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Review

Transition metals in organic synthesis: Highlights for the year 2003

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Abstract

A review with 1807 references to transition metal-catalyzed or mediated reactions and functional group preparations.

Keywords: Transition metals; Organic synthesis; Catalysis

1. General comments

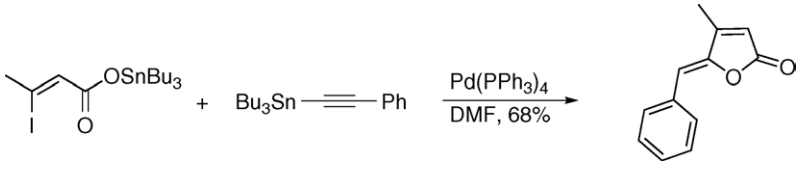
This survey highlights the use of transition metals in organic synthesis for the year 2003. Comprehensive literature coverage with extensive citations is presented. The field of transition metal chemistry continued to expand in 2003 and have a significant role in functional group transformations and 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. Some emphasis was put on reactions used in total synthesis of biologically active compounds. Alkylations and most Michael type reactions of pre- or intermediately formed copper reagents have not been reviewed. The chemistry of metal carbon multiple bonds, e.g., reactions of Fischer and Schrock carbenes, are covered in a separate review although decompositions of diazo compounds and metathesis reactions were included herein. Papers describing new chiral ligands for palladium-catalyzed allylic alkylations and allylic aminations of, primarily, 1,3-diphenylallylacetate and ligands for metal-catalyzed asymmetric cyclopropanations were not included. Most hydroformylations were not included apart from some reactions used in total synthesis.

2. Reactions of alkyl, alkenyl, alkynyl, benzyl, and aryl halides, tosylates, triflates, and nonaflates

2.1. Carbon–carbon bond-forming reactions via transmetallation

Palladium-catalyzed cross-coupling reactions of organotin reagents (Stille couplings) with a large variety of electrophiles continued to be developed and extensively used in organic synthesis. The accelerating effect of added copper salts was studied using spectroscopic and kinetic methods [1]. A variety of palladium catalyst systems were described [2–4] including systems used in water [5], and reagents and reactants on solid support [6,7]. Alkyl bromides having β-hydrogens were coupled with alkenyltin reagents [8].

Thiol esters [9], 3,4-dithioaryl-3-cyclobuten-1,2-diones [10], and alkyl bromides and iodides [11], electron-deficient aromatic fluorides [12], sulfonyl chlorides [13] were used as electrophiles in Stille-type couplings. Some more unusual tin reagents were used including 1-stannyl-1,2-dienes [14] and 4-stannyl-3-alkenoic acid tributyltin esters [15]. An interesting tandem coupling intramolecular Diels–Alder sequence to give complex polycyclic compounds was described [16]. Stille coupling of tin-substituted alkynes with (*Z*)-3-iodo-2-enoic acid tributyltin esters furnish either 5- or 6-membered lactones depending on the alkyne (Eqs. (1) and (2)) [17].



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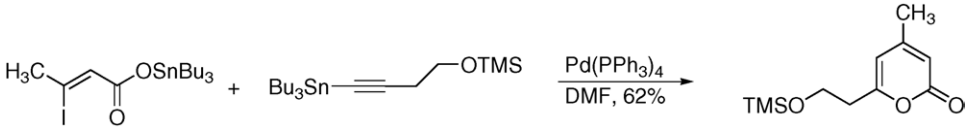

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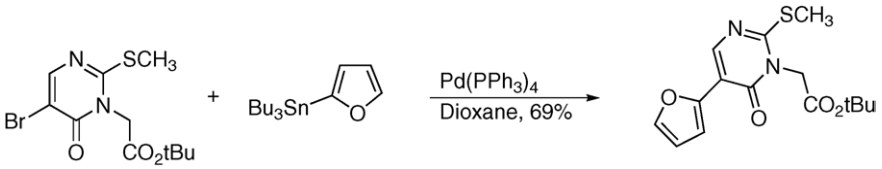
A large variety of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and alkenyl-tin reagents. Example of ring systems include, cyclic carbamate derived alkenyl phosphonates [18], heteroaromatic thioethers [19,20], 5-bromo-dihydropyran-4-ones [21], 3-iodoimidazopyridine in a synthesis of imidazo[1,2-*a*]pyridines [22], iodoimidazoles [23–25], 5- and 6-halo-3(2*H*)-pyrazinones [26,27], 3,5-dichloro-1,4-oxazin-2-ones [28], 3-chloro-4-halo-1,2,5-thiadiazoles [29], 2-chlorobenzoxazole [30], 2-iodopyrrolo[2,3-*b*]quinolines [31], 6-haloimidazo[1,2-*a*]pyridine [32], 8-iodoadenosine [33], and 4-bromo-2*H*,10*H*-azepino[3,4-*c*]indole-1,5-dione [34]. Selective coupling in the 4-position of 3,4-dichloro-2(5*H*)-furanone was observed [35]. Depending on the solvent and temperature, either 3- or 5-substitution of 3,5-dibromo-2-pyrone can be achieved [36]. Depending on the catalyst system, either sulfide or halide substitution of 5-

bromo-2-methylthiouracil was obtained (Eqs. (3) and (4) [37].



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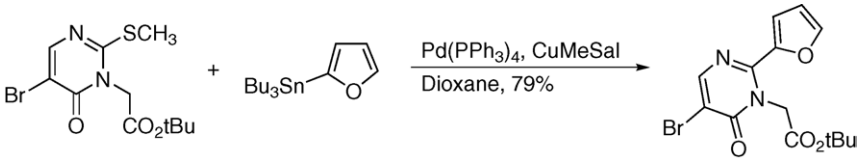

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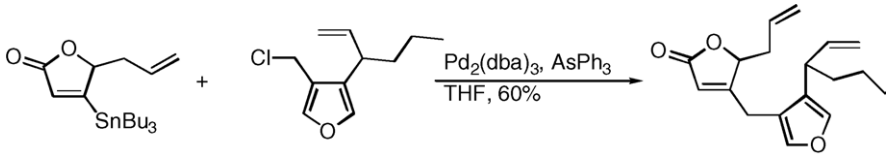
A variety of heterocyclic stannanes such as 4-stannyl-3-thiomethylpyrimidine used in a synthesis of imidazo[1,2-*a*]pyridines [22], 2-stannylpyrrole [38], 2-stannylpyridines [39], 2-stannylpyrazines [40], 4-stannyl-4-pyrone [41], 2-stannylindole [42], 5-stannylpyrimidines [25], and a sugar-derived alkenyl stannane [43], or derivatives thereof, were used in Stille-type couplings with a variety of aromatic and heteroaromatic halides and triflates.

The intermolecular Stille reaction continued to be extensively used in organic total synthesis. Synthetic targets include, steroids [44], salicylihalamides A and B [45], ailanthoidol [46], barbacenic acid [47], C21–C26 fragment of superstolide A [48], epi-indolizidine 223A [49], louisianin C [50], reserpine [51], (−)-allocolchicine [52], dumsin [53], variolin B and deoxyvariolin B [54], carbocyclic core of cornexistins (Eq. (5)) [55], mappacine ketone [56], panepophenanthrin (Eq. (6)) [57], himbacine [58], FR182877 [59], haterumalide NA methyl ester [60], idiospermuline [61], hodgkinsines [62], epothilone analogs [63,64], azaspiracid-1 (Eq. (7)) [65,66], lactonomycin [67], carbazomadurin A [68], SB-242784 [69], variolin B [70], nafuredin-γ [71], macrocyclic lactones [72], TMC-120B [73], trisubstituted pyridine skeleton of thiostrepon-type antibiotics [74], (−)-acromelobic acid [75], 6,7-dihydroeponemycin [76], (−)-apicularen A [77], medermycin analogs [78], (+)-rottnestol, (+)-raspailol A and B [79], oxazolomycin antibiotics [80], dynemicin analogs [81], 3-arylisocoumarins [82], mycothiazole [83], (−)-idiospermuline [84], epolactaene [85], gambierol [86], (+)-deoxyneodolabelline [87], elisabethin A [88], (+)-torreyanic acid [89], FR182877 [90], oximidine II [91], lobatamide C [92], fostriecin [93], gambierol and 16-epi-gambierol [94], (+)-macquarimicin A [95], and apoptolidin [96,97]. Selective coupling of the *trans*-bromide of a 1,1-dibromo-1-alkene was used in a synthesis of (+)-himbacine [58] and disorazole A_1 (Eq. (8)) [98].



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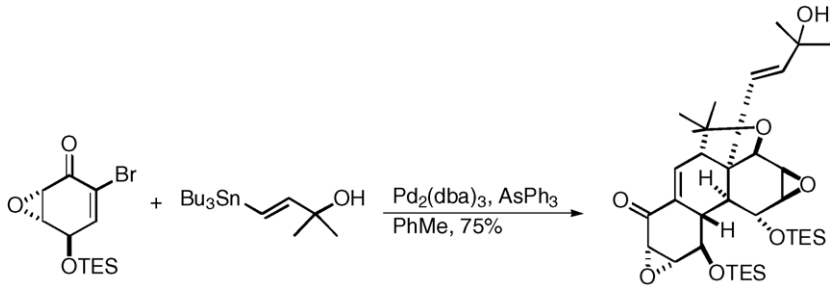

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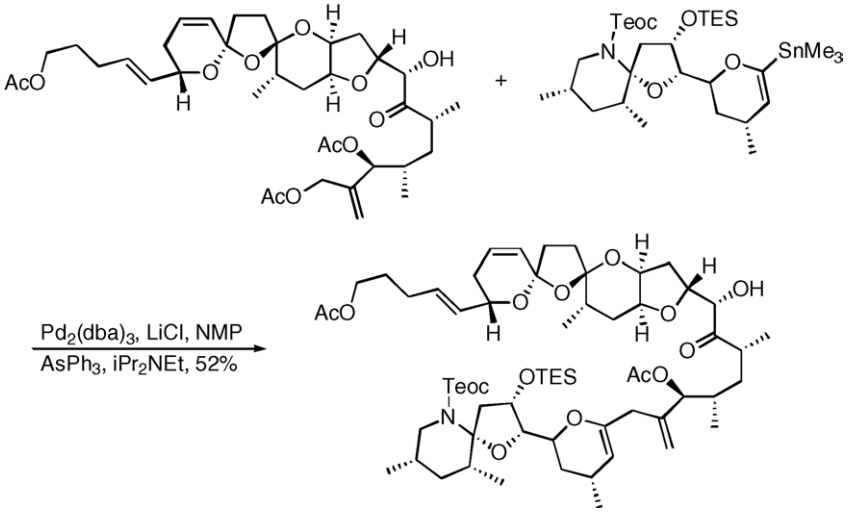

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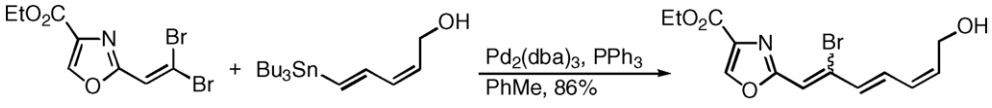

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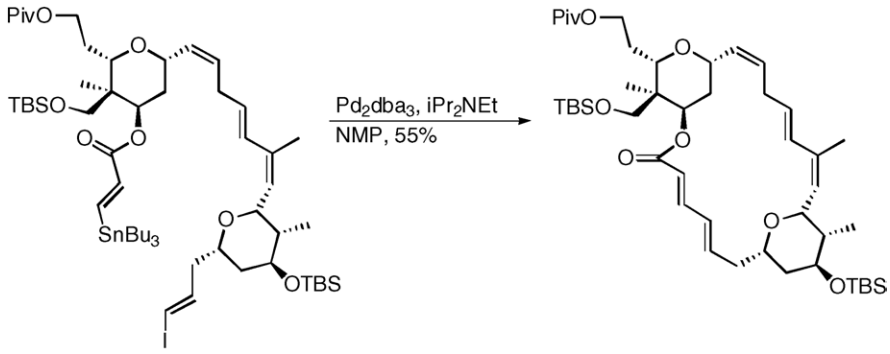
Stereoselective synthesis of *E*- and *Z*-1,1-diaryl or triarylalkenes was reported [99]. Intramolecular Stille-type macrocyclization were used in synthesis of lasonolide (Eq. (9)) [100], isocembrene [101], (+)-aphidicolin [102], and chloropeptin I (Eq. (10)) [103]. Intermolecular formation of an aromatic stannane using hexamethylditin followed by intramolecular coupling with an aryl halide (Stille–Kelly reaction) was used to prepare polyheterocyclic systems [104]. Reaction of 3-iodo-2-enecarboxylic acid amides with allenylstannanes furnished 4,6-disubstituted-2-pyridones (Eq. (11)) [105]. A copper promoted Stille-type coupling was used in a synthesis of formamicinone (Eq. (12)) [106].



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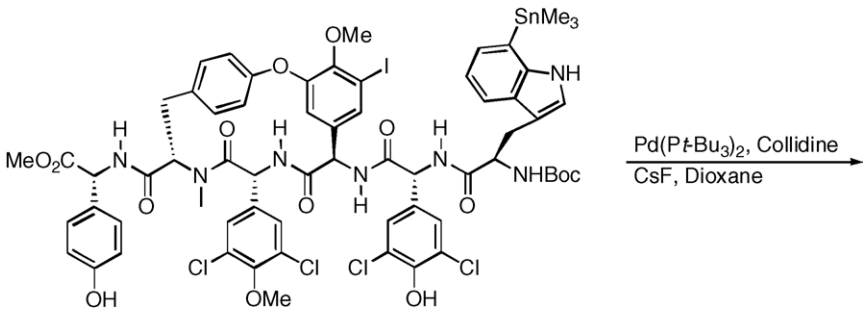

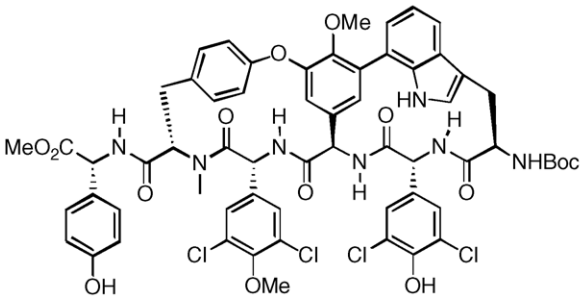

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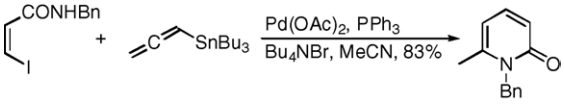

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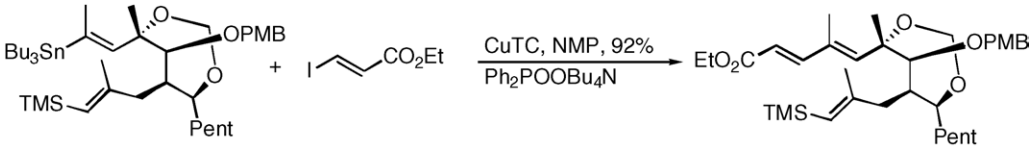
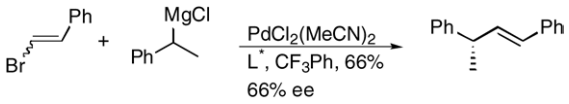


(12)

Nickel catalyzed the coupling of arylmagnesates with heteroaromatic bromides [107]. Palladium catalyzed the coupling of lithium tri(quinolyl)magnesates [108]. Stereoselective synthesis of *E*- and *Z*-1,1-diaryl or triarylalkenes was reported [99]. Nickel catalyzed the coupling of a *Z*-alkenyl(diisopropyl)carbamate with Grignard reagents [109]. Nickel catalyzed the coupling of 1,1-dibromo-alkenes with silylmethylmagnesium halides to give *Z*-β-bromoallylsilanes [110]. Nickel catalyzed the coupling of alkyl arenesulfonates with aryl Grignard reagents [111]. Couplings using nickel on carbon is most likely of a homogenous nature [112].

Palladium catalyzed the coupling of 1-chloro-1-en-3-ynes [113] and alkyl chlorides [114] with Grignard reagents. Palladium and nickel catalyzed the coupling of alkyl tosylates and bromides with Grignard reagents in the presence of 1,3-dienes [115]. Palladium catalyzed an asymmetric cross-coupling of Grignard reagents with alkenylbromides (Eq. (13)) [116]. Three different aromatic groups can be introduced using 1-butynyldimethyl(2-pyridyl)silane (Eq. (14)) [117].



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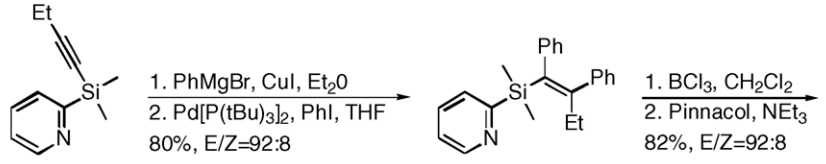
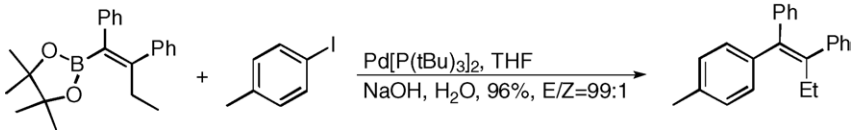




(14)

Nickel catalyzed the cross-coupling of aromatic nitriles with alkyl and alkenyl Grignard reagents [118].

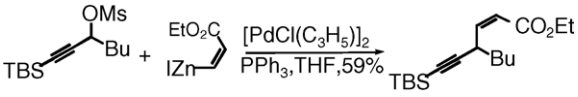
Nickel and copper catalyzed the coupling of alkyl fluorides with Grignard reagents [119]. Cobalt catalyzed the coupling of *N*-heteroaromatic chlorides with aromatic Grignard reagents [120]. Iron catalyzed the methylation of 2,6-dichloropurine with methyl magnesium chloride [121].

Nickel and palladium complex-catalyzed cross-couplings of Grignard reagents with halides, phosphates and triflates were used in organic synthesis [122] of guanacasterpene A [123], (*Z*)-tamoxifen [124], and leuscandrolide A [125], and cryptotanshinone [126]. A nickel-catalyzed ring-opening coupling of methyl magnesium bromide with a 2,3-dihydrofuran was used in a synthesis of dihydrorhipochephalin [127].

Palladium- or nickel-catalyzed couplings of halides and triflates with organozinc reagents (Negishi couplings) continued to grow [99,128–130] and a variety of new ligands [131,132] have been reported. Polymer-bound organohalides were used in coupling reactions with organozinc reagents [133]. Couplings using nickel on carbon as the catalyst is most likely of a homogenous nature [112,134]. Arylzinc halides were readily prepared from aryl halides, zinc powder, and a catalytic amount of trimethylsilyl chloride and the reagents were coupled with allyl halides using a palladium catalyst [135]. An efficient method for the preparation of alkyl zinc reagents from unactivated alkyl bromides and chlorides followed by coupling with aromatic halides was reported [136]. Palladium- and nickel-catalyzed couplings of alkyl halides and tosylates with alkenyl-, aryl-, and alkyl-zinc reagents [137,138]. Palladium catalyzed the coupling of trimethylsilylethynylzinc chloride and B-halocobaltacarborane [139].

The yields of Negishi couplings of alkynylzincs with alkenyl halides compared favorably with the corresponding Sonogashira coupling [140]. The rate of decomposition of β-aminoalkylzinc reagents was reduced using TFA in place of a BOC protecting group for the amine [141]. A few example of cobalt-catalyzed Negishi-type coupling were also described [142]. Cobalt catalyzed the coupling of acid chlorides and arylzinc reagents to give unsymmetrical ketones [143].

trans-1-Iodo-2-bromoethene was selectively coupled on the iodine bearing carbon [144]. 1,1-Dihaloalkenes were stereoselectively coupled with two different zinc reagents [145]. 1-Chloro-1-en-3-ynes [113] and anhydrides [146] were coupled with organozinc reagents. The regioselectivity of the cross-coupling between organozinc reagents and propargylic mesylates was controlled using ligands (Eqs. (15) and (16)) [147]. Inversion of stereochemistry was observed upon reaction of 2-brom-1,3-dienes with zinc reagents (Eq. (17)) [148].



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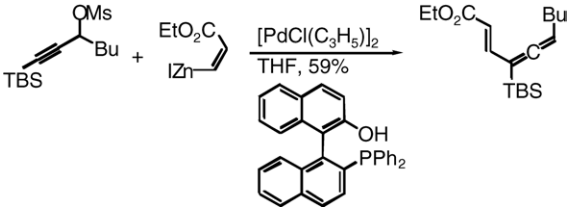

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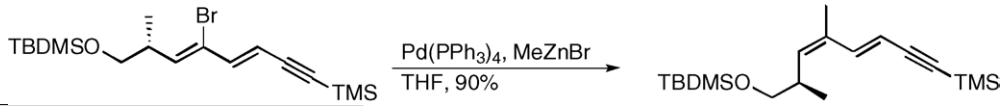

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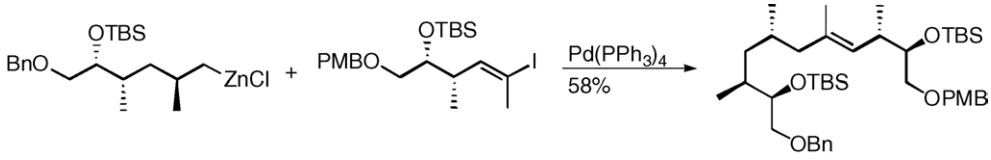
Chiral ferrocenyl [149,150], oxiranyl [151], sugar-derived alkenyl [43], 4-imidazolyl [152], 2-pyridyl [153,154], and 2-furyl, in a synthesis of a HIV protease inhibitor [155], zinc reagents were coupled in the presence of a palladium catalyst. Palladium catalyzed the cross-coupling of a cycloalkyl zinc reagent with iodobenzene [156]. Heterocyclic halides and triflates also participated in Negishi couplings. For example, chlorotetrazines [157] 2-trifloxyquinolines [158], 4-bromo-2-pyranones [159], and 2-iodopyrazines [40] were used. Interestingly, attempted Negishi coupling of zinc reagents derived from 6-haloimidazo[1,2-*a*]pyridine with phenyl iodide gave 5-phenylimidazo[1,2-*a*]pyridine [32]. Either the 2- or 4-position of 2,4-dichloropyrimidine can be substituted with some selectivity using conventional or microwave heating [160]. 2,6-Dichloro-9-(1-methylethyl)purine was selectively coupled in the 6-position [161].

Synthetic targets using the Negishi coupling include kallolide A and pinnatin A [162], (+)-discodermolide [163], (−)-delactonomycin [164], NVP-ACQ090 [165], (+)-rottnestol, (+)-raspailol A and B [79], phomactin A [166], 3-arylisocoumarins [82], borrelidin (Eq. (18)) [167], hemibrevetoxin B [168], elisabethane diterpenes [169], and amphidinolides (Eq. (19)) [170]. Three consecutive cross-couplings were used in the synthesis of eupomatenoids (Eq. (20)) [171]. Thio esters [172] were used in Negishi-type reactions and were utilized in a synthesis of phomoidride B [173].



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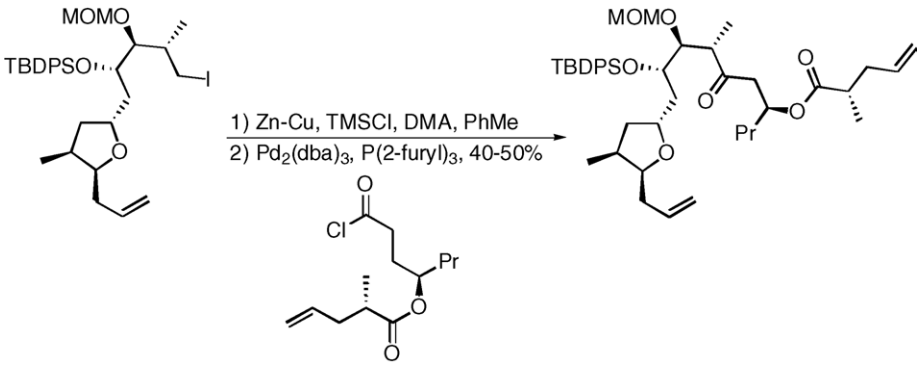

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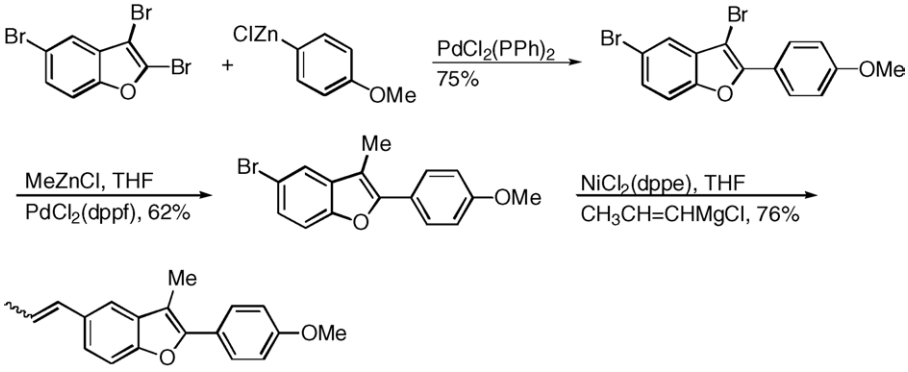

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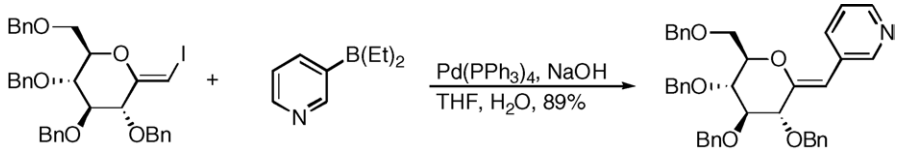
The Suzuki cross-coupling, i.e., palladium-catalyzed coupling of organoboronic acids and esters with organic halides and triflates, continue to see substantial use in organic chemistry. A number of ligands [174–183], catalyst systems [4,172,184–201], and reactions in water, aqueous solutions, or ionic liquids [202–208] were described. Couplings using nickel on carbon as the catalyst is most likely of a homogenous nature [112]. Nickel catalyzed the cross-coupling of aromatic boronic acids with bromoporphyrins [209]. Palladium-catalyzed reactions using reagents on solid support [6,210–217] and Suzuki reactions facilitated by grinding [218] or microwave irradiation [219,220] were described. Palladium-catalyzed Suzuki reactions of fluoroarenes [221] and aryltrimethylammonium salts [222]. Dialkylaminoorganoboranes were used in Suzuki couplings [223].

Acid chlorides were coupled with aromatic boronic acids using a palladium catalyst to give unsymmetrical ketones [224–226]. 2-Bromo-*N*,*N*-dimethylethane amide was coupled with aryl and alkenyl boronic esters [227,228], aryl boronic acids were coupled with α-bromoamides [229] and 1-exo-iodomethylene pyranoses (Eq. (21)) [230], α-bromo-sulfoxides [231], electron-deficient aromatic fluorides [12] and aromatic tosylates [232], and allylic acetates were coupled with aryl- and alkenyl boronic acid esters [233]. Bromobenzylbromides were sequentially coupled in the benzylic and aromatic positions [234].

A chromium tricarbonyl complexed aromatic bromide [235] and a (4-bromo-η^4-2-pyrone)tricarbonyl iron complex [236] were coupled with boronic acids. An interesting tandem coupling intramolecular Diels–Alder sequence to give complex polycyclic compounds was described (Eq. (22)) [111,116]. Functionalized styrenes were obtained via a palladium-catalyzed coupling of aromatic boronic acids with 1,2-dibromoethane [237].



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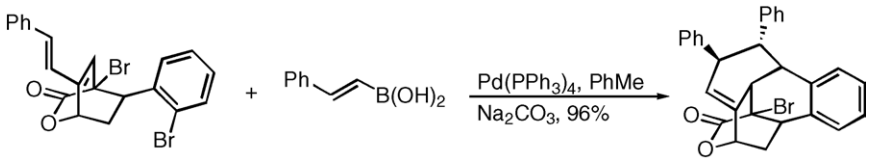
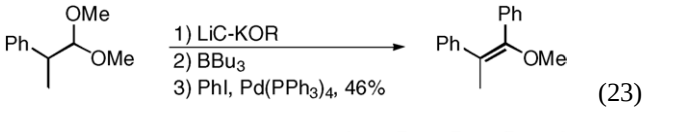


(22)

Suzuki couplings of potassium alkyl-, aryl-, and heteroaryltrifluoroborates [238,239], 2,2-difluoroalkenylboranes [240], potassium diaryldifluoroborates [241], and boronates derived from acetals (Eq. (23)) [242] were described. Coupling of an alkyl boronate was used in a synthesis of leptofuranin D (Eq. (24)) [243]. Suzuki–Heck-type cascade reactions were described (Eq. (25)) [244].



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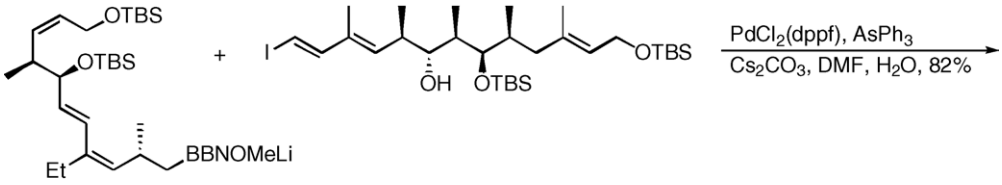

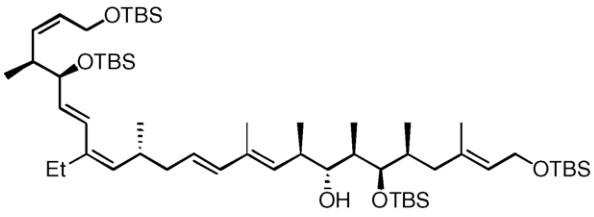

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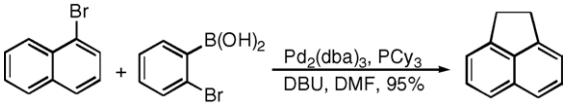

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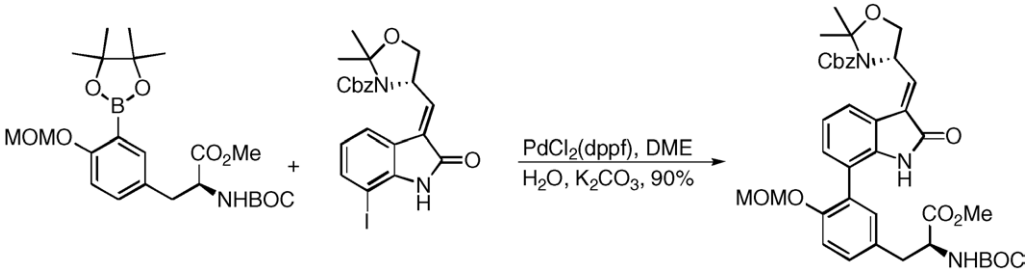
A large variety of functionalized heterocyclic halides and triflates were employed in the palladium-catalyzed Suzuki reaction. For example, 4-tosyloxy-2(5*H*)-furanone [245], 2-, 3-, and 7-haloindoles [246–248], chloropurines [121], a 4-chloropyrimidine [249], 5-iodo-2′-deoxyuridine, 5-iodo-2′-deoxycytidine, 8-bromo-2′-deoxyadenosine, 8-bromo-2′-deoxyguanosine [250], 4-iodoimidazole [152], cyclic carbamate derived alkenyl phosphonates [18], 3-iodopyrrole [251], lactam and lactone derived alkenyl phophonates [252], 5-bromodihydropyran-4-ones [21], 4-bromo- and 4-trifloxy-2-pyranones [159], 2- and 5-halo-oxazoles and -thiazoles [253], 2-bromobenzothiazole [254], 5-bromopyrazolopyrimidine [255], 6-bromothiazolo[4,5-*b*]pyridine [256], 5-bromopyrid-2-ylhydrazine [257], 4-bromo-2*H*,10*H*-azepino[3,4-*c*]indole-1,5-dione [34], 3-bromofuran [258], 3-bromofuran[3,2-*b*]pyrroles [259], 4,5-dihalo-2-hydroxymethy-3(2*H*)-pyrazinones [26], 4-chloro- and 5-trifloxypyridazin-3(2*H*)-ones [260], bromopyrazines [261,262], 4-halo-1,8-naphthyridin-2(1*H*)-ones [263], a kojic acid derived triflate [41], halo pyrazines [40,264], 4-iodo-1*H*-imidazo[1,2-α]imidazole-2-one [265], 3-chloro-4-halo-1,2,5-thiadiazoles [29], 2-iodopyrrolo[2,3-*b*]quinolines [31], and 5,6,7,8-tetrahydro-2-iodoimidazo[1,2-*a*]pyridine [266] were used in Suzuki type cross-couplings. Dehalogenation was observed as the major product using 4-bromopyrrole-2-carboxylates [267]. The undesired ipso-substitution was suppressed using silver(I) oxide in couplings of pentafluorophenylboronic acid and 2-iodopyridine [268].

2,6-Dichloronicotinamide was selectively coupled in the 2-position [269] and 2,6-dichloro-9-(1-methylethyl)purine in the 6-position [161] using organoboronic acids. Selective coupling in the 4-position of 3,4-dichloro-2(5*H*)-furanone [35] and in the 5-position of 3,5-dichloroisothiazole-4-carbonitrile [270] were observed. Depending on the catalyst system, either sulfide or halide substitution of 5-bromo-2-methylthiouracil was obtained [37].

A variety of heteroaryl boronic derivatives were used such as, 2- and 3-thienylboronic acids [271,272], 3-pyridyl- and 3-quinolinylboronic acids [273], 7-indolylboronic esters and 3-indolyl boronic acid [274], used in the synthesis of pyrrolophenanthridone alkaloids [247], 3-, 5-, and 7-benzothienylboronic acids [275], 2- and 3-furylboronic acids and esters [276,277], a boronate derived from 5-bromopyrid-2-ylhydrazine [257], and halo-2-pyridinylboronic acids and esters [278].

A number of syntheses wherein aryl or alkenyl boronic acids or esters were coupled with an aryl-, heteroaryl-, alkenyl-halides, -triflate, and phosphonates were reported. Synthetic targets included, UB-165 [279], a cathepsin K inhibitor [280], glucocorticoid receptor ligand A-224817.0 [281], angiotensin-(1–7)-receptor agonist AVE 0991 [282], deoxyfusapyrone [283], raloxifen analogs [284], 17α-substituted estradiol-pyridine-2-ylhydrazine conjugates [285], 1α,25-dihydroxyvitamin D_3 and analogs [286], nemertelline [287], TMC-95A/B proteasome inhibitors (Eq. (26)) [288], pyridomacrolidin [289], spirotoxin C [290], ancistrotanzanine B and ancistroealaine A [291], dipeptide β-turn mimetics [292], norbadione [293], diazonamide A (Eq. (27)) [294], TMC-95A [295], toward azadirachtin [296], RP-66453 [297], 2-arylsubstituted 1,2-unsaturated pyrrolobenzodiazepines [298], an aporphine alkaloid [299], dehydroaltenusin [300], 13-aryl substituted (11*Z*)-retinal [301], RP

66453 [302], imidazo[1,2-*a*]pyridines [303], gymnocin A [304], permethyl storniamide A, ningaline B, and lamelarine G trimethyl ether [305], buflavine [306], pyrido[2,3,4-*kl*]acridin-4-one [307], 3-substituted prolines [308], bupleurynol [309], korupensamines (Eq. (28)) [310], indatraline [311], factor Xa inhibitors [312], tissue factor VIIa inhibitors (Eq. (29)) [313], (9*Z*)- and (11*Z*)-8-methylretinals [314], rhazinal [315], crisamicin A [316], khafrefungin [317], furanoeudesmanes [318], halitulin [319], nakadomarin A [320], (−)-FR182877 (Eq. (30)) [321], gymnocin A [322], (−)-lemonomycin [323], and apoptolidin [96]. Substituted terphenyls were prepared via a double Suzuki cross-coupling [324]. Intramolecular couplings were used in synthesis toward a TMC-95A analog (Eq. (31)) [325] and (+)-phomactin A (Eq. (32)) [326]. Asymmetric Suzuki reactions to afford axially chiral biaryl were described [327].



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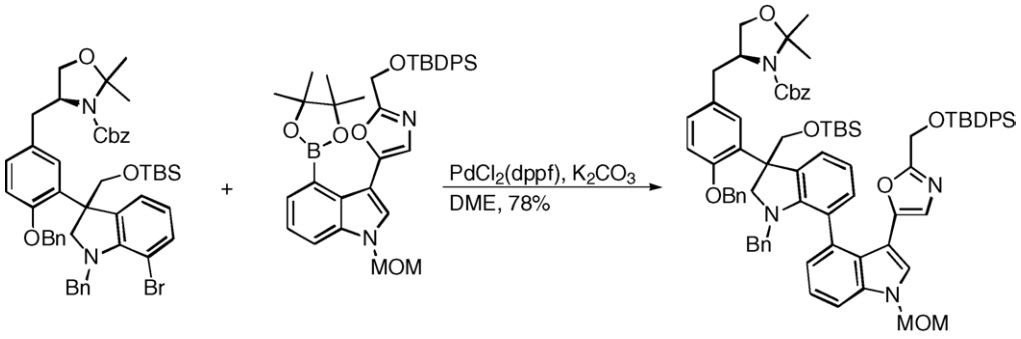

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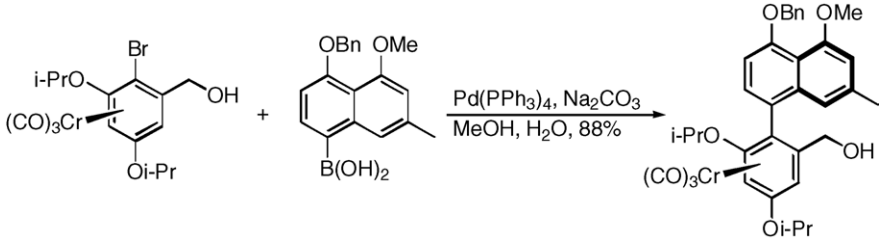

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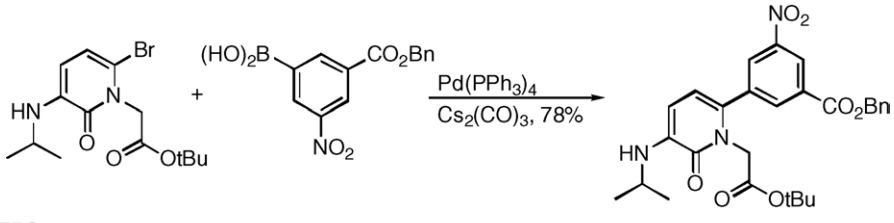

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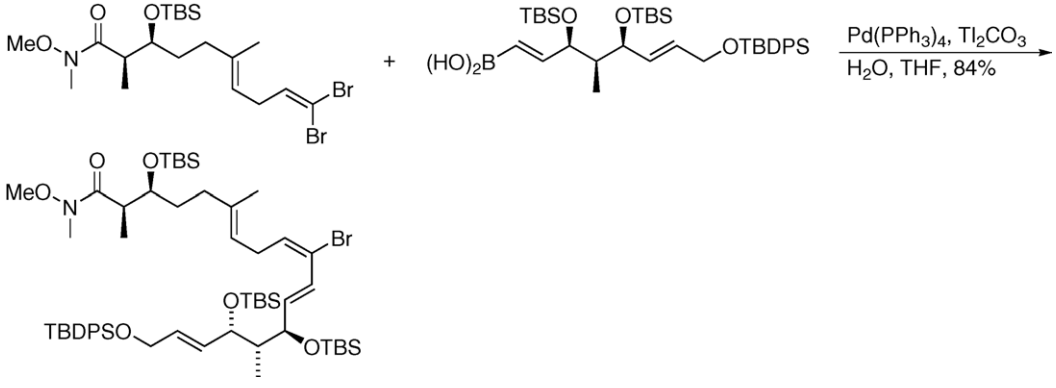

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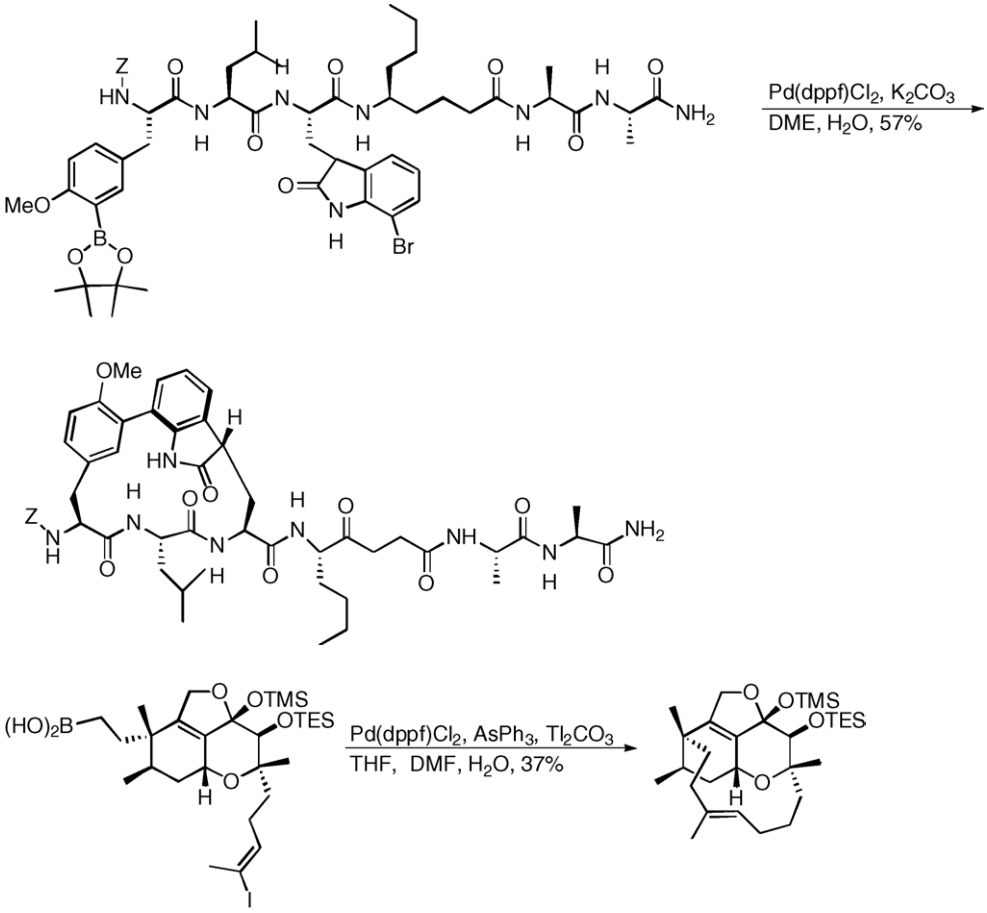

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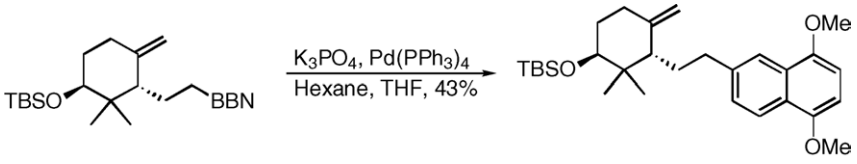
(32)

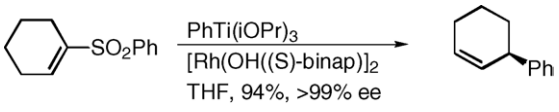
Alkyl boron reagents derived from hydroboration of terminal alkenes or addition of alkyl lithiums to B-OMe-9-BBN were used in synthesis of for example monomorine [328], salicylhalamides [329], core of halichlorine and pinnaic acid [330], ABC rings of phomactin A [331], and cordiaquinone K (Eq. (33)) [332].



(33)

Silicon reagents continue to be developed as alternatives to organotin, -boron, and -zinc compounds in palladium-catalyzed cross-coupling reactions with organic halides and triflates. Palladium-catalyzed cross-couplings of alkylidenesiloxycyclopentanes [333], 6-ethylidenedioxadisilacyclohexane [334], 1-fluoro-1-methyldiphenylsilylethene [335], phenylsilatrane and aryltrimethoxysilanes [336–338], alkynylsilanols [339], arylsilanols [340], *t*-butyldifluoroarylsilane [341], ethenyltrimethylsilane [342], 1-trimethylsilyl-1,3-diynes [343], and poly(diorganosiloxane) [344] with arylhalides and triflates. Alkyl bromides and iodides were coupled with arylsilanes using a palladium catalyst [345].

Some more unusual cross-coupling reactions employing organometallic reagents were described. Palladium–iron or –cobalt catalysts were used in cross-couplings of aluminum, gallium, and indium reagents with aromatic halides and triflates [346]. Palladium catalyzed the coupling of an alkenylzirconium compound derived from hydrozirconation of a 1-alkynyl sulfone with allyl bromide [347]. Alkenylzirconium compounds were coupled with (*Z*)-α-bromostannanes to give 1,3-dienyl-2-stannanes [348]. Alkynylgermatranes were coupled with aromatic chlorides and triflates [349]. Palladium-catalyzed coupling reactions of alkenylindiums [350], dihydropyranylindiums [351], dimethylindium reagents [352], and tetraorganoindates [353] with a variety of organic halides. Rhodium catalyzed an asymmetric formation of allylarenes from alkenyl sulfones and aromatic titanium reagents (Eq. (34)) [354].



(34)

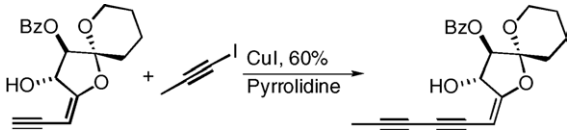
Palladium also catalyzed cross-coupling reactions of acid chlorides and α-haloketones with phenyl tributylstannyl selenide affording phenylseleno-substituted products [355]. Palladium catalyzed the coupling of ethynyl-1,5-azastibocines with aromatic iodides and bromides [356]. Palladium-catalyzed coupling of haloarenes and chloroarene tricarbonyl chromium complexes with alkenyl- and allylaluminum reagents [357]. Palladium also catalyzed the coupling of trimethylaluminum with 2,6-dichloropurine [121].

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

The palladium-catalyzed coupling between terminal alkynes and aryl and alkenyl halides or triflates, usually called the Sonogashira coupling, was one of the more frequently utilized carbon–carbon coupling reaction. A number of catalyst systems [131,172,188,189,358–369] including catalysts and reagents on solid support [6,216,370–376], and microwave-assisted couplings were described [220,377,378]. A polymer-bound Sonogashira macrocyclization was reported forming a cyclic peptide [379]. Unwanted homo-coupling of the alkyne can be suppressed using a 2% hydrogen in nitrogen or argon atmosphere [380]. Copper free reactions were described [381].

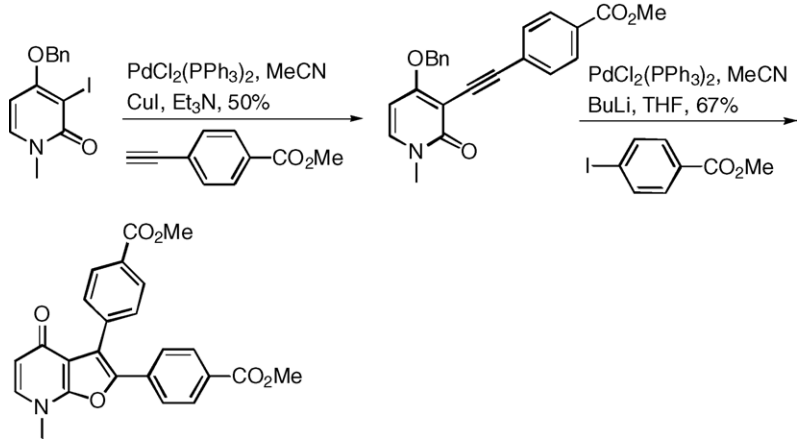
Palladium catalyzed the coupling of aromatic acid chlorides with TMS-ethyne [382]. Acid chlorides [383] and thiol esters [384] were coupled with terminal alkynes affording alkynones. Alkyl bromide and iodides were used in Sonogashira couplings [385].

Related coupling reactions using copper and nickel catalysts have also been reported. Copper catalyzed the coupling of terminal alkynes with monooxallyl chloride [386] and alkynyl iodide [387]. A copper-catalyzed coupling of an alkynyl iodide with and alkyne was used in a synthesis of (−)-AL-2 (Eq. (35)) [388] and callyberynes [389]. A copper-catalyzed intramolecular terminal alkyne–alkenyl iodide coupling was used in a synthesis of oximidines [390]. Copper catalyzed the coupling of a terminal alkyne with a TMS-alkyne in a synthesis of (+)-crocacin A [391]. Nickel catalyzed the cross-coupling of aromatic iodides with terminal alkynes [392].



(35)

Heterocyclic ring systems were shown to participate readily in Sonogashira-type couplings. Reactions of cyclic carbamate derived alkenylphosphonates [18], halofuranopyrimidines [393], 2-iodopyrroles [394,395], 2-bromo-3-formylindole [396], chlorotetrazines [157], 6-chloro-2(2*H*)-pyranone [397], 3-iodo-4*H*-4-chromenones [398], 4-iodoindole [399], 4-iodoimidazole [152], iodoimidazole-fused azasugars [400], 5-bromo- and 4,5-dihalo-3(2*H*)-pyridazinones [26,401], 2′-deoxy-5-iodouridines [402,403], 8-bromoadenine [404], 4-bromo-3-phenylsydnone [405], 4-iodopyrazole [406], 4- and 5-trifloxy-pyridazine-3(2*H*)-ones [407], and 2-iodopyrazine [40] derivatives were reported. Palladium-catalyzed regioselective coupling in the 2-position of 2,4-dibromoquinolines [408] and in the 4-position of 3,4-dibromo- and 3,4-dichloro-2(5*H*)-furanone [409]. A tandem Sonogashira coupling of 3-iodopyridones followed by hydroxy-palladation arylation producing furo[2,3-*b*]pyridones was described (Eq. (36)) [410]. A related tandem Sonogashira coupling annulation of 2-iodophenols with terminal alkynes forming benzofuranes was described [411]. Coupling of organic halides and triflates with 2-propynyl-1,3-dicarbonyl compounds furnished 2,3,5-trisubstituted furanes (Eq. (37)) [412].



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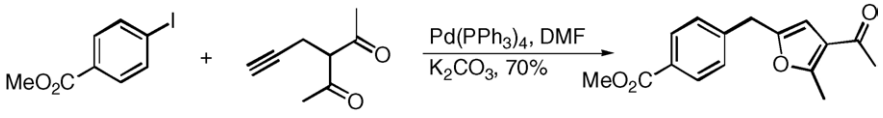
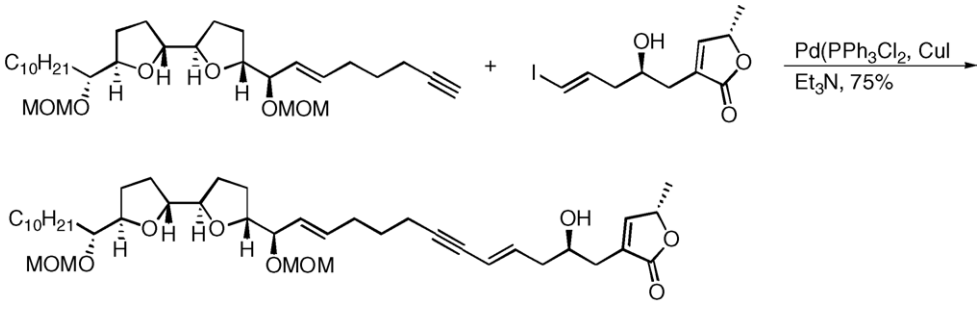


(37)

The Sonogashira reaction continued to be extensively used in organic synthesis [413]. Examples of synthetic applications include analogs of 1α,25-dihydroxyvitamin D_3 [414–416], annonaceous acetogins (Eq. (38)) [417,418], XH14 [46], 17α-substituted estradiol-pyridin-2-ylhydrazine conjugates [285], leptofuranin D [243], wedelolactone [419], a novel antitumor agent [420], lamellarin U and L on solids support [421], (+)-rogioloxepane A [422], vasopeptidase inhibitor BMS-189921 [423], callyberynes [389], α-substituted serine analogs [424], callipeltoside A [425], (+)-leucascandrolide A [426], 6*Z*,8*E*-heneicosadien-11-one [427], (+)-quinolizidine 207I [428], SB-242784 [69], imidazo[1,2-*a*]pyridines [303], (+)-neoisoprelaurefucin [429], 3-substituted prolines [308], bupleurynol [309], (15*S*,16*S*,21*R*)-4-deoxyrollicosin analog [430], an uPA-inhibitor [431], disorazole A_1 [98], (+)-virol [432], (−)-heliannuol E [433], epothilone analogs [434], 1α,25-dihydoxyvitamin D analogs [435], muconin [436], hachijodine G [437], (+)-leucascadrolide A [438], disorazole A_1 [439], cicerfuran [440], (−)-aspidophytine (Eq. (39)) [441], borrelidin [167], (+)-obtusenyne [442], tetrodotoxin [443], and (*E*)- and (*Z*)-(+)-pinnatifidenyne [444]. A palladium-catalyzed Sonogashira-type coupling mediated by polymethylhydrosiloxane was used in a synthesis of (−)-akolactone A [359].



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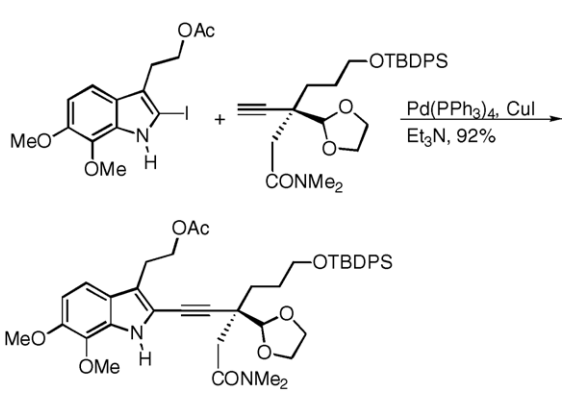
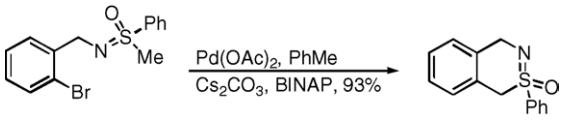


(39)

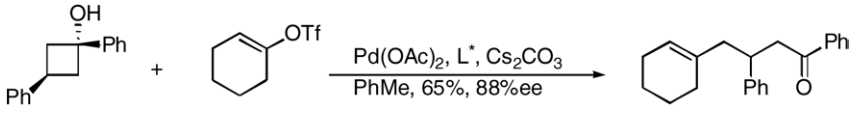
Other carbon nucleophiles were also employed in palladium-catalyzed carbon–carbon bond-forming reactions of halides and triflates. Palladium-catalyzed α-arylations of ketone enolates [232,445–448], zinc enolates of esters and amides [449], *N*-protected 2-piperidones [450,451], ethyl cyanoacetate and nitriles [452,453], azalactones [454], and malonitrile [455,456] using aryl halides and tosylates. Palladium catalyzed the reaction of sodium dimethyl malonate with heterocyclic benzylic substrates [457].

Palladium catalyzed an intramolecular coupling of ketone enolates with a pendant alkenyliodide [458] and of sulfoximines with pendant aromatic halides (Eq. (40)) [459]. Intramolecular palladium-catalyzed α-arylations/alkenylations [460] were used in the synthesis of a number of sarpagine alkaloids [461–463], the tricyclic skeleton of FR901483 [464], and γ-lycorane [465].



(40)

Palladium-catalyzed asymmetric arylations, alkenylation, and allenylation of cyclobutanols (Eq. (41)) [466]. Palladium catalyzed the formation of diarylethyne from aromatic bromides and 1-bromo-2-chloroethane under phase-transfer catalysis conditions (Eq. (42)) [467].



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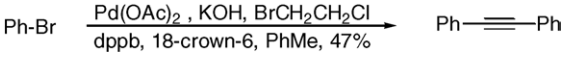
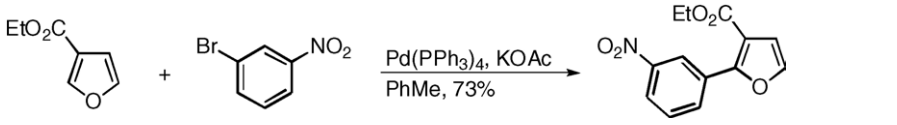


(42)

Heterocyclic substrates can also be used as carbon nucleophiles [260,468–470], at least in a formal sense, without initial deprotonation. Palladium catalyzed the coupling of 3-carbalkoxyfuran and -thiophene with aromatic bromides (Eqs. (43) and (44)) [471]. Cobalt and palladium catalyzed the arylation of thiazoles, oxazoles, and imidazoles using aromatic bromides or iodides [472]. The regioselectivity of the arylation was reversed depending on choice of catalyst system (Eq. (45)) [473]. Palladium catalyzed the C2-arylation of an imidazole with 2-iodopyridine

[152]. Depending on the catalyst and additives either C2- or C4-arylation of imidazoles can be obtained (Eq. (46)) [474,475]. Intramolecular palladium-catalyzed reaction of α-chloroacetanilides furnished oxindoles (Eq. (47)) [476]. Rhodium catalyzed a direct intermolecular *ortho*-arylation of phenols (Eq. (48)) [477–479], and imidazol-2-ylbenzenes [475] using aryl halides. An unusual arylation of α,α-disubstituted arylmethanols with alkyl halides was described (Eq. (49)) [480].



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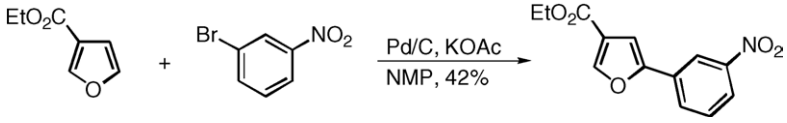

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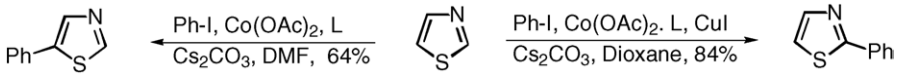

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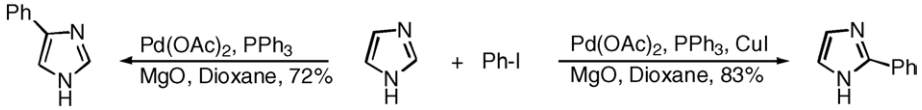

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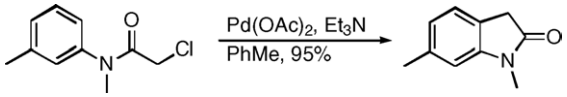

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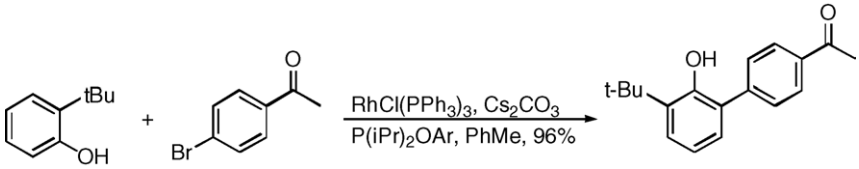

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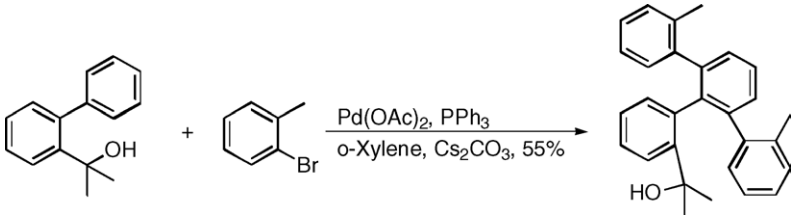
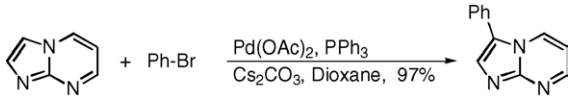


(49)

The palladium-catalyzed reaction of aromatic carbon nucleophiles with halides and triflates was used to prepare pyrazolo[1,5-*f*]phenanthridines [481], an oxazolo[4,5-*c*]quinoline-4(5*H*)-one [253], 3-arylimidazo[1,2-*a*]pyrimidines (Eq. (50)) [482], lirinidine and nuciferin [483], isocryptolepine [484], fagaridine and decarine [485], anhydrolycorinone and hippadine [486], tetrahydrophenanthridone [487], pyrrolophenanthridines [488], pyrrolophenanthridones [489], 3*H*-benzo[*e*]cyclo[3.3.2]azin-3-one [490], naphth[3,2,1-*cd*]indole [491], heteroannelated indoles and carbazoles [492], and 6*H*-naphtho[2,3-*c*]chromene-7,12-diones [493].



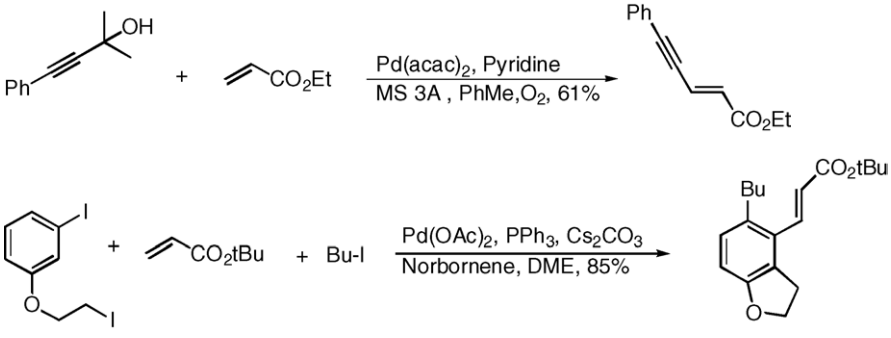
(50)

Palladium catalyzed the cyanation of aromatic halides with zinc dicyanide or potassium cyanide [494–497]. This reaction was accelerated under microwave irradiation in a synthesis of 3-cyano-3-desoxy-10-ketomorphinanes [498]. Trimethylsilylcyanide [499] and acetone cyanohydrin [500] were used as the cyanide source in palladium-catalyzed cyanations of aromatic halides. Copper catalyzed a cyanation of aromatic bromides via initial bromide to iodide exchange [501].

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

The Heck (or Mizoroki–Heck) reaction continued to be one of the most versatile methods for the alkylation of alkenes. A variety of ligands [131,502–504], catalyst systems [2,188,199,200,505–522], catalysts and reagents on solid support [216,523–529], and reaction conditions including unusual solvents [530,531] were described. Added halide was shown to accelerate Heck reactions in ionic liquids [532]. Examination of the Heck reaction indicated that the oxidative addition step is not the rate determining step at high pressures [533]. The regioselectivity of the Heck reaction can be controlled to some extent using different bidentate ligands [534].

Enamides [535], 1-alkoxy-1,3-dienes [536], and alkenylboronic acid esters [537] were used as the alkene in Heck reactions. Reaction of aromatic iodides and bromides with acrolein diethyl acetal furnished cinnemaldehydes after hydrolysis [538]. Tertiary propargylic alcohols were used as precursors for alkynylation of alkenes (Eq. (51)) [539]. In situ prepared allylic mesylates, and triflates were used in place of halides [540], and 2-chloroacetamides and bromoacetonitrile [541] were used Heck reactions. An intriguing annulation-Heck reaction was reported in the presence of norbornene (Eq. (52)) [542].



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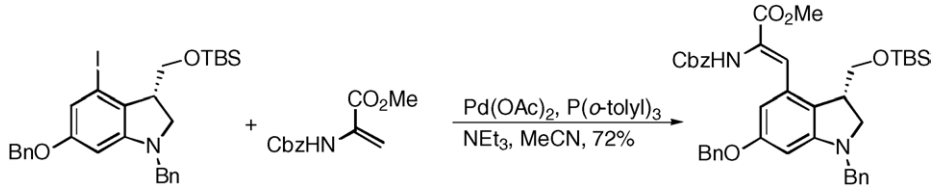
(52)

Aromatic carboxylic acid chlorides were used in place of halides via an initial decarboxylation and this type of reaction was applied to the synthesis of resorcylic acid [543]. Arylphosphonic acids can also be used in palladium-catalyzed Heck-type reaction in place of halides and triflates [544]. Halides and triflates of a variety of heterocyclic ring systems were used, such as 4-iodoimidazole [152], a triflate derived from kojic acid [41], 2-iodopyrrolo[2,3-*b*]quinolines [31], and 5,6,7,8-tetrahydro-2-iodoimidazo[1,2-*a*]pyridine [266].

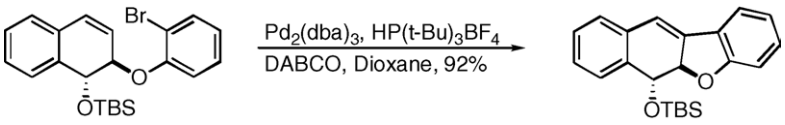
A regio- and stereoselective Heck reaction of cyclic enol ethers with aryldiazonium salts was described [545]. A palladium–rhodium two-catalyst system effected both an intermolecular Heck reaction followed by a hydroformylation using a supported liquid phase system [546]. Domino Heck-Diels–Alder reaction of 1,3-dicyclopropyl-1,2-propadiene was described [547].

The intermolecular Heck reaction was used in organic synthesis of for example, a novel COX-2 inhibitor [548], (+)-cryptophycins [549], unnatural nucleosides [550,551], toward beraprost [552], tangutorine [553], (+)-panepophenathrine [554], macrocycles [555], a glycosidase inhibitor [556], crocacin [413], flavonoids [557], epiuleine [558], melatonin [559], (−)-callystatin A [560], lyngbyatoxin A [561], annoretine and litebamine [562], an HIV reverse transcriptase-selective nucleoside chain terminator [563], protein tyrosin phosphatase 1B inhibitor [564], ducarmycins (Eq. (53)) [565], and (−)-ephedradine [566].



(53)

An intramolecular Heck reaction involving an anti-β-hydride elimination, in the presence of DABCO, was used to prepare tetracyclic compounds (Eq. (54)) [567]. Intramolecular Heck reaction of a 1,1-dibromo-1,5-diene followed by a nickel-catalyzed cross-coupling was described (Eq. (55)) [568]. An oxidative addition-1,4-migration-annulation sequence furnished fused polycyclic compounds (Eq. (56)) [569]. Depending on the catalyst system either a Heck reaction or an amination was observed for some indole derivatives [570].



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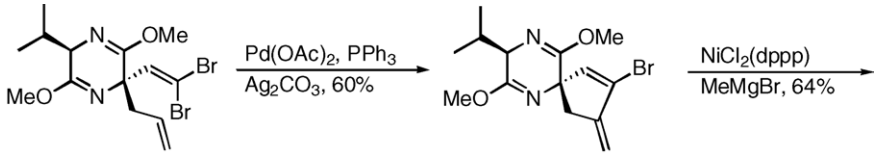

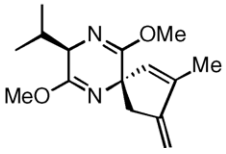

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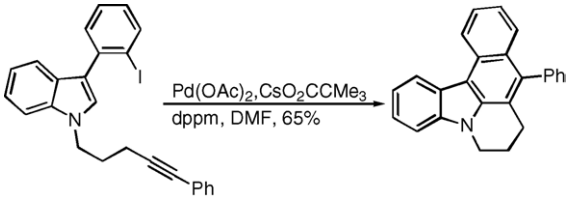
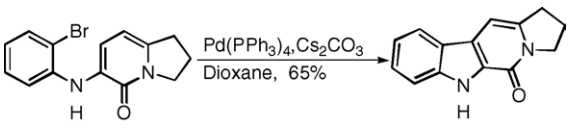


(56)

The intramolecular variation of the Heck reaction [571–573] was used as the key step toward an array of synthetic targets for example, maritidine [574], ring analogs of ergot alkaloids [575], taxol analogs [576], toward guanacastepenes [577], β-carbolines (Eq. (57)) [578], idiospermuline (Eq. (58)) [61], hodgkinsines [62], cyclic peptides [579], 2,3,5-trisubstituted-furo[3,2-*b*]pyridines [580], 6-epi-(−)-hamigeran B [581], lennoxamine and chilenine [582], galanthamine ring system [583], curcumene ether [584], huperzine A [585], oxerine [586], and indole alkaloids [587]. A sequential intra- and intermolecular Heck sequence was used to prepare B-nor-steroids [588].



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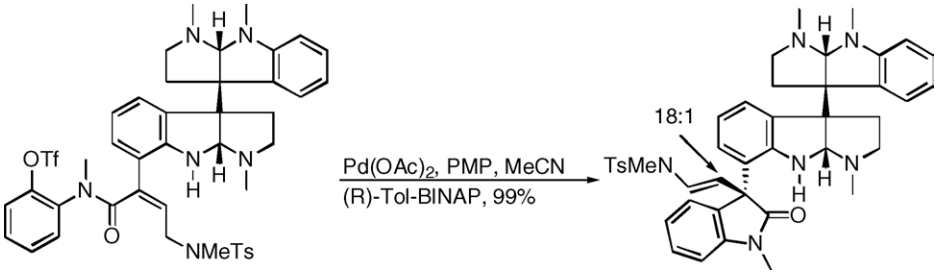
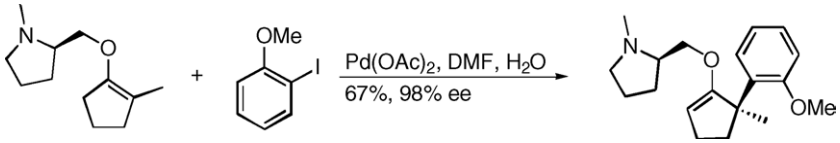


(58)

A number of catalyst systems for asymmetric Heck reactions of cyclic alkenes were developed [589–593]. Palladium-catalyzed intramolecular asymmetric Heck reactions to give *cis*-decalines [594] and 3-alkyl-3-aryl oxindoles [595]. An asymmetric reductive Heck reaction using a chiral enecarbamate and diazonium salts was used in a synthesis (2*S*,5*R*)-phenylproline methyl ester and Schramm's C-azanucleoside [596]. Palladium catalyzed an asymmetric chelation-controlled intermolecular Heck reaction (Eq. (59)) [597]. Reductive Heck reactions [598] were used in syntheses of dichroanal B and dichroanone (Eq. (60)) [599], analogs of epibatidine [600], and furaquinoncins [601].



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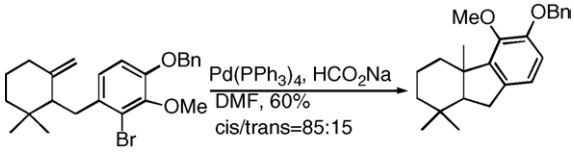
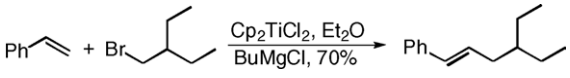


(60)

A nickel-catalyzed coupling of aromatic bromides with acrolein diethyl acetal to give arylated enol ethers was reported [602]. Titanium catalyzed Heck-type reactions of arylsubstituted alkenes with alkyl bromides and chlorides (Eq. (61)) [603]. Electrochemically driven cobalt-catalyzed

coupling of aromatic halides with alkenyl acetates was reported (Eq. (62)) [604].



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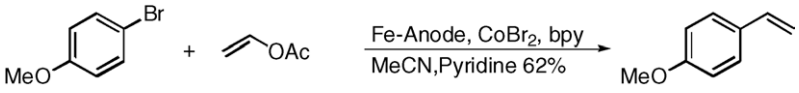
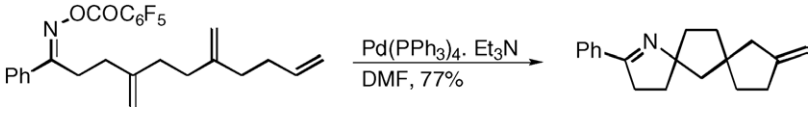


(62)

Reactions involving an oxidative addition of palladium to organic halides and triflates followed by an alkene insertion and a termination step, different from the β-hydrogen elimination observed in the Heck reaction, were extensively examined. Insertion of a second or third alkene was used to prepare polycyclic compounds from pentafluorobenzoyloximes (Eq. (63)) [605]. Multiple insertions were used to prepare macrocycles from two dienes and two diiodides [606]. A Heck reaction initiated multiple carbon–carbon bond coupling forming tricyclic compounds was described (Eq. (64)) [607]. Palladium catalyzed a reaction of 1,6-dienes with benzyl halides to give substituted cyclopentenes [608].



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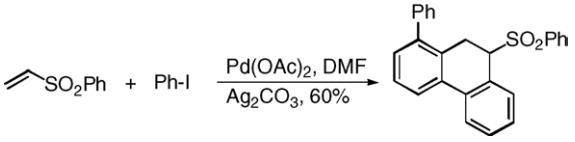
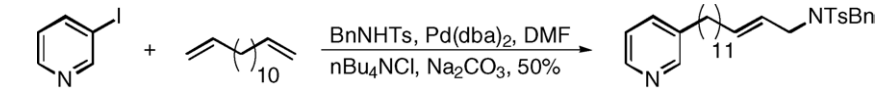


(64)

Oxidative addition of palladium to 3-iodopyridine followed by insertion of a non-conjugated diene, migration of palladium, and intermolecular amination was used in synthesis of pyridine alkaloids (Eq. (65)) [609]. A related reaction of 2-iodobenzenamines with conjugated a sulfur-substituted diene terminated in an intramolecular amination was used to prepare 2,3-dihydroindoles [610]. A tandem Heck reaction intramolecular allylic amidation was described (Eq. (66)) [611].



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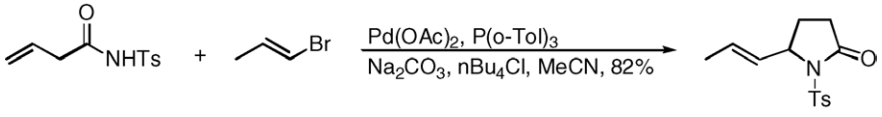
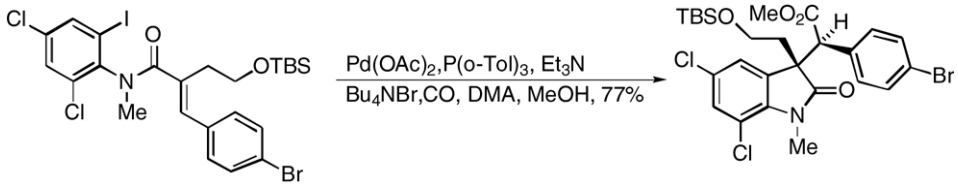


(66)

A palladium-catalyzed oxidative addition, intramolecular alkene insertion, and carbonylation sequence was used in an approach to perophoramidine (Eq. (67)) [612]. Oxidative addition followed by insertion of a conjugated diene and intramolecular alkoxylation was used to prepare dihydrofurocoumarins (Eq. (68)) [613]. An oxidative addition to benzyl halides followed by an intramolecular alkene insertion and an intermolecular alkene insertion of *N*-allyl-*N*-(2-butenyl)-*p*-toluenesulfonamides was used to prepare 2,3-dihydropyrroles [614]. Cobalt catalyzed the reaction of alkyl bromides with conjugated dienes and trimethylsilyl magnesium chloride furnishing 4-trimethylsilyl-1-enes (Eq. (69)) [615]. Nickel catalyzed the coupling of oxa- and azabicyclicalkenes with aromatic and alkenyliodides to give annulated coumarins and lactams [616].



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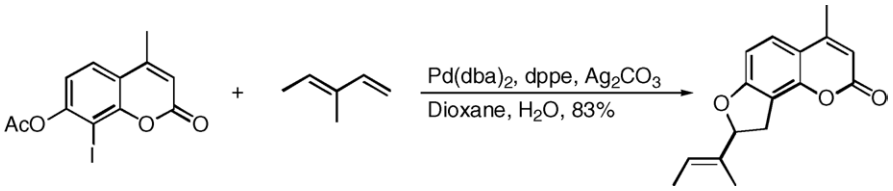

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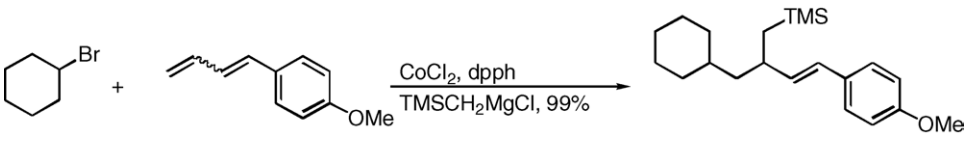
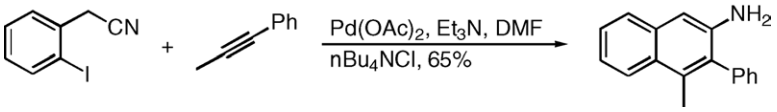


(69)

2.4. Carbon–carbon bond formation via insertion of alkynes

Reactions wherein σ-palladium complex intermediates are formed by oxidative addition of palladium(0) to aryl iodides, bromides, and triflates followed by sequential insertion of an alkyne and a termination step, usually a nucleophilic addition, are discussed in this section. Palladium-catalyzed the reaction of 2-iodobenzylnitriles with alkynes to give 3,4-disubstituted 2-aminonaphthalenes and 1,3-benzoxazine derivatives (Eq. (70)) [617]. Palladium catalyzed the formation of 3,4-disubstituted isoquinolines from 2-(1-alkynyl)benzaldimines and organic halides (Eq. (71)) [618]. Related formation of 4-(1-alkenyl)isoquinolines from 2-(1-alkynyl)benzaldimines and alkenes [619], and polycyclic heteroaromatic compounds via intramolecular reactions with pendant alkynes [620] were reported. Similar reactions to give pyridine fused compounds were described (Eq. (72)) [621]. Tetrahydroquinoline alkaloids were prepared from 2-iodobenzaldehyde imines and 3-benzyloxy-1-propyne via an oxidative addition, alkyne insertion, and cyclization sequence (Eq. (73)) [622].

Oxidative addition followed by insertion of two alkynes and cyclization produced tetrasubstituted naphthalenes (Eq. (74)) [623]. Related annulations of internal alkynes with aromatic iodides having a pendant alkene furnished naphthalenes and carbazoles (Eq. (75)) [624]. An oxidative addition-alkyne insertion annulation sequence was used to prepare pyridopyrrolo[2,1,−*a*]isoindoles (Eq. (76)) [625]. Dioxolanes formed from 6-bromo-3,4-dihydroxy-5-en-1-ynes either undergo a palladium-catalyzed cyclization Stille coupling or direct Stille coupling with tin reagents. The mode of reaction depends on the dioxalane stereochemistry (Eqs. (77) and (78)) [626]. Related reactions affording bicyclic 1,2-cyclobutendiols using 5-bromo-3,4-dihydroxy-5-en-1-ynes were described [627].



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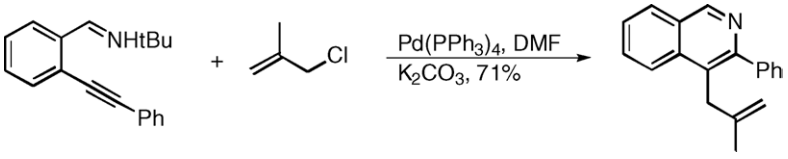

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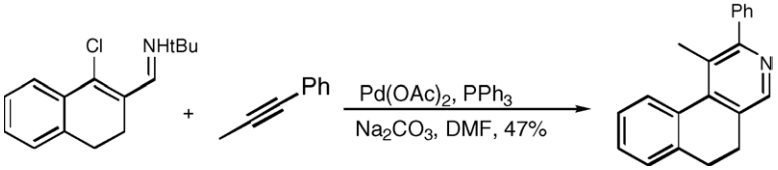

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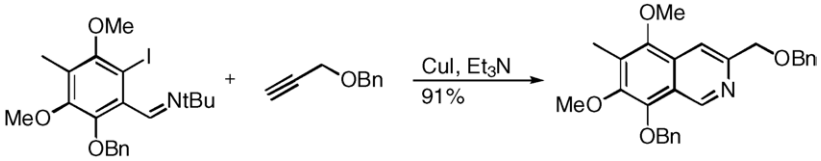

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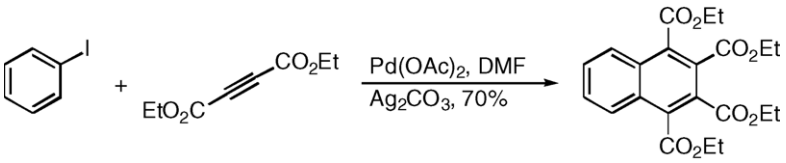

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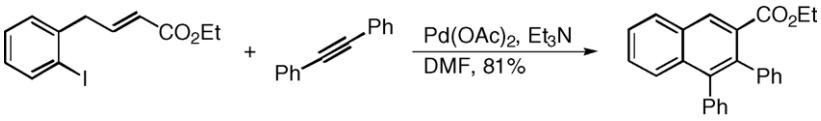

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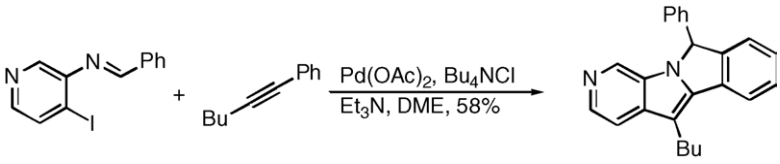

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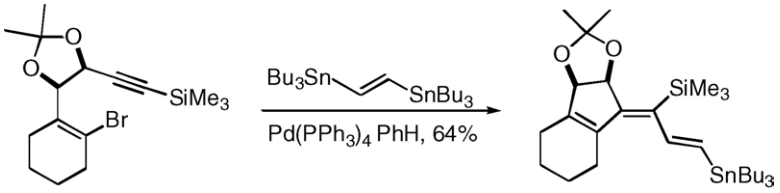

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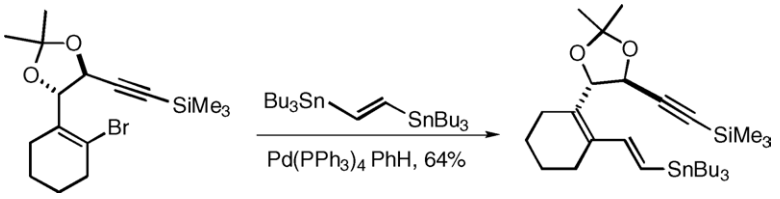
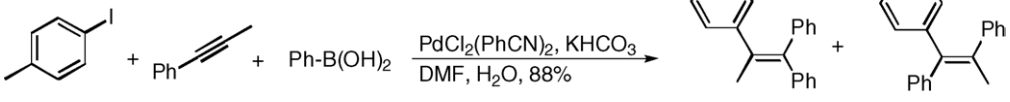


(78)

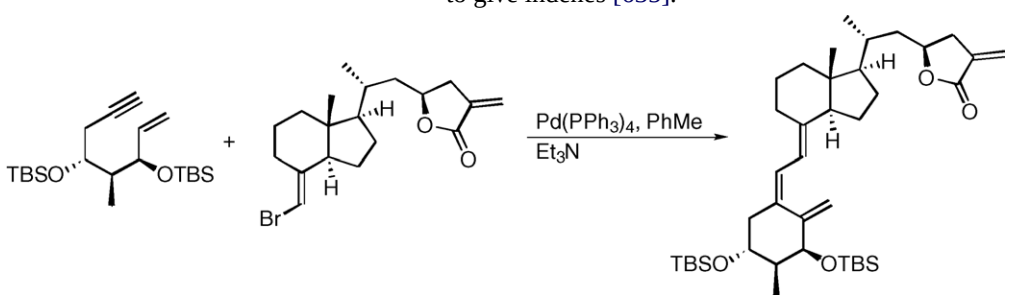
Highly substituted 1,3-dienes and 1,3,5-trienes were prepared via a palladium-catalyzed oxidative addition, alkyne insertion, and boronic acids termination sequence [628].

Tetrasubstituted alkenes were prepared by a palladium-catalyzed reaction of aromatic iodides, an internal alkyne, and an aromatic boronic acid (Eq. (79)) [629].



(79)

Palladium catalyzed an oxidative addition, intramolecular alkyne insertion, intramolecular Heck-type reaction of (*Z*,*Z*)-diaryl-1,5-hexadien-3-ynes to give (*E*)-1,1′-biindenylidene derivatives [630]. A related reaction was used to prepare 1α-hydroxyvitamin D_3-26,23-lactones (Eq. (80)) [631]. A related intramolecular reaction terminated in a Suzuki-type reaction was also reported [632]. A sequence consisting of an oxidative addition-alkyne insertion, and trapping of the formed σ-palladium intermediate with boron or tin reagents was described [633]. Cobalt catalyzed an oxidative addition, alkyne insertion, and carbocyclization sequence of 2-iodobenzaldehydes and 2-iodophenylketones to give indenole derivatives (Eq. (81)) [634]. Nickel catalyzed an oxidative addition, alkyne insertion, and intramolecular ketone alkylation of an intermediately formed alkenylnickel species to give indenes [635].



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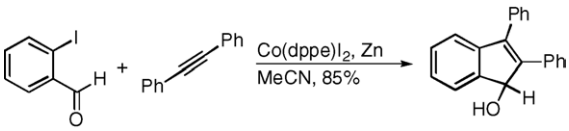
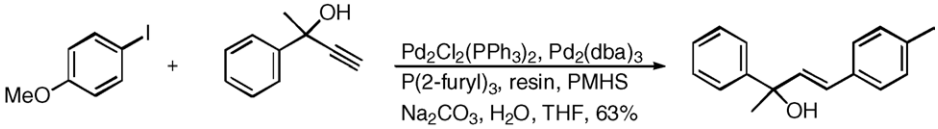


(81)

A palladium-catalyzed oxidative addition to 2-iodoaniline derivatives followed by alkyne insertion and intramolecular amination to give indoles was used to prepare psilocin [636] and in a synthesis toward celogentin C [637]. Palladium catalyzed a reductive Heck-type reaction of terminal alkynes with aromatic and alkenyl halides (Eq. (82)) [638]. A reductive intramolecular Heck-type reaction of aromatic halides having a tethered alkyne using triethylsilane or ammonium formate as the reducing agent was described (Eq. (83)) [639]. Palladium-catalyzed oxidative addition followed by insertion of an alkyne and carbonylative cyclization of 2-iodophenols or 2-iodobenzenamines furnished coumarins and 2-quinolones [640,641].



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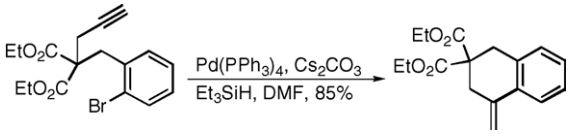
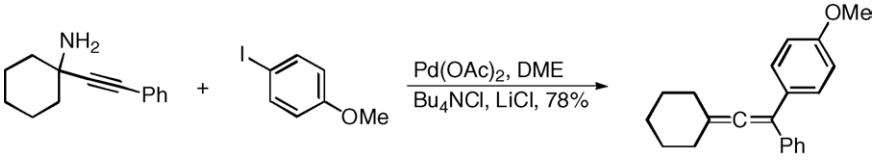


(83)

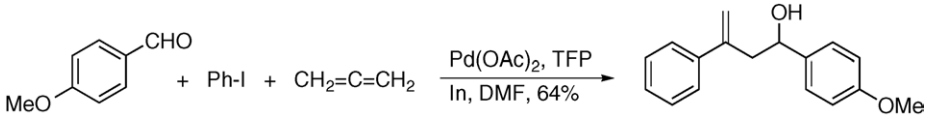
A palladium-catalyzed reaction of aromatic halides with 1-(1-alkynyl)cyclobutanols to give α-alkylidene cyclopentanones was described [642]. Palladium catalyzed the coupling of tertiary propargylic amides with aromatic halides to give allenes (Eq. (84)) [643].



(84)

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 terminations. Oxidative addition, intermolecular allene insertion, followed by transmetallation to indium and intermolecular aldehyde alkylation was described (Eq. (85)) [644]. An interesting oxidative addition, intermolecular allene insertion, and intramolecular alkene insertion reaction was reported (Eq. (86)) [645]. Nickel catalyzed an oxidative addition, allene insertion, and allenylzirconocene termination reaction (Eq. (87)) [646]. Palladium catalyzed a related oxidative addition to acid chloride followed by allene insertion and termination with dipinacolatoboron, hexamethyldisilane, or hexabutylditin [647]. Palladium catalyzed a three-component reaction of aryl iodides, allenes, and a stannylgermane to afford 2-arylallylgermanes (Eq. (88)) [648].



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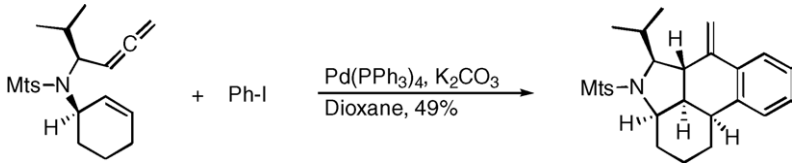

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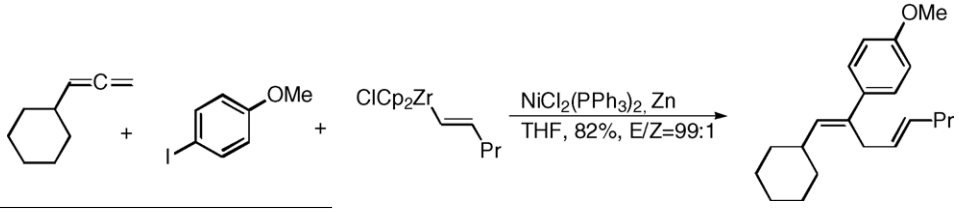

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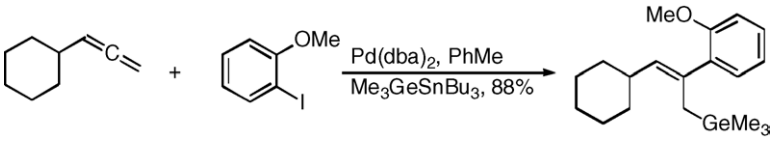
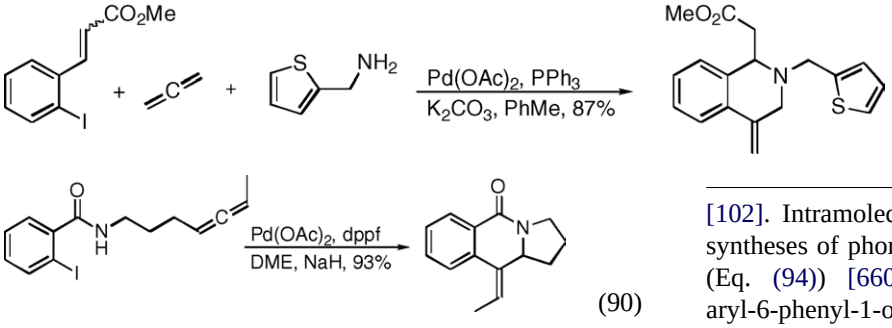


(88)

Oxidative addition, intermolecular 3- or 4-amino-1,2-diene insertion followed by intramolecular amination furnished 2,5-dihydropyrroles or 1,2,3,6-tetrahydropyridines, respectively [649]. Related reactions of 2,3-diene-1-ones to give 2,3,4-, 2,3,5-trisubstituted, and 2,3,4-tetrasubstituted furanes [650] and reactions of 2,3-dienoic acids affording butenolides [651] were described. A sequence of oxidative addition, intermolecular allene insertion, intermolecular amination or alkylation followed by an intramolecular Michael addition reaction was described (Eq. (89)) [652]. An oxidative addition, intramolecular allene insertion, and allylic amidation sequence was used to prepare isoquinolone derivatives (Eq. (90)) [653].



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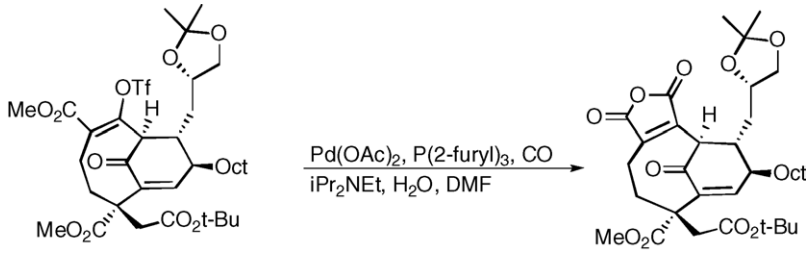
2.6. Carbon–carbon bond formation via insertion of carbon monoxide

A variety of palladium complexes and catalyst systems catalyzed the carbonylation of alkenyl and aryl halides and triflates to afford carboxylic acids and derivatives [654]. *N*-hydroxysuccinimido esters were prepared via a palladium-catalyzed carbonylation of aromatic triflates and halides in the presence of *N*-hydroxysuccinimide [655].

The palladium-catalyzed alkoxy-carbonylation of halides and triflates was used in synthetic applications toward martinellic acid [656], steroids [44], phomoidride B (Eq. (91)) [173], toward azadirachtin [296], macrosphelide A (Eq. (92)) [657], calcitrol A-ring [658], and (+)-aphidicolin [102]. Intramolecular alkoxy-carbonylations were used in syntheses of phomoidrides (Eq. (93)) [659], (+)-manoalide (Eq. (94)) [660], a macrosphelide library [661], 3-aryl-6-phenyl-1-oxa-7-azaspiro[4.5]decanes [662], and 2-substituted pyridazino[4,3-*h*]psoralenes [663]. A nickel-catalyzed intramolecular alkoxy-carbonylation of a 4-hydroxy-2-bromoalkene was used to prepare a 3-methylene-4,5-dihydro-2-furanone [110].



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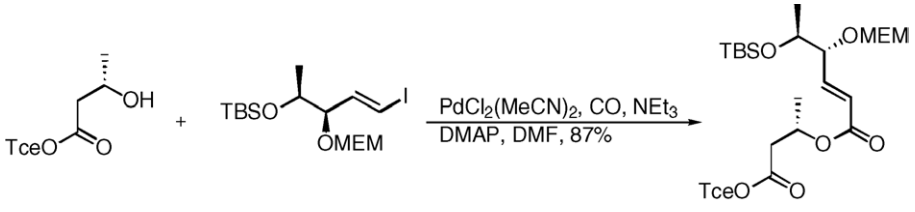

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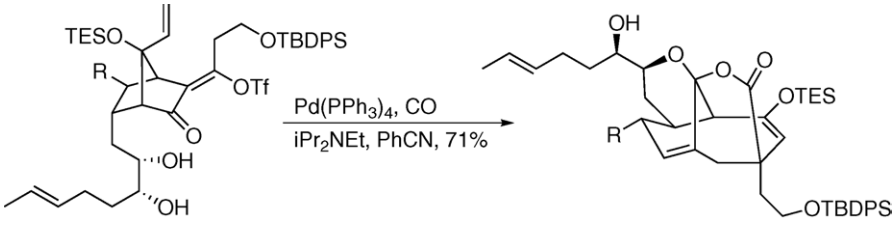

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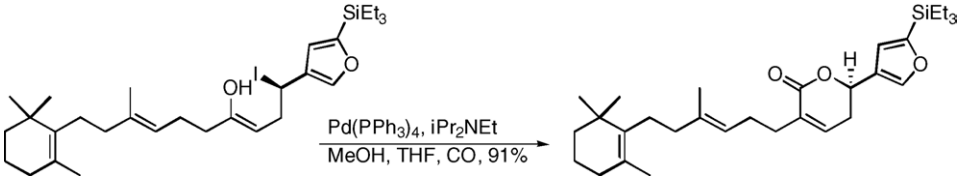
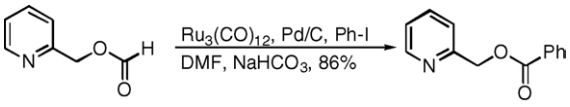


(94)

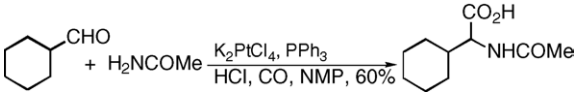
An enantioselective palladium-catalyzed methoxycarbonylation of 1,3-dichloroarenetricarbonyl chromium complexes was reported [664]. Palladium catalyzed the hydroxycarbonylation of aryl and alkenyl halides and triflates using acetic anhydride and formate ions as the carbon monoxide source [665]. A palladium–ruthenium catalyst couple was used to prepare pyridylmethyl esters from iodides or triflates and pyridylmethyl formate (Eq. (95)) [666].



(95)

Palladium catalyzed the formation of isoindolinones from 2-iodobenzylbromide [667] and 2-halobenzylamines [668].

Related reactions of 2-iodostyrene derivatives forming 3-substituted isoindolinones were described [669]. Iodoferrocene was used to prepare ferrocene-substituted amides and ketoamides [670]. Platinum catalyzed amidocarbonylations of aldehydes to give amino acid derivatives (Eq. (96)) [671]. Double carbonylation of aromatic iodides in the presence of diethylamine [672] and an asymmetric palladium-catalyzed double carbohydroamination of aromatic iodides to give α-aminoamides (Eq. (97)) [673] were described. Isotopically labeled amides were prepared using ^{11}C carbon monoxide [674]. Direct formation of amides from alkenyl halides was observed using a palladium catalyst and carbamoylsilane as the amide source [675]. Aldehydes furnished the carbonyl carbon in ruthenium-catalyzed intramolecular aminocarbonylations to form lactams [676].



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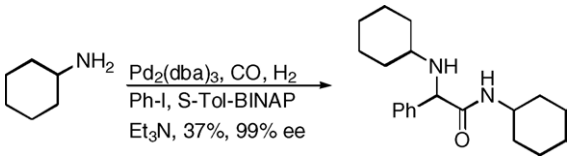
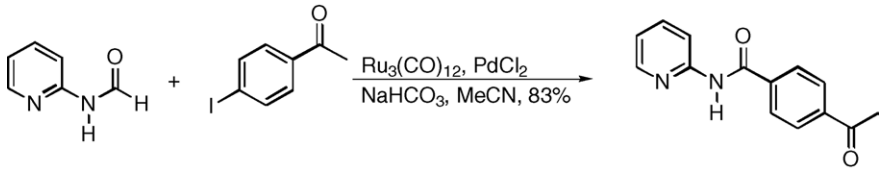


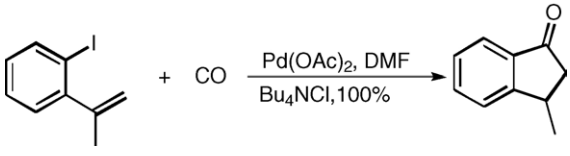
(97)

Palladium catalyzed the carbonylation of aromatic iodides in the presence of alkynes to give aryl alkynyl ketones [677]. Cobalt catalyzed the formation of symmetrical ketones by carbonylation of aromatic iodides under microwave irradiation [678]. A palladium–ruthenium catalyst system was used in aminocarbonylations of aromatic iodides using *N*-(2-pyridylmethyl)formamide (Eq. (98)) [679].



(98)

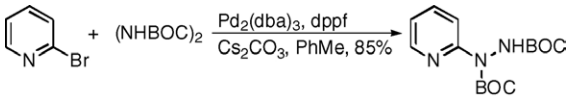
A palladium-catalyzed oxidative addition, carbonylation, allene insertion, intermolecular amination sequence was developed affording functionalized unsymmetrically substituted ketones [680]. Palladium catalyzed an oxidative addition, carbon monoxide insertion, alkene insertion sequence leading to indanones and 2-cyclopenenones (Eq. (99)) [681].



(99)

Palladium catalyzed the coupling of triorganoindiums with aryl, benzyl, alkenyl, and acyl halides and triflates in the presence of carbon monoxide to give unsymmetrical ketones. All three groups on indium were coupled [682,683]. Palladium-catalyzed carbonylative couplings of halopyridines and phenylboronic acid [684,685] and an aromatic iodide and 4-stannyl-4-pyrone [41] to give unsymmet-

rical ketones. Palladium catalyzed the coupling of aryl diazonium salts with aromatic and alkenylic boronic acids in the presence of carbon monoxide to produce amides [686].



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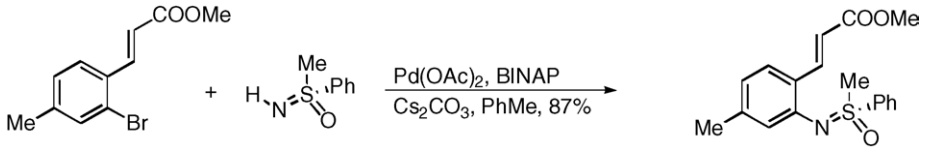
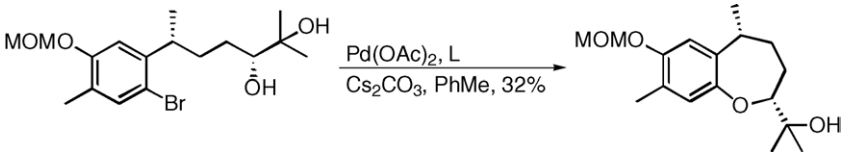


(102)

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

Palladium catalyzed the coupling of alkenyl triflates with phenols to give aryl enol ethers [687]. An intramolecular palladium-catalyzed alkoxylation was used in a synthesis of (+)-heliannulol D (Eq. (100)) [688]. Palladium catalyzed an S_N1-type ether-formation of secondary benzylic alcohols to give symmetric (homo-coupling) and unsymmetric ethers (cross-coupling) with a second molecule of alcohol [689]. Copper-catalyzed intermolecular alkoxylations of aromatic trifluoroborates with alcohols [690]. Copper-catalyzed Ullmann-type couplings of phenols with aromatic halides were reported [691–694]. Microwave-accelerated reactions were described [695] using water as the solvent [696]. Copper catalyzed the coupling of water with aromatic chlorides under microwave irradiation to form phenols [697].



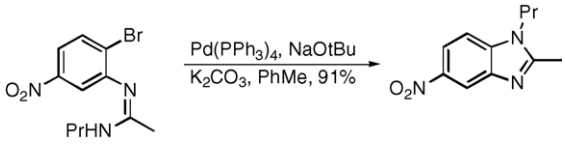
(100)

A variety of aryl halides, triflates, and benzenesulfonates were aminated or amidated using a number of palladium or copper catalysts [2,198,275,445,698–705]. Aminations using reagents on solid supports were reported [706]. Microwave-assisted palladium-catalyzed aminations [707] of 1-bromonaphthalenes and 5- and 8-bromoquinolines [708] and of nitrogen-containing aromatic chlorides [709] were described. Nickel catalyzed the amination of aromatic nitriles in place of halides or triflates [710].

Palladium catalyzed the intermolecular *N*-arylation of aziridines [711], di-*t*-butylhydrazodiformate (Eq. (101)) [285], aromatic hydrazides [712], 17α-substituted estradiol-pyridine-2-ylhydrazine conjugates [285], oxazolidinones [713], benzophenone imine [714], sulfonamides [715], chiral sulfoximines [716], imidazoles [475], ureas [717], linear polyamines [718], and 8-amino-2′-deoxyguanosine [719]. A chiral sulfoxime was used in a synthesis of (+)-curcumene and (+)-curcuphenol (Eq. (102)) [720].

Palladium catalyzed the amination of aromatic nonaflates [721], electron-deficient aromatic fluorides [12], and amidation of enol triflates [722]. Aminations of 5-membered heterocyclic halides [723], 6-haloimidazolo[1,2-*a*]pyridines [724,725], 6-chloropurine deoxynucleoside [726], triflates derived from 2(1*H*)-pyridones [578], 4-chloro-1*H*-pyrrole[2,3-*c*]pyridine [727], 3- and 6-bromobenzothiophens [492], 2-substituted azulenes [728], and a 7-chloropyrazolo[1,5-*a*]pyridine [729] were described. High selectivity was observed using some dihalogenated heterocycles. 5-Bromo-2-chloropyridine was selectively aminated in the 5-position, 2,5-dibromopyridine in the 2-position [730], and 3.5-dibromo-2-pyrone in the 3-position [731] using palladium catalysts.

Intramolecular palladium- or copper-catalyzed aminations or amidations were used in the synthesis of heterobenzazepine ring system [732], indoles [733], indolines, indolizidines, and pyrrolizidines [734,735], benzimidazoles (Eq. (103)) [736], 3,4-dihydro-2(*H*)-quinazolinones [737], benzimidazoles [738], and cryptocarya alkaloids [739]. Intramolecular palladium-catalyzed amidations were used to prepare fumiquinazolines (Eq. (104)) [740], 3-alkoxycarbonyl-1β-methylcarbapenem [741], and 2,3,4,5-tetrahydro-1-benzazepines [742]. A palladium-catalyzed Heck reaction intramolecular amination furnished indoles [733].



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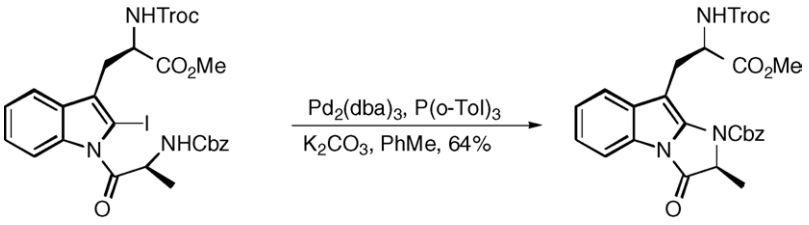
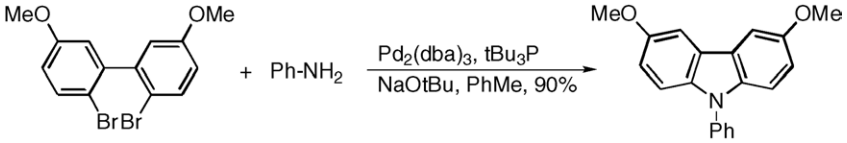


(104)

Palladium-catalyzed intermolecular aminations were used to prepare an src kinase inhibitor [743], carbazomadurin A [68], and isocryptolepine [484]. A combination inter–intramolecular amination was used to prepare fused heterocyclic systems (Eq. (105)) [744]. Palladium catalyzed an S_N1-type amination of secondary benzylic alcohols with benzeneamines to give unsymmetric amines [689]. An iron catalyzed sulfimidation-rearrangement of propargylic sulfides using BOC-azide was described (Eq. (106)) [745].



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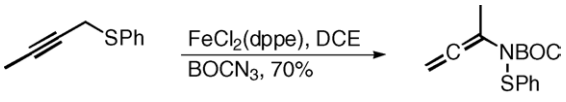
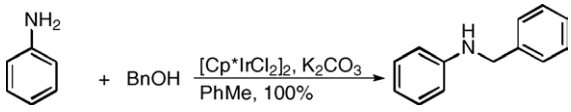


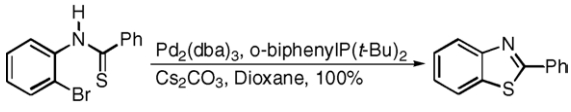
(106)

Transition metals other than palladium, mainly copper, also catalyzed amination reactions. Ullmann-type couplings of amines and amides with aromatic halides using a copper catalyst are receiving more and more attention [746–750]. Reactions accelerated by microwave irradiation in water were described [751,752]. Copper catalyzed the *N*-alkylation of a variety of *N*-heteroaromatic compounds [753]. Copper-catalyzed intramolecular amination was used to prepare an aporphine alkaloid [299]. Copper-catalyzed amidation of aromatic bromides was used to prepare linezolide and toloxatone [754] and ducarmycins [565]. Copper-catalyzed intermolecular amidations [755] were used in a synthesis of lobatamide C [92], and in amination [756] of alkenyl halides. Copper-catalyzed *N*-alkynylation of carbamates, ureas, sulfonamides, oxazolidinones, and imidazolidinones using 1-bromo-1-ynes [757]. Copper catalyzed the *N*-arylation of sulfonamines using aryl bromides [758] and *N*-arylation of indigo using aromatic bromides and iodides [759]. Nickel-catalyzed intramolecular aminations of aryl chlorides [760]. Couplings using nickel on carbon is most likely of a homogenous nature [112,761]. Iridium catalyzed a direct N-alkylation of amines with alcohols (Eq. (107)) [762].



(107)

Palladium catalyzed the coupling of chloroquinolines with thiols to form thioethers [2]. Palladium catalyzed the intramolecular coupling of 2-bromophenylthioureas and 2-bromophenylthioamides to give 2-substituted benzothiazoles (Eq. (108)) [763]. Cystein derivatives were coupled with aryl-, alkenyl-, and alkynyl-iodides [764]. A palladium-catalyzed alkylation of a sulfide with an alkenyl iodide was used in a synthesis toward griseoviridin [765]. Palladium catalyzed the coupling of aromatic bromides, iodides, and triflates with potassium triisopropylsilanethiolate (Eq. (109)) [766]. Copper catalyzed microwave-accelerated couplings of aromatic bromides and iodides with thiols [767]. Copper catalyzed the coupling of an alkenyl bromide with an aromatic thiol [756], of sugar thiols with aromatic iodides [768], and sodium 4-methylphenylsulfinate and an aromatic iodide [769].



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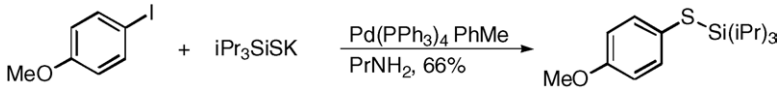


(109)

Nickel and palladium catalyzed the coupling of ester derived enol triflates and phosphates with thiophenoxide [770]. Palladium catalyzed the coupling of aromatic sulfinates with aromatic bromides and triflates to give diarylsulfones [771]. Palladium catalyzed the formation of propargylic thioethers from reaction of propargylic bromides and thiols [772]. Palladium-catalyzed S_N1-type thioether-formation from reaction of secondary benzylic alcohols with thiols [689].

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

Palladium-catalyzed reactions of hexaalkylditins with alkenyl and aryl triflates or iodides to give alkenyl and aryltin reagents were employed in the synthesis of the carbocyclic core of cornexistins [55], fasicularin [773], haterumalide NA methyl ester [60], epothilone analogs [63], azaspiracid-1 [65], FR182877 [90], briarellins E and F [774], and chloropeptin I [103]. A triflate formed from kojic acid was stannylated using the same type protocol [775].

Copper catalyzed the formation of triarylphosphines from diarylphosphines and aromatic iodides [776]. Copper catalyzed the coupling of secondary phosphines and phosphates with aromatic and alkenyl halides [777]. Nickel catalyzed the formation of triaryl- or diarylalkylphosphines from aryltriflates and diaryl- or arylalkylphosphines [778,779]. Palladium catalyzed the coupling of aryl triflates with diarylphosphines to give triarylphosphines [780]. Palladium catalyzed the formation bis-1,1-(phosphono)alkenes using dialkylphosphites and 1-phosphono-1-nonaflates [781]. Palladium catalyzed the coupling of ethyl benzyloxymethylphosphinate with aromatic halides to form ethyl arylbenzyloxymethylphosphinates [782]. An alkenyl diethylphosphonate was prepared from an alkenyl bromide using a palladium catalyst and diethylphosphite [783]. Palladium catalyzed the coupling of 2-oxo-1,3,2-dioxaphospholanes and 2-oxo-1.3.2-dioxaphosphorinanes with bromoethene [784]. Nickel, copper, and palladium catalyzed the formation of phosphinoalkynes coupling of terminal alkynes and chlorophosphanes [785,786].

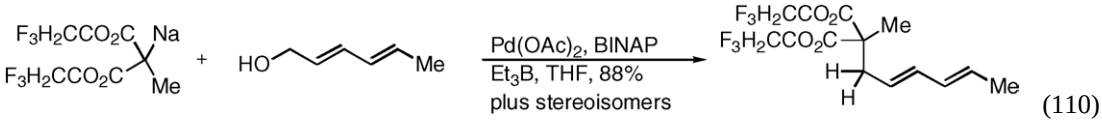
Palladium catalyzed the coupling of aryl iodides with tributyltin arsenic and antimony compounds to give triaryl arsene and stibines [787]. Copper catalyzed the formation of diarylselenides from aromatic halides and tributyltin aryl selenides [788]. Reaction of aromatic bromides and iodides with 1,2-diethoxy-1,1,2,2-tetramethyldisilane in the presence of a palladium catalyst gave aryldimethylmethoxysilanes [789].

Palladium catalyzed the cross-coupling of aryl or alkenyl halides and triflates, allylic acetates, diazonium salts, and benzyl halides with dipinacolyldiborane or pinacolborane producing aromatic pinacolyl boronic esters [790–799] and this reaction was used in Suzuki couplings toward axially chiral 7,5-fused bicyclic lactams [271], spirotoxin C [290], cross-linked peptide dimers [800], norbadione [293], diazonamide A [294], TMC-95A [295], RP-66453 [297], iodotyrosine derivatives [801], β-substituted dehydroamino acids [802], dicyclobutylidene derivative [803], terbenzimisdazole [804], (*R*)-rolipram [805], cyclooctadiene lignans [806], melatonine analogs [807], and crisamicin A analogs [808]. Reaction of 2-bromopyridines with dipinacolylborane gave homo-coupled 2,2′-bipyridines [809].

3. Reactions of allyl, propargyl, and allenyl halides, sulfonates, alcohols 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. Chloride ions were shown to have structural and kinetic effects on palladium-catalyzed allylic substitutions [810]. Regioselective allylic alkylations of silicon substituted substrates were described [811]. A silicon substituent on the central carbon of the η^3-allyl complex was shown to stabilize the complex [812]. Allylic alkylation using allylic carbonates was found to be significantly slowed down in ionic liquids due to trapping of the formed base [813]. A palladium catalyst for the direct alkylation of allylic alcohols with stabilized carbon nucleophiles in water was developed [814]. Palladium catalyzed a regio- and stereoselective alkylation of bis(trifluoroethyl) malonates with dienyl alcohols promoted by triethyl borane (Eq. (110)) [815]. Related reactions of a number of stabilized nucleophiles with allylic alcohols in the presence of triethyl borane were described [816].



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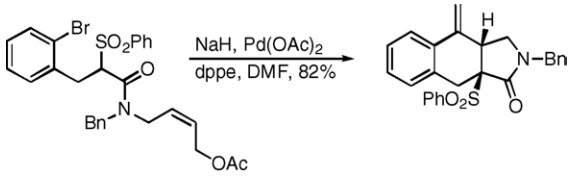
Palladium catalyzed an allylic alkylation-Heck reaction sequence to afford polycyclic compounds (Eq. (111)) [817]. A Michael addition initiated intramolecular allylic addition was developed (Eq. (112)) [818]. Palladium-catalyzed Michael addition initiated, intramolecular carbopalladation followed by trapping with allyl chloride sequence to give tetrahydrofuran derivatives (Eq. (113)) [819]. A tandem palladium-catalyzed intermolecular allylic alkylation intramolecular Michael addition was employed to prepare methylenecyclohexanes [820]. Allenes acts as nucleophiles in intramolecular allylic alkylations [821]. Pendant heteroatoms can assist in intramolecular alkylations (Eq. (114)) [822]. A palladium-catalyzed deconjugative allylation of alkylidenemalonates was described [823].



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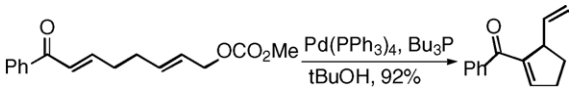

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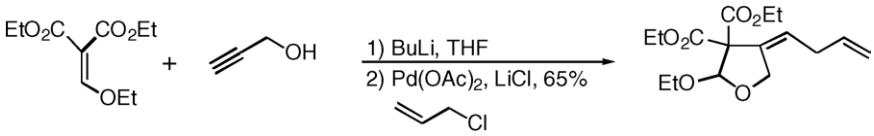

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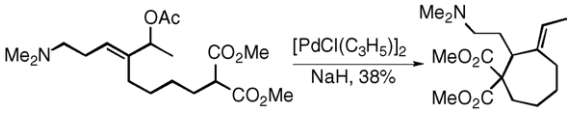
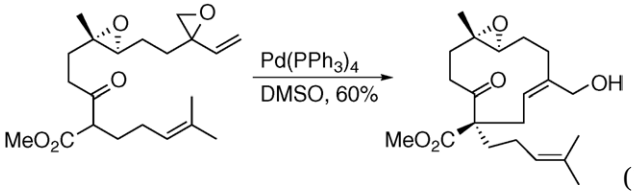


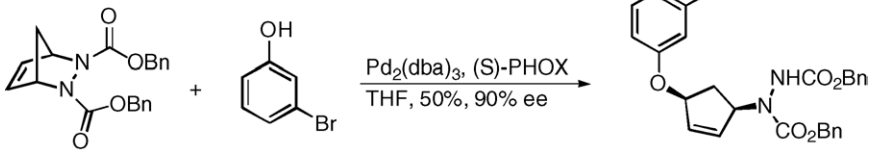
(114)

Intermolecular palladium-catalyzed allylic alkylations were used to prepare the DEF-ring core of hexacyclinic acid [824], brevione B [825], indanofan [826], and BCX-1812 [827]. A sequential inter- and intramolecular allylic alkylation was used in a synthesis of huperzine A analogs [828]. Palladium-catalyzed intramolecular allylic alkylation were employed in the synthetic applications toward vibsanin F (Eq. (115)) [829], silyl-substituted 4-ethenyl-2-pyrrolidinones [830], ambruticin [831], and (+)-macquarimicin A [95].



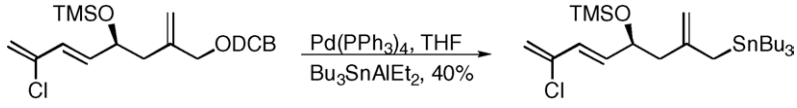
(115)

Palladium-catalyzed asymmetric allylic alkylations were used in the synthesis of viridenomycin [832], TMC-69-6H [833], 3-aminoaspartic acid derivatives [834], and (−)-chaulmoogeric acid [835]. A palladium-catalyzed asymmetric allylation of unsymmetrical 1,3-diketones was described [836]. Palladium catalyzed a diastereoselective ring-opening of meso bicyclic hydrazines using stabilized carbanions (Eq. (116)) [837]. An enantioselective α-allylation of a ketone enolate was used in a synthesis of benzomorphans [838]. A microwave-accelerated molybdenum-catalyzed asymmetric allylic alkylation using a polymer-bound ligand was used in a synthesis of baclofen [839]. Palladium catalyzed an enantioselective alkylation of (3-bromopenta-2,4-dienyl)trimethylsilane to afford axially chiral (allenylmethyl)silanes [840].



(116)

A palladium-catalyzed reaction of tributylstannyl diethylaluminum and an allylic ester was used in a synthesis of (+)-spongistatin 1 (Eq. (117)) [841]. Palladium-catalyzed intermolecular allylation of an allylic carbonate with an aromatic tin reagent was employed in a synthesis of (+)-ar-tumerone [842]. Palladium catalyzed the coupling of allylic acetates with aryl and alkenyl trifluoroborates [843]. A palladium-catalyzed coupling of an allylic acetate with an alkenylaluminum reagent, derived from a terminal alkyne and trimethylaluminum in the presence of a zirconium catalyst, was used in a synthesis of geraniol [844].



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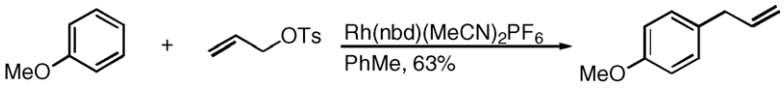
β-Substituted amino acids were prepared via an iridium catalyzed asymmetric allylic substitution using diphenylimino glycinate and allylic phosphates or benzoates [845,846]. Addition of lithium chloride was shown to improve the regio- and enantioselectivity in iridium catalyzed asymmetric allylic alkylations of allylic acetates [847]. Iridium and rhodium catalyzed the allylation of electron-rich aromatic compound using allyl tosylate (Eq. (118)) [848]. A nickel-catalyzed allylic alkylation of an allylic trimethylsilyl ether with methylmagnesium bromide was used in a synthesis of hirsutene [849].



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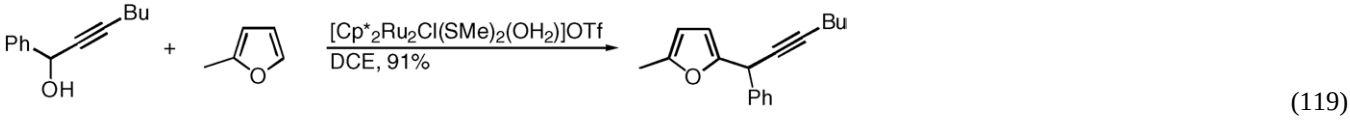
Ruthenium catalyzed regioselective allylic alkylation of allylic carbonates to give the more substituted product [850–852]. Ruthenium catalyzed the direct propargylation of furanes, pyrrole, 2-methylthiophene, electron-rich benzenes, and azulene with propargylic alcohols (Eq. (119)) [853]. Rhodium catalyzed arylations of fluorinated acyclic allylic carbonates with inversion of stereochemistry (Eq. (120)) [854]. Rhodium catalyzed regio- and enantioselective allylic alkylations of enolates [855]. This reaction was used in a synthesis of (−)-sugiresinol dimethyl ether [856]. Rhodium catalyzed the direct alkylation of allylic alcohols using aryl or alkenyl boronic acids [857]. Palladium and rhodium catalyzed nucleophilic ring-opening reactions of oxabicyclic alkenes [858,859].



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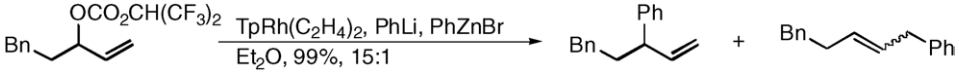
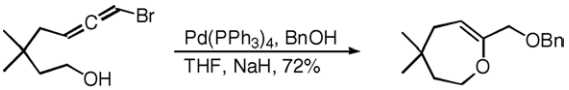


(120)

phenyl ether substituted cyclic carbonates (Eq. (123)) [865]. Palladium-catalyzed glycosylation reactions of 6-*t*-butoxycarboxy- (Eq. (124)) [866] and 6-acetoxy-2*H*-pyran-3(6*H*)-one [867].



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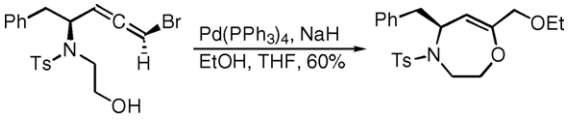

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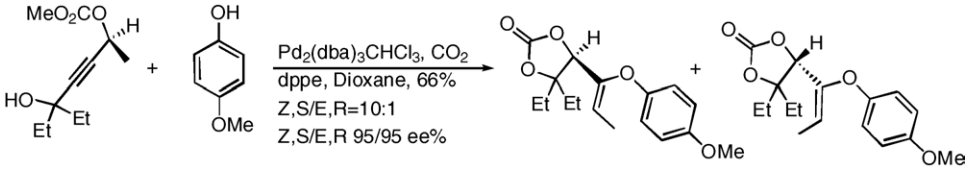

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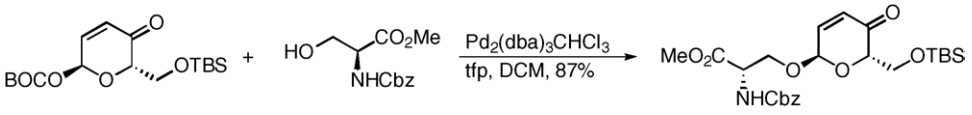

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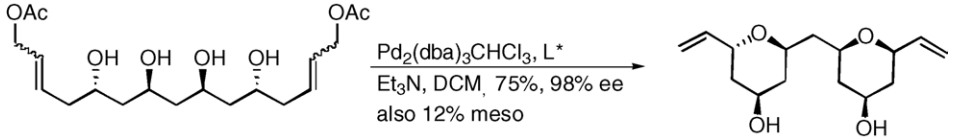
3.2. Carbon–O, –N, and –S bond-forming reactions using O-, N-, and S-nucleophiles

A number of palladium catalysts for the allylation of alcohols and phenols were reported [860,861]. Palladium and iridium catalyzed *O*-allylations of hydroxylamines having an electron-withdrawing substituent on the nitrogen [862]. Palladium catalyzed a diastereoselective ring-opening of meso bicyclic hydrazines using phenols or acetate ion [837]. A palladium-catalyzed acetoxylation of an alkenyl epoxide was used in a synthesis of guanacasterpene A [123]. Palladium catalyzed the formation of oxygen-containing rings from 7- and 8-hydroxy-1-bromo-1,2-dienes (Eq. (121) and (122)) [863,864]. Palladium catalyzed the reaction of optically active propargylic carbonates with phenols affording

Palladium-catalyzed asymmetric allylic alkylations of alkoxides or alcohols were used in the synthesis of a segment of phorboxoles A and B (Eq. (125)) [868], ustiloxin D (Eq. (126)) [869], and (+)-clusigoliol [870]. An asymmetric intermolecular followed by an intramolecular alkoxylation of propargylic carbonates using benzene-1,2-diols was used to prepare 2,3-dihydrobenzo-1,4-dioxin derivatives [871]. An intramolecular palladium-catalyzed allylation was used in the synthesis of (−)-siccanin [872]. Intermolecular allylation of phenols were used in a synthesis of (−)-heliannuol A [873,874] and (−)-heliannuol E [433], (+)-aflatoxin B_1 and B_{2a} [875], and furaquinoncins [601]. Palladium catalyzed dynamic kinetic resolution of racemic epoxides with unsaturated alcohols [876]. Oxidative addition of palladium to an allylic bromide followed by intermolecular alkoxylation using 2-iodophenol, intramolecular alkene insertion, and cross-coupling with an aromatic boronic acid furnished 3,3-disubstituted-2,3-dihydrobenzofuranes (Eq. (127)) [877].



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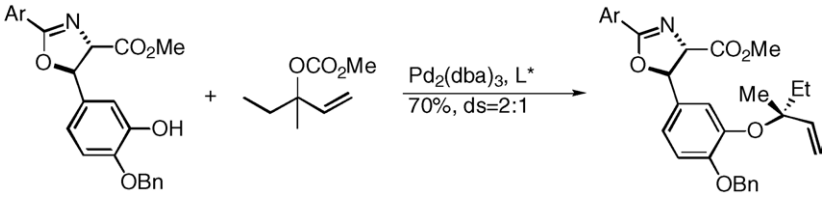

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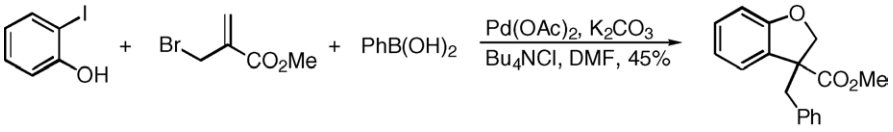

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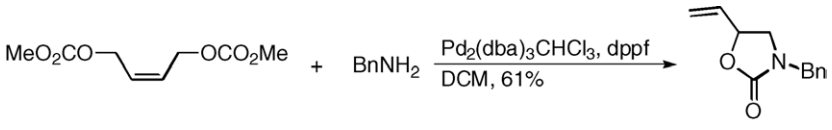

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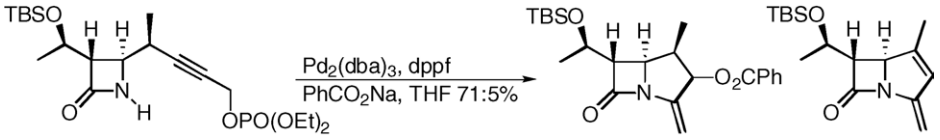

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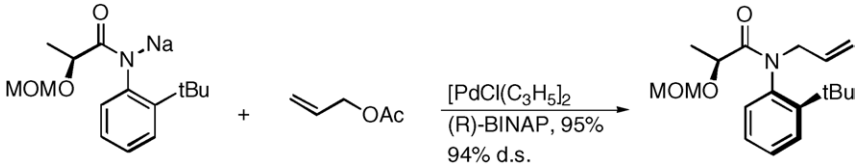
A regio- and enantioselective rhodium-catalyzed allylic alkoxylation using copper alkoxides, affording the more substituted ether, was described [855]. Iridium catalyzed allylic alkoxylations [878] and regio- and enantioselective iridium-catalyzed alkoxylation of allylic carbonates was reported [879].

Palladium catalyzed a variety of alkylations of amines using allylic substrates. Pyrimidines and purines were *N*-allylated [880]. Palladium-triethylboron [881] or -titanium tetraalkoxide [882] catalyst systems were used for the direct alkylation using allylic alcohols. Palladium catalyzed a diastereoselective ring-opening of meso bicyclic hydrazines using phthalimide [837].

Palladium-catalyzed allylic amination was used to prepare 3,5-disubstituted piperazinones [883], 1,2,3,4-tetrahydro-β-carbolines [884], and 3-*N*-alkyl-5-alkenyloxazolidinones (Eq. (128)) [885]. Palladium catalyzed the formation of carbapenams via intramolecular amination of propargylic substrates (Eq. (129)) [886]. Intramolecular amination using polymer-bound allylic esters to give 4-methylene pyrrolidines was reported [887].

The allyl palladium complex formed from 1-alkenyl-1-tosyloxycyclopropane was aminated [888].

A palladium-catalyzed diastereoselective amination leading to atropisomeric anilide derivatives was described (Eq. (130)) [889]. Axially chiral *N*-allyl anilides were prepared by asymmetric *N*-allylation [890]. Palladium catalyzed an enantioselective amination of (3-bromopenta-2,4-dienyl)trimethylsilane to afford axially chiral (allenylmethyl)silanes [840]. Regioselective aminations of 5-alkenyloxazolidinones with phthalimide affording the more substituted product was shown to be the result of a hydrogen-bond directed addition [891].



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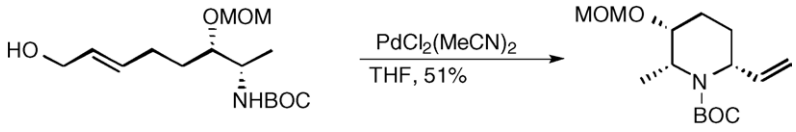
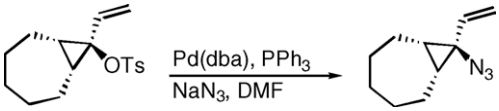
Palladium-catalyzed regio- and enantioselective allylic aminations were used in synthesis of (+)-broussonetine G [892], catecol *O*-methyltransferase inhibitors [893], ^{13}C-labeled aristeromycylcobalamin [894], and indole alkaloids [587]. Intramolecular enantioselective allylic aminations of allylic alcohol were used to prepare (−)-cassine (Eq. (131)) [895], and C1-substituted tetrahydroisoquinolines [896]. Palladium catalyzed the amidation of methylenecyclopropanes [897].



(131)

A palladium-catalyzed azidation of an allylic tosylate was used in a synthesis of an unnatural amino acid (Eq. (132)) [898]. An iridium catalyst for allylic aminations was described [878]. Iridium catalyzed a sequential intermolecular–intramolecular amination of substrates having two allylic carbonates (Eq. (133)) [899]. A palladium-catalyzed dynamic kinetic resolution of allylic carbonates using lithium sulfinate nucleophiles was developed (Eq. (134)) [900].



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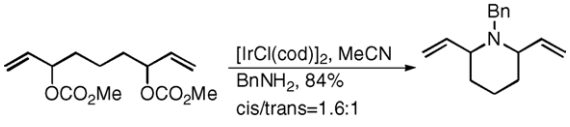

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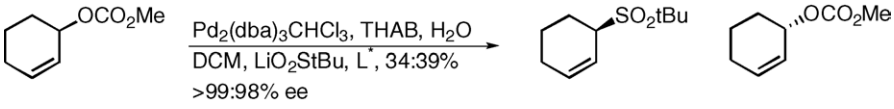

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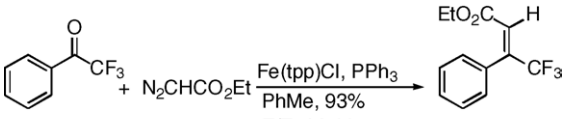
4. Metal-catalyzed diazo decompositions (including other cyclopropanations)

Iron catalyzed the alkenylation of trifluoromethyl ketones using ethyl diazoacetate (Eq. (135)) [901]. An iron porphyrin complex catalyzed the alkenylation of ketones with ethyl diazoacetate [902]. Copper catalyzed the formation of 1,1-dibromo-1-alkenes by reaction of hydrazones with aldehydes and ketones in the presence of carbon tetrabromide (Eq. (136)) [903]. Rhodium catalyzed the decomposition of cyclic 2-diazo-1,3-dicarbonyl compounds in the presence of an acid halide to give β-acyloxy α-haloenones [904]. A protocol for in situ generation of diazo compounds and subsequent rhodium-catalyzed decomposition and further reactions was developed [905].



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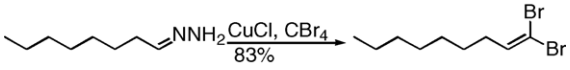

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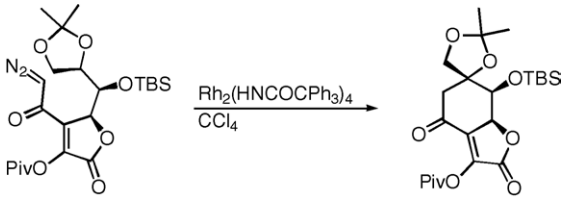
Rhodium catalyzed the formation of enamines via decomposition of α-diazo-β-amido esters [906]. Rhodium catalyzed the formation of isomünchone 1,3-dipoles from α-diazoimides followed by subsequent trapping with alcohols [907]. Related formation of thioisomünchnones followed by cycloaddition was used to prepare analogs of dehydrogliotoxin [908].

A polymer-supported rhodium catalyst was used in carbon–hydrogen-bond insertion and cyclopropanation reactions employing diazo compounds [909]. Reactions in an ionic liquid were reported [910]. Intramolecular rhodium-catalyzed carbon–hydrogen-bond insertions via decomposition of diazo compounds were used in a number of synthetic applications forming 4–6-membered rings [911–915]. For example, griseolic acid analogs [916], methyl-L-callipeltoside [917], rolipram [918], L-vancosamine [919], gabapentin [920], (−)-ephedradine [566], and (−)-tetrodotoxin (Eq. (137)) [921] were prepared. Intramolecular diastereoselective (Eq. (138)) [922] or enantioselective [923–925] insertions were described. A substrate-directed asymmetric intramolecular carbon–hydrogen-bond insertion was used in a synthesis of 7-episordidin [926]. Intramolecular insertions forming lactams and lactones were described [927].



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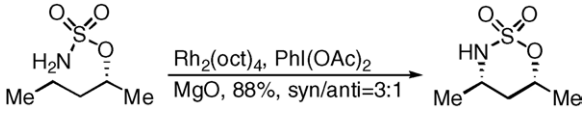

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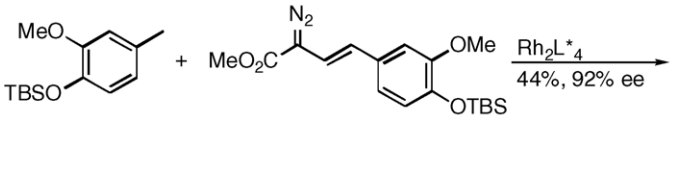
A polymer-bound rhodium catalyst was used in asymmetric intermolecular carbon–hydrogen insertion reactions [928]. Rhodium catalyzed a diastereoselective carbon–hydrogen-bond insertion of allyl *t*-butyldimethylsilyl ethers [929]. Rhodium catalyzed the carbon–hydrogen-bond insertion by decomposition of an iodonium ylide in the presence of pyrroles [930].

Intermolecular indium- and copper-catalyzed carbon–hydrogen-bond insertions of α-diazocarbonyl compounds with pyrroles and indoles were reported [931]. (+)-Imperanene and (−)-α-condendrine were prepared using an asymmetric intermolecular carbon–hydrogen insertion as the key step (Eq. (139)) [932]. Rhodium catalyzed a carbon–hydrogen-bond insertion of β-disulfone iodonium ylides into flavones [933].

The carbenoids derived from rhodium-catalyzed decomposition of diazo compounds insert into nitrogen–hydrogen-

bonds. Intramolecular nitrogen–hydrogen-bond insertions to give substituted substituted prolines [934] and intermolecular insertions to afford imidazolones [935], and β-fluoropyrroles [936] were reported. Asymmetric rhodium-catalyzed silicon–hydrogen-bond insertions were described [937].



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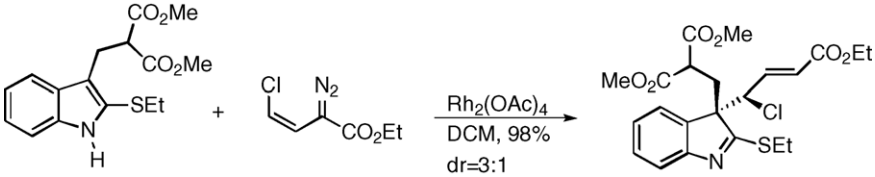
Rhodium catalyzed the 1,2-aryl migration of β-(*N*-tosyl)amino diazo carbonyl compounds [938]. Rhodium catalyzed a sulfur ylide-initiated thio-Claisen rearrangement to give indolines (Eq. (140)) [939]. Copper catalyzed a [2,3]sigmatropic rearrangement of *N*-methyltetrahydropyridinium ylides (Eq. (141)) [940,941]. Rhodium-catalyzed asymmetric formation of sulfonium ylides followed by [2,3]sigmatropic rearrangements [942–944]. Silver catalyzed a tandem Wolff–Cope rearrangement to give fused carbocyclic compounds (Eq. (142)) [945].



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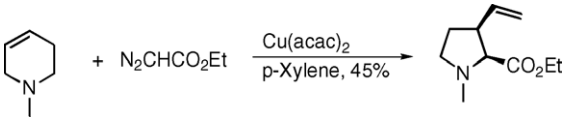

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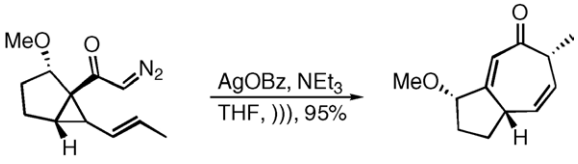

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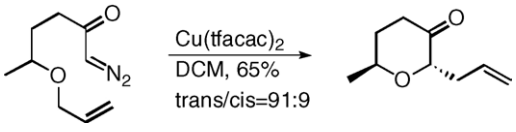
A stereoselective formation of tetrahydropyranes via a rearrangement of an oxonium ylide formed using a rhodium or copper catalyst was described (Eq. (143)) [946]. An enantioselective, copper-catalyzed, ammonium ylide-[1,2]rearrangement to give bicyclic morpholinones was described [947]. A stereospecific [1,2]rearrangement of ammonium ylides generated from metal carbenoids to give enantiomerically pure bicyclic 1,4-oxazepinones was reported [948]. Copper catalyzed an oxonium ylide formation [1,2]shift (Eq. (144)) [949].



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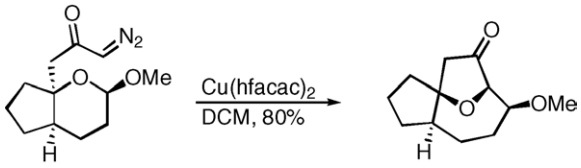
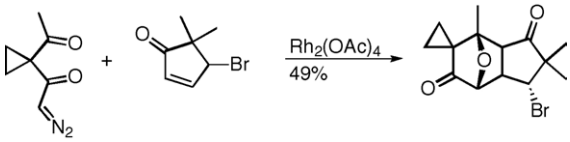


(144)

A variety of cycloaddition reactions of carbonyl ylides formed by rhodium- or copper-catalyzed decomposition of α-diazocarbonyl compounds were described [950]. Rhodium-catalyzed asymmetric intermolecular carbonyl-ylide alkyne and alkene [3+2]cycloadditions were described [951,952]. Copper catalyzed the formation of carbonyl ylides from α-diazocarbonyl compounds followed by cyclization with 2-alken-1-ones to give dihydrofuran derivatives [953]. Enantioselective intramolecular [3+2]cycloadditions via rhodium-catalyzed diazo decomposition-carbonyl ylide formation were described [954–956]. Rhodium-catalyzed intermolecular carbonyl ylide-ketone [3+2]cycloadditions were described [957].

Intermolecular carbonyl ylide-alkene [3+2]cycloadditions were used to prepare illudinoids (Eq. (145)) [958], 3,4-fused bicyclic-2-bromofuran [959], pyridones [578], and mappacine ketone [56]. Formation of a carbonyl ylide followed by intramolecular [3+2]cycloaddition with a pendant alkene was used in an approach to pseudolaric acid [960]. A related rhodium-catalyzed reaction was used to form furo[3,4-*c*]pyridines [961]. Rhodium catalyzed the formation of furo[3,4-*c*]furanes via an intramolecular cycloaddition to a pendant alkyne (Eq. (146)) [962].



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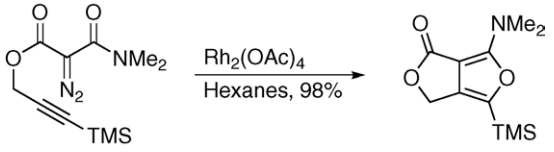
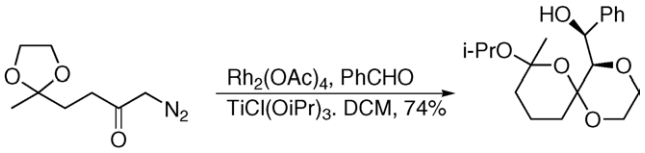


(146)

Oxonium ylides derived from decomposition of diazo compounds using a rhodium catalyst react with aldehydes to give cyclic products (Eq. (147)) [963]. Related reactions of azomethine ylides with alkenes or alkynes afford pyrrolidines [964]. An intermolecular azomethine ylide formation–cycloaddition was used to prepare functionalized pyrrolidines (Eq. (148)) [965]. Depending on the catalyst, either 2,3- or 2,5-dihydropyrroles can be obtained from alkenyldiazoacetates and imines (Eqs. (149) and (150)) [966].



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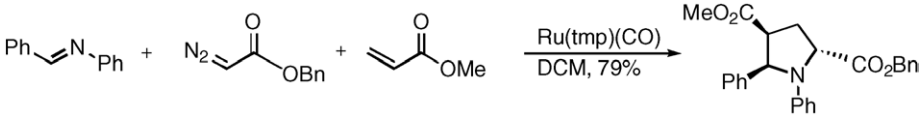

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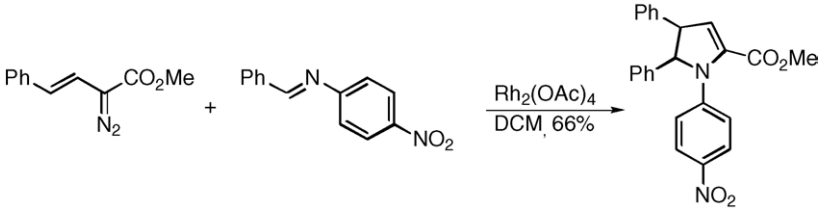

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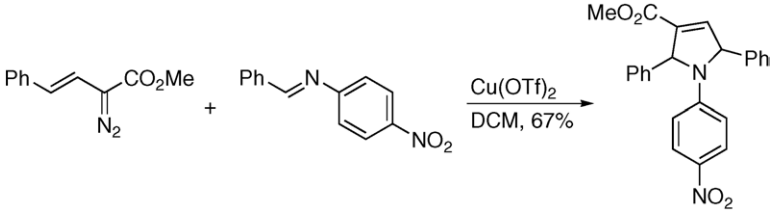
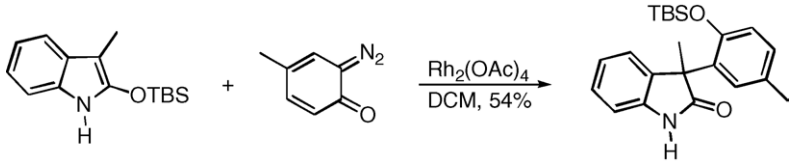


(150)

Cyclopropanations of alkenes via transition metal-catalyzed decomposition of diazo compounds continued to be developed. Rhodium catalyzed a distereotopic intramolecular cyclopropanation of bisallylic α-diazophosphonates [967]. Copper-catalyzed intramolecular alkene cyclopropanation of chiral α-diazocarbonyl compounds [968]. Iron catalyzed the cyclopropanation of dehydroamino acids using tosylhydroazone salts as the diazo precursor [969]. Chiral rhodium, cobalt, or copper complexes catalyzed asymmetric intramolecular cyclopropanations using α-diazo carbonyl compounds [970–973]. Rhodium-catalyzed intermolecular cyclopropanation was used in a synthesis toward CP-225,917 and CP-263,114 [974], sabina ketone [975], dispirocyclic cyclopropane systems [976], and rhazinal [315].

Ethyl 2-nitro-2-diazoethanoate was used in cyclopropanations of alkenes in the presence of a rhodium catalyst [977]. Rhodium catalyzed a cyclopropanation rearrangement sequence employed in an application toward diazonamide A (Eq. (151)) [978]. A rhodium-catalyzed intermolecular cyclopropanation with high enantiocontrol was used to prepare bicyclic ring systems [979]. Cyclopropanated spirocyclic ring systems were obtained from decomposition of diazoalkanes in the presence of 1,6-enynes (Eq. (152)) [980]. Rhodium-catalyzed intramolecular cyclopropanation followed by ring-expansions (Eq. (153)) [981] and this type of reaction was utilized in an approach to toward guanacastepenes (Eq. (154)) [577], and (+)-10-hydroxy-10,11-epoxythapsan-5-yl seneicoate [982]. Iodonium ylides were used as an alternative in rhodium-catalyzed cyclopropanations [983,984].



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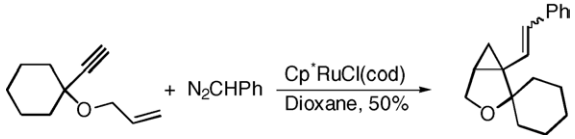

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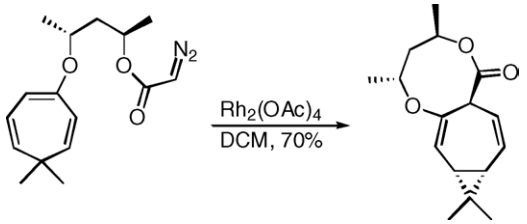

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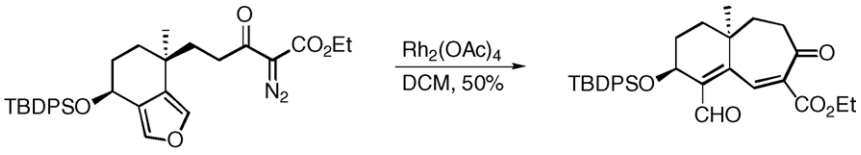

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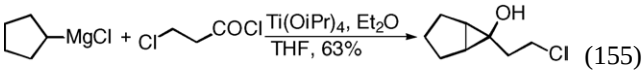
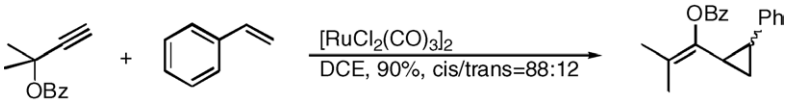
Copper-catalyzed enantioselective intermolecular [985] and intramolecular [986] cyclopropanations using α-diazocarbonyl compounds. Copper-catalyzed intermolecular cyclopropanation of furanes was used in the synthesis of the core of xanthonolides, guaianolides, and eudesmanolides [987]. A copper-mediated intramolecular cyclopropanation was used in a synthesis of cucumin H [988]. Palladium-catalyzed cyclopropanation of alkenes using diazomethane [989–991].

Titanium mediated an intramolecular aminocyclopropane formation using alkene-tethered carboxamides and a Grignard reagent [992]. A titanium-mediated formation of fused chloroethyl-substituted cyclopropanols was described (Eq. (155)) [898]. The mechanism of the formation of cyclopropanols from titanacyclopropanes and carboxylic acid esters (Kulinkovich reaction) was examined. The intramolecular variation using *N*-(3-alkene-1-yl)amides proceeded with almost complete diastereoselctivity to form 1-azabicyclo[3.1.0]hexanes [993]. Related reactions using alkene tethered to an ester via an ether linkage were also described affording oxabicyclo[4-6.1.0]alkanols [994]. Addition of ethene was shown to significantly increase the yield of product [995].



(155)

Ruthenium and a number of other transition metals catalyzed the cyclopropanation of styrenes using propagylic esters (Eq. (156)) [996,997]. 1-Arylcyclopropyl amines were prepared from aromatic nitriles and diethylzinc in the presence of titanium (Eq. (157)) [998]. Zirconium mediated the transformation of homoallylic ethers to cyclopropane derivatives (Eq. (158)) [999]. An interesting addition of alkenylzirconocenes to aldimines in the presence of dimethylzinc affording allylic amines or cyclopropanated products was described (Eqs. (159) and (160)) [1000]. A stereoselective iodocyclopropanation of terminal alkenes using iodoform, chromium dichloride, and 1,2-bis(*N*,*N*-dimethylamino)ethane was reported (Eq. (161)) [1001].



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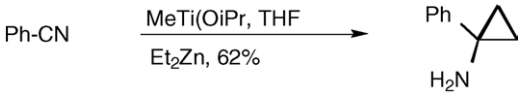

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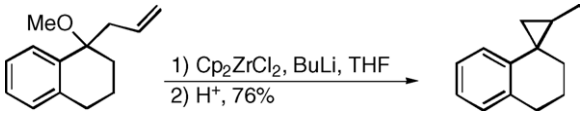

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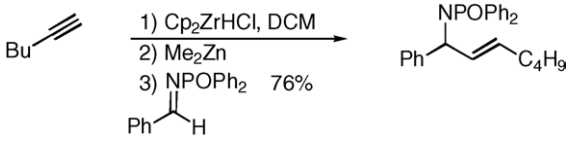

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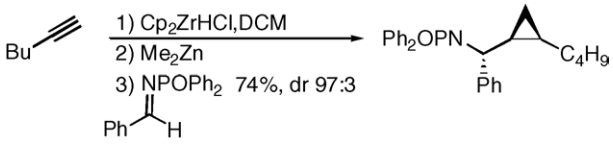

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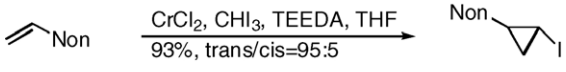
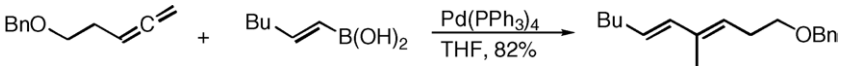


(161)

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

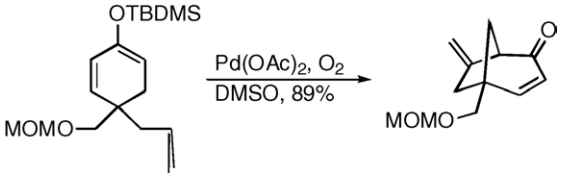
5.1. Addition of C-nucleophiles to alkene and alkynes

Palladium catalyzed an oxidative coupling of organoboron reagents with alkenes using oxygen to reoxidize the catalyst [1002,1003]. Palladium also catalyzed the oxidative coupling of triarylantimony reagents with terminal alkenes [1004] and indium catalyzed related reactions using organosilicon reagents [1005]. Palladium-catalyzed regioselective additions of organoboronic acids to allenes (Eq. (162)) [1006,1007]. Rhodium-catalyzed related reactions of arylboranes [1008]. Palladium catalyzed the intramolecular hydroalkylation of alkynes using organoboronic acids forming trisubstituted alkenes [1009].



(162)

Other carbon nucleophiles were used to alkylate alkenes. A palladium-catalyzed cyclization of 5-allylsubstituted 2-silyloxy-1,3-cyclohexadienes was used in a synthesis of aphidicolin (Eq. (163)) [1010]. Palladium catalyzed an intramolecular oxidative alkylation of pendant alkenes (Eq. (164)) [1011]. Palladium-catalyzed intramolecular oxidative hydroarylations of activated alkenes (Eq. (165)) [1012]. Palladium also catalyzed intramolecular oxidative carbocyclization of 1,2,6-trienes (Eq. (166)) [1013,1014] and alkene-tethered indoles (Eq. (167)) [1015,1016].



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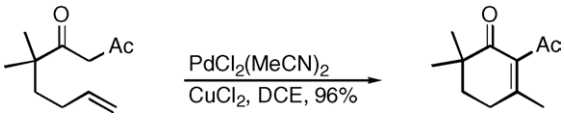

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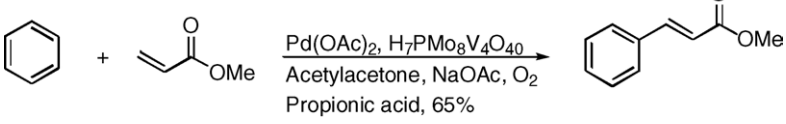

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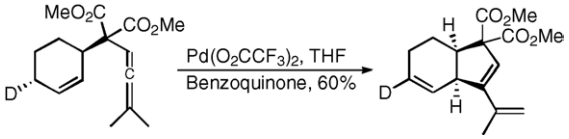

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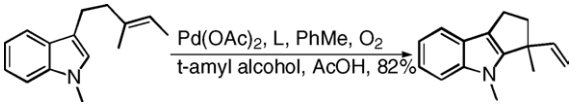
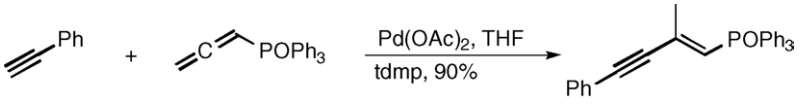


(167)

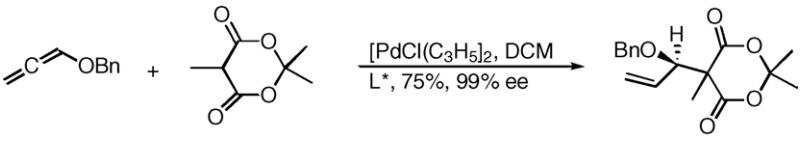
Reaction of a terminal alkyne with trimethylaluminum and a catalytic amount of zirconocene dichloride followed by quenching with oxirane was used in a synthesis of (+)-manoalide [660]. Copper mediated the addition of Grignard reagents to propargyl alcohol to form 2-substituted-3-hydroxy-1-alkenes [1017].

Reductive carbon–carbon bond formations of alkynes were described. Rhodium catalyzed a regioselective hydroarylation of *N*-heteroaromatic alkynes in water using arylboronic acids [1018]. Rhodium catalyzed a hydroarylation of alkynes using aromatic ketimines [1019]. Palladium catalyzed the coupling of terminal alkynes with allenylphosphine oxides (Eq. (168)) [1020].



(168)

Palladium-catalyzed hydrocarbonations of 3,3-dicyclohexylcyclopropene using pronucleophiles [1021]. Palladium-catalyzed asymmetric hydroalkylation of allenes (Eq. (169)) [1022]. A palladium-catalyzed intramolecular hydroalkylation of 4- or 5-en-1-ones was reported (Eq. (170)) [1023]. A related palladium-catalyzed lanthanide-promoted intramolecular hydroalkylation of alkenyl tethered β-keto esters and amides to give 6–8-membered rings was developed [1024]. Rhodium-catalyzed hydroalkylation of myrcene with ethyl acetoacetate [1025].



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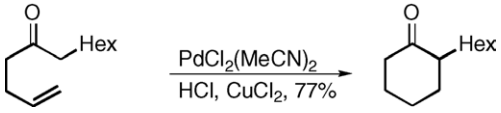
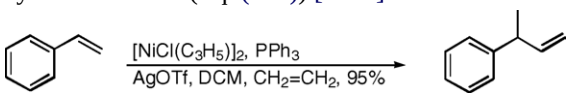


(170)

A palladium–indium catalyst system was used to hydroalkenylate alkenes [1026]. Nickel catalyzed a hydroalkenylation of styrenes using alkenes (Eq. (171)) [1027]. Ruthenium catalyzed the hydroalkenylation of 1,3-dienes using ethene (Eq. (172)) [1028]. A palladium–copper-catalyzed hydroalkynylation of allenes using terminal alkynes was developed (Eq. (173)) [1029]. Ruthenium catalyzed alkyne–alkyne coupling to give conjugated enynes [1030]. Ruthenium catalyzed alkyne–alkene intermolecular cross-metathesis to give 1,3-dienes in the presence of ethene [1031,1032]. This type of reaction was used to prepare macrocyclic dilactones (Eq. (174)) [1033].



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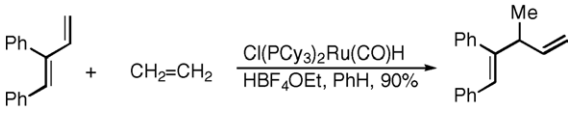
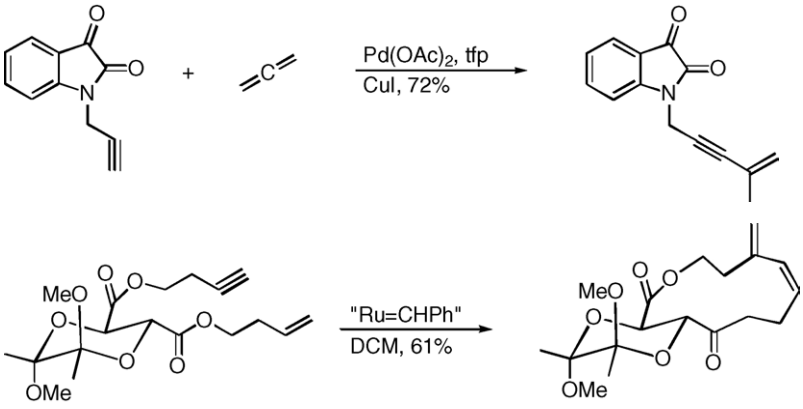


(172)

Chelation-assisted rhodium-catalyzed hydroesterifications of alkenes were reported [1042,1043]. Hydroacylation and carbon–carbon bond cleavage of alkynes was observed using Wilkinson's catalyst (Eq. (178)) [1044]. Rhodium-catalyzed chelation-assisted intermolecular hydroacylations of 2-hydroxy-substituted aromatic aldehydes with terminal alkenes [1045].



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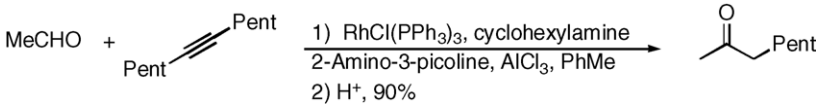
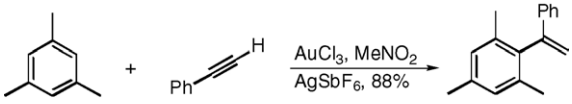
(174)

Iridium-catalyzed hydroarylation of norbornene using phenol [1034]. A gold-silver catalyst system was used in hydroarylations of arylethynes (Eq. (175)) [1035]. Palladium-catalyzed hydroarylation of alkynes [1036]. Palladium-catalyzed hydroarylation of propinoic esters with phenols was used to prepare coumarins (Eq. (176)) [1037,1038].



(178)

Ruthenium- and rhodium-catalyzed intra- and intermolecular hydroarylations of alkenes using aromatic compounds (Eq. (177)) [1039,1040]. The mechanism of the intramolecular palladium-catalyzed hydroarylation of 7-octene-2,4-dione was examined [1041].



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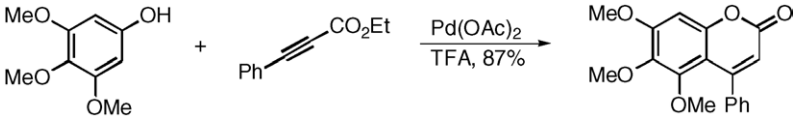

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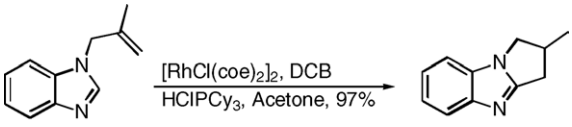
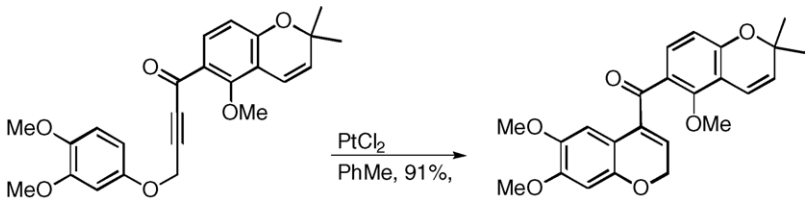


(177)

Platinum catalyzed a hydroarylation of arene–alkyne substituted substrates to form a variety of heterocycles including chromenes, dihydroquinolines, and coumarins [1046,1047]. This type of reaction was used in a synthesis of deguelin (Eq. (179)) [1048]. Rhodium catalyzed an intramolecular hydroarylation of alkenyl phenyl ethers (Eq. (180)) [1049]. Rhodium catalyzed an intramolecular hydroacylation of 4-methyl-4-pentenal to give 3-methylcyclopentanone [1050].

A kinetic resolution was observed in a related rhodium-catalyzed hydroacylations of 4-ynals (Eq. (181)) [1051]. Ruthenium catalyzed a hydroamidation of alkenes using *N*-(2-pyridylmethyl)formamide (Eq. (182)) [1052].



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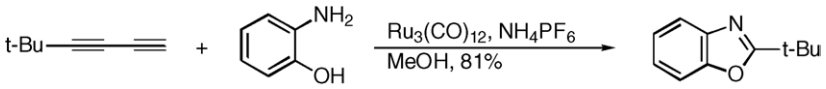
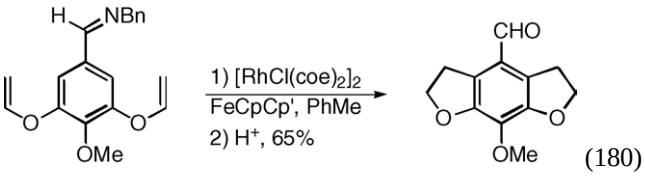


(184)

Copper and palladium catalyzed the formation of indoles from 2-alkynyl-substituted aminobenzenes [1065,1066] and this type of reaction was used to prepare a GnRH antagonist [1067] and a PDZ protein interaction domain inhibitor [1068]. Polymer-supported 2-(1-alkynyl)benzenamides under microwave irradiation were also used [1069].



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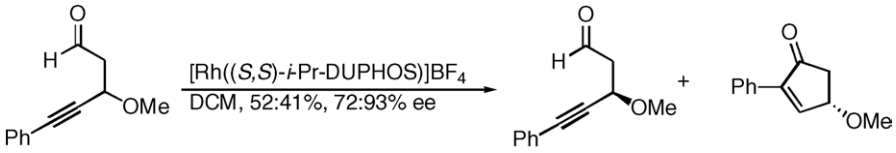

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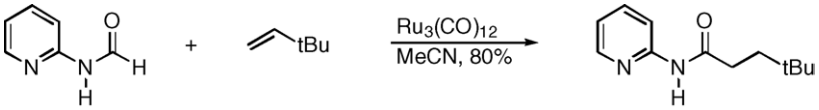
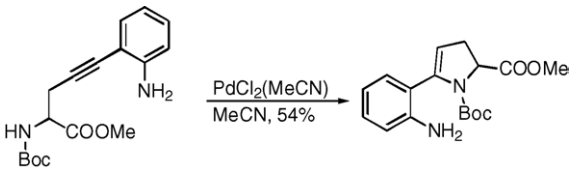


(182)

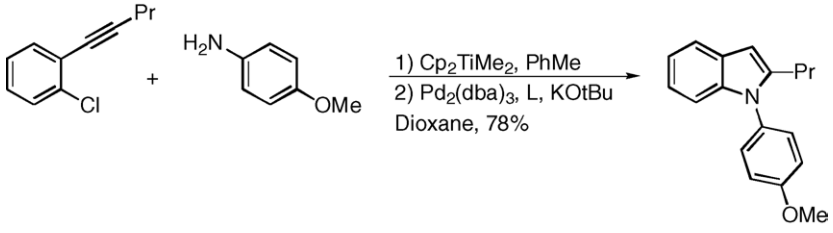
5.2. Addition of N-nucleophiles to alkenes and alkynes

Palladium catalyzed the formation of 2,5-disubstituted 2,3-dihydropyrroles from 4-amino-1-ynes via hydroamination (Eq. (183)) [1053,1054]. Carbon–carbon bond cleavage was observed upon hydroamination of terminal 1,3-diynes catalyzed by palladium or ruthenium (Eq. (184)) [1055]. Gold, titanium, and zirconium catalyzed intramolecular hydroaminations of alkynes [1056,1057]. Iridium catalyzed a tandem intramolecular hydroamination–hydrosilylation [1058]. Titanium catalyzed intermolecular hydroamination of alkynes with primary amines to give imine intermediates [735,1059]. Both imines and enamines were obtained from internal alkynes [1060]. Anti-Markovnikov hydroaminations of terminal alkynes catalyzed by titanium [1061] and rhodium [1062] were described. Titanium catalyzed hydroaminations of alkynes affording imines [1063,1064].



(183)

Copper catalyzed the coupling-cyclization of terminal alkynes with 2-iodotrifluoroacetanilide to give 2-substituted indoles [1070]. Palladium catalyzed the formation of 2-aryl- or -heteroaryl-substituted indoles from 2-alkynyltrifluoroacetanilides and aromatic halides [1071]. A combination of sequential titanium catalyzed amination and palladium-catalyzed aminopalladation was used to prepare indoles (Eq. (185)) [1072]. Palladium catalyzed an asymmetric intramolecular aminopalladation carbonylation sequence (Eq. (186)) [1073]. Palladium and copper catalyzed the reaction of (*Z*)-1-amino-2-en-4-ynes to give pyrolles [1074]. Gold catalyzed the formation of isatogens or anthranils from 2-(1-alkyn-1-yl)-1-nitrobenzenes (Eq. (187)) [1075].



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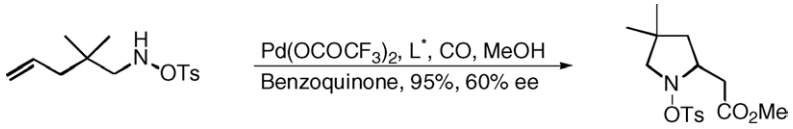

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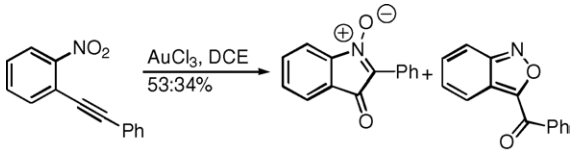
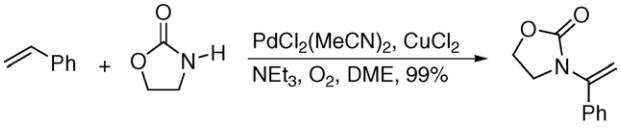


(187)

Palladium-catalyzed hydroaminations [1076] of 3,3-dicyclohexylcyclopropene [1021], and of acyclic alkenes [1077]. An oxidative palladium-catalyzed hydroamination of

terminal alkenes was developed (Eq. (188)) [1078]. Palladium catalyzed a dichloroamination of alkynes (Eq. (189)) [1079]. Iron catalyzed the amination of alkenes with aromatic nitroso-compounds to give allylic amines (Eq. (190)) [1080].



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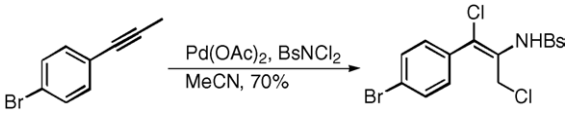

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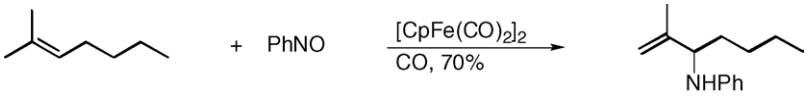
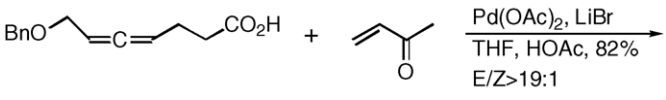


(190)

5.3. Addition of O- and S-nucleophiles to alkenes, allenes, and alkynes

Palladium catalyzed an intramolecular lactonization-reductive Heck-type sequence using allene-tethered carboxylic acids (Eq. (191)) [1081]. Intramolecular hydroxy-metallations using a variety of transition metals was described (Eq. (192)) [1082]. Oxidative intramolecular hydroxy-palladation of an alkene was used in an approach to the C-ring of Vitamin B_{12} (Eq. (193)) [1083]. Intramolecular hydroxy-palladation of an in situ formed hemiacetal was used to prepare a novel antitumor agent (Eq. (194)) [420].



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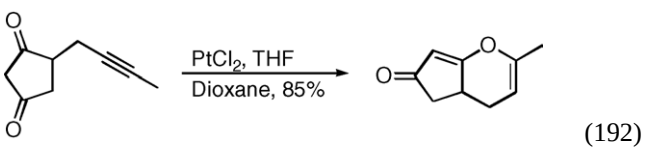

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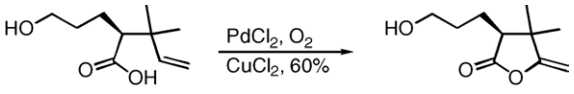

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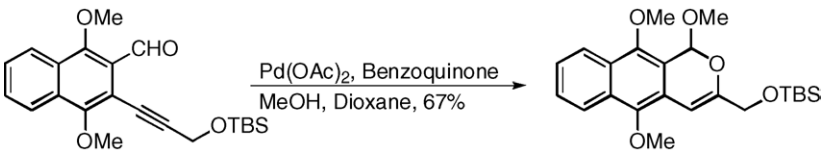
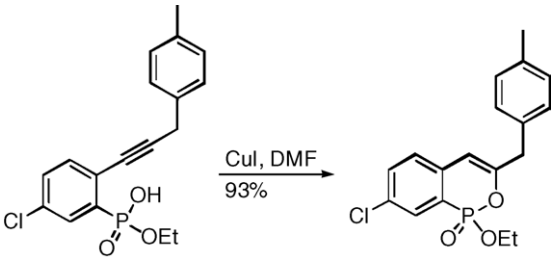


(194)

An intramolecular alkyne hydroxy-palladation was used to prepare 5-(4-aryl)-2-phenyl-5,6-dihydrobenzo[*b*][1,5]oxazocin-4-ones [495]. Copper catalyzed an intramolecular hydroxy-metallation to give phosphaisocoumarins (Eq. (195)) [1084]. Palladium catalyzed an asymmetric aerobic cyclization via intramolecular hydroxy-palladation-β-hydride elimination to give dihydrobenzofuranes [1085]. Ruthenium catalyzed intramolecular hydroxy-ruthenations of 4-pentene-1-ols to form 2,3-dihydrofuranes. Palladium catalyzed an asymmetric reaction of benzene-1,2-diols with propargylic carbonates to give 2-alkylidene-1,4-benzodioxanes [1086]. Palladium catalyzed an intramolecular alkoxy-palladation arylation sequence affording 2-(diarylmethylene)3-benzofuranones (Eq. (196)) [1087]. A photolytic tungsten-catalyzed cycloisomerization of alkynols forming dihydrofuranes and dihydropyrans was reported (Eq. (197)) [1088].



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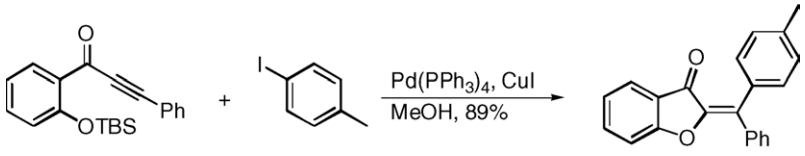

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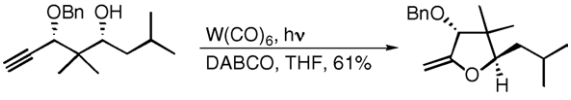
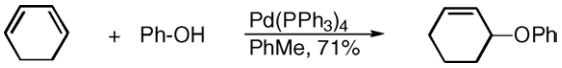


(197)

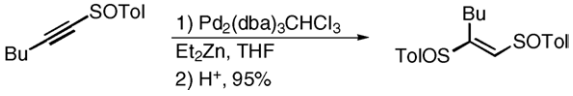
A palladium-catalyzed asymmetric 1,4-diacetoxylation of 1,3-cyclohexadienes was described [1089]. Palladium catalyzed the reaction of phenols with 1,3-dienes to give

allylic alcohols (Eq. (198)) [1090]. Palladium catalyzed the formation of an alkenyl ether from an alcohol and ethyl alkenyl ether [1091]. Ruthenium catalyzed a hydroacyloxylation of terminal alkynes forming either Markovnikov or anti-Markovnikov alkenyl esters depending on the choice of base [1092]. Iridium also catalyzed this type of reaction [1093].



(198)

Palladium-catalyzed sulfinylzincation of activated alkynes using 1-alkynyl sulfoxides (Eq. (199)) [1094]. An asymmetric palladium-catalyzed sulfinylzincation of alkynoates was also reported [1095]. The mechanism of the palladium-catalyzed addition of disulfides and diselenides to alkynes was examined leading to the development of a new catalyst system [1096].

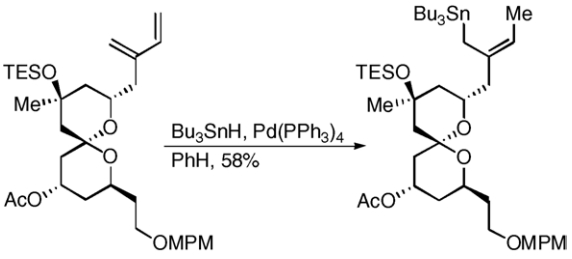


(199)

5.4. Additions of hydrogen–B, –Sn, and –Zr and miscellaneous heteroatom reagents to alkynes and alkenes

Palladium-catalyzed hydrostannation of terminal alkynes [1097], using tributyltin hydride, to give alkenylstannanes was used in total syntheses of reserpine [51], nafuredin-γ [71], leptofuranin D [243], and apoptolidin [96]. Palladium-catalyzed regioselective hydrostannations of propargylic alcohols [396,1098], an internal alkyne in a synthesis of formaminicinone [106], of hindered propargylic esters [1099], and 1-alkynylphosphonates [1100].

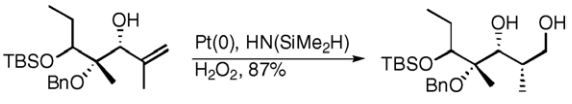
Regioselective α-hydrostannation of an ynone using trineophyltin hydride catalyzed by palladium was described [1101]. Regioselective β-hydrostannation of ynones followed by Stille coupling was reported [1102]. Palladium-catalyzed hydrostannation of bromoalkynes to give alkenylstannanes was used in a synthesis of lobatamide C [92], and (+)-macquarimicin A [95]. Palladium catalyzed a mild bisstannylation of alkynes [1103]. A platinum catalyzed intramolecular double hydrostannylation of tethered alkynes was used in a synthesis of (−)- and (+)-membrenone C [1104]. A palladium-catalyzed regioselective hydrostannation of a 1,3-diene was used in a synthetic application toward of altothyrtin (Eq. (200)) [1105]. Palladium catalyzed the 2,3-distannation of 1,4-dichloro-2-butyne using hexabutylditin [1106].



(200)

Platinum catalyzed regioselective hydrosilylations of styrenes [1107]. A related platinum-catalyzed intramolecular alkene hydrosilylation was used in a synthesis of (9*S*)-dihydroerythronolide A (Eq. (201)) [1108]. Platinum catalyzed the intramolecular hydrosilylation of pendant alkynes [334].

Rhodium, iridium, and cobalt catalyzed hydrosilylations of alkynes [1109]. Platinum catalyzed mild regio- and stereoselective hydrosilylation of terminal alkynes [1110,1111]. Titanium catalyzed a regioselective *syn*-hydrosilylation of alkynes [1112]. A rhodium-catalyzed intramolecular alkyne hydrosilylation was used in a synthesis toward dolabelides A and B [1113]. Ruthenium-catalyzed regioselective hydrosilylations of 1-yne-3-ols [1114], 1-yne-4-ols and 1-yne-5-ols (Eq. (202)) [1115], and terminal alkynes [1116] were described.



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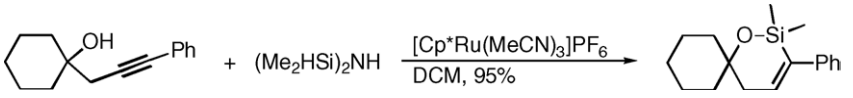
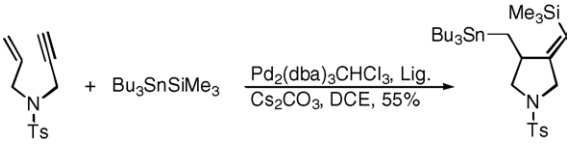


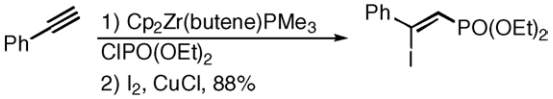
(202)

Palladium catalyzed a tandem silastannylation–cyclization of 1,6-enynes to give dimetallated 5-membered rings (Eq. (203)) [1117–1119]. Rhodium catalyzed an enantioselective cyclization–silylation of 1,6-enynes affording silylalkylidene substituted cyclopentanes [1120]. Racemic reactions were also described [1121].



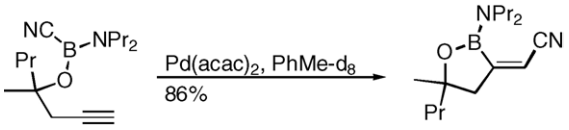
(203)

Palladium catalyzed the hydrophosphination of alkynes using phosphine-boranes [1122]. Alkene alkyl ethers were regioselectively hydrophosphinated using palladium or nickel catalysts [1123]. Ruthenium catalyzed regioselective hydrophosphinations of 3-hydroxy-1-alkynes [1124]. A variety of β-functionalized alkenylphosphonates were prepared from reaction of alkynes with zirconocenes, and chlorophosphates in the presence of phosphines (Eq. (204)) [1125].



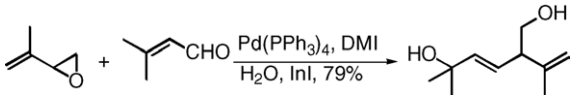
(204)

Platinum catalyzed carboselenation of alkynes using selenoesters [1126]. Palladium- and nickel-catalyzed intramolecular cyanoborations of alkynes (Eq. (205)) [1127]. Zirconium catalyzed hydroboration of conjugated en-ynes to give 1-boron-substituted-1,3-dienes [1128]. Platinum catalyzed the diboration of conjugated dienes [1129]. Enantioselective silaboration of allenes was reported [1130].



(205)

Umpolung of η^3-allyl palladium complexes by transmetallation to indium was used in a number of cases [1144]. Palladium-catalyzed diasteroselective alkylation of aldehydes by chiral propargylic mesylates in the presence of indium iodide was used in the synthesis of (−)- and (+)-membrenone C [1104]. A related reaction using an alkenyl epoxide was used in a synthesis of lavandulol (Eq. (207)) [1145]. η^3-Allylpalladium intermediates derived from *N*-acylnitroso Diels–Alder adducts and 1-amino-4-acetoxycyclopentenes were transmetallated to indium and reacted with aldehydes and ketones (Eq. (208)) [1146]. Addition of related allylic indium intermediates, catalyzed by palladium, to glyoxylic oxime ether and *N*-sulfonylimine was described [1147].



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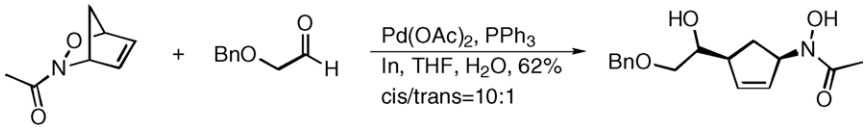
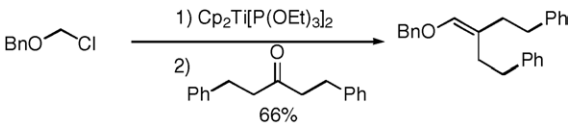


(208)

5.5. Alkylations of carbonyl compounds and imines

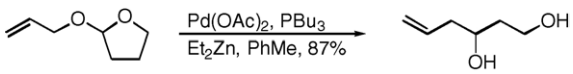
A number of carbonyl to alkene transformations was reported. Reaction of CH_2Br_2–Zn–$TiCl_4$ or CH_2Br_2–Zn–$TiCl_4$–$PbCl_2$ was used in the synthesis of indolizidines [1131], ottelione A and B [1132], and F–H gambierol subunit [1133]. Tebbe's reagent, $Cp_2TiCH_2ClAlMe_2$, was used to methylenate ketones in synthetic applications toward conformationally restricted glutamic acid analogs [1134] and pogostol and epi-pogostol [1135]. Tebbe's reagent was also used to transform esters to alkenyl ethers [1136,1137] and this reaction was employed in a synthetic approach to scleophytin A [1138]. Petasis reagent, Cp_2TiMe_2 was used to form alkenyl ethers from 2-alkylidene-β-lactones [1139] and pyranose derived lactones [230]. A related reagent, dicyclopropyl titanocene was used to introduce a cyclopropylalkylidene group [1140]. Titanium benzylidenes were used in solid-phase synthesis of 2-substituted benzofuranes and indoles [1141,1142]. Titanium mediated an alkoxymethylenation of aldehydes, ketones, and esters (Eq. (206)) [1143].



(206)

Oxidative addition followed by intramolecular insertion of a conjugated diene, transmetallation to indium from the intermediately formed η^3-allyl palladium complex and aldehyde [1148] or imine alkylations [1149] were reported. Related reactions of allenes were described [1150].

Palladium-catalyzed allylations of aldehydes and ketones in the presence of diethylzinc (Eq. (209)) [1151,1152]. The allyl palladium complex formed from 1-alkenyl-1-tosyloxycyclopropane was transmetallated to zinc and reacted with aldehydes [888]. Reaction of 5-ethenylpyranosides with diethylzinc and a catalytic amount of palladium furnished 5-membered rings (Eq. (210)) [1153]. Related reactions using 5-ethynylpyranosides and samarium diiodide were described [1154]. Palladium catalyzed a diastereoselective alkylation of aldehydes using 1-aryl-substituted-3-chloro-1-propenes in the presence of hexamethylditin [1155]. Palladium catalyzed the formation of bis-allylpalladium complexes and subsequent reaction with electrophiles (Eq. (211)) [1156]. Cobalt catalyzed the allylation of aldehydes and ketones using allylic acetates in the presence of zinc [1157]. Epoxides were transformed into homoallylic alcohols via a palladium-catalyzed epoxide rearrangement–allylation sequence mediated by indium (Eq. (212)) [1158].



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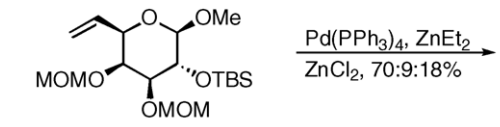

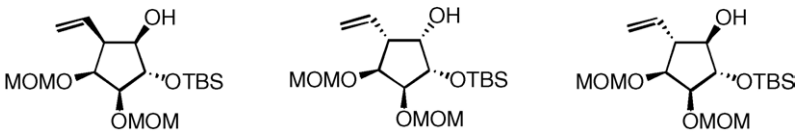

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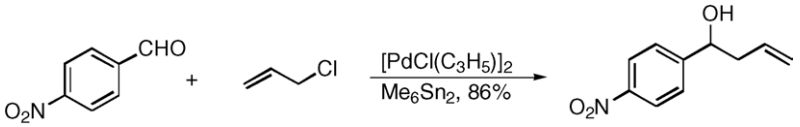

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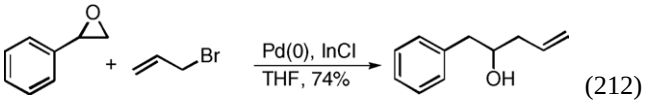
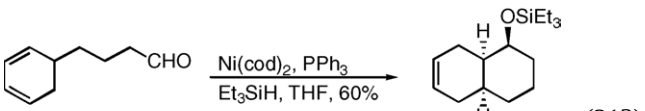


(212)

Nickel-catalyzed intramolecular allylations of 1,3-dienes having a tethered aldehyde in the 2-position [1159]. Nickel catalyzed the cyclization of 5,7- and 6,8-dienals in the presence of silanes affording 5- and 6-membered rings (Eq. (213)) [1160]. Copper-catalyzed carbonyl-ene reactions [1161]. Palladium catalyzed an intramolecular ketone alkylation to form heterocyclic compounds (Eq. (214)) [460]. Vanadium catalyzed an anti-selective alkylation of imines using 4-hydroxy-1,2-dienes (Eq. (215)) [1162]. Rhodium catalyzed a reductive alkylation of 1,2-diones using 1,3-dienes and hydrogen gas [1163]. A related regio- and enantioselective reaction of 1,3-diynes was reported (Eq. (216)) [1164].



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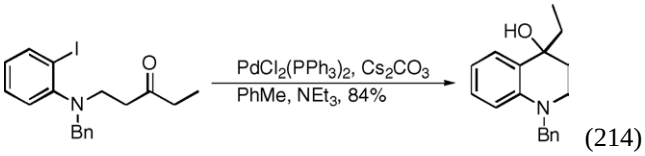

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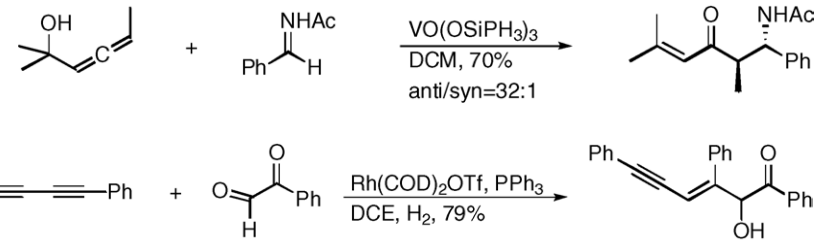

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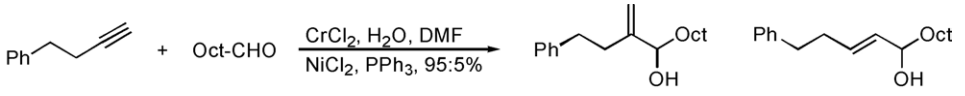
Nickel catalyzed a reductive coupling of terminal alkynes with aldehydes, in the presence of $CrCl_2$ to give allylic alcohols (Eq. (217)) [1165]. Nickel-catalyzed enantioselective reductive couplings of alkynes with aldehydes [1166,1167] and imines, and a reductive alkylation of epoxides (Eqs. (218) and (219)) [1168,1169]. This reaction was employed in a synthesis of (−)-terestacin (Eq. (220)) [1170]. Polycyclic compounds were obtained from acylic yne-dials using a nickel catalyst (Eq. (221)) [1171]. Titanium mediated a coupling of ynamides with aldehydes or alkynes to give enamides or dienamides, respectively (Eq. (222)) [1172]. An asymmetric synthesis of allyl- and allenylamines mediated by titanium was developed (Eq. (223)) [1173].



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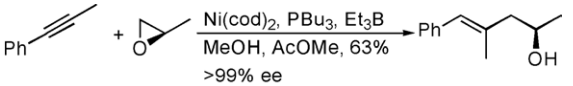

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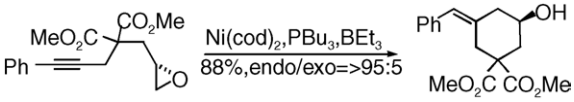

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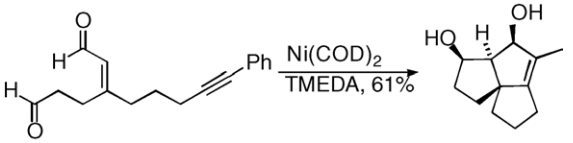

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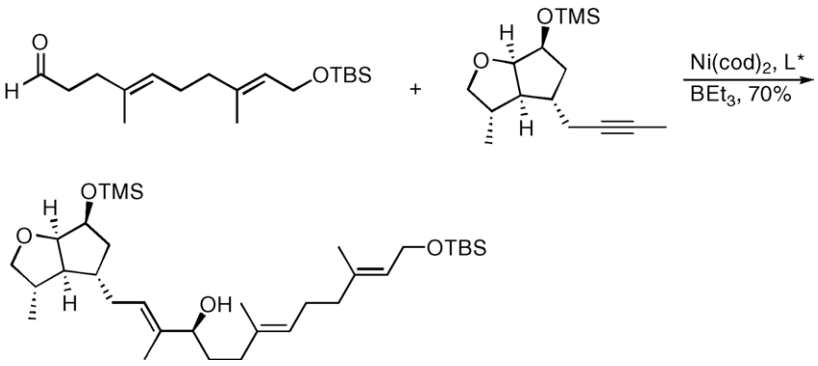

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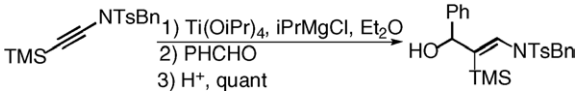

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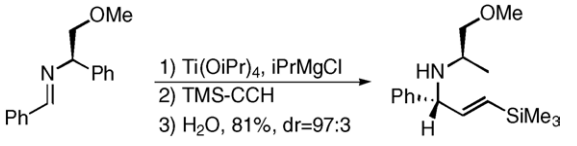
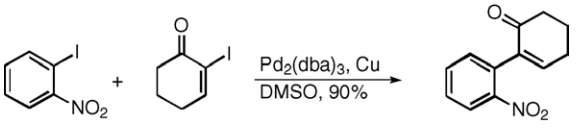


(223)

6. Miscellaneous carbon–carbon bond-forming reactions

Cobalt catalyzed the cross-coupling of aromatic halides with allylic acetates [1174]. A palladium-catalyzed copper-mediated coupling of 2-nitro-1-halobenzenes with 2-halo-2-cycloalken-1-ones was described (Eq. (224)) [1175]. Palladium catalyzed the coupling of aromatic iodides with acetic anhydride to give aromatic methyl ketones (Eq. (225)) [1176]. Nickel catalyzed the homo-coupling of 2-chloro or 2-trifloxypyridines [1177], palladium catalyzed the homo-coupling of 1-iodoalkynes [1178], aromatic halides [1179], and imidoyliodides [1180], and ruthenium catalyzed the homo-coupling of benzylic halides [1181]. Carbon dioxide was found to promote homo-coupling reactions using a palladium catalyst and zinc in water [1182]. Ruthenium catalyzed intermolecular *ortho*-arylation of aromatic ketones using aromatic boronic acid esters (Eq. (226)) [1183]. An intramolecular nickel-catalyzed aryl iodide-aryl bromide coupling was used in a synthesis of analogs of the protease inhibitor TMC-95A [1184].



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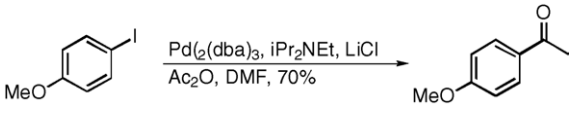

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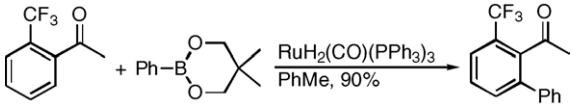

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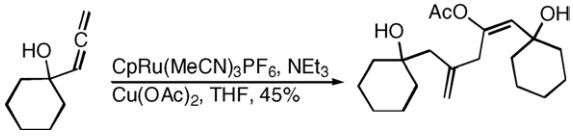
Copper [1185] and palladium [1186] catalyzed the homo-coupling of organoboronic acids. Palladium catalyzed the homo-coupling of aromatic zinc reagents in the presence of *N*-chlorosuccinimide [1187], of organosilanes [1188], and of organostannanes using air or allyl acetate as the oxidant [1189].

A palladium-catalyzed intramolecular oxidative arene–arene coupling was used in a synthesis of carbazomadurin A [68] and murrayaquinone A, koeniginequinone A, and koeniginequinone B [1190]. Palladium and copper catalyst systems were used to dimerize terminal alkynes to 1,3-diynes [387,1191–1193]. A copper-mediated intermolecular coupling, was used to prepare (+)-isoschizandrin [468], and polyfunctionalized biphenyls [1194]. Titanium mediated a reductive head-to-head coupling of homoallylic alcohols [1195]. Rhodium catalyzed the dimerization–acetoxylation of 1,2-diene-3-ols (Eq. (227)) [1196].



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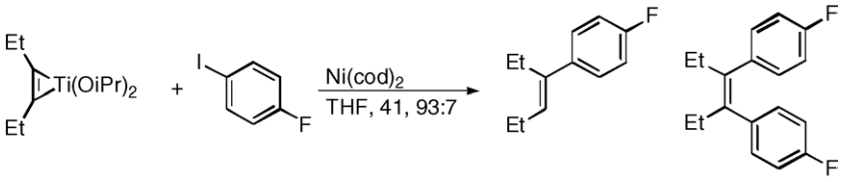
Nickel catalyzed a stereoselective cross-coupling of dialkenyl chalogenides with terminal alkynes to give enynes [1197]. Palladium catalyzed the coupling of terminal alkynes with 2,5-bis-(butyltelluro)-thiophene [1198] and -furan [1199], alkenyl-, furyl-, and thiophenyl-tellurium reagents [1200,1201], and aryl boronic acids [1202]. Titanium–alkyne complexes were coupled with allylic bromides in the presence of a nickel catalyst (Eq. (228)) [1203].

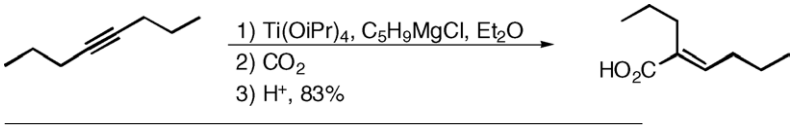


(228)

7. Carbonylations

7.1. Carbonylations of alkenes, allenes, and arenes

A palladium-catalyzed bis(methoxycarbonylation) of terminal alkenes was developed [1204]. Palladium catalyzed an asymmetric hydrocarbomethoxylation of styrene [1205]. Titanium mediated a hydrocarboxylation of alkynes using carbon dioxide to form α,β-unsaturated carboxylic acids. Further functionalization of the intermediately formed titanacyclopropene with electrophiles was also reported (Eq. (229)) [1206].



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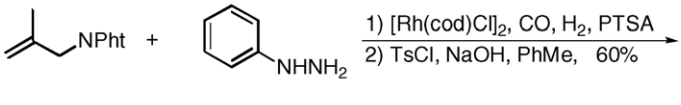
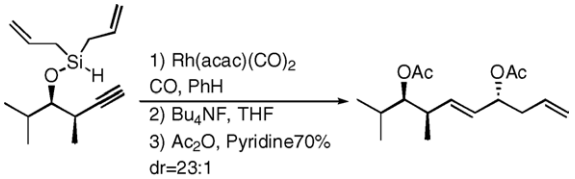
A tandem rhodium-catalyzed alkene hydroformylation Fischer indole synthesis was described (Eq. (230)) [1207]. Rhodium-catalyzed intramolecular silylformylations of pendant alkenes [333]. Rhodium catalyzed a related silylformylation and alkynes followed by an allylation (Eq. (231)) [1208]. A silylcarbocyclization formylation of 1,6-enynes using a rhodium–cobalt catalyst was described [1209].





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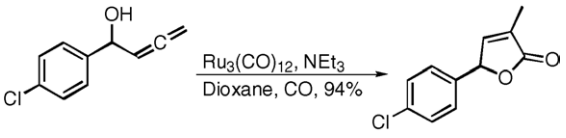
Ruthenium catalyzed the formation of lactones and lactams from hydroxy- and amino-substituted allenes (Eq. (232)) [1210]. Palladium catalyzed a double carbonylation of homoallylic amine derivatives to give γ-lactams (Eq. (233)) [1211]. Nickel mediated a sequential carbonylation–alkylation of terminal allenes using carbon dioxide (Eq. (234)) [1212].



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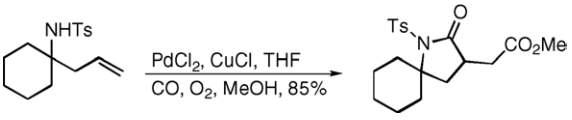

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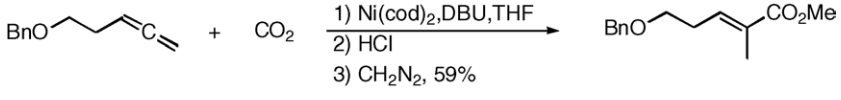
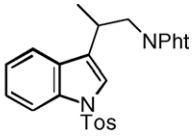


(234)

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

Metal-mediated [2+2+1]cycloadditions forming cyclopentenones continued to be extensively studied, in particular the cobalt-mediated, Pauson–Khand reaction. Loss of carbon monoxide from the alkyne dicobalt hexacarbonyl complex was show to precede alkene coordination in the Pauson–Khand reaction [1213]. Pauson–Khand reactions in water using surfactants were described [1214]. Polymer-supported reagents were used [1215]. Ruthenium–cobalt nanoparticles [1216], rhodium and iridium [1217], and an entrapped rhodium complex [1218] were also used as catalyst. Rhodium also catalyzed a Pauson–Khand-type reaction using formaldehyde as the carbon monoxide source [1219]. Rhodium-catalyzed asymmetric intramolecular Pauson–Khand reactions in aqueous solutions [1220]. Molybdenum mediated a novel Pauson–Khand-



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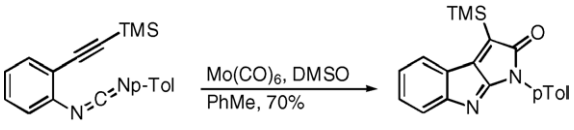
type reaction of alkyne-substitute carbodiimides (Eq. (235)) [1221].



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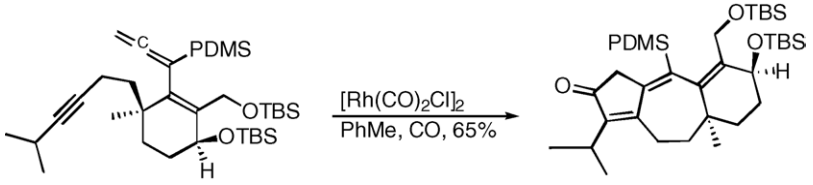
Intermolecular cobalt mediated Pauson–Khand reactions of 7-oxabicyclo[2.2.1]heptenes and heptadienes [1222] and of exo-methylene compounds [1223] were described. An unusual endo-addition was observed using chiral ynamides and norbornadiene [1224]. The intermolecular Pauson–Khand reaction was used in a synthesis of (−)-terestacin [1170]. Intramolecular Pauson–Khand reactions of electron-deficient alkenes (Eq. (236)) [1225], sugar-derived 1,6-enynes [1226], 1,3-diene-8-ynes (Eq. (237)) [1227], and 1,17-diene-7,11-diynes [1228] were reported. A sequential [5+1]cycloaddition–Pauson–Khand reaction was developed (Eq. (238)) [1229]. The intramolecular Pauson–Khand reaction was used in synthetic applications toward the carbocyclic core of palau'amines and styloguanides (Eq. (239)) [1230], magellanine [1231], tricycloillicinone [1232], and tecomanine [1233].

A precatalyst for asymmetric Pauson–Khand was described [1234]. Cobalt mediated intramolecular asymmetric Pauson–Khand reactions of chiral (2-dimethylaminophenyl)sulfinyl-substituted alkenes [1235]. Diastereoselective intramolecular Pauson–Khand reactions using optically active 1,6-enynes [1236] and enantioselective reactions of optically active 1-sulfonimidoyl-substituted 5-aza-1-en-7-ynes [1237] were described.

Rhodium-catalyzed intramolecular allenic Pauson–Khand reactions were used to prepare the carbon skeleton of guanacastepene A (Eq. (240)) [1238] and a bicyclo[5.3.0]decenone skeleton [1239]. Iridium-catalyzed intramolecular allenic Pauson–Khand reactions of 1,2-diene-7-ynes and 1,2-diene-8-ynes [1240]. A related double intramolecular allenic Pauson–Khand reaction catalyzed by molybdenum was used to prepare a dicyclopenta[*a*,*e*]pentalene [1241].



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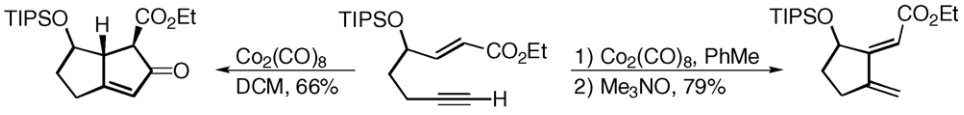
Intramolecular palladium-catalyzed hydroxy-palladation carbonylations of alkynes was used to prepare wedelolactone [419] and (+)-cystothiazole A [1242]. An intramolecular alkyne hydroxy-palladation of in situ formed hemiketals followed by carbonylation was used to prepare (−)-AL-2 (Eq. (241)) [388]. An asymmetric intramolecular hydroxy-palladation carbonylation of 2-propargyl-1,3-diones was described [1243].



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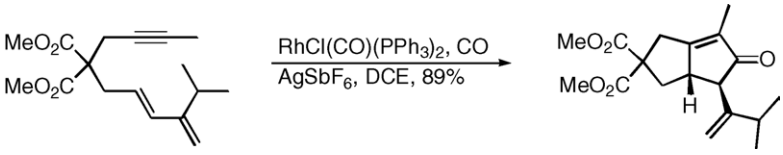

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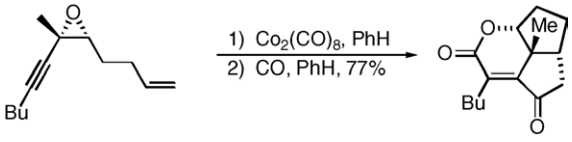

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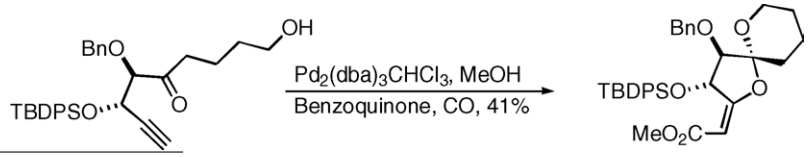

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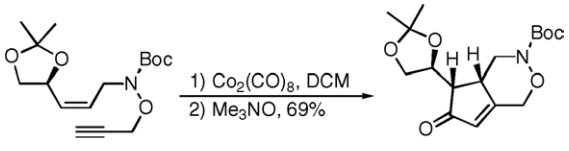
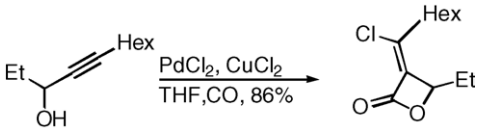
A palladium-catalyzed carbonylation–lactonization of a propargylic alcohol was used in a synthesis of (+)-(−)-akolactone A [359]. (*Z*)-α-Chloroalkylidene-β-lactones were prepared via a palladium-catalyzed cyclocarbonylation



(239)

(Eq. (242)) [1244]. Nickel catalyzed an alkylative carbonylation of alkynes and this reaction was used in a synthesis of erythrocarine (Eq. (243)) [1245]. Palladium catalyzed a [2+2+1]cyclization of 1,6-diynes and carbon monoxide to give bicyclo[3.3.0]octa-1,5-dien-3-ones (Eq. (244)) [1246]. Cobalt also catalyzed carbocyclizations of 1,6-diynes in the presence of carbon monoxide (Eq. (245)) [1247]. Platinum catalyzed a thiocarbonylation of terminal alkynes to give α,β-unsaturated thioesters (Eq. (246)) [1248]. In contrast, rhodium-catalyzed thioformylations of alkynes (Eq. (247)) [1249].



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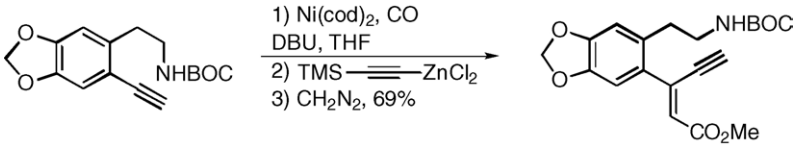

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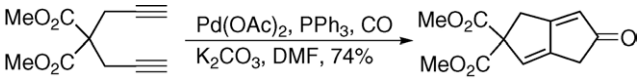

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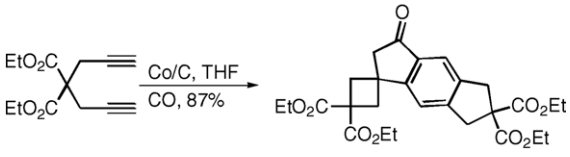

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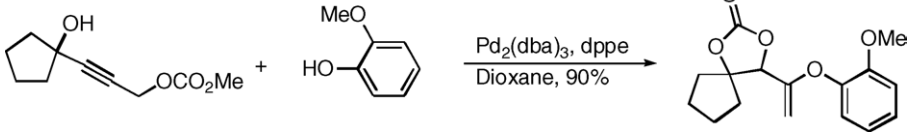

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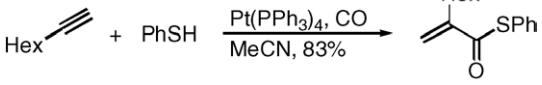

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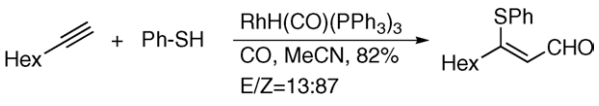

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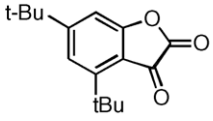
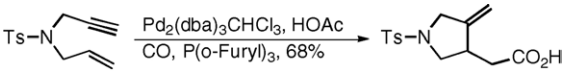


7.3. Miscellaneous carbonylations

Carbonylation of a stable β-hydrogen containing σ-alkyl palladium complex was described [1250]. Palladium catalyzed an oxidative carbonylation of amines forming unsymmetrical ureas [1251]. Palladium catalyzed a cyclization–carbonylation of 1,6-enynes (Eq. (248)) [1252]. Palladium catalyzed a decarboxylation–carbonylation of 5-alkenyloxazolidine-2-ones to give 3,6-disubstituted 3,6-dihydripyridin-2-ones (Eq. (249)) [1253]. This reaction was used in syntheses of deoxymannojirimycin and D-mannolactam [1254]. Palladium also catalyzed a decarboxylation–carbonylation of 4-methoxycarbonyloxy-2-butyn-1-ols with phenols to give functionalized cyclic carbonates (Eq. (250)) [1255]. Palladium catalyzed the double carbonylation of 2-(propargyl)allyl phosphates to give unsaturated lactones (Eq. (251)) [1256].



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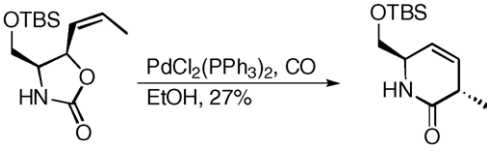

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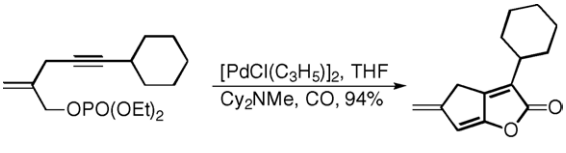
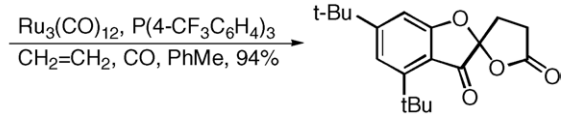


(251)

Ruthenium catalyzed a carbonylative cycloaddition of α-ketolactones with alkynes or alkenes (Eq. (252)) [1257]. Cobalt catalyzed a carbonylative ring-expansion of 2-aryl-2-oxazolines (Eq. (253)) [1258]. Cobalt also catalyzed a carbonylation of cyclic imino esters (Eq. (254)) [1259] and the formation of thioisobutyrolactones from thiethanes and carbon monoxide [1260]. Nickel catalyzed the reaction of epoxides with carbon dioxide to form cyclic carbonates [1261].



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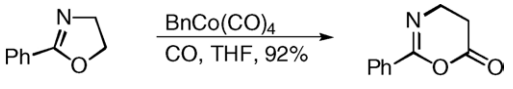

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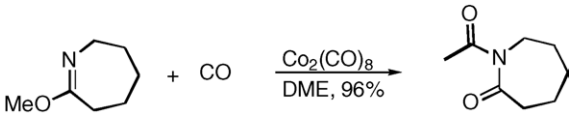
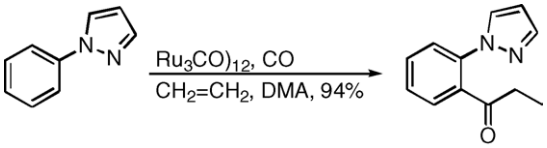


(254)

Ruthenium catalyzed a carbon–hydrogen-bond insertion, carbonylation, and alkylation sequence of *N*-arylpyrazoles (Eq. (255)) [1262]. Ruthenium catalyzed an intermolecular insertion of ethene and carbon monoxide into α,β-unsaturated imines to form pyrrolidones (Eq. (256)) [1263].



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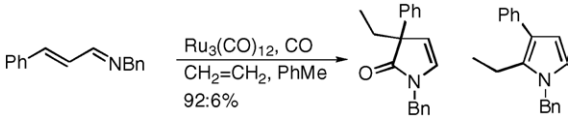
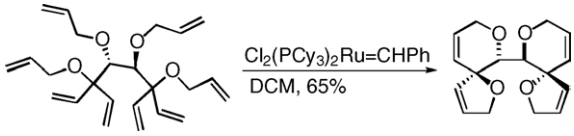


(256)

8. Metathesis reactions

A large number of reports on ring-closing metathesis reactions using Grubbs'-type ruthenium catalysts were published. The rate and mechanism of ring-closing metathesis using ruthenium allylidene complexes were studied [1264]. New, highly efficient, and sometimes air stable, or recyclable ruthenium catalysts [1265–1272] and reaction conditions [1273] were developed. Ruthenium byproducts were removed by sequential treatment with silica gel, activated carbon, and chromatography [1274]. Microwave-accelerated reactions were developed [1275–1278]. A higher reaction temperature was shown to improve the yield of monomeric macrocycles [1279]. In addition to ruthenium, molybdenum catalysts were also described [1280].

A quadruple ring-closing metathesis was reported (Eq. (257)) [1281]. A ring-closing metathesis was used to cleave a polymer alkene linker and simultaneously form oxacycles [1282]. A sequential ring-closing metathesis double bond isomerization using a ruthenium catalyst was reported (Eq. (258)) [1283]. Tandem metathesis-hydrogenations were described (Eq. (259)) [1284,1285]. A ring-closing metathesis Pauson–Khand sequence was developed (Eq. (260)) [1286]. 2-Chloro-1,*n*-dienes were shown to undergo ring-closing metathesis to give chloroalkene functionalized products (Eq. (261)) [1287]. Related reactions of 2-fluoro-1,*n*-dienes were also described [1288]. Double bond isomerization was observed prior to cyclization in some cases (Eq. (262)) [1289]. Domino metathesis reactions were used to prepare functionalized pyrones (Eq. (263)) [1290].



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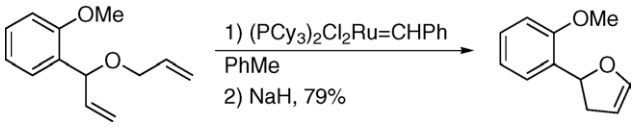

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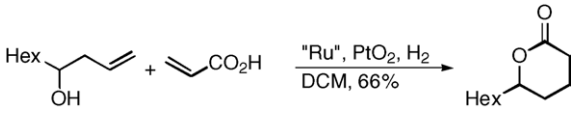

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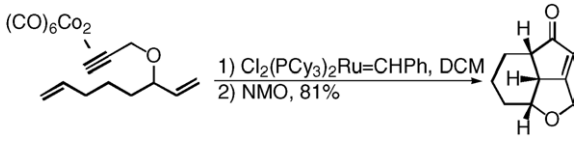

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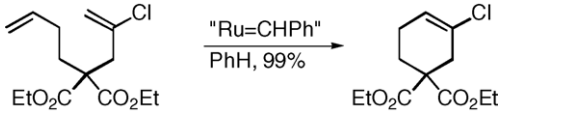

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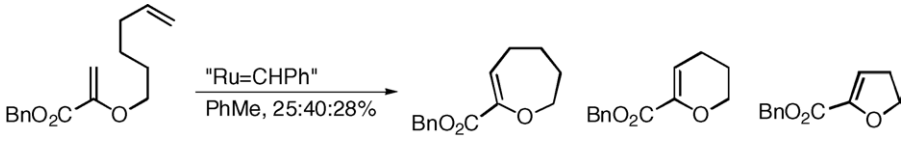

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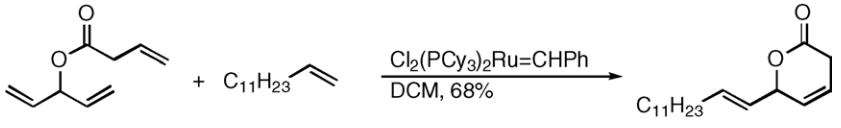
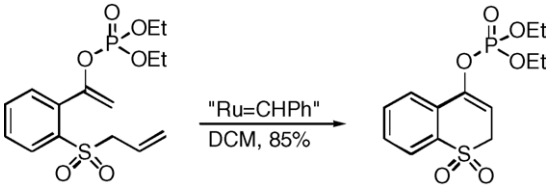


(263)

A large variety of ring systems were prepared via alkene– alkene ring-closing metathesis using ruthenium catalysts. Ring systems including chromenes [1291], 7-membered cyclic amine [1292], 5–8-membered cycloalkenes [1293–1298], spirocycles [1299], *trans*-hydrindanes [1300], 7- and 8-membered unsaturated ethers [1301–1303], oxepines [1091], oxepinones [1304], 2-oxa-1-azabicyclo[5.3.0]-5-alkenes [1305], 6–10-membered lactams [1306], fused piperazines [1307], chiral 1,2,5,6-tetrahydropyridines [1308], chiral cyclic β-amino acids and cyclic amines [1309], fused tricyclic β-lactams [1310], bicyclic lactams [1311], fused bicyclic amines [49,1312], fused bicyclic glutarimides [1313], fused bicyclic imidazoles [23], fused bicyclic sulfonamides [1314], α-trifluoromethylated

cyclic amines and amides [1315], 2,5-dihydropyrroles [680], 1,2,3,6-tetrahydropyridines [1316,1317], carbazoles [1318], bicyclic peptides [1319], 7-membered cyclic β-aminoacids [1320], macrocyclic peptides [1321,1322], indolizidinones [1323], indolizidines and quinolizidines [1324], 4-dihydrobenzo[4,5]imidazo[1,2-*a*]pyridine [1325], 8–9-membered cyclic carbamates [1326], 4*H*-chromenes, a naphthol, and an indenol [1327], [1,2]oxazines [1328], enantiomerically pure arylated dihydropyran derivatives [1329], coumarins [1330], silacyclopent-3-enes [1331], 2-sila-1,3-dioxcycloalkenes [1332,1333], polymer-supported cyclic sulfamides [1334], unsaturated sultones [1335], 1,1-dioxo-1,2,7-thiadiazepines [1336], unsaturated 5–6-membered lactones [1337], cyclic enol phosphates (Eq. (264)) [1338], borane protected cyclic phosphines [1339], macrocycles [1340–1344], [3]catenane [1345], carbazole-linked cyclic peptoids (Eq. (265)) [1346], macrocyclic paracyclophanes [1347], macrocyclic crown amides [1348], macrocyclic dilactones [1349], 'inside–outside' medium-sized rings [1350], medium ring alkynes complexed to dicobalt hexacarbonyl [1351], ferrocene containing cyclophanes [1352], dicobalt hexacarbonyl complexed macrocyclic yne-lactones (Eq. (266)) [1353], macrocyclic metallacycles [1354], fused dinucleotides (Eq. (267)) [1355], 2′-*O*,3′-*O*-bicyclic adenosine analogs [1356], benzofused heterocycles [1357], unsaturated, 2-oxazepines, 1,2-oxazines, 1,2-oxazonine, and 1,2-oxazecine [862,1358,1359], nucleosides [1360,1361], and quinolone-fused hetereocycles [1362] were all prepared using ring-closing metathesis methodology.



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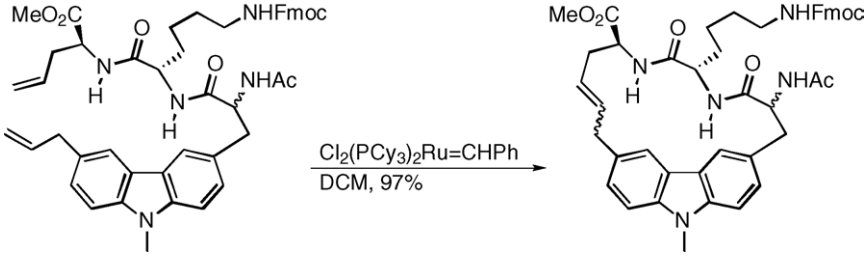

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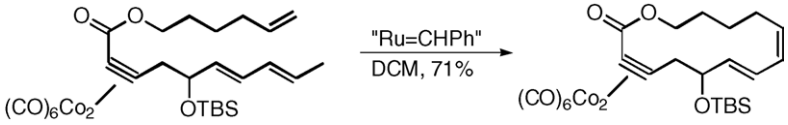

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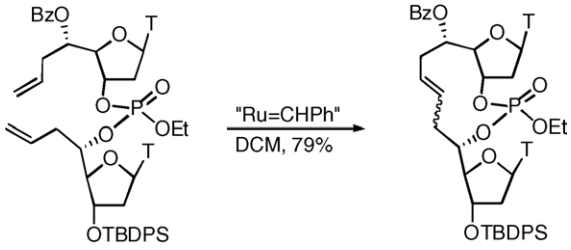
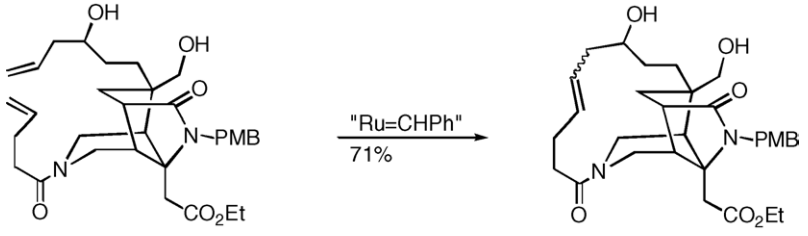


(267)

Synthetic targets using alkene–alkene ring-closing metathesis include, guanacasterpene A [123], F-ring of halichondrin B [1363], (+)-patulolide A and patulolide B [1364], calystegine alkaloids [1365], sarain A (Eq. (268)) [1366], indolizidine skeleton [1367], an aziridinomitosane [1368], laulimalide [1369], (+)-diplodialimide A [1370], spicigerolide [1371], antiviral nucleosides [1372], (+)-salicylihalamides [1373], carbocyclic core of cornexistins [55], naturally occurring α-pyrones [1374], fostriecin [1375], F–H gambierol subunit [1133], the core of xanthonolides, guaianolides, and eudesmanolides [987], the proposed structure of passiflorin A [1376], cryptocarya triacetate, cryptocaryolone, and cryptocaryolone diacetate [1377], (+)-strictifolione [1378], iroids [1379], (−)-antofine [1380], muscothiazole A and B [1381], (−)-agelastatin A [1382], macrosphelides A and B [1383], (+)-rogioloxepane A [422], ring analogs of ergot alkaloids [575], (−)-peduncularine [1384], salicylhalamides A and B [1385], (−)-PF1163B [1386], malayamycin A [1387], (+)-muscopyridine [1388], broussonetine G [892], thapsigarins [1389], (−)-heliannuol A [873], isofagomine [1390], eleutherobin and simplified analogs [1391,1392], spicigerolide [1393], α-herbetenol, and β-herbertenol [1394], tricycloclavulone [1395], (+)-hyptolide [1396], 1-deoxymannojirimycins [1397], TEI 9826 [1398], microcarpalide [1399],

peloruside A [1400,1401], (−)-carbovir [1402], guanacastepene A [1403], the proposed and correct structure of pasciflorin A [1404,1405], pentenomycins [1406], (2*S*,3*R*,4*S*)-3,4-dihydroxyproline [1407], eudesmane, eremophile, and agarofuran sesquiterpenes [1408], dolabellane-type diterpenoids [1409], pacifigorgianes (Eq. (269)) [1410], eleutheside analogs [1411], (−)-heliannulol C [874], repinotan [1412], (*R*)-argentilactone and (*R*)-goniothalmin [1413], lepadiformine [1414], lentiginosine [1415], constanolactone A and B [1416], (+)-rhopaloic acid [1417], G-ring of brevetoxin A [1418], amino-1,4-anhydro-D-pentitols and amino-1,5-anhydro-D-hexitols [1419], epothilone analogs [434,1420], (−)-epi-deoxoprosopinine [1421], phomactin A [166], (+)-malyngolide, (−)-malyngolide, and (+)-tanikolide [1422], d4T [1423], Δ^7-protilludene [1424], (+)-ambruticin S [1425], sarcodictyin and eleutherobin analogs [1426], halitulin [319], gambierol (Eq. (270)) [86], (+)-deoxyneodolabelline [87], oximidine II [1427], (+)-migristatin (Eq. (271)) [1428], nakadomarin A (Eq. (272)) [1429], (+)-obtusenyne (Eq. (273)) [442], merrilactone A [1430], gambierol and 16-epi-gambierol [94], podophyllotoxin [1431], (−)-borrelidin [1432], (−)-mucocin (Eq. (274)) [1433], and amphidinolides [1434].



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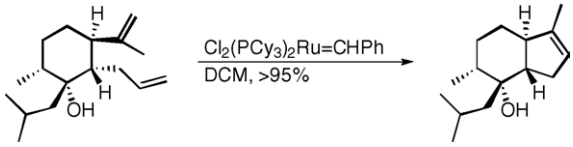

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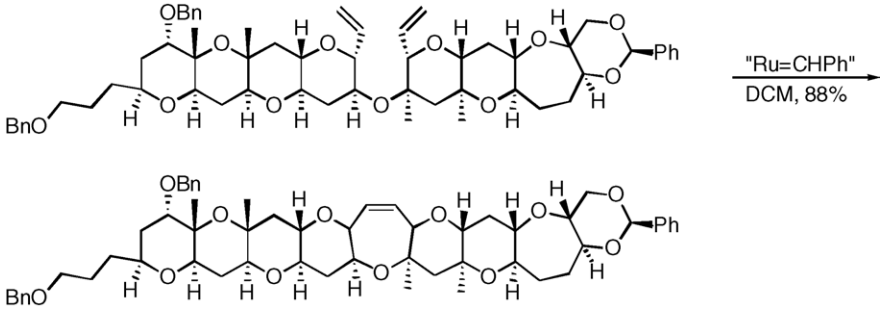

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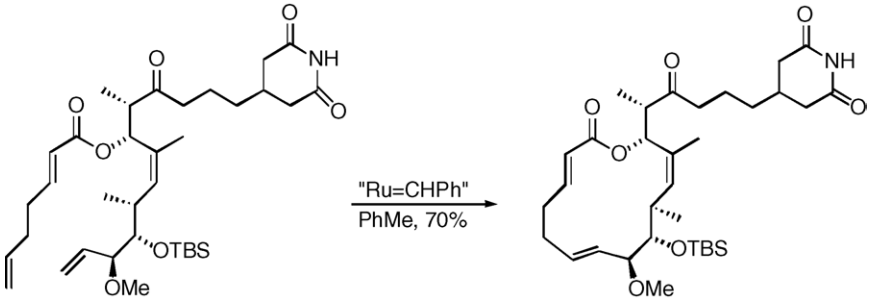

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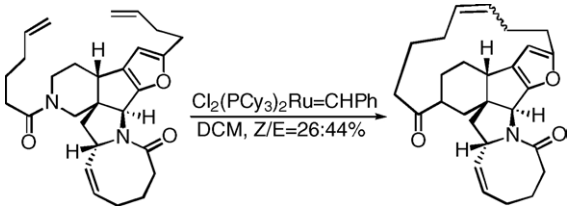

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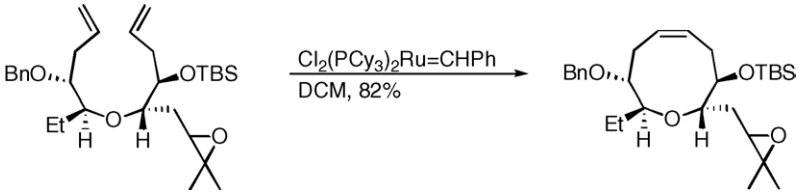

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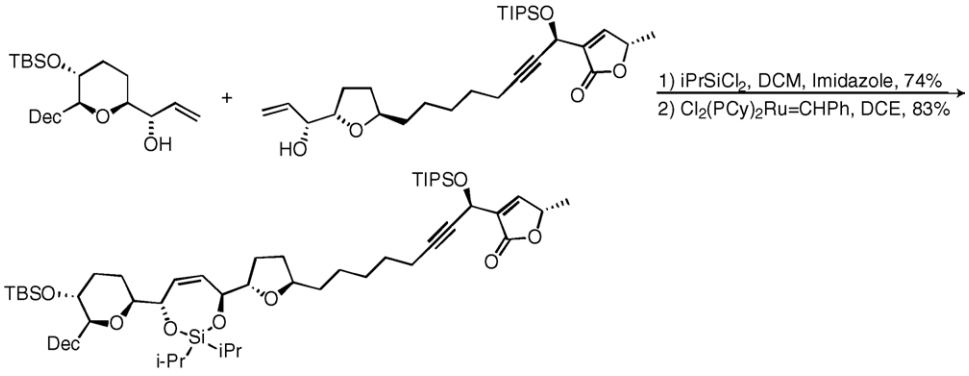
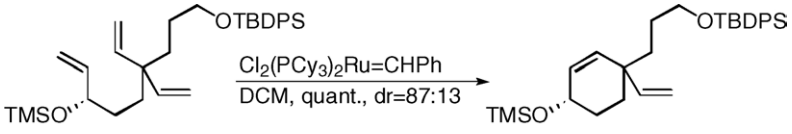


(274)

A diastereoselctive ring-closing metathesis was used in a synthesis of (−)-aspidospermine (Eq. (275)) [1435]. An interesting ring-contraction metathesis was used to reduce the size of a macrolide by two carbons (Eq. (276)) [1436]. Chiral molybdenum and tungsten complexes were used in asymmetric ring-closing metathesis [1437–1439]. Ruthenium catalyzed asymmetric ring-closing metathesis reactions of silicon-tethered ene-dienes [1440]. Schrock type molybdenum carbenes were used to prepare azabicylic amines [1441].

Efficient catalysts for ring-opening alkene–alkene cross-coupling metathesis were developed [1442–1444]. A ring-opening cross-coupling metathesis of a 2-azabicyclo[2.2.1]ring-system with allyl silanes was reported [1445]. Ruthenium catalyzed an enyne ring-opening, ring-closing, cross-metathesis (Eq. (277)) [1446]. A ring-opening ring-closing metathesis was used to prepare (+)-astrophylline (Eq. (278)) [1447].

Tungsten and molybdenum-catalyzed alkyne–alkyne ring-closing metathesis were used in a synthesis of (+)-



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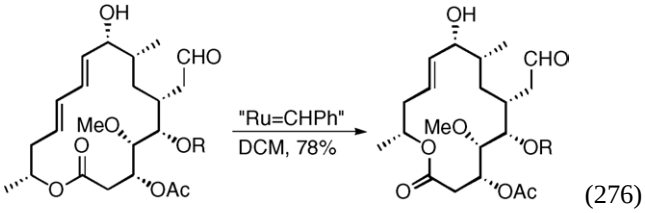
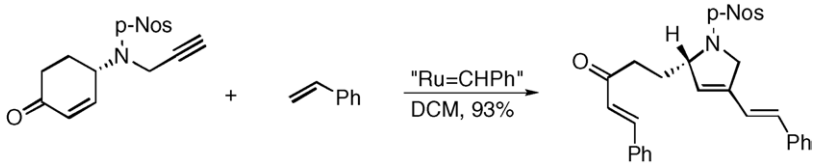


(276)

citreofuran (Eq. (279)) [1448], twisted tolanes [1449], latrunculin B [1450], macrocyclic diynes [1451], dehydrobenzannulenes [358], and metallamacrocycles (Eq. (280)) [1452]. Ene-yne-ene metatheses (Eq. (281)) [1453,1454] were used in synthesis of erythrocarine [1455] and erythravine [1456]. A domino ring-closing cross-metathesis was described (Eq. (282)) [1457].



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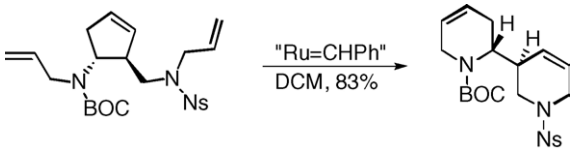

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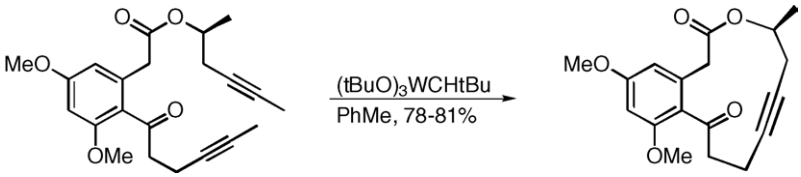

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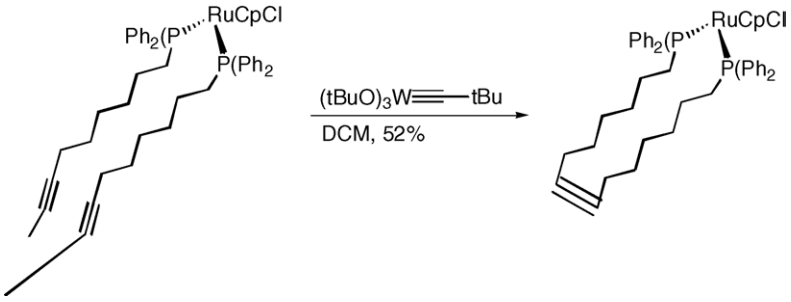

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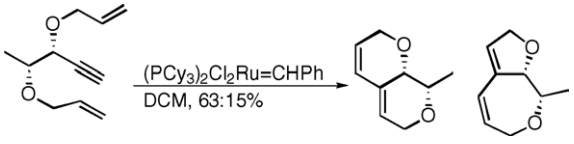

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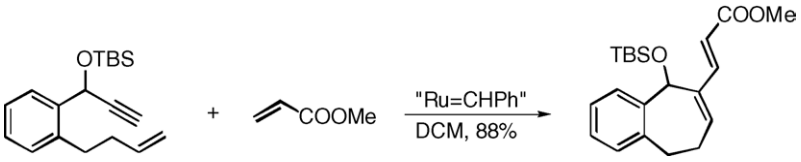
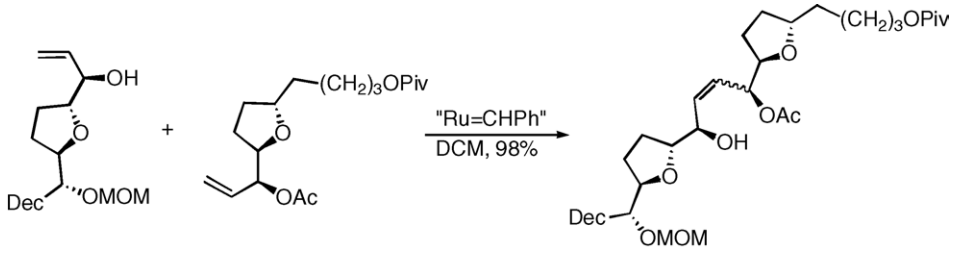


(282)

Ruthenium catalysts were used in alkene–alkene cross-metathesis reactions [880,1458–1462]. A general model for the selectivity of alkene–alkene cross-metathesis for different types of alkenes was developed [1463]. Cross-metathesis of alkenyl boronates [991,1464], alkenyl and allyl phosphine oxides [1465,1466], α- and β-1-C-allylglycosides [1467,1468], polymer-bound azide-containing sugars [1469], allyl silanes [1470], alkenylsilanes [1471], allyl-zirconocenes and -titanocenes [1472], and alkenyl sulfones and sulfoxides [1473] with terminal alkenes were described. A polymer-bound alkene was used [1474]. An α-2-propenylglycoside was transformed into an α-ethenylglycoside via a cross-metathesis using ethene [1475].

Alkene–alkene cross-metathesis was used to prepare salicylihalamides A and B [1476], 3,5-dialkyl-substituted indolizidine alkaloids [1477], acetogenins (Eq. (283)) [1478], (−)-centrolobine [1479], a segment of phorboxoles A and B [868], paraconic acids [1480], (+)-amphidinolide T1 (Eq. (284)) [1481], prostaglandin derivatives [1482], and furaquinoncins (Eq. (285)) [601].



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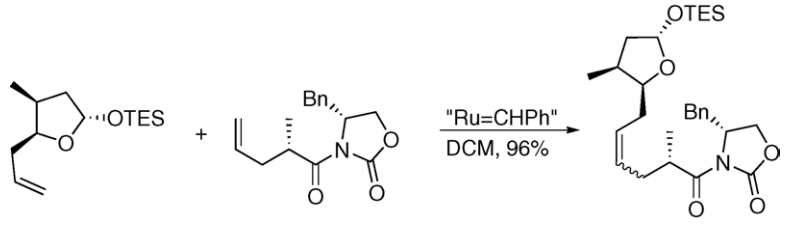

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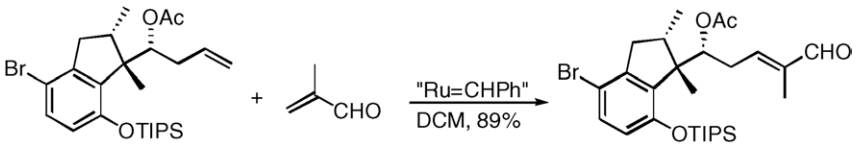
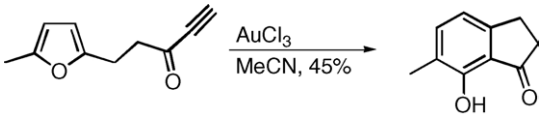


(285)

9. Cycloisomerizations

Reactions wherein all elements of the starting material were found in a cyclic product are grouped in this section. An intermolecular copper-catalyzed [2+2]cycloadditions was used in a synthesis of tricycloclavulone [1395]. Ruthenium catalyzed intermolecular [2+2]cycloadditions of bicyclic alkenes and chiral propargylic alcohols [1483], and of disubstituted alkynes [1484].

A gold catalyzed intramolecular Diels–Alder reaction aromatization sequence was used in synthesis of jungianol and epi-jungianol (Eq. (286)) [1485]. Platinum catalyzed the cyclization of 5-(2-furyl)-1-alkynes [1486]. Gold and copper also catalyzed benzannulations of 2-(1-alkynyl)-substituted aromatic aldehydes and ketones and enynals (Eq. (287)) [1487]. High level of stereoselectivity was observed in rhodium-catalyzed intramolecular Diels–Alder reactions compared to non-catalyzed [1488].



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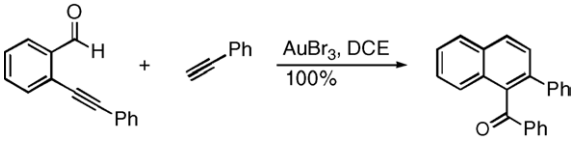

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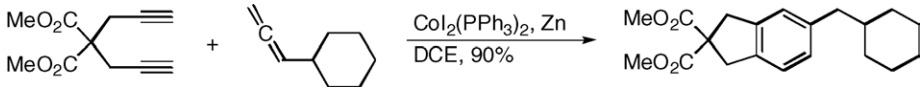
Cobalt–cycloheptyne complexes were used in intramolecular [2+2+2]cycloisomerizations with tethered alkynes [1489]. Cobalt mediated a diastereoselective intermolecular [2+2+2]cycloaddition of linear enediyne phosphine oxides [1490]. Intermolecular linear enediyne cycloadditions were also described [1491]. Hexahelicenols [1492] and (+)-rubiginone B_2 [1493,1494] were prepared by an intramolecular cobalt-catalyzed [2+2+2]cycloisomerization of triynes. Rhodium-catalyzed inter- and intramolecular alkyne [2+2+2]cycloisomerizations [1495]. Cobalt catalyzed a regio- and stereoselective [2+2+2]cycloaddition of 1,6-heptadiynes with 1,2-dienes (Eq. (288)) [1496] and of 6-alkynyl purines with 1,6-diynes [1497]. An intramolecular cobalt-mediated [2+2+2]cycloaddition of 8-en-1,13-diynes to give 2-hydroxy-substituted decahydrophenanthrenes was reported [1498].



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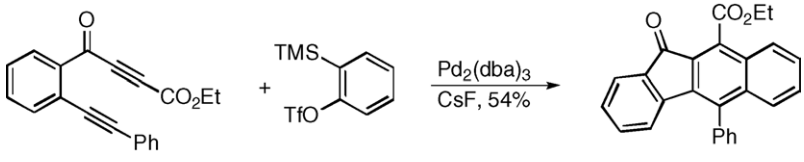
The mode of iridium-catalyzed annulation of DMAD with alkynes was controlled by the choice of catalyst system [1499]. Ruthenium catalyzed [2+2+2]annulations of 1,6-diynes with nitriles to give pyridines [1500]. Ruthenium catalyzed the [2+2+2]cyclotrimerization of terminal alkynes [1501]. A water-soluble rhodium catalyst was used for [2+2+2]cyclotrimerizations [1502]. Cobalt catalyzed the [2+2+2]cycloaddition of 1,*n*-diynes with isocyantes forming pyridophanes [1503]. Titanium mediated [2+2+2]cyclobenzannulations of diynes with homoallylic alcohols affording fused 1,3-cyclohexadienes [1504]. A regioselective [2+2+2]cycloaddition of a nickel benzyne complex with 1,3-diynes was reported [1505]. Palladium catalyzed a [2+2+2]cycloaddition of intermediately formed benzyne with 1,6-diyn-3-ones to give benzofluorenones (Eq. (289)) [1506]. Both intra- and intermolecular palladium-catalyzed [2+2+2]cycloisomerizations were described [1507].



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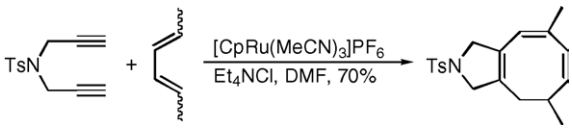
A ruthenium-catalyzed formal [4+2+2]cycloaddition of 1,6-diynes with 1,3-dienes was reported (Eq. (290)) [1508]. Rhodium catalyzed the intramolecular [5+2]cycloaddition of alkynes-tethered alkenylcyclopropanes to give [5.3.0]ring-systems with good to high diastereoselectivity (Eq. (291)) [1509]. Rhodium catalyzed a [5+2]cycloaddition of alkene-tethered alkenylcyclopropane in water [1510]. Palladium catalyzed an intramolecular [3+2]cycloaddition of alk-5-

ynilidenecyclopropanes to give bicyclo[3.3.0]octenes (Eq. (292)) [1511].



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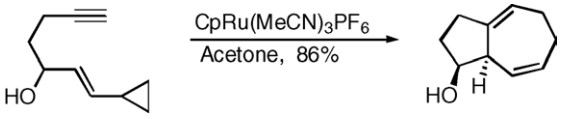

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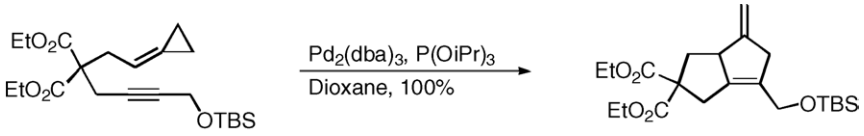

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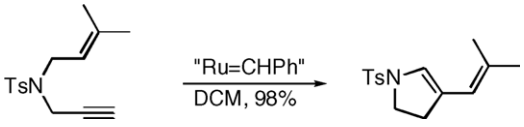
Ruthenium catalyzed ring-opening ring-closing metathesis of 1,6- and 1,7-dienes [1512,1513]. Ruthenium-catalyzed 1,7-enyne ring-closing metathesis reactions were reported forming unsaturated tetrahydropyridines [1514], and 5-ethenyl-1-oxa-2-silaspiro[5.5]undec-4-enes [1515]. Ruthenium catalyzed intramolecular 1,6-enyne metathesis to give dihydropyrolles (Eq. (293)) [1516]. Ruthenium catalyzed enyne ring-closing metathesis of 1,6-enyne or 1,7-enyne sulfonates furnished unsaturated sultones [1335]. Interesting metathesis of 1,10-diene-3-yne in the presence of ethene (Eq. (294)) [1517] and intermolecular enyne metathesis affording cycloheptadienes (Eq. (295)) [1518] were described. An enyne metathesis of a functionalized epoxide was reported (Eq. (296)) [1519].



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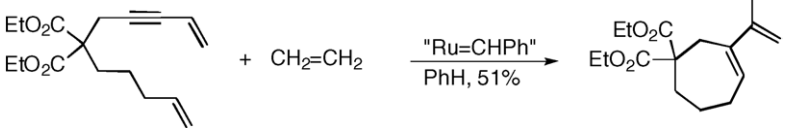

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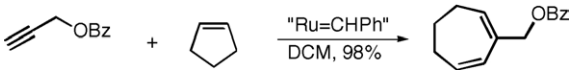
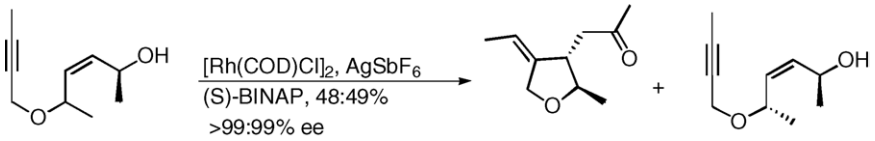


(295)

Dihydrofuranes and dihydropyranes were prepared via a rhodium-catalyzed cycloisomerization of 1-yne-4-ols and 1-yne-5-ols, respectively [1520]. Palladium catalyzed cycloisomerizations of 2-(1-alkynyl)benzylalcohols to give (*Z*)-1-alkylidene-1,3-dihydroisobenzofuranes and 1*H*-isochromenes [1521]. Cobalt catalyzed the cycloisomerization of 1,6-enynes to give alkenyl substituted 5-membered rings. Double bond isomerization was also observed is some cases [1522]. Closely related asymmetric palladium-catalyzed cycloisomerizations of 1,6-enynes were also reported [1523,1524]. Rhodium catalyzed a kinetic resolution of 1-hydroxy-2,8-enynes (Eq. (297)) [1525]. Rhodium-catalyzed cycloisomerizations of 1,3,8-trienes to give alkenyl-substituted 5-membered rings. The Diels–Alder reaction is a competing reaction [1526]. Rhodium catalyzed the cycloisomerization of 1,2,7-trienes to give substituted 6-membered rings (Eq. (298)) [1527]. Migration of an aryl group or a halogen was observed in ruthenium-catalyzed cycloisomerizations of aromatic 1,5-enynes (Eq. (299)) [1528]. A halogen shift was observed upon related cycloisomerizations of 1-chloro-2,7-enynes (Eq. (300)) [1529]. Palladium catalyzed an asymmetric cycloisomerization of 4-acetoxy-2-buten-1-yl 2-alkynoate to give α-methylene-γ-butyrolactones (Eq. (301)) [1530] and of 8-acyloxy-1,2,6-trienes to give fused bicyclic compounds (Eq. (302)) [1014]. Palladium catalyzed cyclizations of 1-stannyl-2,7-enynes with migration of tin (Eq. (303)) [1531]. A ruthenium-catalyzed cycloisomerization hydroxylation of a 1,6-diyne was used in a synthesis of (+)-α-kainic acid (Eq. (304)) [1532]. L-Vancosamine was prepared using a tungsten-catalyzed cycloisomerization of a 1,6-diyne [919].



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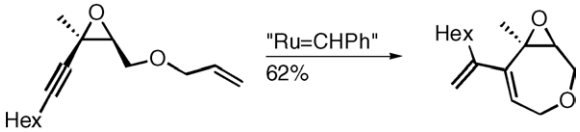

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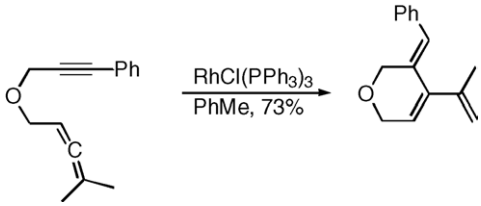

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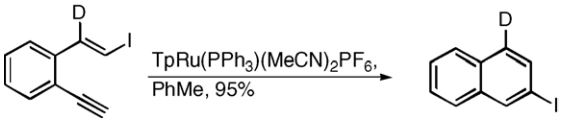

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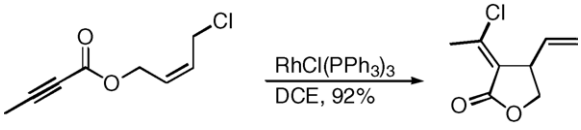

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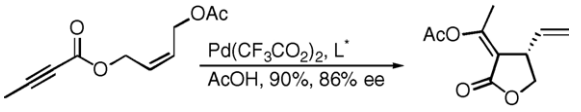

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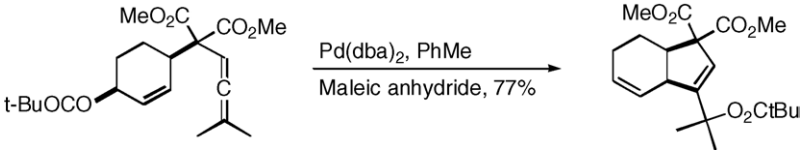

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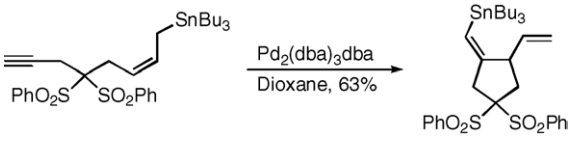

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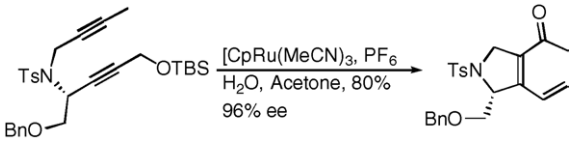
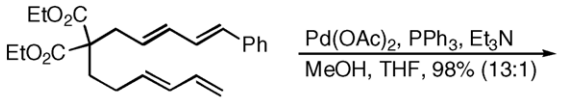
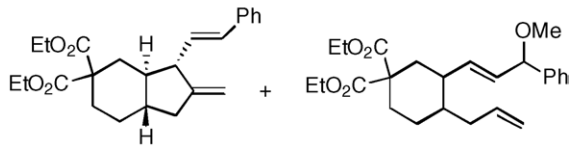


(304)

Palladium catalyzed a cycloisomerization of 1-aryl-substituted 1,3,9,11-tetraenes to give bicyclo[4.3.0]ring-systems (Eq. (305)) [1533] and of 1,3,5,7-tetraenes to give bicyclo[4.2.0]octa-1,3-dienes [1534]. Palladium and ruthenium catalyzed cycloisomerizations of 1,6-dienes to give cyclopentene or exo-methylene cyclopentane derivatives [1268,1535]. Substitution on the diene chain had a large effect on the reaction rate of the cycloisomerization of 1,6-dienes [1536]. A related cycloisomerization of 1,6-dienes catalyzed by ruthenium was reported affording exo-methylene spirolactones [1537]. Rhodium catalyzed the cycloisomerization of 1,2,7-trienes and 1,2,8-trienes to 5- and 6-membered compounds, respectively (Eqs. (306) and (307)) [1538]. In the presence of carbon monoxide, a 7-membered ring was obtained.





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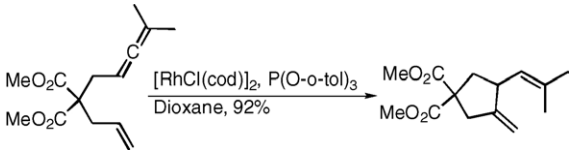

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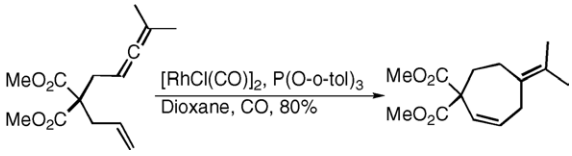
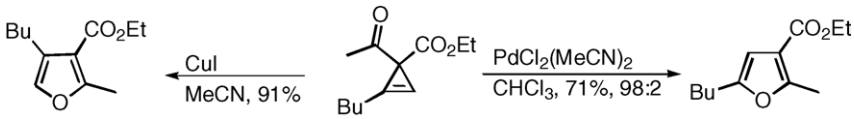


(307)

Palladium and copper-catalyzed regio-complementary ring-opening cycloisomerizations of cyclopropenyl ketones to give furanes (Eq. (308)) [1539]. Palladium catalyzed cycloisomerizations of alkylidenecyclopropyl ketones gave furanes in the presence and 4*H*-pyrans in the absence of sodium iodide (Eq. (309)) [1540]. Copper catalyzed a cycloisomerization with concomitant 1,2-migration of the sulfur group of 4-thiophenoxy-2-yne-1-ones and of the corresponding imine to give 3-thiophenoxyfuranes and pyrroles (Eq. (310)) [1541].



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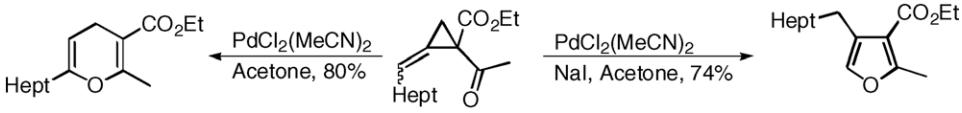

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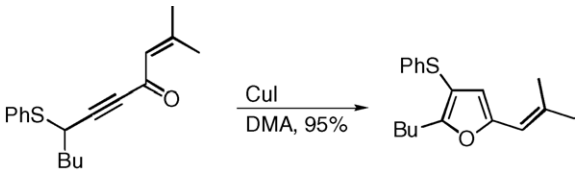
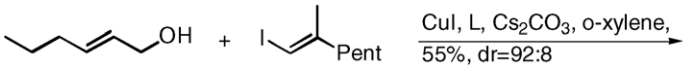
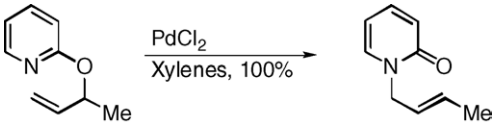


(310)

10. Miscellaneous isomerizations

Palladium catalyzed an enantioselective aza-Claisen rearrangement of allylic imidates [1542]. Copper catalyzed a sequential carbon–oxygen coupling-Claisen rearrangement (Eq. (311)) [1543]. Palladium catalyzed isomerization of alkenyl epoxides to 4-hydroxy-2-alkenoic acid esters [1544] or β,γ-unsaturated ketones depending on the substitution pattern [1545]. Palladium catalyzed a rearrangement of 2-allyloxypyridines to *N*-allyl-2-pyridones (Eq. (312)) [1546]. Palladium catalyzed the isomerization of *N*-sulfonylaziridines to *N*-sulfonyl ketimines (Eq. (313)) [1547]. Iron catalyzed an asymmetric rearrangement of *O*-acylated benzofuranones to give a new quaternary center (Eq. (314)) [1548].





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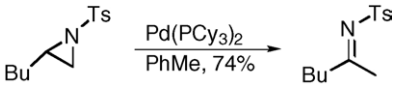
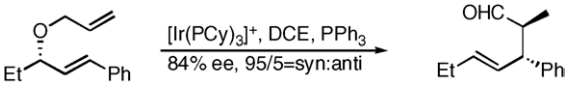


(313)

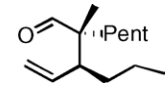
(314)

Ruthenium and iridium catalyzed the isomerizations of allylic ethers to *E*-alkenyl ethers, nickel catalyzed the isomerization of allylic ethers to *Z*-alkenyl ethers [1357,1549], and ruthenium catalyzed isomerization of allylic amines to enamines [1550]. Iridium catalyzed the isomerization of allylic ethers to alkenyl ethers followed by a highly stereoselective Claisen rearrangement (Eq. (315)) [1551]. Ruthenium and palladium catalyzed the isomerization of 2-propen-1-ylarenes to 1-propen-1-ylarenes [1552] and allylic amines to enamines [1312]. Palladium and iridium were used as catalysts to isomerize 2-propen-1-yl-C-glycosides to 1-propen-1-yl-C-glycosides [1475]. The latter reaction was used in a synthesis of repinotan [1412]. Isomerization to form a more substituted double bond catalyzed by iridium was described in a total synthesis of anhydromarasmone [1553].



(315)

Rhodium catalyzed the isomerization of allylic primary alcohols to aldehydes in water [1554]. Ruthenium catalyzed the isomerization of allylic alcohols to ketones followed by aldol and Mannich reactions in ionic liquids (Eq. (316)) [1555]. The mechanism of iron-catalyzed allyl alcohol to aldehyde isomerization was examined by DFT calculations [1556]. Iron catalyzed a tandem allylic alcohol to metal enolate isomerization followed by an aldol condensation (Eq. (317)) [1557]. A related reaction catalyzed by ruthenium was also reported [1558]. Ruthenium catalyzed an isomerization of *cis*- to *trans*-epoxides (Eq. (318)) [1559]. Alkene–alkyne couplings (Alder-ene reactions) to give 1,4-dienes were catalyzed by ruthenium [1560]. Palladium catalyzed a related asymmetric intramolecular reaction to give chiral spirocyclic quinolines (Eq. (319)) [1561]. Ruthenium catalyzed intermolecular alkyne-enol ether cross-metathesis reactions to give dienol ethers (Eq. (320)) [1562].



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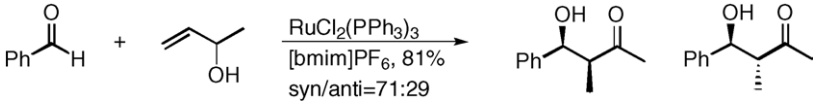

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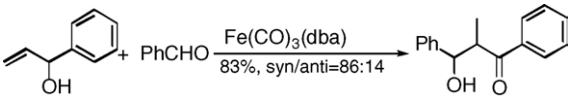

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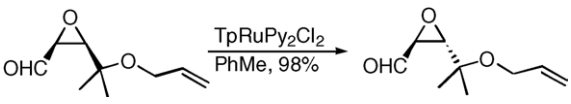

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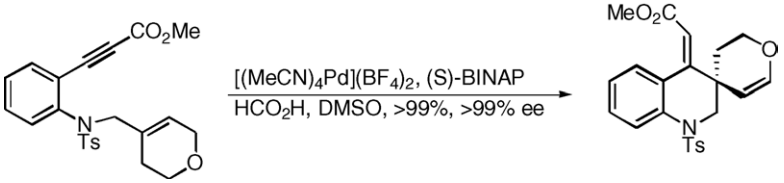

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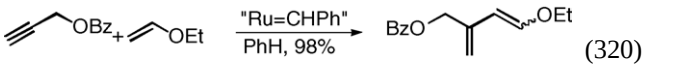

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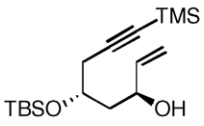
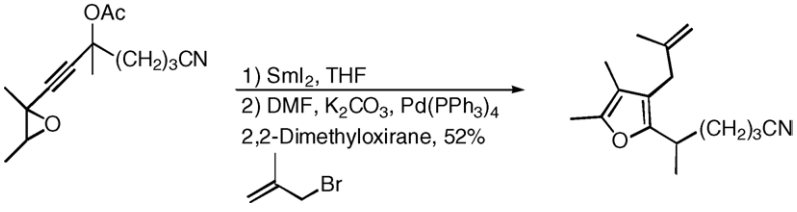


11. Miscellaneous carbocyclizations

Palladium catalyzed benzannulation of alkynes with allylic substrates to give polyfunctionalized benzenes [1563]. Palladium catalyzed a cyclization–allylation of 1-hydroxy-2,3,4-pentatrienes in the presence of an allylic bromide (Eq. (321)) [1564]. Depending on the palladium catalyst, either 2-hydroxycyclopentenones or cross-conjugated cyclopentenones are obtained from 2-alkoxy-1,4-diene-3-ones (Eq. (322)) [1565]. Copper-catalyzed asymmetric cyclizations of dialkenyl ketones to form 4,5-disubstituted-2-cyclopenten-1-ones [1566]. Indene derivatives were formed via hydrolysis of a zirconium-mediated coupling of aromatic ketones and alkynes (Eq. (323)) [1567].



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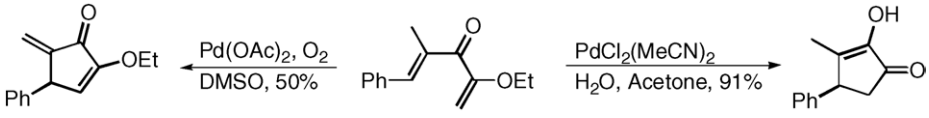

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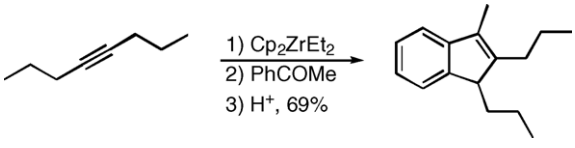
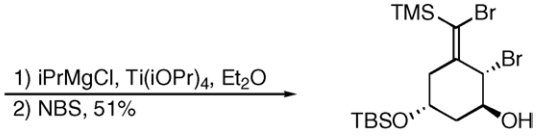


(323)

A titanium-mediated cyclization of 1,6-enynes was used to prepare 1α,25-dihydroxyvitamin D_3 and analogs (Eq. (324)) [286]. An asymmetric titanium-mediated cyclization of 2,7- and 2,8-enyn-1-ol derivatives followed by electrophilic quenching was described (Eq. (325)) [1568]. Titanium mediated a cyclization of enyne enol esters to give stereodefined bicyclo[3.3.0]octenes (Eq. (326)) [1569]. Tungsten catalyzed an endo-selective annulation of 6-silyloxy-1,2,5-trienes to give fused compounds (Eq. (327)) [1570]. Zirconium catalyzed an enantioselective formation of hydrindanes from trienes (Eq. (328)) [1571]. A zirconium mediated reductive cyclization of 1,7-dienes was used to prepare chiral cyclohexanes [1572].



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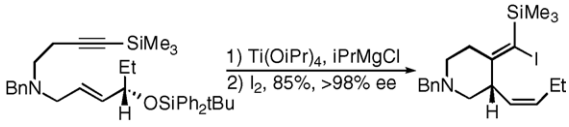

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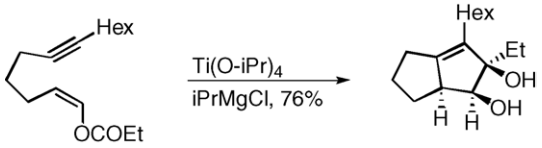

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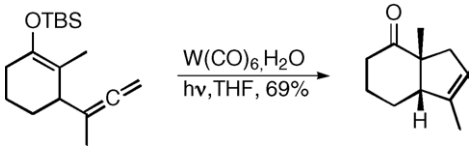

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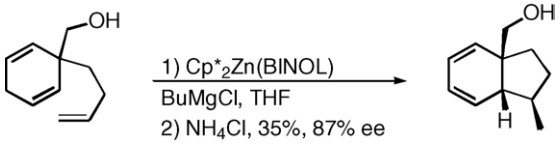
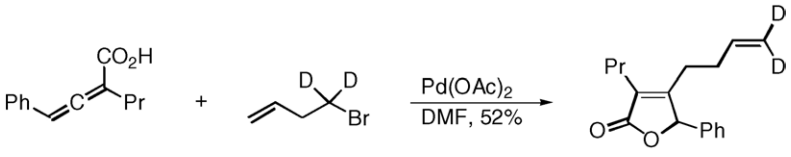


(328)

Palladium catalyzed a cyclization–Suzuki coupling of 1-bromo/chloro/methylcarbonate-2-en-7-ynes with aromatic boronic acids to give 5-membered products having stereodefined exocyclic double bonds [1573]. An interesting cyclization–alkene insertion of 2,3-dienoic acids with terminal alkene having a remote bromine was described (Eq. (329)) [1574]. Copper and gold catalyzed an interesting cycloisomerization of 2-(1-alkynyl)benzaldehyde with alkenes to give 1,2-dihydronaphthalenes (Eq. (330)) [1575].



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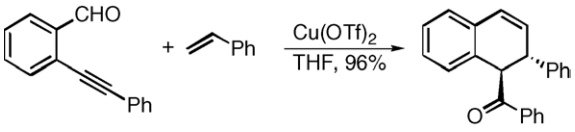
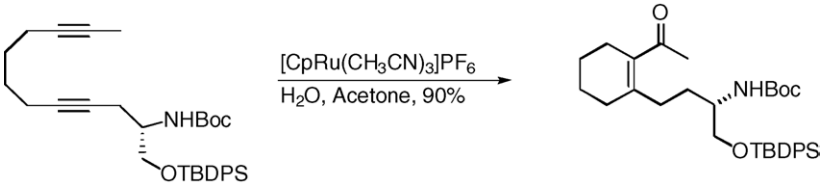


(330)

Ruthenium catalyzed annulations of 1,6- and 1,7-diynes in the presence of water to give ketone substituted unsaturated 5- and 6-membered rings [1576]. This reaction was used in a synthesis of (+)-cylindicines (Eq. (331)) [1577]. Cyclization of 1,6-enynes in the presence of ethene using a rhodium catalyst gave 5-membered ring having an exocyclic conjugated diene (Eq. (332)) [1578]. An optimization of carbocyclization nucleophilic trapping versus cycloisomerization of 1,3,8,10-dienes, catalyzed by palladium, was reported [1579]. Palladium catalyzed an asymmetric carbocyclization–coupling reaction affording spirocyclic compounds [1580].



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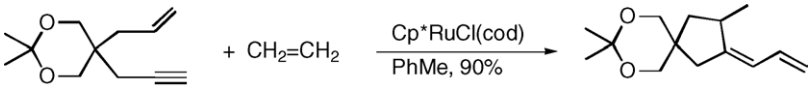
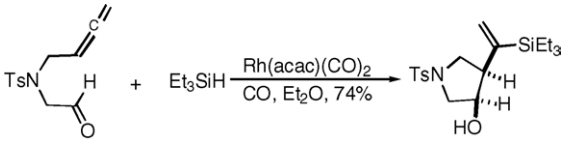


(332)

Rhodium catalyzed a silylative carbocyclization of 1,2-diene-7-ones or -als in the presence of triethylsilane (Eq. (333)) [1581]. Palladium catalyzed hydrosilylative cyclization of 1,6- and 1,7-diynes to form (*Z*)-1-methylene-2-silylmethylenecycloalkanes [1582]. The mechanism of the palladium-catalyzed cyclization of 1,6-dien-8-ol esters to give 5-membered rings was examined computationally [1583]. A palladium-mediated annulation of allene-tethered cyclohexadienes to give bicyclic compounds with defined stereochemistry was reported (Eq. (334)) [1584].



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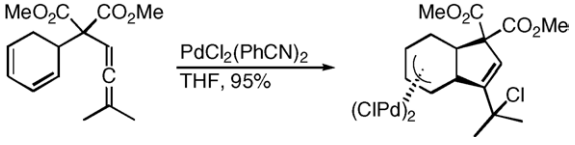
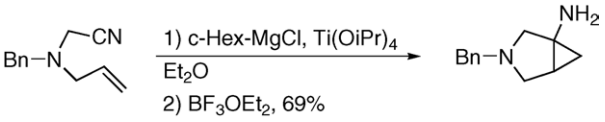


(334)

Titanium catalyzed a cyclization of unsaturated nitriles to give bicyclic cyclopropyl amines (Eq. (335)) [1585]. Platinum, palladium, gold, and copper-catalyzed hydroxy- or alkoxy-annulations of 1,6-enynes and 1-alkoxy-1,6-enynes (Eq. (336)) [1586–1588].



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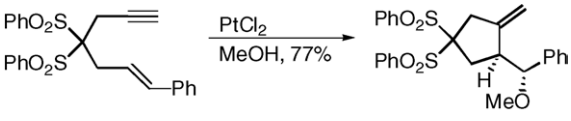
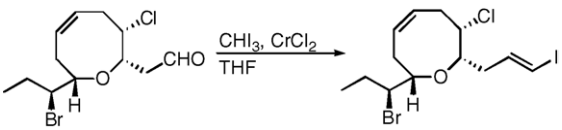


(336)

12. Formation of carbon–halogen bonds

Alkenyl iodides were prepared by the chromium-mediated Takai olefination of aldehydes using iodoform [1589]. This reaction was used in the synthesis of annonaceous acetogenins [418], C21–C26 fragment of superstolide A [48], lasonolide [100], E type I phytoprostanes [1590], oxazolomycin antibiotics [80], disorazole A_1 [439], and (*E*)- and (*Z*)-(+)-pinnatifidenyne (Eq. (337)) [444]. Tributyltindiiodomethane was also used to introduce an alkenyl stannane in a synthesis of isocembrene (Eq. (338)) [101].



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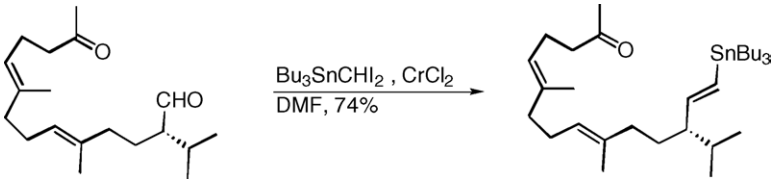


(338)

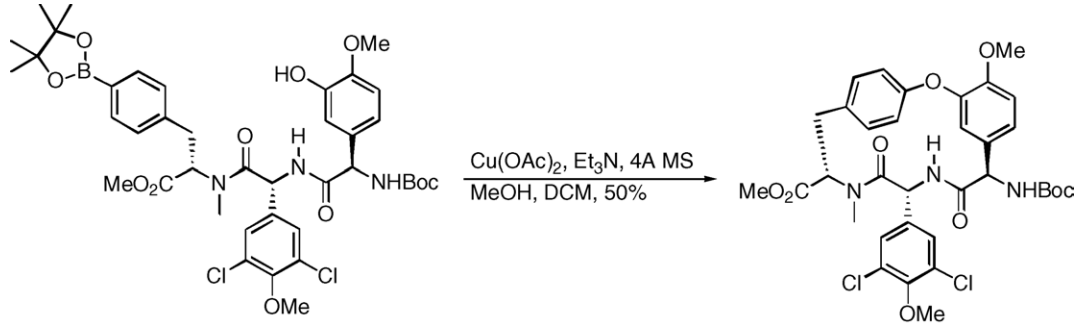
Palladium catalyzed an asymmetric dibromination of alkenes [1591]. An enantioselective fluorination of β-ketoesters using a palladium complex was described [1592]. Hydrozirconation of a terminal alkyne followed by addition of iodine or NBS to produce alkenyl iodides or bromides was used in synthetic applications toward (−)-akolactone A [359], 6*Z*,8*E*-heneicosadien-11-one [427], phorboxazole B [1593], khafrefungin analogs[317], and (+)-phomactin A [326]. Regioselective hydrozirconation–iodinations of internal alkynes were used in a synthesis of deoxyfusapyrone [283], (−)-callystatin A [5609], and apoptolidin [97].

Methylalumination of a terminal alkyne using trimethylaluminum and $ZrCp_2Cl_2$, followed by treatment with iodine to give a trisubstituted alkenyl iodide was used in synthesis of C1–C11-fragment of bafilomycin A_1 [1594], carbazomadurin A [68], 2-hydroxy-substituted decahydrophenanthrenes [1498], (9*Z*)- and (11*Z*)-8-methylretinals [314], (+)-rottnestol, (+)-raspailol A and B [79], phomactin A [166], and epolactaene [85]. A palladium-catalyzed hydrostannation–iodination sequence of a terminal alkyne was use in the synthesis of macrosphelide A [657], and a macrosphelide library [661].

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

Copper catalyzed the amination of aryl boronic acids and esters with aziridines [711], α-amino esters [1595], *N*-heteroaromatic compounds [1325,1596], 6*H*-pyrido[3,4-*b*]pyrazine-5-one [1597], and of aromatic boronic acids and trifluoroborates with amines [1598]. Copper mediated the *N*-9 arylation of purines using arylboronic acids [1599].

Copper catalyzed the coupling of phenols with aromatic boronic acids and esters to give diaryl ethers [1325,1596]. An intramolecular copper-mediated reaction was used to prepare L,L-cycloisodityrosine [796] and chloropeptin I (Eq. (339)) [103]. Copper also catalyzed the formation of symmetrical diaryl ethers via coupling of aromatic boronic acids in the presence of water [1600].



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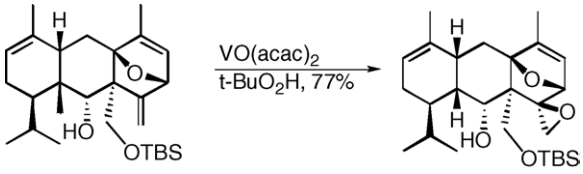
14. Formation of epoxides

Vanadyl acetylacetonate-catalyzed epoxidations of allylic and in some cases homoallylic alcohols using peroxides continued to be an invaluable tool in organic total synthesis [122]. Synthetic targets include, phytuberin [1601], cyclopentenone protaglandins [1602], (−)-aphanorphine [1603], eleutherobin (Eq. (340)) [1604], Sch 49028 [1605], and (−)-herbertenediol [1606]. Asymmetric epoxidation of homoallylic alcohols using $VO(OiPr)_3$ and a chiral ligand was employed in a synthesis of (−)-a-bisabolo and (−)-8-epi-a-bisabolol [1607]. Vanadyl acetylacetonate-catalyzed epoxidation-ring-expansions of furyl alcohols to give pyranones [1608] were used in synthesis of phomactin A [1609].



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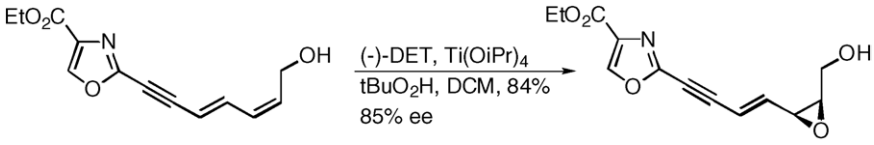
Sharpless enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continue to be extensively used in organic synthesis. For example, this reaction was used in synthetic approaches to laulimalide and analogs [1369,1610], (−)-nakamurol [1611], (−)-cassine [895], (−)-aspidospemine [1435], (+)-rogioloxepane A [422], scytophycin [1612], (+)-prelactone B [1613], 6-hydroxy-4*E*-sphingenines [1614], (+)-laurallene [1615], (−)-chrysolic acid and (+)-isofregenedol [1616],

nafuredin-γ [71], A–F segment of yessotoxin and adriatoxin [98], disorazole A_1 (Eq. (341)) [98], pseudoplexaurol [1617], heptopyranosides [1618], taurospongin A [1619], and (+)-1-deoxynojirimycin and (+)-castanospermine [1620]. Zirconium catalyzed a related enantioselective epoxidation of homoallylic alcohols using cumene hydroperoxide (Eq. (342)) [1621].



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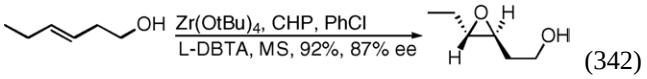

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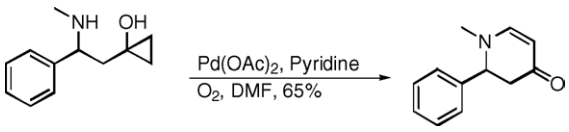

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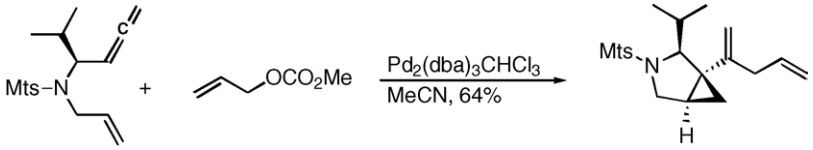

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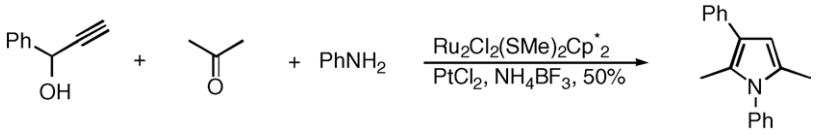

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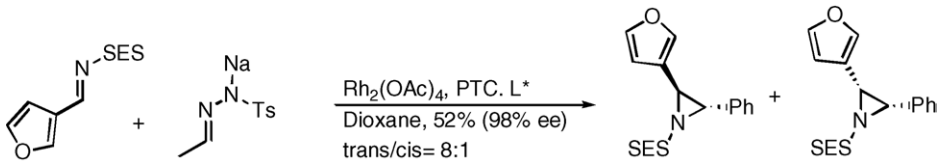

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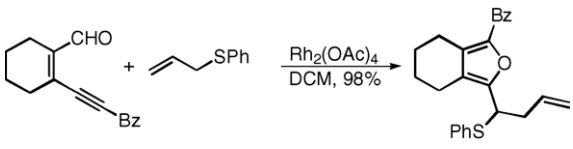

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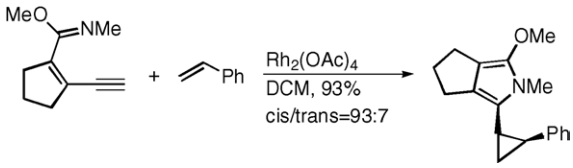
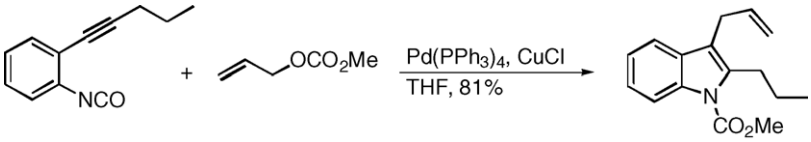


(348)

15. Formation of heterocycles

Miscellaneous reactions forming heterocycles not included in the previous sections are discussed here. Palladium catalyzed the oxidative transformation of β-aminocyclopropanols to 2,3-dihydro-1*H*-pyridin-4-ones (Eq. (343)) [1622]. Palladium catalyzed the formation of a chiral oxazoline via intramolecular *O*-alkylation of a 3-acetoxy-4-*N*-benzoylamino-1-alkene in a synthesis of (+)-spectaline 2 [1623]. Palladium catalyzed an interesting formation of 3-azabicyclo[3.1.0]hexanes from 1,2,7-trienes and allylic carbonates (Eq. (344)) [1624]. A ruthenium–platinum catalyst system was used to prepare furanes and pyrroles from reaction of 3-hydroxy-1-alkynes with ketones (Eq. (345)) [1625]. A rhodium-catalyzed asymmetric sulfur ylide mediated aziridination was developed and applied in a synthesis of the side chain of taxol (Eq. (346)) [1626]. Rhodium catalyzed the formation of (2-furylyl)carbenoids from ene-yne-carbonyl compounds followed by reaction with allylic sulfides and a [2,3]sigmatropic rearrangement of the formed sulfur ylide (Eq. (347)) [1627]. A related rhodium-catalyzed formation of (2-pyrrolyl)carbenoids from ene-yne-iminio ethers followed by intermolecular cyclopropanation was developed (Eq. (348)) [1628].

N-alkoxycarbonyl indoles were obtained upon reaction of 2-(1-alkynyl)arylisocyanates and allylic carbonates in the presence of a palladium–copper catalyst system (Eq. (349)) [1629]. A palladium–copper catalyst system was used to make a thienoindole (Eq. (350)) [1630]. Palladium catalyzed the annulation of 2-halo-*N*-alkynylbenzenamines in the presence of disubstituted amines to give 2,3-disubstituted indoles (Eq. (351)) [1631]. Ruthenium catalyzed a regioselective formation of indoles from an epoxide and an aromatic primary amine (Eq. (352)) [1632].



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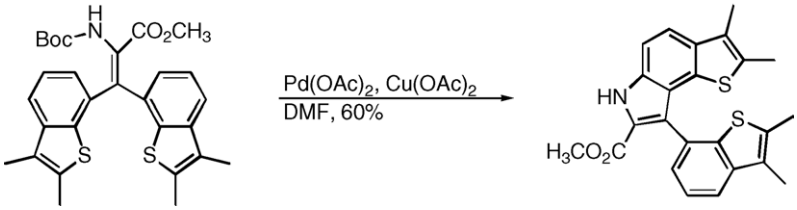

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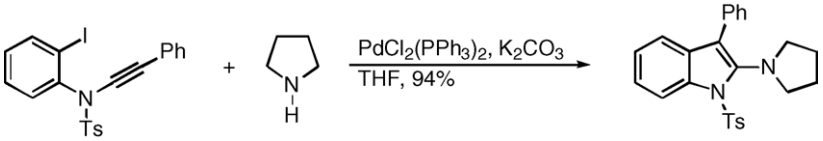

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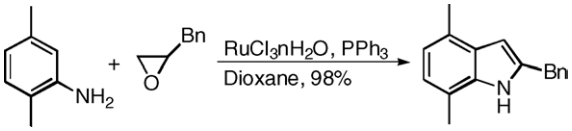
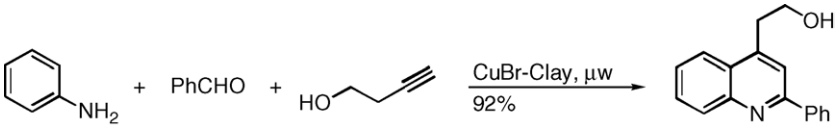


(352)

Copper catalyzed a microwave-accelerated annulation of intermediately formed benzaldehyde imines with terminal alkynes to give 2,4-disubstituted quinolines (Eq. (353)) [1633]. Ruthenium also catalyzed the formation of quinolines from 2-aminobenzylalcohols and secondary alcohols (Eq. (354)) [1634]. An interesting formation of quinolines from *N*,*N*-diallylbenzenamines in the presence of a cobalt catalyst and carbon monoxide was reported (Eq. (355)) [1635].



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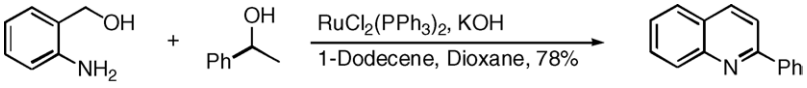

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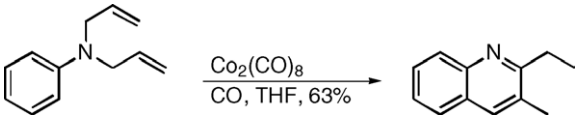

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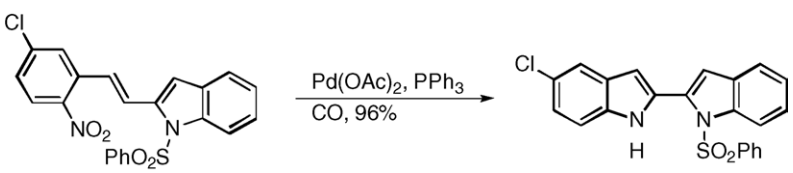
Palladium-catalyzed reductive *N*-heteroannulations of nitrostyrene derivatives were used to prepare tjipanazoles (Eq. (356)) [1636], 1*H*-indol-2-yl-1*H*-quinolin-2-ones [1637], 1,2-dihydro-4(3*H*)-carbazolones [1638], and bauerine A [1639]. Reductive annulation of Baylis–Hillman products using iron affords indoles and 4-quinolones (Eq. (357)) [1640]. Iron catalyzed an interesting annulation of 3-acetoxy-3-(2-nitrophenyl)-2-substituted-1-propenes to give 3-substituted quinolines (Eq. (358)) [1641]. Ruthenium catalyzed an annulation of aromatic nitro-compounds with conjugated dienes, in the presence of carbon monoxide, to give oxazines and *N*-arylpyrroles [1642]. Ruthenium catalyzed a reductive cyclization of nitrobenzenes with tris(3-hydroxypropyl)amine in aqueous solvent to form quinolines [1643]. A zirconium mediated cyclization to form a substituted tetrahydroquinoline was reported (Eq. (359)) [572].



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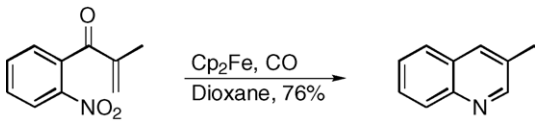

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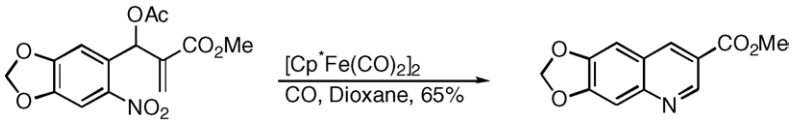

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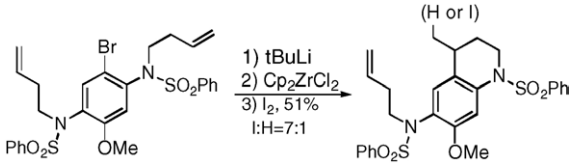
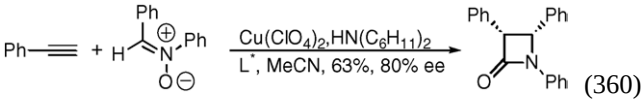


(359)

Copper catalyzed an asymmetric formation of β-lactams from reaction of alkynes with nitrones (Eq. (360)) [1644]. Gold and copper were efficient catalysts for the formation of functionalized pyridines from carbonyl compounds and propargyl amines (Eq. (361)) [1645]. Palladium catalyzed the formation of diazepine-2-ones from cyclization of 2-alkenylpyrrolidines and aryl isocyanates (Eq. (362)) [1646]. A dynamic kinetic asymmetric cyclization reaction between alkenyl aziridines and arylisocyanates was also reported [1647]. Palladium catalyzed a [3+3]cycloaddition of aziridines with intermediately formed trimethylenemethane complexes to give functionalized piperidines [1648]. This reaction was used in a total synthesis of (−)-doexynupharidine (Eq. (363)) [1649]. A palladium catalyzed [3+2]cycloaddition of trimethylenemethane to an α,β-unsaturated amide was used to prepare 5,5-fused proline derivatives [1650]. A related reaction of methylenecyclopropanes with *N*-tosylimines was used to prepare 3-methylenepyrrolidines [1651]. Palladium catalyzed the formation of polyheterocycles via intramolecular *N*-arylation–carbon–hydrogen-bond activation–aryl–aryl bond coupling (Eq. (364)) [1652].



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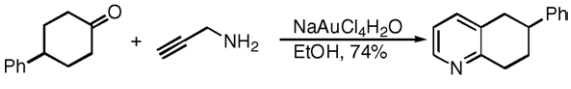

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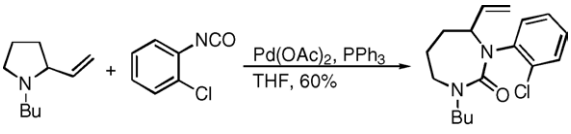

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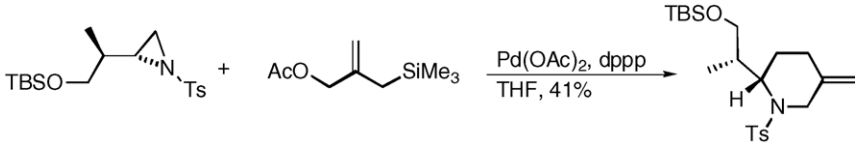

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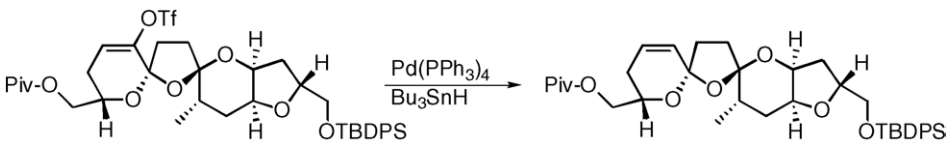
16. Formation of carbon–hydrogen bonds

A variety of functional groups were replaced by a hydrogen atom using palladium-catalyzed methodologies. Palladium complexes, together with a hydride source, such as ammonium formates and triethylsilane, catalyzed the reduction triflates and halides. Synthetic applications include, (−)-blestriarene C [1653], lignans [1654], azaspiracid-1 (Eq. (365)) [65], 12-alkoxybenzo[*c*]phenanthridine bases [1655], 3-arylisocoumarins [82], fragranol and grandisol (Eq. (366)) [1656], merrilactone A [1430], and altohyrtins [1657,1658].



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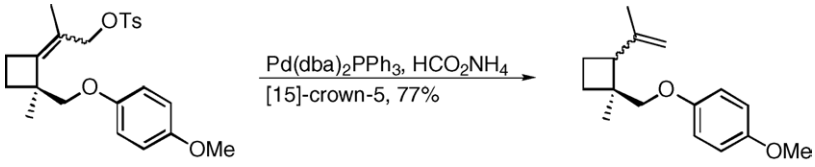

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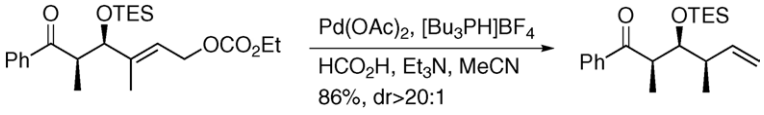
Palladium catalyzed regioselective lithium triethylborohydride reduction of an allylic mesylate [243]. Reductive removal of an allylic sulfonate employing lithium triethylborohydride was used in a synthesis of ubiquinones and menaquinones [1659]. A diastereoselective reductive removal of an allylic carbonate was described (Eq. (367)) [1660].



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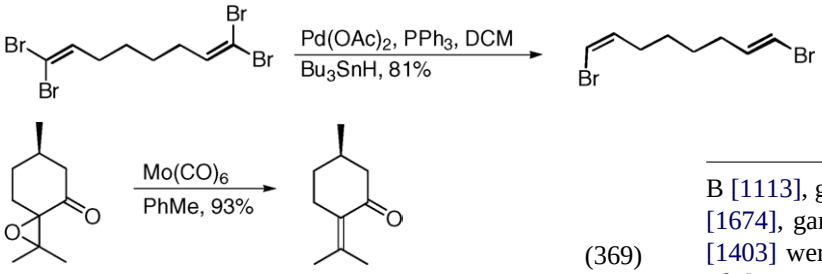
Monodebromination of 1,1-dibromo-1-alkenes was achieved using tributyltin hydride in the presence of a palladium catalyst forming *cis*-substituted bromoalkenes. This type of reaction was used in the synthesis of callyberynes (Eq. (368)) [389], oximidines [390], (+)-neoisoprelaurefucin [429], oxazolomycin antibiotics [80], and (+)-macquarimicin A [95]. A ruthenium catalyst was used to remove an aldehyde (decarbonylation) in a synthesis of indolizinoindole ring system [1661]. Molybdenum catalyzed a deoxygenation of α,β-epoxy ketones and esters to give α,β-unsaturated ketones and esters (Eq. (369)) [1662].



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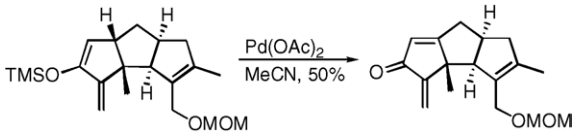
(369)

17. Formation of carbon–oxygen double bonds

A number of synthetic applications of the Saegusa reaction were published, for example applications toward illudins [1663], gymnocin A [304], pleurotellol and pleurotellic acid (Eq. (370)) [1664], illudosin [1665], (−)-xialenone A [1666], and galubulima alkaloid GB 13 (Eq. (371)) [1667].



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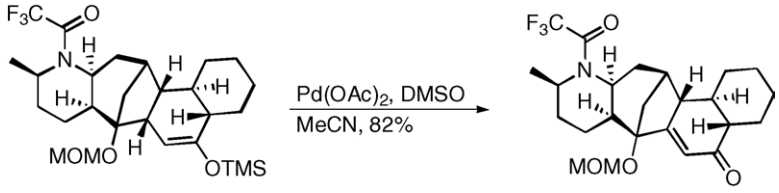
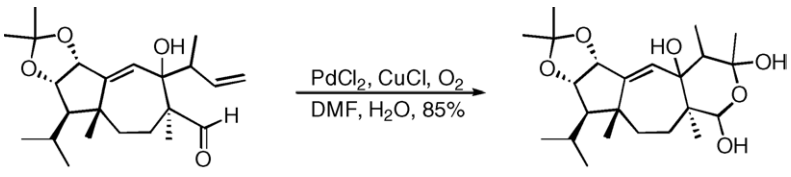


(371)

Titanium dioxide supported palladium nanoclusters were used in Wacker oxidations [1668]. Wacker-type oxidation, i.e., reaction of monosubstituted terminal alkenes with palladium(II) and water to afford methyl ketones, was utilized in a number of syntheses. For example, guanacasterpene A [123], buergerine F and G [1669], (−)-nakamurol [1611], leucascandrolide A [1670], (−)-cassine [895], toward reidispongiolide A [1671], cephalotaxine [1672], dolabelides A and B [1113], gelastanins [1673], JKLM fragment of ciguatoxin [1674], gambierol [86], and guanacastepene A (Eq. (372)) [1403] were prepared using a Wacker-type reaction in one of the steps. A regioselective Wacker-type oxidation of a cyclic alkene was used in a synthesis of calystegine alkaloids (Eq. (373)) [1365]. Wacker oxidation of an internal alkene was used in a synthesis of annuionone A (Eq. (374)) [1675].



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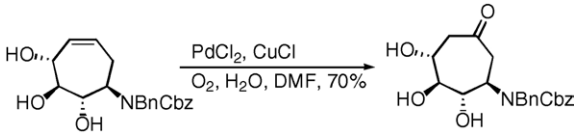

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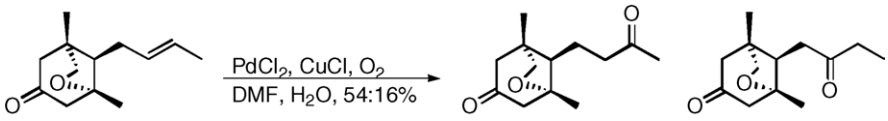
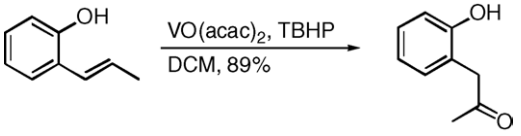


(374)

Abnormal Wacker oxidations to form aldehydes were observed using 1,5-dienes [1676]. This mode of reaction was used in a synthesis of *trans*-(+)-laurediol [1677] and (+)-rottnestol, (+)-raspailol A and B [79]. $VO(acac)_2$ catalyzed the epoxidation of 2-(1-alkenyl)phenols to give 2-hydroxybenzyl ketones (Eq. (375)) [1678]. Palladium catalyzed an oxidative rearrangement of 2-alkyl-3-hydroxy-3-alkenyl-isoindolin-1-ones to give 3-(2-oxoethylidene)isoindolin-1-ones (Eq. (376)) [1679].



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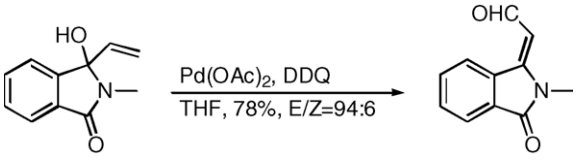
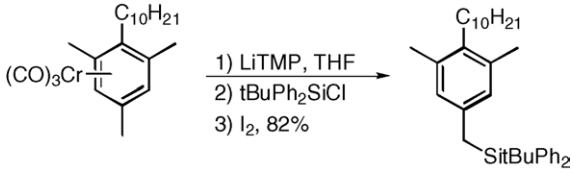


(376)

benzaldehyde imines were reported [1681]. Chelation-controlled nucleophilic addition to chromium tricarbonyl complexed benzaldehydes was developed [1682]. Complexed benzaldehyde imines were used in stereoselective [2+2]cycloadditions forming 2-azetidinones [1683]. Asymmetric deprotonation and benzylic alkylation of chromium tricarbonyl complexed benzylamine derivatives were reported [1684]. An enantioselective ring lithiation was used in a synthesis of korupensamine (Eq. (378)) [1685]. Axially chiral anilides were prepared in a related fashion [1686].



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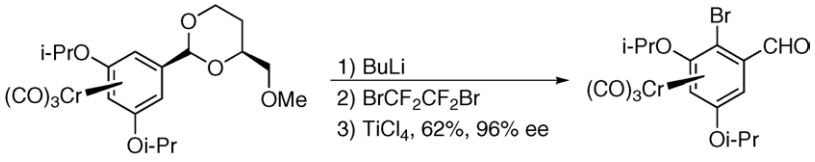
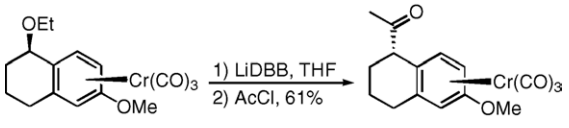


(378)

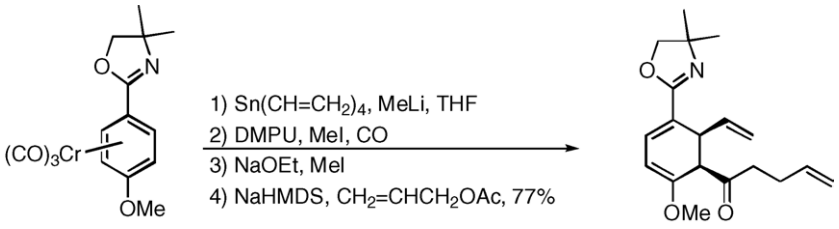
Electrophilic addition to a tetraline tricarbonyl chromium complex was a key step toward norcalamenenes (Eq. (379)) [1687]. Dearomatizations of arene tricarbonylchromium complexes by nucleophilic addition to the ring were reported (Eq. (380)) [1298]. This type of reaction was used to prepare both enantiomers of acetoxytubipofuran (Eq. (381)) [1688]. Substituted 1,4-benzodiazepines were prepared by complexation and aromatic nucleophilic substitution (Eq. (382)) [1689]. Intramolecular nucleophilic additions to arene iridium and ruthenium complexes were described (Eq. (383)) [1690].



(379)

18. Reactions of isolated transition metal complexes and titanium- and zirconium-intermediates

A number of transition metal-catalyzed reactions were published wherein metal complexes were allowed to react with 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. Selective functionalization of a tetralkylbenzene complex was described (Eq. (377)) [1680]. Diastereoselective additions of organozinc reagent to complexed



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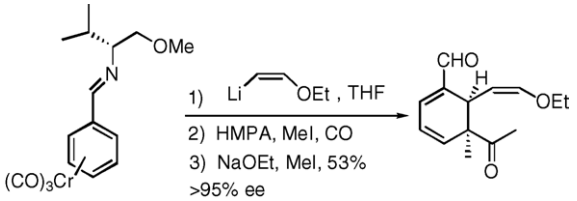

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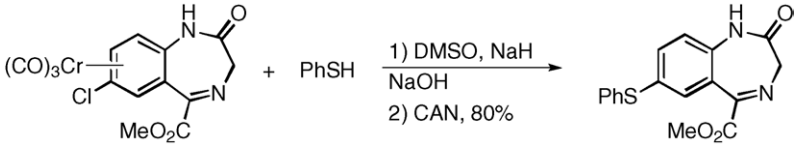

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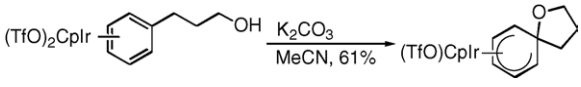
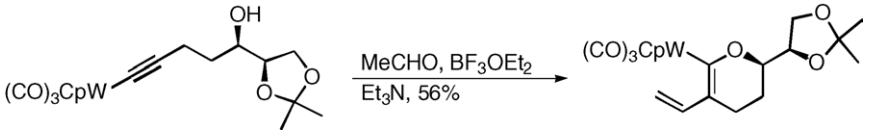


(383)

Tungsten–σ-alkyne complexes were used to prepare enantiopure tricyclic furanyl and pyranyl derivatives (Eq. (384)) [1691], (−)-epilitsenolide C2 and (−)-isodihydromahubanolide B [1692], and bicyclic lactone derivatives [1693]. Chromanes were prepared from osmium–η^2-arene complexes (Eq. (385)) [1694].



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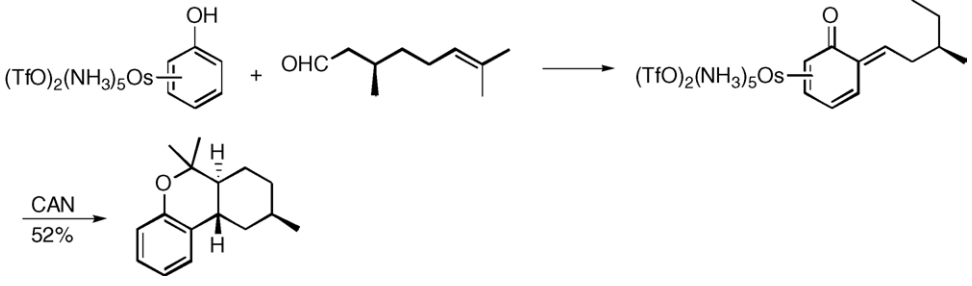
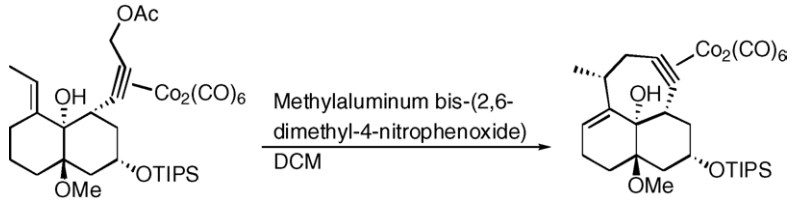


(385)

Intramolecular Nicholas-type reactions of cobalt alkyne complex stabilized cations [1695] were used to prepare cyclic ethers [1696], E′FGH fragment of ciguatoxin 1B [1697], JKLM fragment of ciguatoxin [1674], and ingenol (Eq. (386)) [1698]. Reduction of an ynone cobalt complex with triethylsilane was used in a synthesis toward polyprenylated acylphloroglucinols (Eq. (387)) [1699]. Double nucleophilic additions of Grignard reagents [1700] and enolates [1701] to dicobalt hexacarbonyl complexed 2-butyn-1,4-dial were described. Cobalt 2-ynal complexes undergo stereoselective pinacol coupling [1702]. Hexacarbonyl dicobalt complexed alkyne substituted cyclic enol ethers were rearranged to α-alkyl–β-alkynyl substituted cyclohexanones (Eq. (388))



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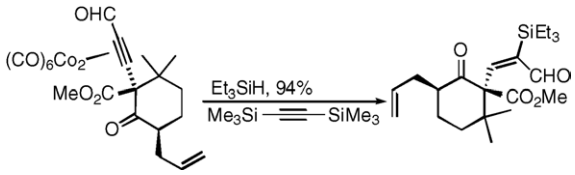
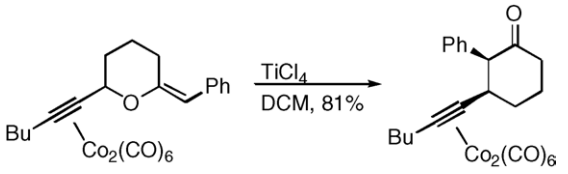


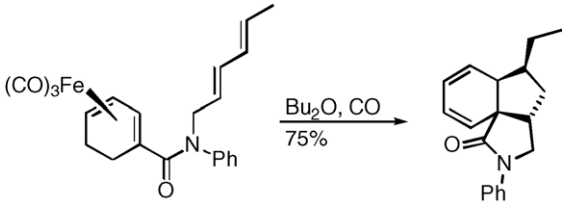
(387)

[1703]. Epimerization from the α- to the β-anomer of dicobalt hexacarbonyl complexed trimethylsilylethynyl substituted sugars was described [1226].



(388)

Iron tricarbonyl complexes continued to be used in organic synthesis. Cyclohexadiene iron tricarbonyl complexes undergo double cyclization with pendant conjugated dienes (Eq. (389)) [1704]. An ene-type cyclization using cyclohexadiene iron tricarbonyl complexes was reported (Eq. (390)) [1705]. Allylic zirconium reagents were added to cationic η^5-iron pentadienyl complexes [1706]. A sequential nucleophilic and electrophilic addition to iron cyclooctatetraene complex furnished *cis*-5,7-substituted cycloocta-1,3-dienes (Eq. (391)) [1707].



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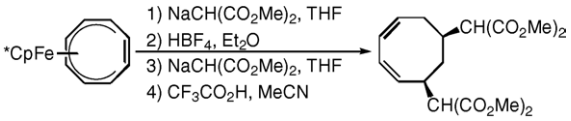
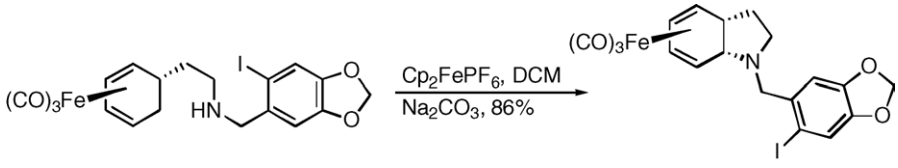


(391)

An iron tricarbonyl diene complex served as template for the synthesis of anhydrolycorinone and hippadine (Eq. (392)) [480] and a 2,4,6-cycloheptatrien-1-one complex was used for the synthesis of spirocyclic cycloheptatrienes (Eq. (393)) [1708]. Acyclic diene iron complexes were used in syntheses of cyclopropyldihydrophenylalanine (Eq. (394)) [1709] and segments of macrolactin A [1710,1711]. Carbazomycin G and H, mukonine, and mukonidine were prepared using cationic cyclohexadienyliron tricarbonyl complexes (Eq. (395)) [1712,1713].



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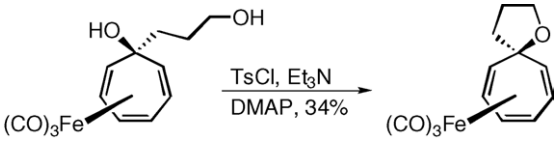

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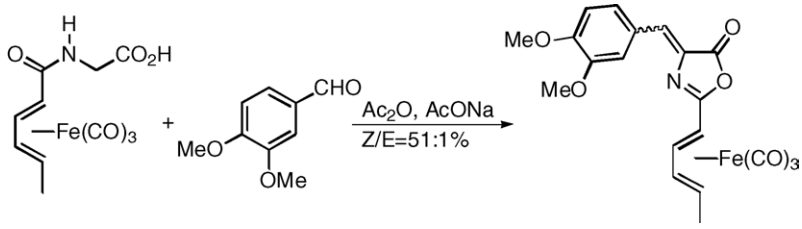

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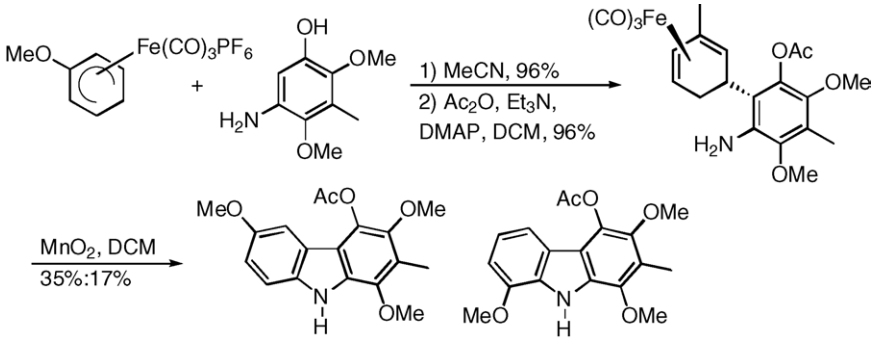

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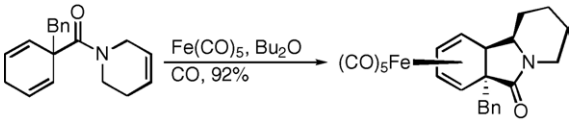
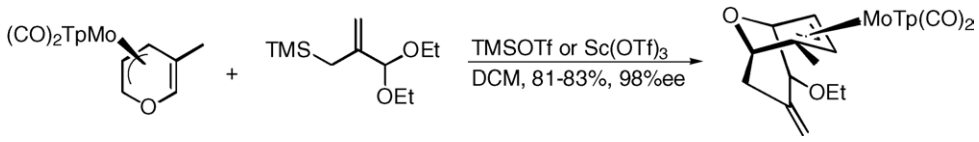


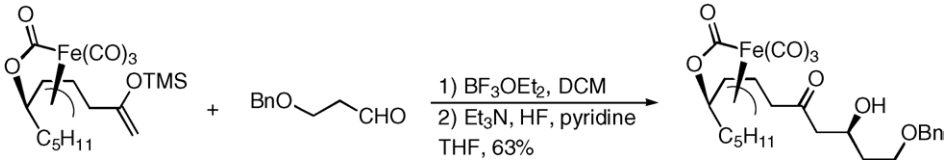
(390)

Iron η^3-allyl lactone complexes were used as templates in syntheses of taurospongin A [1714] and (−)-gloesporone (Eq. (396)) [1715]. Stereodefined 1,7-diols and 2,3-diene-1,7-diols can be obtained by reductive decomplexation of iron η^3-allyl lactone complexes using sodium naphthalenide



(400)

[1716]. A diastereoselective hydroxylation of an iron η^3-allyl lactone complexes having a silyl enol ether was reported (Eq. (397)) [1717]. Iron cyclopentadienyl complexes were used as templates for the preparation of alkenylcyclopropanecarboxylates Eq. (398)) [1718].



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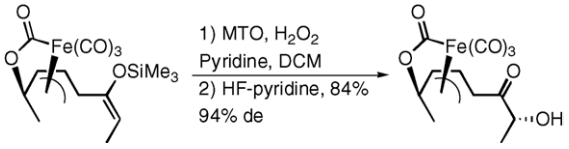

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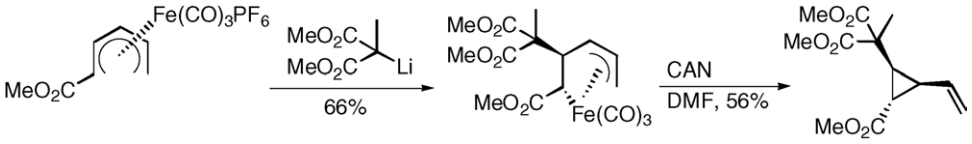
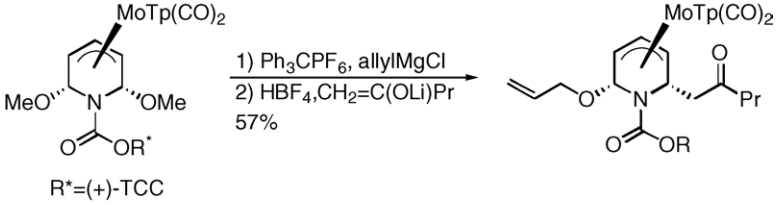
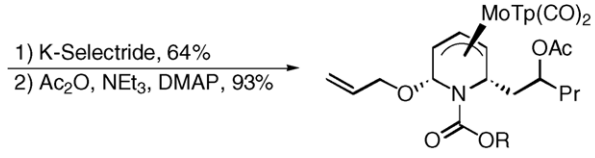


(398)

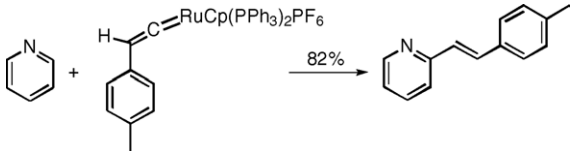
Chiral cyclic η^3-allyl molybdenum complexes were used as scaffolds for enantiocontrolled synthesis of (−)-dihydropinidine and (−)-andrachcinidine (Eq. (399)) [1719]. Related chiral pyranyl and pyridinyl η^3-allyl molybdenum complexes were used in regio- and enantioselective [5+3]cycloaddition reactions (Eq. (400)) [1720]. Palladium catalyzed the introduction of nitrogen, sulfur, and phosphorus substituents employing an η^5-1-chlorocyclohexadienyl manganese complex [1721].





(399)

Ruthenium mediated a regio- and stereoselective alkenylation of pyridine (Eq. (401)) [1722]. Cyclopalladated pyridine derivatives produced heterocyclic compounds upon reactions with allenes and alkynes (Eq. (402)) [1723].



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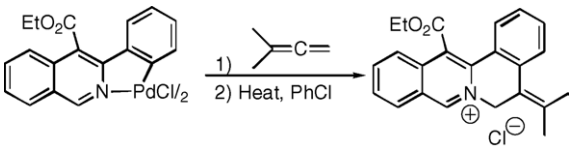
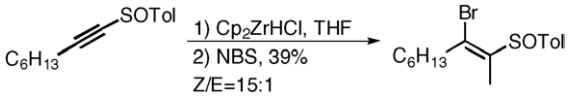


(402)

(Eq. (408)) [1729]. Synthesis of acetoxyodontoschismenol was achieved using sequential reactions of zirconium intermediates (Eq. (409)) [1730].



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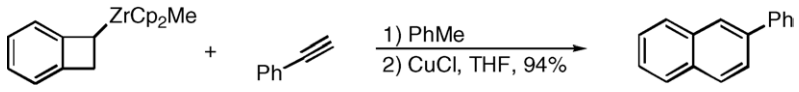

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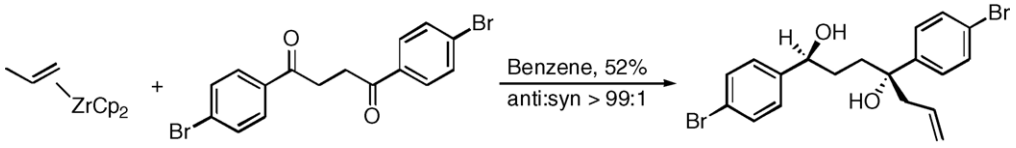

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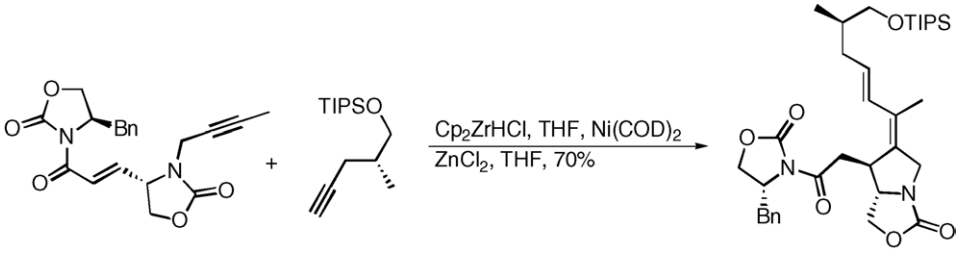
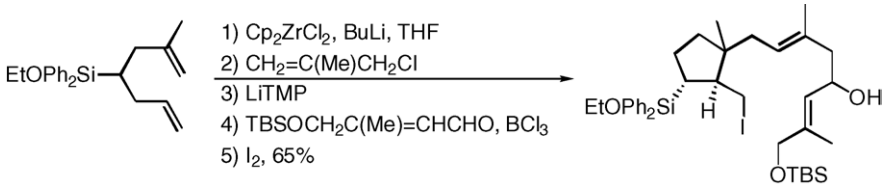
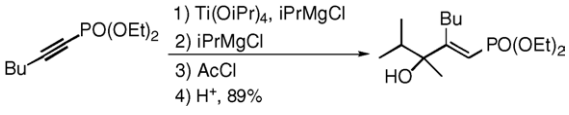


(408)



(409)

Intermediately formed titanacyclopropene phosphonates affords di- or trisubstituted alkenylphosphonates with high regio- and stereoselectivity upon reaction with Grignard reagents (Eq. (403)) [1724]. Coupling of alkynylphophonate titanium complexes with imines furnished 3-amino-1-alkenylphosphonates (Eq. (404)) [1725].



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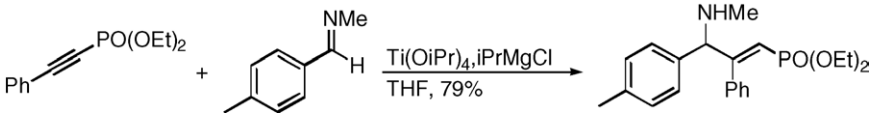
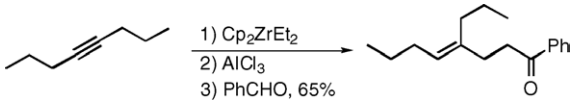


(404)

Stereodefined alkenyl sulfoxides and sulfones were prepared via hydrozirconation of 1-alkynyl sulfoxides and sulfones, respectively (Eq. (405)) [1726]. A zirconocene–copper-mediated coupling of benzocyclobutadiene with nitriles and alkynes affording naphthalenes, isoquinolines, and benzocines was developed (Eq. (406)) [1727]. Zirconocene–alkene complexes participated in a diastereoselective reduction–alkylation reaction of 1,4-diketones (Eq. (407)) [1728]. A nickel-catalyzed cyclization of alkynyl enones with an intermediately formed alkenylzirconium species was used in a synthesis of isodomic acid G

Zirconacyclopentenes undergo coupling reactions with aldehydes to give homoallylic ketones (Eq. (410)) [1731] and in the presence of a copper catalyst tetrahydrofuran derivatives (Eq. (411)) [1732]. Optically active indan-2-ols were prepared via reaction of zirconacyclopentadienes with alkynes [1733].



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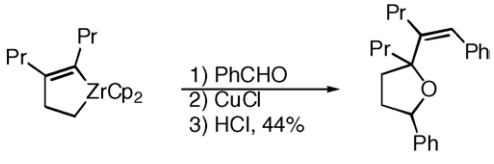

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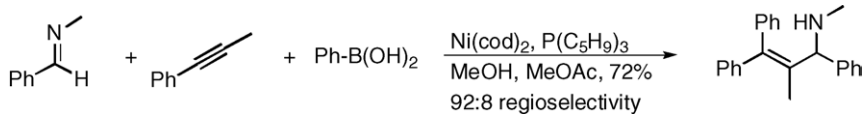

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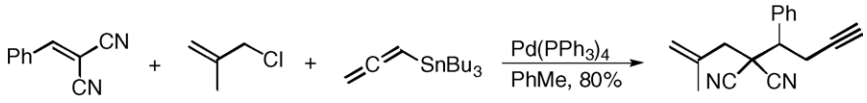

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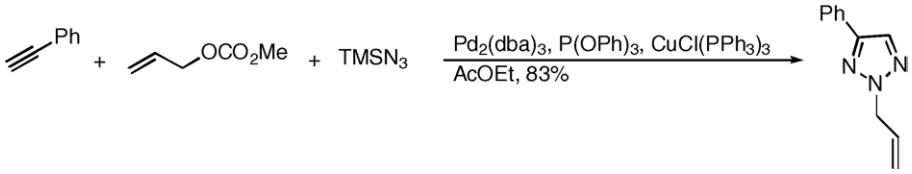

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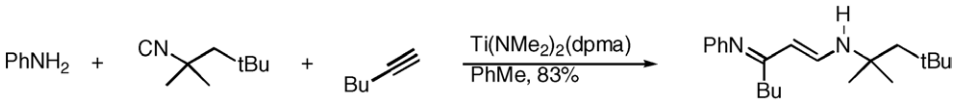
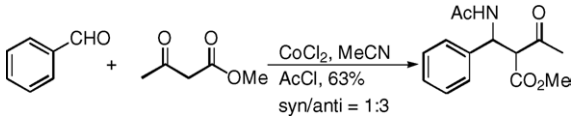


(416)

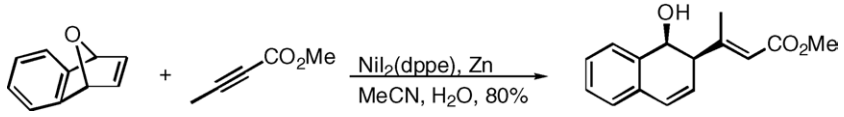
19. Miscellaneous reactions

Cobalt catalyzed a three-component coupling between an aromatic aldehyde and 1,3-dicarbonyl compound, and a nitrile to give β-acetamido carbonyl compounds (Eq. (412)) [1734]. Nickel catalyzed a coupling of alkynes, imines and organoboron reagents to give allylic amines (Eq. (413)) [1735]. A three-component palladium-catalyzed coupling of arylethylidene malonitriles, allylic chlorides, and allenylstannanes to give 1,7-enynes was described (Eq. (414)) [1736]. A palladium–copper catalyst system was used in a three-component reaction of a terminal alkyne, trimethylsilylazide, and an allylic carbonate affording allyltriazole (Eq. (415)) [1737]. Titanium catalyzed a three-component coupling of an amine, an alkyne, and an isocyanide to give α,β-unsaturated β-iminoamines (Eq. (416)) [1738].



(412)

Nickel catalyzed a regio- and diastereoselective reductive coupling of propiolates with bicyclic alkenes (Eq. (417)) [1739]. Palladium catalyzed an oxidative dimerization ic cyclization coupling reaction to give bibutenolides (Eq. (418)) [1740]. A copper-mediated 1,2-metalate rearrangement was used in a synthesis of (+)-manoalide [660]. Nickel catalyzed the coupling of 1,3-dienes with Grignard reagents and chlorosilanes (Eq. (419)) [1741].



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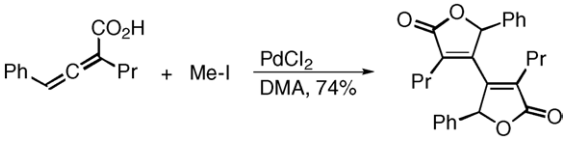

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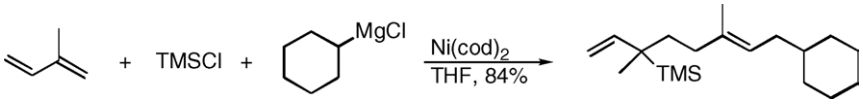
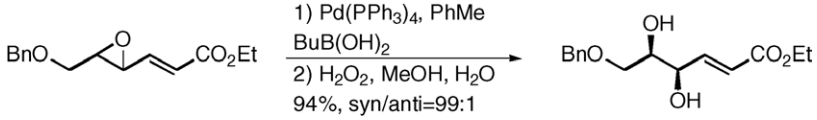


(419)

Palladium catalyzed an interesting epoxide ring opening in the presence of an alkyl boronic acid leading to vicinal diols having two inverted stereocenters (Eq. (420)) [1742]. Iridium-catalyzed direct borylations of aromatic compounds using pinacolborane or dipinacolborane [475,1743–1746]. A direct borylation of terminal alkenes catalyzed by rhodium was developed (Eq. (421)) [1747]. Palladium catalyzed the stereospecific formation of *E*- or *Z*-alkenylphosphonates from reaction of *E*- or *Z*-alkenylboronic esters with triethylphosphite [1748].



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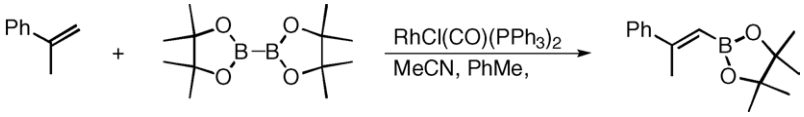
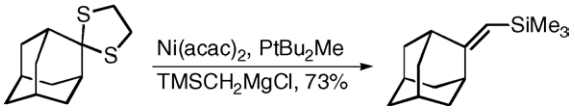


(421)

Iridium catalyzed a direct *t*-butyldifluorosilylation of aromatic carbon–hydrogen bonds [341]. A related *ortho*-silylation of benzaldehyde imines catalyzed by rhodium was described [1749]. Palladium catalyzed disilylation of aromatic compounds with cyclic disilanes [1750].

A palladium–indium catalyst system was used to form alkenes from halohydrins [1751]. Nickel catalyzed the formation of alkenes from dithioacetals and Grignard reagents (Eq. (422)) [1752]. Iridium catalyzed the dehydrogenation of tertiary amines to form enamines [1753]. Palladium catalyzed asymmetric Bayer–Williger reactions [1754]. Ruthenium catalyzed a β-alkylation of secondary alcohols using a primary alcohol (Eq. (423)) [1755].



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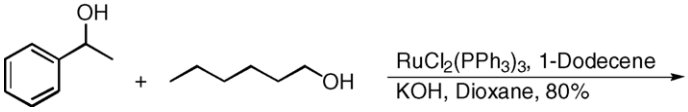
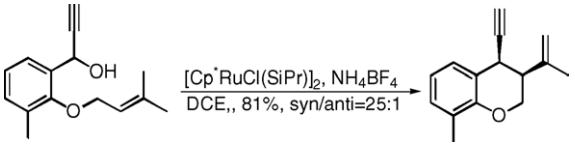


(423)

Rhodium catalyzed the formation of 1,5-enynes from propargylic alcohols and alkenes (Eq. (424)) [1756]. Ruthenium catalyzed a carbon–carbon cleavage of propargylic alcohols to form a terminal alkene and carbon monoxide [1757]. Palladium catalyzed an oxidation–reduction reaction of aromatic iodides and bromides having a tertiary benzylic group (Eq. (425)) [1758].



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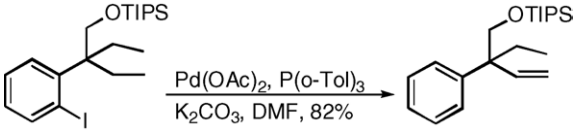

(425)

20. Reviews

The following reviews appeared in 2003:

- Transposition of allylic alcohols into carbonyl compounds mediated by transition metal complexes [1759].
- Rhodium-catalyzed carbon–carbon bond-forming reactions of organometallic compounds [1760].
- Palladium-assisted routes to nucleosides [1761].
- Palladium-catalyzed alkynylations [1762].
- Asymmetric hydroalkenylation reaction [1763].
- Catalytic enantioselective C–H activation by means of metal-carbenoid-induced C–H insertion [1764].
- Asymmetric transition metal-catalyzed allylic alkylations: applications in total synthesis [1765].
- The asymmetric intramolecular Mizoroki–Heck reaction in natural product synthesis [1766].
- Enantioselective catalytic aziridinations and asymmetric nitrene insertions into CH bonds [1767].
- Combination of enzymes and metal catalysts. A powerful approach in asymmetric catalysis [1768].
- Polymer-supported organic catalysts [1769].
- Construction of pyridine rings by metal-mediated [2+2+2]cycloaddition [1770].
- Transition metal-catalyzed reactions in steroid synthesis [1771].
- Nucleophilic transition metal-based cyclization of allenes [1772].
- Evolution in the palladium-catalyzed cross-coupling of sp- and sp^2-hybrizized carbon atoms [1773].
- The Pauson–Khand reaction: the catalytic age is here! [1774].
- Recent developments in olefin cross-metathesis [1775].
- Molybdenum and tungsten imido alkylidene complexes as efficient olefin-metathesis catalysts [1776].
- Novel substrates for palladium-catalyzed coupling reactions of arenes [1777].
- Modern synthetic methods for copper-mediated C(aryl)–O, C(aryl)–N, and C(aryl)–S bond formation [1778].
- Palladacyclic catalysts in C–C and C–heteroatom bond-forming reactions [1779].
- Enyne metathesis catalyzed by ruthenium carbene complexes [1780].
- The synthesis and applications of heterocyclic boronic acids [1781].
- Selectivity in rhodium(II) catalyzed reactions off diazo compounds: effects of catalyst electrophilicity, diazo substitution, and substrate substitution. From chemoselectivity to enantioselectivity [1782].
- Cyclizations involving attack of carbo- and heteronucleophiles on carbon–carbon π-bonds activated by organopalladium complexes [1783].

- Catalytic multistep reactions via palladacycles [1784].
- Palladium-catalyzed intramolecular arylation reaction: mechanism and application for synthesis of polyarenes [1785].
- New advances in selected transition metal-catalyzed annulations [1786].
- Renaissance of Ullmann and Goldberg reactions: progress in copper-catalyzed C–N–, C–O–, and C–S-coupling [1787].
- The catalytic hydroamination of alkynes [1788].
- Toggling enantioselective catalysts: a promising paradigm in the development of more efficient and versatile enantioselective synthetic methodologies [1789].
- The Pauson–Khand reaction, a powerful synthetic tool for the synthesis of complex molecules [1790].
- Transition metal-catalyzed borylation of alkanes and arenes via C–H activation [1791].
- Stereoselective accesses to enantioenriched allyl-, allenyl-, and propargyl-silanes via Si–Si bond activation by palladium–isocyanide catalysts [1792].
- Heck reactions with various types of palladium complex catalysts: application of multiphase catalysis and supercritical carbon monoxide [1793].
- Recent development in the synthesis of heterocyclic derivatives by PdI_2-catalyzed oxidative carbonylation reactions [1794].
- Phospha-palladacycles and *N*-heterocyclic carbene palladium complexes: efficient catalysts for CC-coupling reactions [1795].
- Palladium-catalyzed cross-coupling reactions and electrocyclizations: efficient combinations for cascade reactions [1796].
- Reactivity control in palladium-catalyzed reactions: a personal account [1797].
- Efficient coupling of heteroaryl halides with arylboronic acids in the presence of a palladium–tetraphosphine catalyst [1798].
- Triallyl(aryl)silanes serve as convenient agents for silicon-based cross-coupling reactions of aryl halides [1799].
- Carbon–carbon bond formation with electrogenerated nickel and palladium complexes [1800].
- Sequential metathesis in oxa- and azanorbornene derivatives [1801].
- Group-IV metal complexes as hydroamination catalysts [1802].
- Pd-assisted multicomponent synthesis of heterocycles [1803].
- Non-metathetical behavior patterns of Grubbs' carbene [1804].
- Mechanistic aspects of transition metal-catalyzed 1,6-diene and 1,6-enyne cycloisomerization reactions [1805].
- Palladium-catalyzed reactions in solid-phase organic synthesis [1806].
- Palladium-catalyzed oxidation of primary and secondary alcohols [1807].

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