq. (397)) [334].

A tandem isobenzofuran formation–Diels–Alder reaction of 2-(6- or 7-ene-1-yne)-substituted benzaldehydes with saturated Fischer chromium carbenes (Eq. (398)) [1592]. A related reaction of 2-(1-alkynyl)benzaldehydes with γ,δ-unsaturated alkoxychromium carbenes was described [1593]. A benzannulation of *trans–trans*-dienyl Fischer carbene complexes in the presence of iron was reported (Eq. (399)) [1594]. Benzannulation of Fischer carbene complexes and hetereocyclic-bridged enynes was described (Eq. (400)) [1595].



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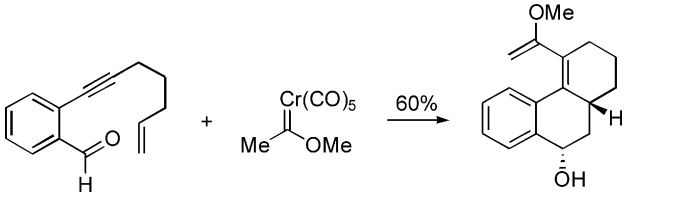

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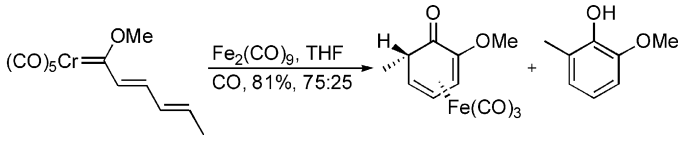

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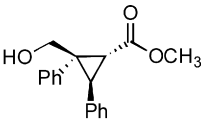

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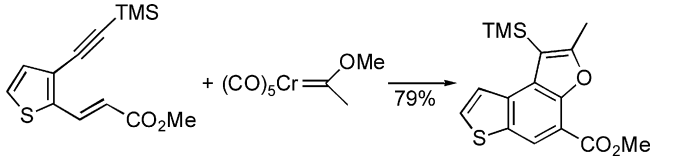
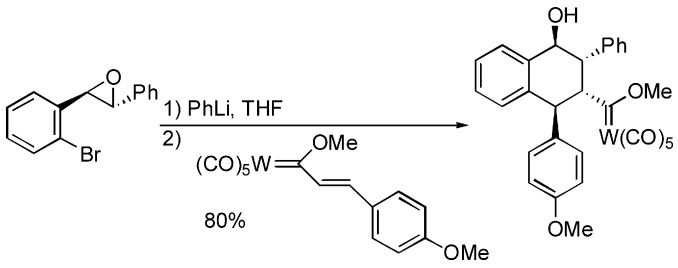


(400)

Addition of *ortho*-lithiated stilbene oxides to α,β-unsaturated Fischer carbene complexes produced tetrahydronaphthols (Eq. (401)) [1596]. Mukaiyama–Michael reactions of chiral α,β-unsaturated Fischer tungsten carbenes were described [1597]. Cyclopenta[*b*]pyrans were prepared by reaction of 2-alkoxy-, 2-amino-, or 2-thioxy-substituted ethenylcarbenes with alkynes (Eq. (402)) [1598]. Functionalized cyclopentadienes were obtained from reaction of β-amino-α,β-unsaturated alkoxy Fischer chromium carbene complexes [1599]. Related reactions of β-amino-α,β-unsaturated alkoxy Fischer chromium carbene complexes with 1,5-dien-3-ynes afforded benzene derivatives [1600]. A [4 + 1]cycloaddition of methylenecyclopropanes with Fischer chromium carbenes was described (Eq. (403)) [1601].



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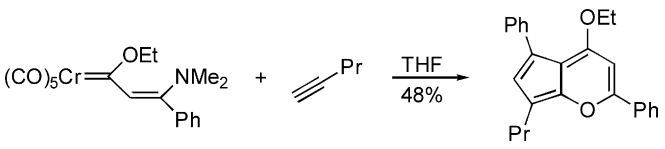

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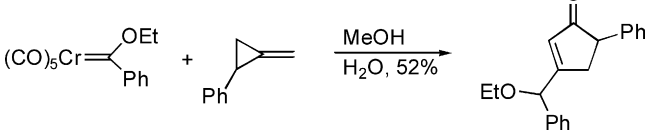
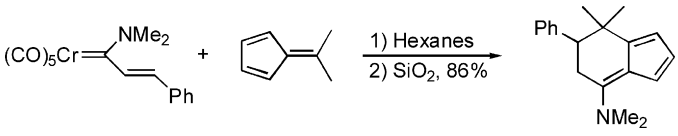


(403)

α,β-Unsaturated Fischer carbenes underwent cycloadditions with fulvenes (Eq. (404)) [1602]. Irradiation of imino Fischer chromium carbenes in the presence of an alkyne affords different products depending on the alkyne (Eq. (405)) [1603]. Rhodium catalyzed cyclizations of alkenyl Fischer carbenes with 1,1-diphenylallene (Eq. (406)) [1604]. Reaction of 2-alkynylphenyl or 3-buten-1-yl substituted alkoxy chromium carbenes with hydride or methyl anion triggered an insertion–annulation reaction [1605]. Reaction of tungsten Fischer carbene complexes with sulfinyl carbanions gave allyl sulfoxides (Eq. (407)) [1606].



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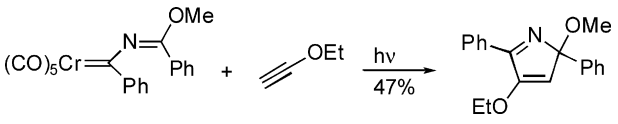

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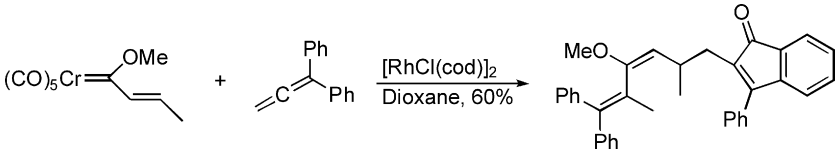

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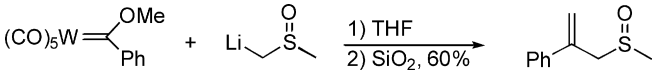
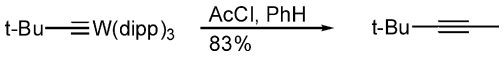


(407)

Chiral ruthenium allenylidene complexes were alkylated with ketone enolates and the diastereomers separated to yield enantiomerically pure products [1607]. Synthesis and reactions of allenylidene iron complexes with nucleophiles was reported [1608]. Chiral cationic iron carbene complexes participated in *cis*- and enantioselective cyclopropanations of unactivated alkenes [1609]. Tungsten acetylide complexes react with iminium ions to form alkenyl carbenes [1610]. Reaction of acid chlorides with tungsten alkylidenes furnished alkynes (Eq. (408)) [1611].



(408)

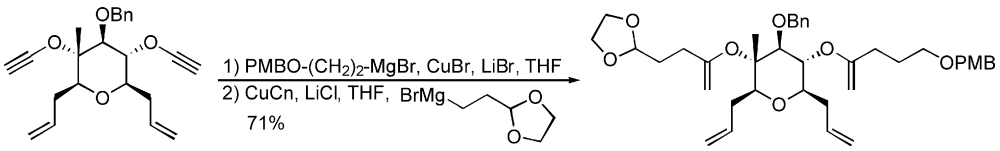
A stereoselective Michael addition of dialkyl cuprates to carbohydrate-derived 2-diethoxyphosphorylhex-1-en-3-uloses was described [1612]. Michael additions of dimethylcuprate were used in synthesis of hamigeran B [202], (+)-allocyathine B_2 [128], umbrosone [85]. Stereoselective Michael additions of organocopper reagents were used in synthetic applications toward cylindramine [324], pumiliotoxin C [1613], fumagalone analogs [503], tridachiahydropyrone [1614], guanacastepenes [1516] and 5-epi-10-epi-vibsanin E [704].

A zirconium-catalyzed methylalumination of a terminal alkyne followed by transmetallation to copper and Michael addition was used in a synthesis of (−)-kendomycin [1615]. A syn-selective sila-cupration of an alkyne was used in a synthesis of lucilactaene [47]. Diarylcopper salts were oxidatively coupled to form biaryls [1616].

Regio- and stereoselective oxirane ring opening using R_2CuLi_2CN or R_2CuLi were used in syntheses of (+)-crocacin D [51], (2*S*,3*S*,4*R*)-1-*O*-(α-galactosyl)-2-tetracosanolyamino-1,3,4-nonanetriol [1617], 1-azasugars [1618] and the C20–C26 fragment of superstolide A [905].

Nucleophilic substitution of an alkyl, alkenyl, and alkyl iodides or triflates with organocopper reagents were used in syntheses of sordaricin [1619], (−)-salicylhalamides A and B [938], (−)-kainic acid [1620], (+)-bistramide C [801] and (+)-discodermolide analogues [1621]. Two consecutive carbocuprations of a diyne was used in a synthesis of F–J ring-fragment of gambieric acid (Eq. (409)) [1121]. An allylic substitution using a cuprate was used in a synthesis of prostacyclin analogs [1622].

Cyclopalladation using an oxime as the directing group was used in a synthesis of rostratone (Eq. (410)) [1623].



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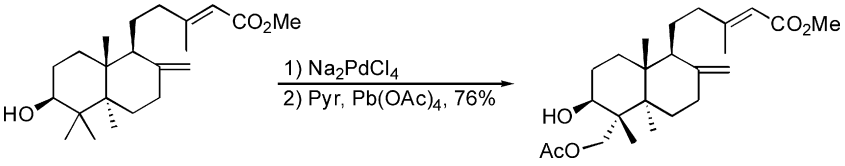
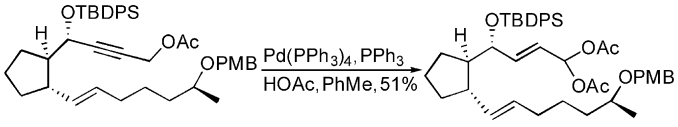


(410)

15. Miscellaneous reactions

Palladium catalyzed an organozinc promoted formation of 1,3-dienes from allylic esters and ethers having a second ester or ether leaving group [1624]. A palladium-catalyzed propargylic acetate to 1,1-diacetoxy-2-ene transformation was used in a synthesis of (+)-brefeldin C (Eq. (411)) [253].

Iron catalyzed an asymmetric coupling of ketenes with aldehydes to form enol esters (Eq. (412)) [1625]. Copper mediated an enantioselective oxidative dearomatization and this reaction was used in the synthesis of azaphilones (Eq. (413)) [1626]. Iridium catalyzed ring-cleavage of cyclobutanone *O*-benzyloximes to give nitriles (Eq. (414)) [1627].



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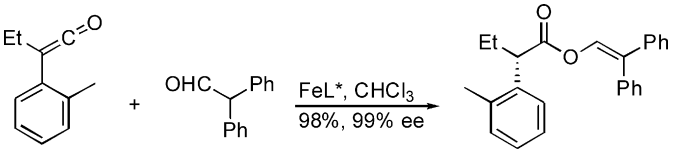

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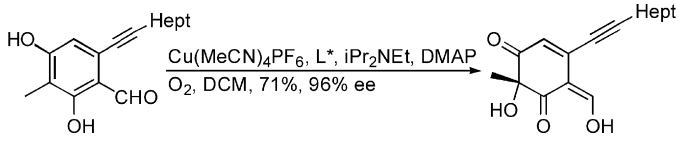

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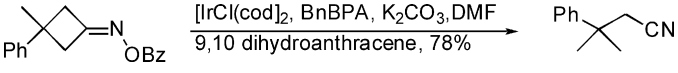

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