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Transition metals in organic synthesis: highlights for the year 1999

Björn C.G. Söderberg *

Department of Chemistry, West Virginia University, Morgantown, WV 26506-6045, USA

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Contents

Abstract

A review with 1621 references to transition metal catalyzed or mediated carbon–carbon bond forming reactions and functional group preparations.

Keywords: Transition metals; Organic synthesis; Catalysis

* Tel.: +1-304-293-3435.
E-mail address: bjorn.soderberg@mail.wvu.edu (B.C.G. Söderberg).

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1. General comments

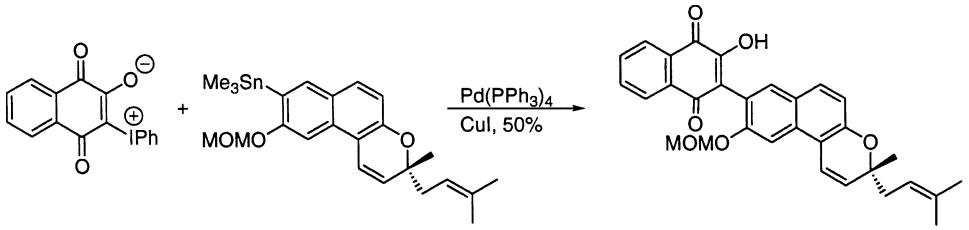
This survey highlights the use of transition metals in organic synthesis for the year 1999. A fairly comprehensive literature coverage with extensive citations is presented. The field of organometallic chemistry continued to expand exponentially in 1999. Considering the large number of papers published, a complete review of all reported reactions employing transition metals is outside the scope of this review. 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 by Professor James Herndon although decompositions of diazo compounds were included herein. Most carbonylations or hydroformylations were included apart from reactions used in total synthesis.

2. Carbon–carbon bond-forming reactions

2.1. Alkylation

2.1.1. Alkylation of organic halides, tosylates, triflates, nonaflates, iodonium salts, epoxides, and aziridines

Coupling reactions of tin reagents with a variety of electrophiles continue to be developed. This reaction is generally called the Stille reaction, and this name will be used herein. Palladium-catalyzed Stille type reactions in supercritical carbon dioxide were reported [1]. A polymer supported palladium catalyst was used in aqueous media for the coupling reaction [2]. A palladium carbene complex catalyzed the coupling of aryl halides with aryl tin reagents [3]. Highly fluorous tin compounds rapidly coupled with aryl halides under microwave irradiation [4]. Addition of tetrabutylammonium fluoride (TBAF) facilitated the palladium-catalyzed coupling of aryl halides to tin reagents [5]. Copper(I) chloride catalyzed the coupling between heterocyclic tin reagents and allylic halides [6]. Aryl chlorides were used in Stille couplings with vinyl stannanes [7]. Coupling reactions of polymer bound aryl halides [8–10] and polymer bound tin reagents [11] were reported. Stille coupling of iodonium [12–14] and heterobenzylic sulfonium [15] salts were developed (Eq. (1)) [16]. Vinyl and aryl nonaflates [17,18] and vinyl dialkyl phosphates [19–21] were used in Stille couplings. Coupling of tin reagents with bromomethylborinates was reported (Eq. (2)) [22].



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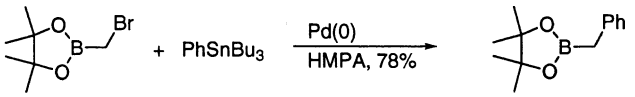
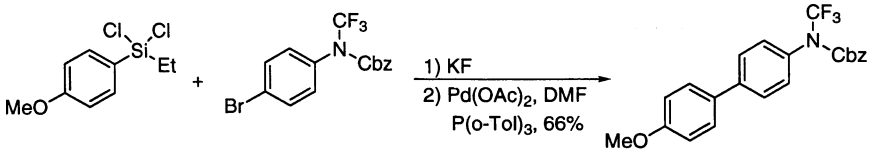


(2)

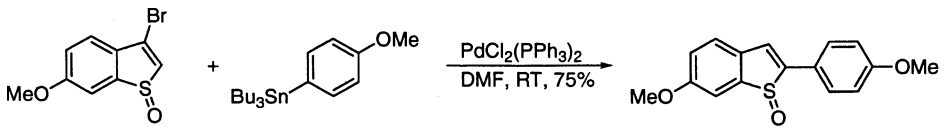
Some more unusual coupling reactions were developed. Palladium complexes catalyzed the coupling reactions of aryl and vinyl triflates with organobismuth dialkoxides [23] and organoindium compounds [24]. All three organic substituents were efficiently transferred in the latter case. Palladium also catalyzed a room temperature coupling of aromatic and heteroaromatic lead triacetates with iodonium salts [25]. Reaction of α-phenyl tributyl stannyl selenide with aryl and alkyl halides afforded diaryl or alkylaryl selenides [26]. Palladium and nickel catalyzed the coupling of trialkyl aluminum reagents with aryl bromides [27]. A number of silicon reagents were developed as alternatives to organotin compounds. Palladium catalyzed the coupling of aryl (fluoro)silacyclobutanes [28], alkenylsilacyclobutanes [29], tetrabutylammonium triphenyldifluorosilicate [30], phenyl, vinyl, and allylsiloxanes [31], dimethyl aryl or alkenyl silanols [32], phenyl trimethylsilane [33], and aryl alkyl dichlorosilanes (Eq. (3)) [34]. The reactions were usually performed in the presence of a fluoride source.



(3)

The palladium(0)-catalyzed coupling of aryl halides and triflates with a variety of aryl, alkynyl, vinyl, methyl, and benzyl tin reagents was extensively used in organic synthesis [35–58]. Vinyltin reagents coupled with acyl chlorides or aryl halides in the presence of carbon monoxide to form vinylketones [59–62]. Vinyl triflates prepared from acetoacetate derivatives were coupled with tin compounds [63]. Palladium catalyzed the coupling of acid chlorides with trimethylsilyltributyltin to give acyl silanes [64]. Two different groups were introduced using 1,2-bis(trialkylstannyl)ethyne [65,66] and 1,2-bis(trialkylstannyl)ethene [67,68]. Unsymmetrically substituted *Z*-1,2-bis(trialkylstannyl)alkenes coupled with iodonium salts, stereo- and regioselectively, at the least substituted terminus of the alkene [69]. Palladium-catalyzed cross coupling of hexaalkylditin reagents with heterocyclic and vinyl halides [70,72] and aryl triflates [73] furnished organotrialkyltin reagents suitable for Stille type reactions. Palladium and copper catalyzed the cross-coupling and carbonylative cross-coupling of organotellurium compounds with organostannanes [74]. Vinyl triflates having an allylic epoxide present were coupled with tin reagents [75].

Heterocyclic halides and triflates were used as electrophiles in reaction with aryl- and vinylstannanes in a number of cases. For example, reactions of 1,2,3,5-tetrahydroindolizine-7-one-6-triflate [76], 5-bromo-2(*H*)-pyridazin-3-one [77], 4-iodo-β-carboline [78], 6-bromo-1,4-dihydro[1,8]naphthyridine-4-one [79], 7-deaza-7-iodopurine-2′-deoxyribose [80], 7-bromo-3-methyl-4,5-dihydroisoxazolo[4,5-*c*]pyridine-4-one [81],7-bromoindole [82], 2-bromothiophene [83], 2-iodoindol [84], 3-bromothiopheneoxide with concurrent rearrangement (Eq. (4)) [85], 7-iodoisoxzolopyridone [86], bromoquinolizinium salts [87], 4-bromofuran-2(5*H*)-one [88], 6-chloropurine [89], and 4-chloromethylene-2-phenyl-5(4H)oxazolone (Eq. (5)) [90] were reported.



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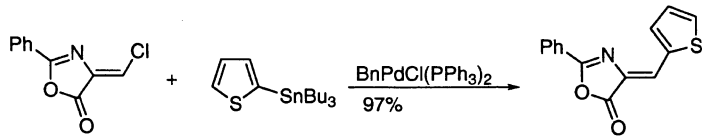
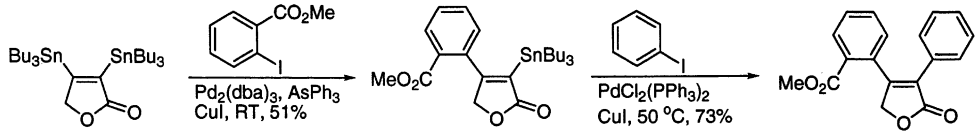


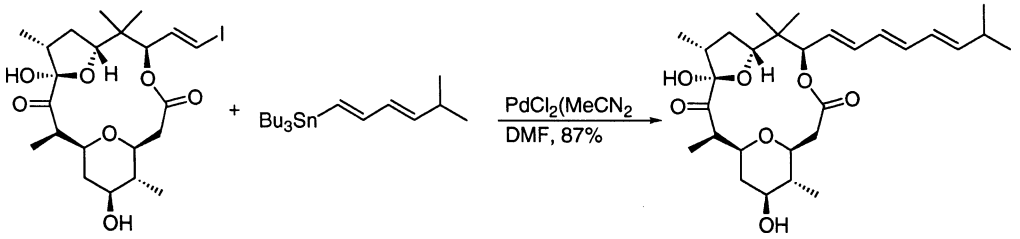
(5)

A plethora of heterocyclic stannanes such as 2-, 3-,and 4-pyridinyl [91–105], 2,6-ditinpyridinyl [106,107], 2- and 5-thiazolyl, 2-benzothiazolyl, 2-indolyl [108,109], 2-oxazolyl [110], 2-furyl [111], 7-aza-3-indolyl [112], 2-thienyl [113–115], 2,5-ditinthienyl [116], 2,5-ditinpyrazinyl [117], tetrathiafulvenyl [118], 3-pyridyl, and 3-quinolyl [119], 2-pyridyl, 2-quinolyl, and 2-thienyl [120], 2- and 3-thienyl, 2-furyl, 2-thiazolyl, 2-benzothienyl, and 2-indolyl [121], 3-chloro-pyrazine [122], and tetrathiafulvalene [123] were used in Stille type couplings with a variety of aromatic and heteroaromatic halides and triflates. 5-Stannyluracil coupled with iodonium salts. The reagent polarity was reversed; 5-iodonium salts coupled with vinyl- and alkynylstannanes [124]. Palladium-catalyzed reaction of an α,β-ditindihydrofuran-2-one was described. The β-position was shown to be the more reactive and two different groups can sequentially be introduced (Eq. (6)) [125].



(6)

Reactions of vinyl halides and triflates with aryl and heteroaryl tin reagents were proven useful in organic synthesis [126]. Stille type cross-coupling of vinyl stannanes with benzyl halides [127], propargyl chlorides [128], and vinyl halides or triflate was used in a large number of synthetic applications [129–155]. A selection of examples can be seen in Eq. (7) [156], Eq. (8) [157], Eq. (9) [158], Eq. (10) [159]. Carbonylative cross-coupling was also been employed in synthesis (Eq. (11)) [160].



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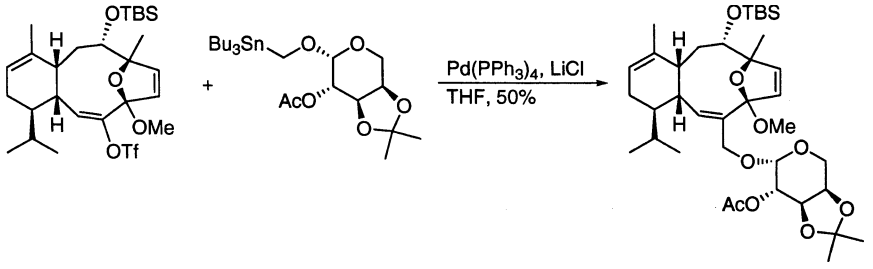

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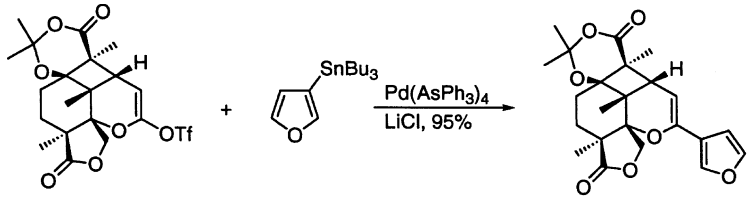

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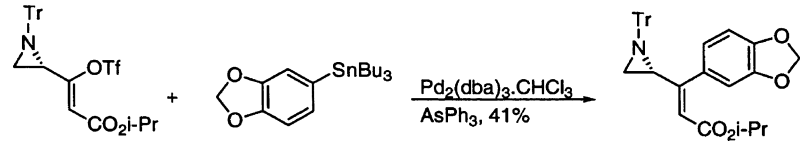

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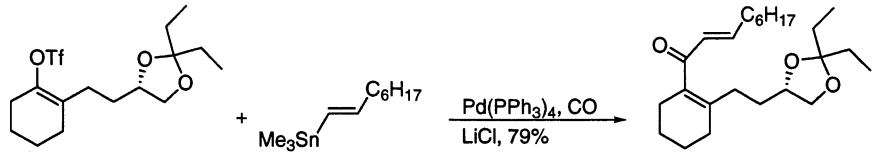
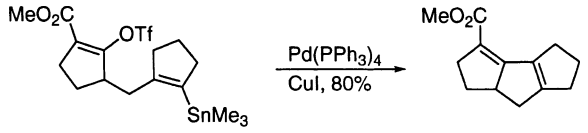


(11)

Intramolecular Stille type cross-couplings were employed in a number of total syntheses [161–163]. Some examples are shown in Eq. (12) [164], Eq. (13) [165], Eq. (14) [166], and Eq. (15) [167].



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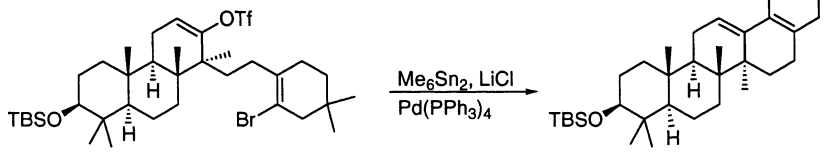

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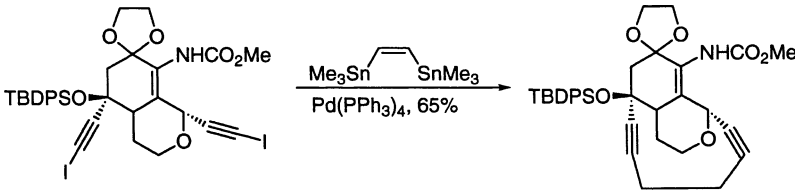

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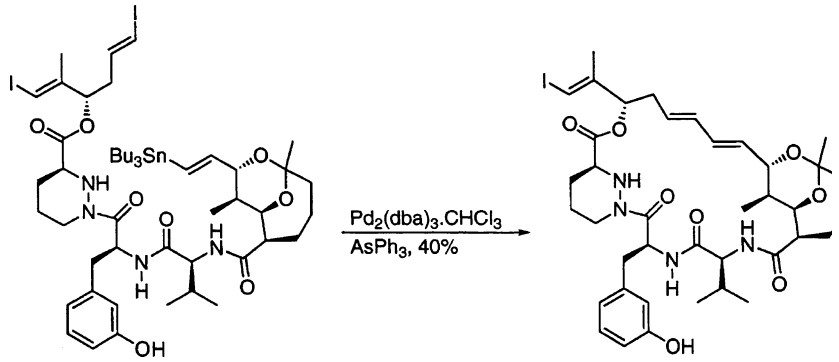
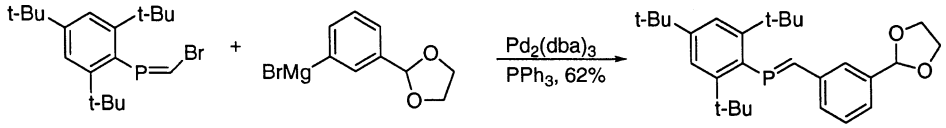


(15)

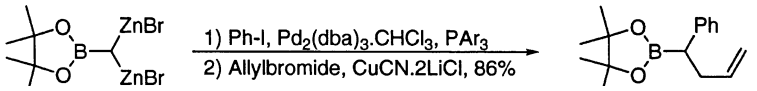
Nickel(0) and palladium(0) complex-catalyzed cross-coupling of Grignard reagents with halides and triflates was extensively used in synthesis. Coupling reactions of aryltriflates [168–171], aryl halides [172–178], lumazine 6-triflate [179], β-carboline-4-triflate [180], thiophene and benzothiophene halides [181–184], vinyl halides [185–187] pyrrole-3-triflates, -phosphonates, and -carbamates [188] were reported. More unusual were the nickel-catalyzed couplings of 2-methoxy quinolines [189] and vinylcarbamates [190] with Grignard reagents. Nickel-catalyzed cross-coupling of alkyl Grignards with vinyl triflates [191,192] without competing β-hydride elimination was reported. A novel phosphorous compound was coupled with a Grignard reagent in the presence of a palladium catalyst (Eq. (16)) [193].



(16)

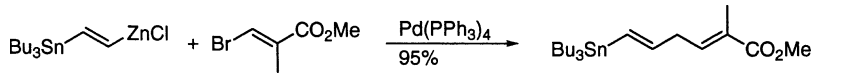
Couplings of halides and triflates with organozinc reagents, the Negishi type coupling, continued to grow as a viable alternative to tin and boron reagents [194,195]. Tetrabutylammonium iodide accelerated the cross-coupling reaction of benzylic zinc compounds [196]. Palladium-catalyzed reactions of lithium *tert*-butylzincates and aryl and heteroaryl iodides were developed [197]. Coupling reactions of alkynyl zinc halides with vinyl halides [198,199] and aryl iodides [200] were reported. A nickel-catalyzed cross-coupling of organozinc reagents with compounds having a novel S–CH_2COOH leaving group was developed [201]. Arylzinc chlorides were coupled with aryl halides [202–204] and heptafulvene bromide [205]. Negishi couplings of heterobenzylic sulfonium salts were published [15]. A number of heterocyclic zinc reagents were used including 2-furyl- [111], 2-pyridinyl- [206–208], 3-pyrazolyl- and 2-imidazolyl- [110], 2-thienyl- [209], 2-indolyl- [210], 2-oxazolyl- [211], 1-benzyloxy-4-pyrazolyl- [212], and 2-silolyl- [213] zinc halides.

Perhaps the most important facet of the Negishi reaction is the cross-coupling of alkyl zinc reagents without β-elimination. Alkyl zinc reagents coupled with aryl halides [214–221], vinyl iodides, and triflates [222–224]. Nickel-catalyzed cross-coupling of aryl chlorides with alkyl zinc iodide [225,226] and of dialkyl zinc reagents with alkyl bromides was developed [227]. Palladium catalyzed the cross-coupling of aryl zinc with methyl chlorooxalate forming β-ketoarylacetic acids [228]. Aryl zinc chlorides were coupled with vinyl iodide [229], allene zinc chloride with allyl bromide [230], vinyl zinc halides with vinyl triflates [231], and alkynyl zinc halides with vinyl halides [88]. Vinyl phosphates were used as electrophiles in Stille and Negishi type couplings [232]. Palladium catalyzed the coupling of optically active iso-propyl alkyl zinc reagents with vinyl iodides or acid chlorides with retention of stereochemistry [233]. Rhodium(I)–dppf complex also catalyzed the coupling between arylzinc reagents and aryl and methyl iodides [234]. An interesting and potentially useful dizinc boronic acid ester was reported. Palladium catalyzed the coupling of one of the zinc bromides; the second was replaced by reaction with a cuprate (Eq. (17)) [235].



(17)

Vinylnonaflates were used in palladium-catalyzed Negishi type couplings [236]. Aryl zinc [237], benzyl zinc [238], and alkynyl zinc [239] halides were coupled with acid chlorides to form unsymmetrically substituted ketones. Selective reaction of the zinc moiety in the presence of a trialkyltin group was reported (Eq. (18)) [240]. A vinylzinc moiety reacted selectively with a vinyl bromide in the presence of a boronic acid ester. The intermediate vinyl boronic ester was transformed to a triene using a Suzuki type coupling (Eq. (19)) [241].



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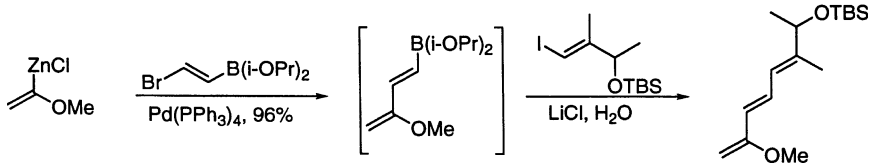
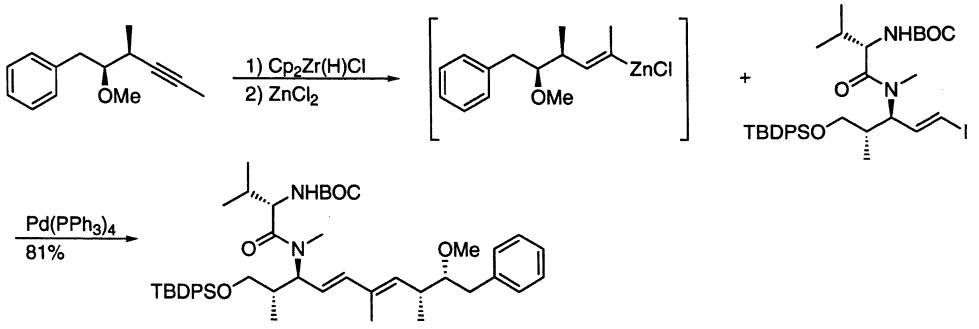


(19)

Hydrozirconation of terminal alkynes using in situ prepared $Cp_2Zr(H)Cl$ gave vinylsubstituted zirconocene chlorides suitable for transmetallation to zinc followed by palladium-catalyzed coupling with aryl iodide [242], vinyl iodide, and allyl bromide [243,244]. An interesting example of this sequence is shown in Eq. (20) [245]. Vinyl zinc reagents were also prepared by hydrotitanation of terminal alkynes followed by transmetallation [246]. The *trans*-bromide in 1,1-dibromoalkenes coupled exclusively with tin reagents to form trisubstituted alkenes (Eq. (21)) [247]. In related reactions of tin reagents, depending on the reaction conditions, either trisubstituted alkenes or internal alkynes were formed. The latter were obtained preferentially in polar solvents [248].



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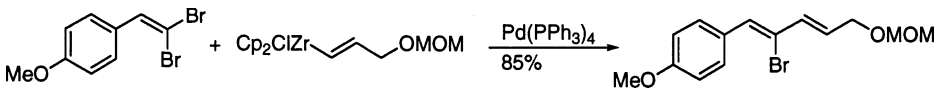
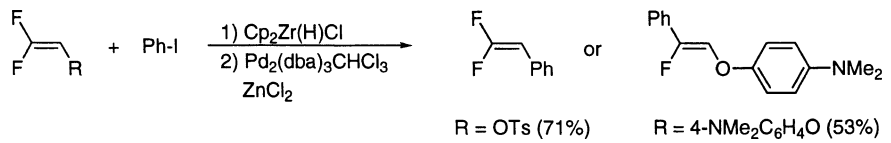


(21)

α-Sulfonyl alkenylzirconocenes, prepared in a similar fashion, were coupled with vinyl and alkenyl halides without prior transmetallation to zinc [249]. Hydrozirconation of 1,1-difluoroalkenes followed by transmetallation to zinc and palladium-catalyzed coupling with iodobenzene afforded either difluoro- or monofluoro products, depending on the substitution pattern of the difluoroalkene (Eq. (22)) [250]. Addition of $Cp_2Zr(H)Cl$ to selenyl alkynes gave α-selenyl alkenylzirconocene suitable for transmetallation to zinc and palladium-catalyzed Negishi type cross-coupling [251]. Palladium catalyzed the cross-coupling of zirconocene benzyne complexes with aryl bromides (Eq. (23)) [252].



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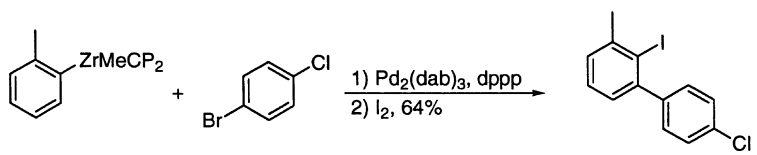


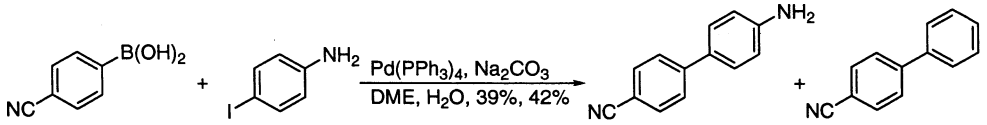
(23)

Copper(I) iodide catalyzed the coupling of vinylzirconocenes with iodonium salts in the presence of carbon monoxide, producing vinyl ketones [253]. Methylaluminoxane catalyzed the coupling of alkynyl iodide and allylzirconocene chloride [254]. Hydroalumination of terminal alkynes followed by nickel-catalyzed coupling with vinyl chloride was reported [186]. Nickel catalyzed the coupling of a vinyl aluminum reagent and a benzylic chloride [255].

The Suzuki cross-coupling, i.e. palladium-catalyzed coupling of organoboronic acids with halides and triflates, continued to see substantial use in organic chemistry. A number of ligands and catalyst systems were developed for the Suzuki reactions of aryl chlorides [256–259]. Coupling of aryl halides with organoboronic acids in water using a water soluble nickel catalyst was reported [260]. A palladium carbene complex catalyzed the coupling of aryl halides with aryl boronic acids [3,171]. A polymer supported palladium catalyst [261], an imidazol-2-ylidene palladium complex [262], a palladium(II) cyclometallated imine [263], a palladium chloride tetraphenyl phosphonium bromide intercalated clay catalyst [264], and palladium complexed to a P–N bidentate ligand [265] were developed for the Suzuki reaction. Suzuki type couplings of aryl halides including chlorides using an air stable triphenylphosphine free nickel complex were reported [266]. Palladium-catalyzed reactions in aqueous solvents [267,268], supercritical carbon dioxide [1], and solventless reactions using palladium doped potassium fluoride/aluminum oxide [269].

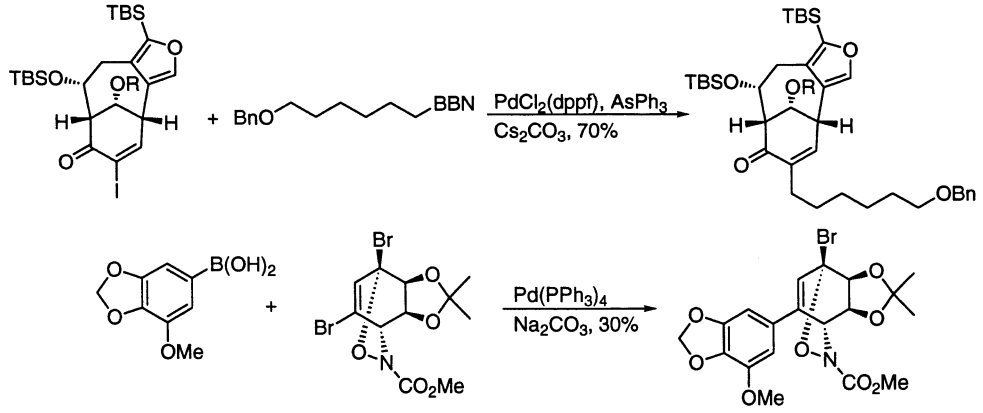
Polymer supported Suzuki reactions have attracted a fair amount of attention. An air and moisture stable resin-bound palladium catalyst was developed [270]. Polymer bound aryl and heteroaryl halides were used together with aryl and thienyl boronic acids [8,9,271–277]. Polymer bound boronic acids and esters were also used [278,279].

Cyclopropyl boronic acids esters were used in the Suzuki coupling [280,281]. Cross coupling of aryl boronic acids and esters with vinyl triflates and halides [282,283] and benzyl halides [284] were reported. Vinyl boronic acids and esters coupled with aryl iodides [285], and allyl boronic esters coupled with aryl triflates [44]. 2-Substituted-1-halo-1-fluoroalkenes [286], and bromoallene [287] were coupled with boronic acids. Vinyl [288] and heteroaryl phosphates [289], vinyl mesylates, phosphates, and tosylates were coupled with aryl boronic acids employing palladium or nickel catalysts [290]. Coupling reactions of heterobenzylic sulfonium salts were reported [15]. Suzuki type coupling of aryl boronic acids with 4-iodoanilines unexpectedly gave a substantial amount of deaminated coupling products (Eq. (24)) [200].



(24)

The Suzuki reaction was used in a plethora of total syntheses wherein vinylboronic acids or esters were coupled with a vinyl halides or triflate [153,199,291–295]. Similar to the Stille reactions of 1,1-dibromo-2-substituted alkenes, the *trans*-bromide reacted selectively with vinyl boronic acids [296]. Aryl halides [170,297–313] were extensively used in Suzuki type coupling reaction of arylboronic acids and esters. Aryl–aryl cross-coupling reactions were used in synthesis [57,188,314–322]. Some synthetic applications of the Suzuki coupling can be seen in Eq. (25) [323], Eq. (26) [324], Eq. (27) [325], and Eq. (28) [326].



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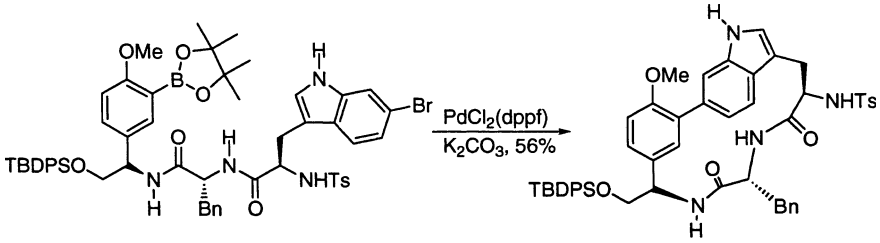
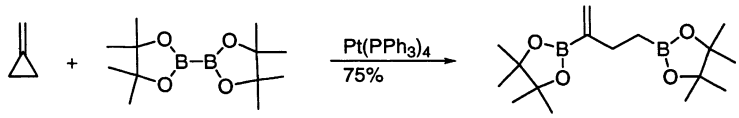


(28)

Palladium catalyzed the cross-coupling of aryl halides with dipinacolylborane producing aryl pinacolyl boronic esters [327–330]. Platinum catalyzed the addition of dipinacolylborane to methylene cyclopropane forming 1,3-dipinacolylboronic esters (Eq. (29)) [331].



(29)

A large variety of heterocyclic halides and triflates were employed in the Suzuki reaction. 6-Bromo-1,4-dihydro[1,8]naphthyridine-4-one [79], 3-iodoindazole [332], 3-iodoindole [333], 2-bromopyrrole [334], 3-iodopyrrole [335], 2-bromoquinoxaline [121], 2-bromothiophene [336], 3-iodopyridine [337], 2-halobenzothiophenes [121], 2-,3-, and 4-halopyridines [104,208,338–341], β-carboline-4-triflate [180], 3-bromo-, 2,9-dibromo-, 3,8-dibromo-1,10-phenanthroline [98,342,343], 1-chloroisoquinoline [344,345], pyridone-3- and -5-triflates [110,346], 2-iodoquinoline [347], 2-chloroquinoline and 5-chloropyrazolo[1,5-*a*]pyrimidine [195], 3-chloropyridazine [348], 4-chloroquinazoline [349,350], uraciliodonium salts [124], 6-halopurines [351], octabromotetraarylporphyrin [352], 2-bromofuran [353], octabromoarylporphyrin [354], quinoline-2-triflate [355] were all used in Suzuki type couplings.

A variety of heteroaryl boronic acids and esters were used, such as 2-thienylpinacolborane [113], thiophene-3-boronic acid [119], pyrrole-2-boronic acid [356], pyridine-3-boronic acid dimethylester [357], indole-4-boronic acid [358], and indoleborates [359].

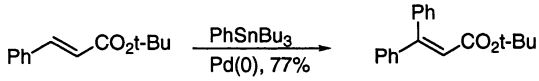
One major advantage of the Suzuki reaction over the Stille reaction is the possibility of using alkyl substituted boronic acids (or esters) without competitive β-hydride elimination [360]. Aryl halides and triflates [221,361,362], vinyl halides [363–367], and vinyl triflates [368] were all coupled with alkylboronic acids.

Finally, palladium catalyzed the alkylation of aryl bromides with ester enolates in the presence of Bu_3SnF [369].

2.1.2. Alkylations of alkenes

The Heck reaction continue to be one of the most versatile methods for the alkylation of alkenes [55,62,126,370–388]. An exploratory study of the Heck reaction showed that solvent polarity, counterion, and ligand all influenced the regiochemistry of addition [389]. A number of papers dealing with different aspects of the catalyst system and the reaction conditions appeared. Several catalyst systems were reported, including a guanidinium phosphine palladium complex supported on glass beads [390], palladium supported on reverse phase silica beads [391], palladium chloride and tertraphenylphosphonium bromide intercalated clay [392], palladium entrapped in zeolite [393], a palladium(II) cyclometallated imine [394], a polymer bound tridentate SCS palladium complex [395], azo-containing phosphine complexes of palladium(II) and platinum(II) [396], a palladacycle with high turnover number [397], and imidazol-2-ylidene palladium complexes [398]. A fluorescence based assay for high throughput screening of ligands for the Heck reaction was developed [399]. Palladium diacetate in the presence of, or absorbed on, calcium carbonate catalyzed the Heck reaction of arene diazonium salts under aerobic conditions [400].

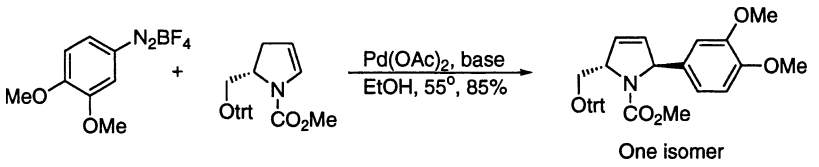
The use of non standard organic solvents for the Heck reaction were described. Catalyst systems and reactions in perfluorinated solvents [401], ionic liquids [402,403] supercritical carbon dioxide [404–406], and aqueous solvents [267,407,408] were developed. Reactions of polymer bound aryl halides were reported [8,409,410]. Arylstannanes reacted with both electron deficient and electron rich alkenes to form Heck type products (Eq. (30)) [411].



(30)

The intermolecular Heck reaction was used extensively in organic total synthesis [130,291,412–414]. Two interesting examples are shown in Eq. (31) [415] and Eq. (32) [416]. Enantioselective reactions of 4,5-dihydrofurans

affording 2-arylated-2,5-dihydrofurans in high enantiomeric excess were reported [417–420]. High asymmetric induction was observed employing an optically active cyclic vinylsulfone (Eq. (33)) [421].



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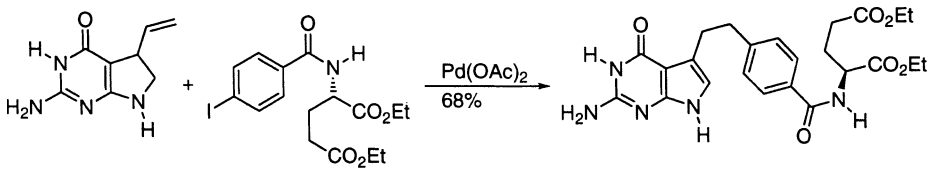

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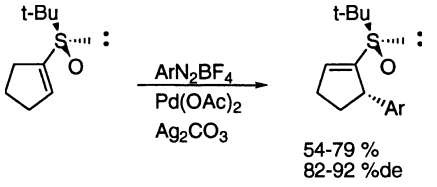
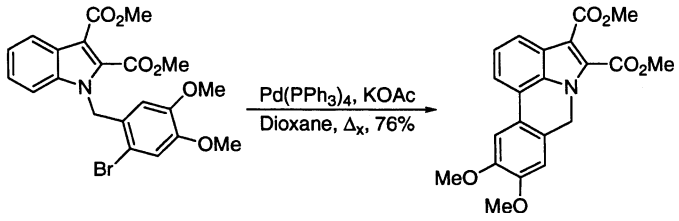


(33)

Aromatic systems were shown to undergo, at least formally, Heck type arylations [163,422,423]. Two examples are shown in Eq. (34) [424] and Eq. (35) [425]. A tandem Heck reaction dicyclopropane insertion was developed (Eq. (36)) [426].



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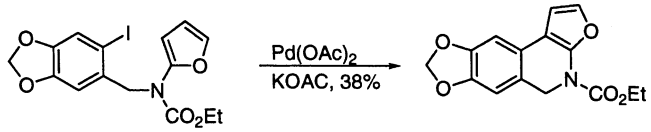

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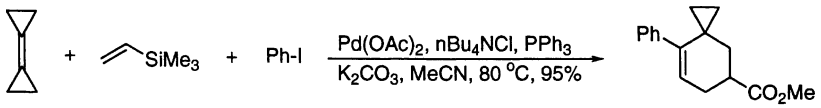
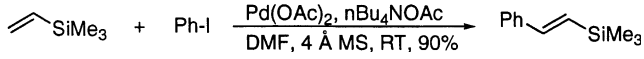


(36)

By proper selection of catalyst system, either styrenes or β-silylstyrenes can be obtained using vinyl trimethylsilane (Eq. (37) and (38)) [427]. Nickel catalyzed the addition of arylhalides to bicyclic alkenes producing ring opened products (Eq. (39)) [428].



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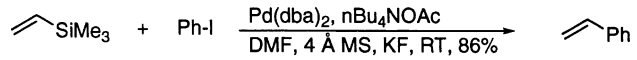

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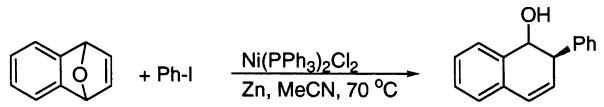
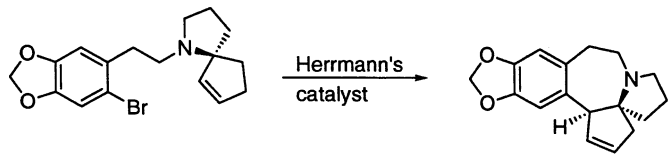


(39)

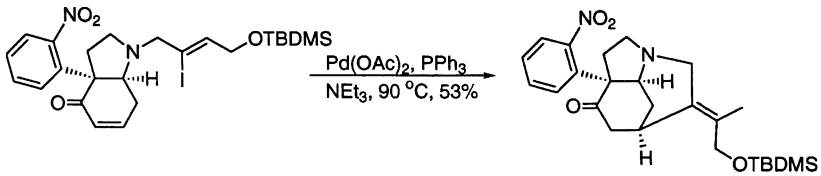
Halides and triflates of a variety of heterocyclic ring systems were used, such as 1-ribofuranosyl substituted 5-iodoimidazole [429], 4-bromotryptophane [430], 3-iodobenzofuran [70], 5-iodouridine [431], 2-bromopyridine [432], 6-bromo-5-deazapteridine (Eq. (28)) [433], 6-bromo-1,4-dihydro[1,8]naphthyridine-4-one [79], 4-iodoquinoline [434], 4-iodo-β-carboline [78], 5-bromo-2(*H*)-pyridazin-3-one [77], pyridone-2-triflate [110], and 1,2,3,5-tetrahydroindolizine-6-triflate [76].

In addition to reactions of electron-deficient alkenes, 4-vinylpyridine [435], 6-vinyl purine [89], vinylbenzotriazoles [436], 4,7-dihydro-1,3-dioxepines [437,438], 2,5-dihydrofuran [439],vinyltriethoxysilanes [440], and vinyl boronic acid pinacol ester [151], were used as acceptor alkenes. In the latter case a small amount of Suzuki type coupling was also observed.

The intramolecular variation [441–445] of the Heck reaction continued to be used as the key step toward an array of natural products [326,446–448]. A number of synthetic applications were reported (Eq. (40)) [449]; (Eq. (41)) [450]; (Eq. (42)) [451]; (Eq. (43)) [452]; (Eq. (44)) [453]; (Eq. (45)) [454]; (Eq. (46)) [455]; (Eq. (47)) [456]; (Eq. (48)) [457]; (Eq. (49)) [458]; and (Eq. (50)) [459].



(40)



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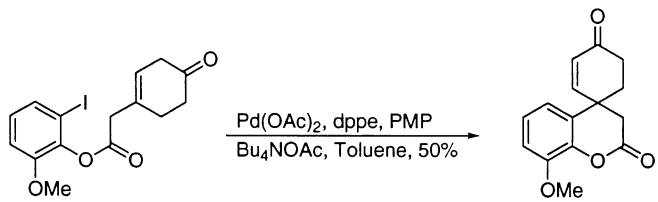
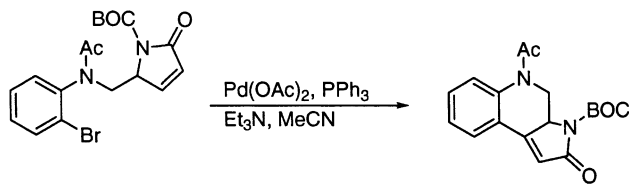
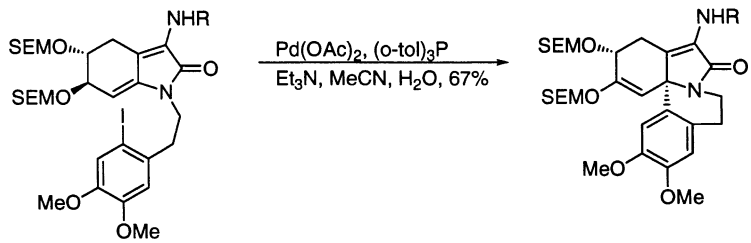


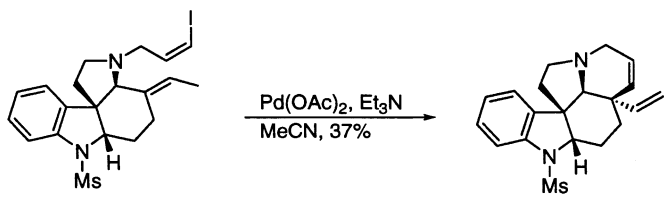
(42)



(43)



(44)



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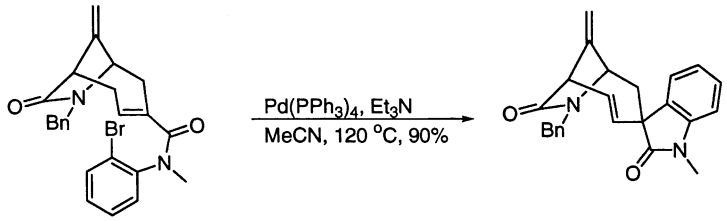

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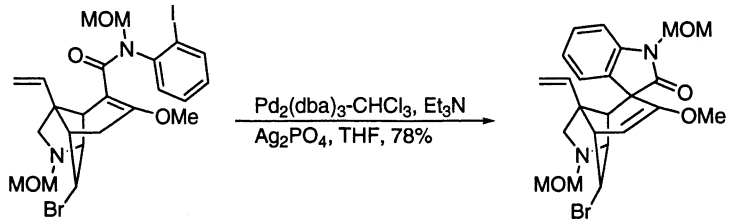
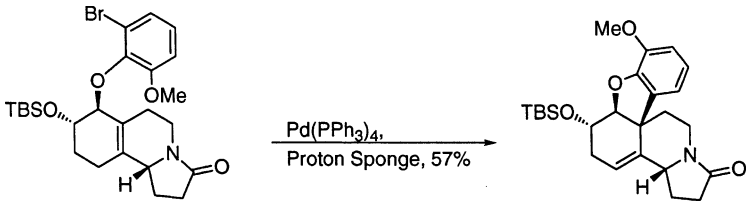
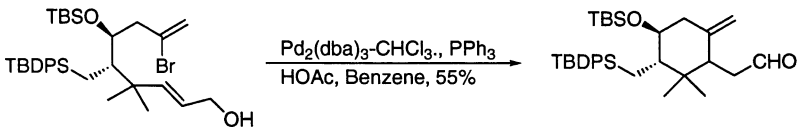


(47)



(48)



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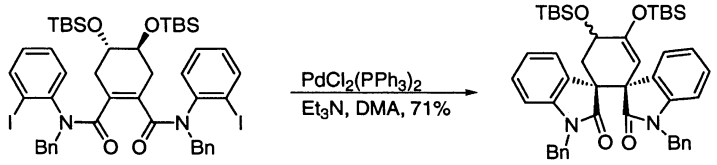
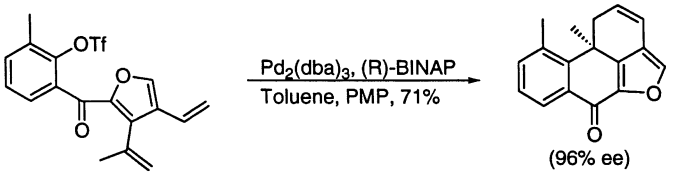
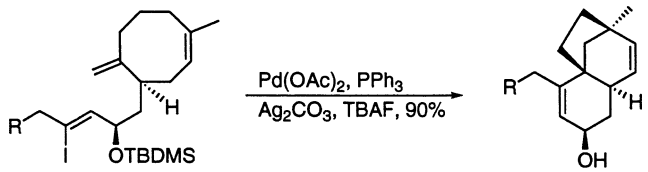


(50)

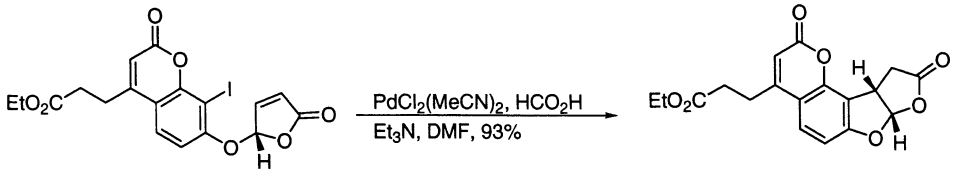
Double intramolecular alkene insertion was achieved (Eq. (51)) [460]; (Eq. (52)) [461]. Catalyst poisoning was observed upon attempted Heck reaction of a tethered 1,4-cyclohexadiene [462]. Reductive Heck reactions were utilized in organic synthesis (Eq. (53)) [463], (Eq. (54)) [464].



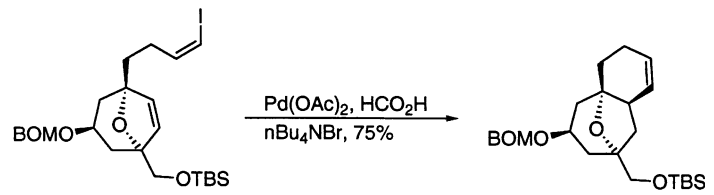
(51)



(52)

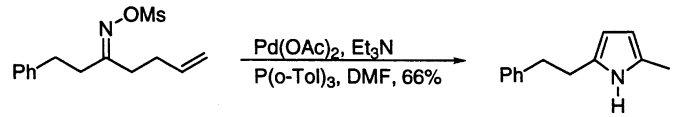


(53)

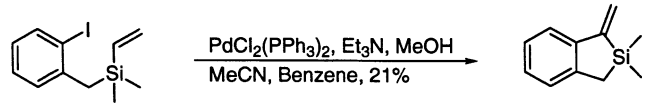


(54)

The intramolecular Heck reaction was used to prepare the pyrrole ring of indoles [465], indolotropanes [466], and pyrrolo[2,3-*b*]quinoxalines [467]. A novel pyrrole synthesis using *N*-mesylated imines was developed (Eq. (55)) [468]. Related cyclizations furnished oxygen containing heterocycles, such as pyranes [469] and benzofurans [470]. Silicon containing heterocycles were also prepared in an intramolecular fashion (Eq. (56)) [471].

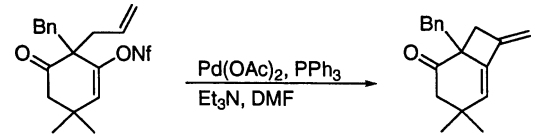


(55)



(56)

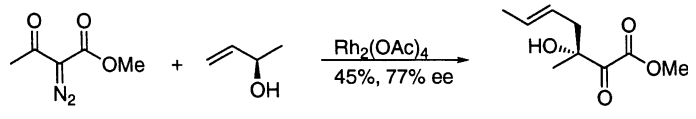
Strained methylenecyclobutanes were prepared from vinylnonaflates (Eq. (57)) [472]. Arylmercury chlorides also participated in insertion reactions with alkenes [473]. Palladium catalyzed the phenylation of unsaturated compounds using phenylantimony chlorides under air [474].



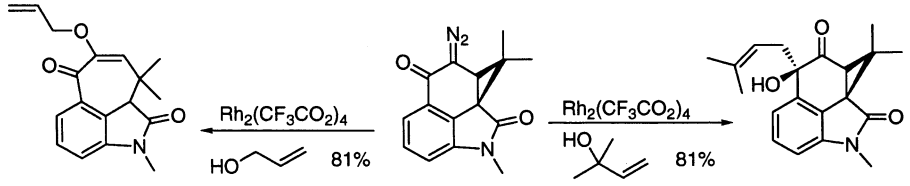
(57)

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

Ruthenium catalyzed the intermolecular insertion of ethyl α-diazoacetate and ethyl diazomalonate into oxygen–hydrogen bonds of alcohols [475,476], and of α-diazoketones into nitrogen–hydrogen and sulfur–hydrogen bonds [477]. Intramolecular rhodium carbenoid nitrogen–hydrogen bond insertion was used to prepare carbapenems on a 2.7 kg scale [52]. An enantioselective rhodium(II)-catalyzed oxygen–hydrogen bond insertion followed by a Claisen rearrangement was reported (Eq. (58)) [478]. A similar insertion-rearrangement sequence can be seen in Eq. (59) [479]. By proper choice of allylic alcohol, the rearrangement was completely suppressed.

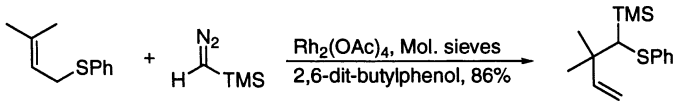


(58)



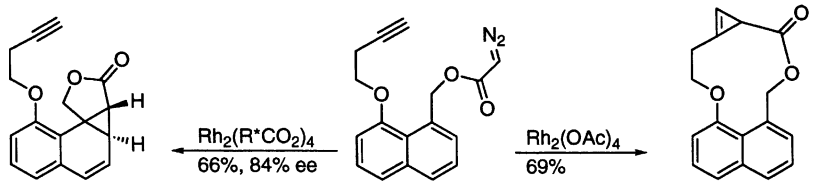
(59)

A few additional examples of rearrangement reactions were reported [480]. α-Silyl-α-diazoketones underwent Wolff type rearrangement in the presence of dirhodium tetraacetate to form silylketenes [481]. Similar rearrangements of β-keto-α-diazophosphonate [482,483] and 4-keto-3-diazo-2-quinolenes, forming oxindoles in the latter case [484], were described. Metal-catalyzed ylide formation and [2,3]sigmatropic rearrangement of allyl sulfides with trimethylsilyldiazomethane were developed (Eq. (60)) [485].



(60)

Proper choice of the catalyst resulted in either alkyne cyclopropanation or arene cyclopropanation (Eq. (61)) [486]. Other examples of synthetic applications of rhodium(II) carboxylate-catalyzed diazodecomposition can be seen in Eq. (62) [487] and Eq. (63) [488].



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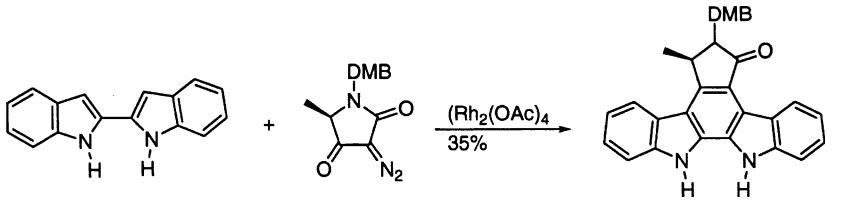
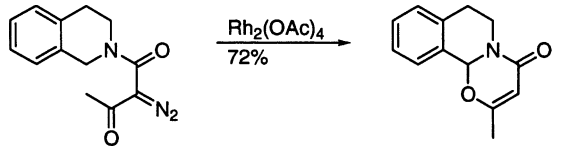
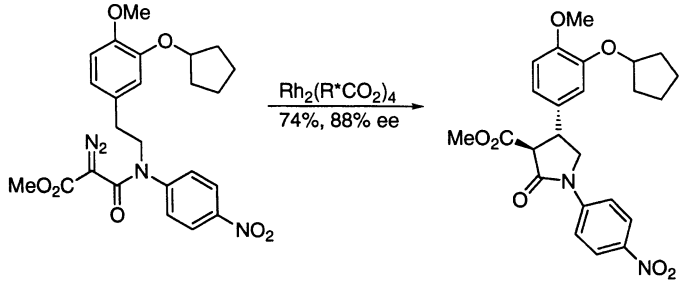


(62)

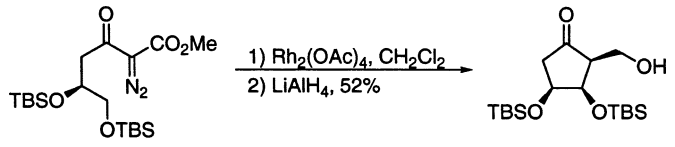


(63)

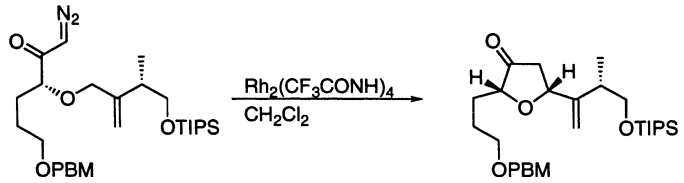
Copper(II) catalyzed the intramolecular carbenoid carbon–hydrogen bond insertion, forming four- to six-membered nitrogen heterocycles [489]. Intramolecular rhodium(II)-catalyzed carbon–hydrogen bond insertions [479,490–492] were used in a number of synthetic applications forming five-membered rings (Eq. (64)) [493]; (Eq. (65)) [494]; (Eq. (66)) [495]; (Eq. (67)) [496].



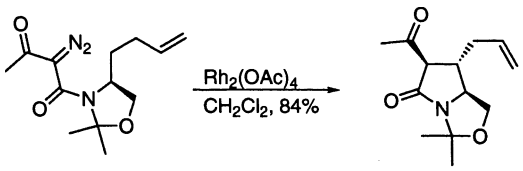
(64)



(65)

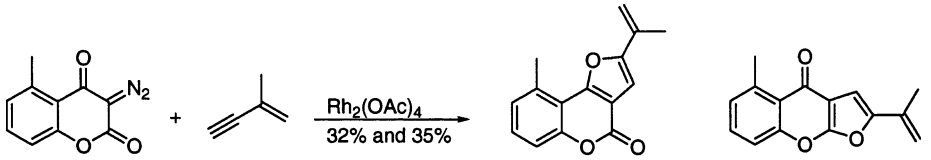


(66)

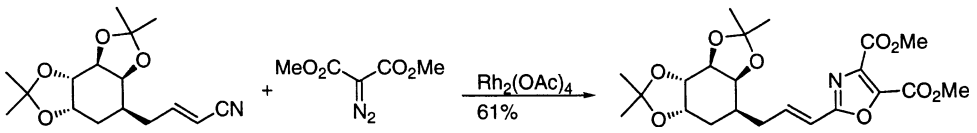


(67)

Rhodium carbenoids participated in a variety of cycloaddition reactions. 2-Diazo-2,4-chromenediones reacted with enynes to form isomeric fused vinylfurans (Eq. (68)) [497]. Oxazoles were prepared by intermolecular cycloaddition of diazocarbenoids to nitriles [498] (Eq. (69)) [499]. Tandem carbonyl ylide formation and intramolecular 1,3-dipolar cycloaddition of diazoketones using chiral rhodium(II) carboxylates were reported (Eq. (70)) [192]. Some additional tandem carbonyl ylide formation cycloaddition reactions were reported [500,501].



(68)



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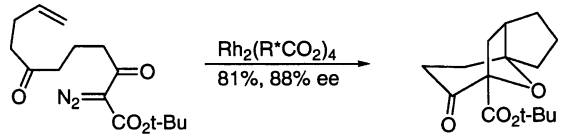
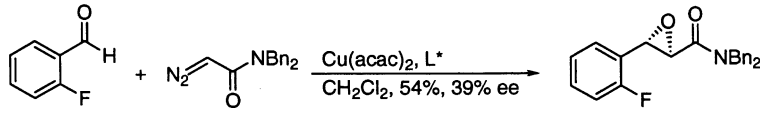


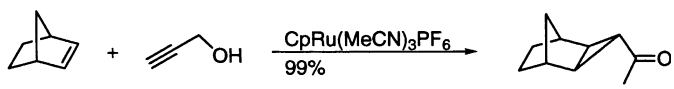
(70)

Copper(II) catalyzed the decomposition of diazo compounds in the presence of a chiral ligand and aromatic aldehydes, producing enantiomerically enriched epoxides (Eq. (71)) [502]. Treatment of rhodium vinylcarbenoids with thioketones gave alkenes or thiiranes, depending on the substitution pattern of the starting diazocompound [503].



(71)

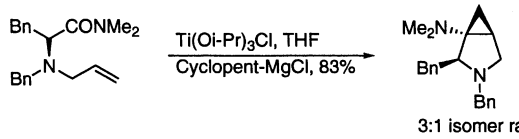
Cyclopropanations of alkenes via transition metal-catalyzed decomposition of α-diazocarbonyl compounds continued to be developed. A variety of catalyst systems were reported, including recyclable rhodium(II) perfluoroalkylcarboxylates [504], dirhodium tetraacetate [505,506], bis(imido)chromium(VI) complexes [507], and ruthenium arene complexes containing phosphines having a pendant aryl group [508]. Palladium catalyzed the cyclopropanation of alkenes using diazomethane [281,509,510]. Asymmetric cyclopropanations using copper(I) triflate and chiral ligands [461,511,512], rhodium(II) complexed to chiral carboxylates [513], ruthenium complexes having an optically active tridentate phosphine [514], or chiral cobalt(II) complexes [515,516] were reported. Sulfonium ylides were also used as carbenoid precursor for asymmetric cyclopropanations [517]. Rhodium(II) catalyzed the intermolecular cyclopropenation of trimethylsilylacetylene using ethyl α-trimetylsilyl-α-diazoacetate [518]. Ruthenium-catalyzed reactions of norbornene, and norbornadienes with propargyl alcohols gave cyclopropanated products (Eq. (72)) [519].



(72)

Cyclopropanation of alkenes using diethylzinc and diiodomethane or chloroiodomethane were employed in organic synthesis by a number of groups [520–527]. Diiodomethane and zinc under ultrasound [528] and mixed diorganozinc reagent were also employed in alkene cyclopropanation [529]. Enantio- [530–532] and diastereoselective [533] cyclopropanations were developed.

An interesting intramolecular titanium-mediated cyclopropanation of amides having a tethered allylamine was reported (Eq. (73)) [534]. Titanium also mediated the intermolecular coupling of vinylsilanes with esters, forming silacyclopropanols (Eq. (74)) [535]. Related cyclopropanations of esters and amides forming hydroxy- and aminosubstituted cyclopropanes were reported [536,537].



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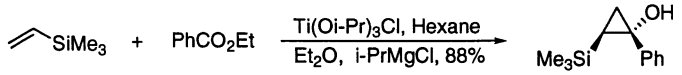
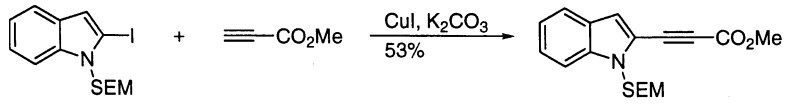


(74)

2.1.4. Alkylation of alkynes

The palladium(0)-catalyzed coupling between terminal alkynes and aryl and vinyl halides or triflates, the Sonogashira reaction, was one of the more frequently utilized carbon–carbon coupling reactions. Reactions of a wide variety of aryl halides and triflates [65,84,279,303,304,382,388,425,538–607], vinyl triflates [21,231,608–611], vinyl halides [62,68,247,381,612–619], acid chlorides [620], alkynyl bromides [621,622], and polymer bound halides [8,9,409,623–626] were reported. Heterocyclic substrates were shown to readily participate in Sonogashira type couplings. Reactions of 4-iodoquinoline [434], 5-iodopyrrolo[2,3-d]pyrimidine [433], 3-bromo- and iodobutenolides [194], 2-bromo and iodothiophenes [627–630], 5-iododeoxyuridine [631], 5-iodouridine [632], iodo- and bromopyrazolo[3,4-d]pyrimidines [633–635], pyridone triflates [110,346], 4-iodo-isoquinoline-1-one [636], 6-iodopurine [89], bromo-, chloro-, and iodopyridines [637–643], pyridine triflates [644,645], 4-iodo-β-carboline [78], 2-iodopyrrole [646], 5-bromo-2(H)-pyridazin-3-one [77], 1,2,3,5-tetrahydroindolizine-6-triflate [76], 3-iodobenzofuran [70], bromo-1,10-phenanthrolines [647,648], 2- and 5-iodo-pyrimidines [649,650], 2,5-dibromofuran [651], 3-iodopyrrolidine-2-one [652], 2-chloro-1,3,5-triazine [653], 2-iodo-1,3-benzotriazole [654], 3- and 5-iodopyrazoles [655], 4,5-diiodoimidazole [656], thiazole-5-triflate [657], 8-iodopyrazolo[3,2-*c*][1,2,4]triazine [658], 2,5-dibromopyrazine [659] were reported.

A guanidinium phosphine palladium complex supported on glass beads was used [390]. Low temperature (-20 °C) reactions using tris(2,4,6-trimethylphenyl)phosphine as a ligand in the presence of tetrabutylammonium iodide [660] were developed. Copper iodide catalyzed the reaction of 2-iodoindole (Eq. (75) [109] and 4-methylbenzoyl chloride (Eq. (76)) [661]. Interesting regioselectivity was observed in a few cases (Eq. (77)) [662]; (Eq. (78)) [663].



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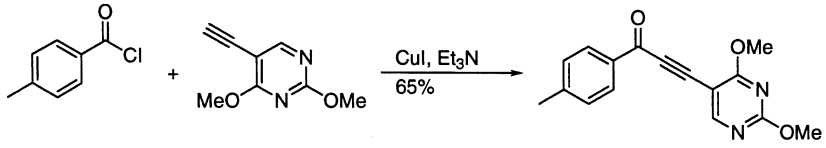

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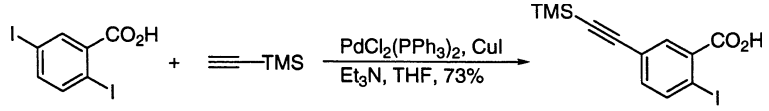

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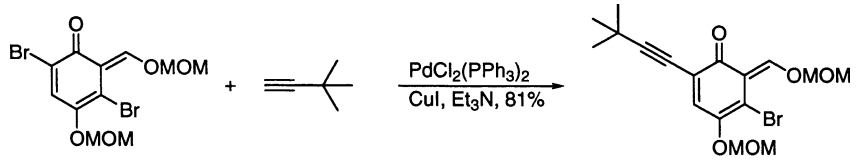
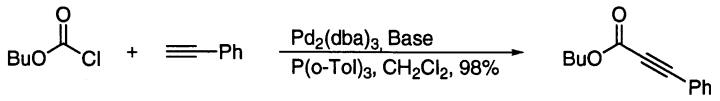


(78)

Chloroformates (Eq. (79)) [664], polymer bound aryldiazo compounds (Eq. (80)) [409], vinyl nonaflates (Eq. (81)) [17], vinyliodonium salts in the presence of a vinyl triflate(Eq. (82)) [665], and vinyliodides in the presence of a vinylstannane (Eq. (83)) [14] were coupled with alkynes.



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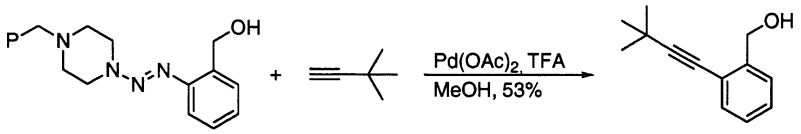

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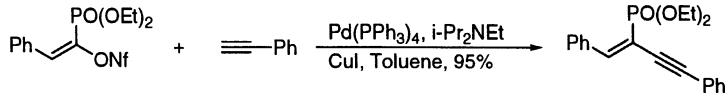

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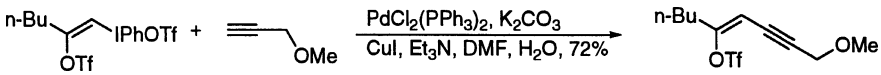

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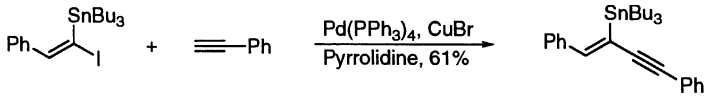
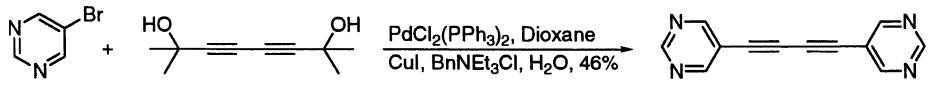


(83)

1,4-Bis(dimethylmethanol)-1,3-butadiyne was used as 1,3-butadiyne precursor (Eq. (84)) [666] and intramolecular transfer of a silicon tethered alkyne was reported (Eq. (85)) [471].



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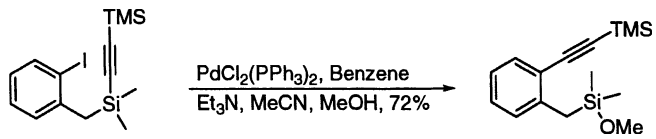
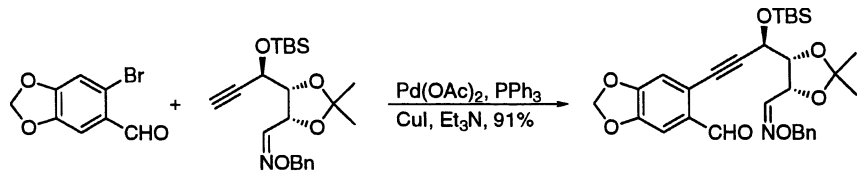


(85)

The Sonogashira reaction continued to be used as the key step toward an array of natural products [186,667–679]. A selection of synthetic applications can be seen below in Eq. (86) [680], Eq. (87) [681], Eq. (88) [682], Eq. (89) [232], Eq. (90) [683], Eq. (91) [684], Eq. (92) [273], Eq. (93) [685], and Eq. (94) [686].



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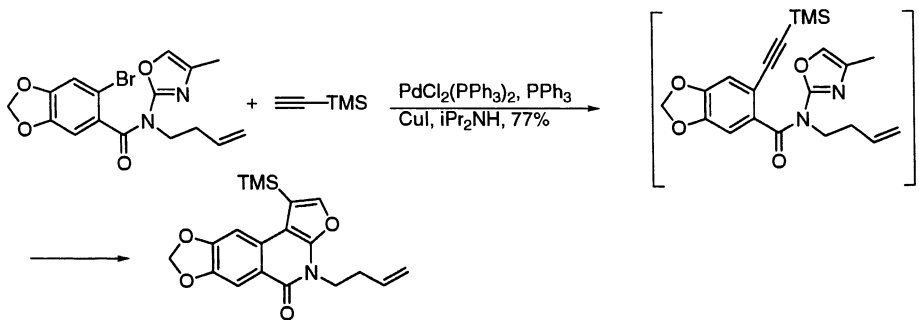

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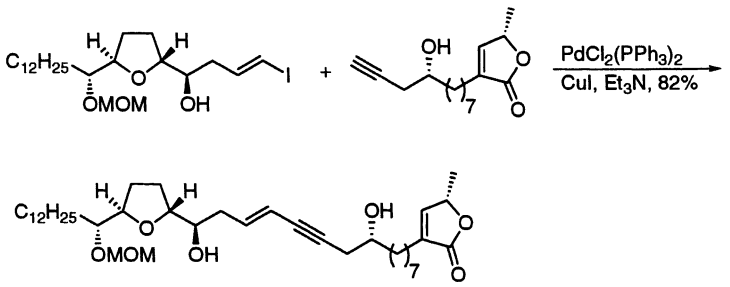

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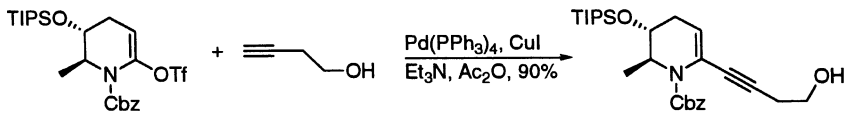

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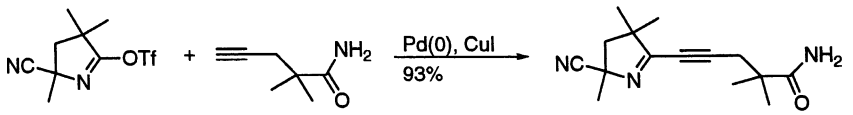

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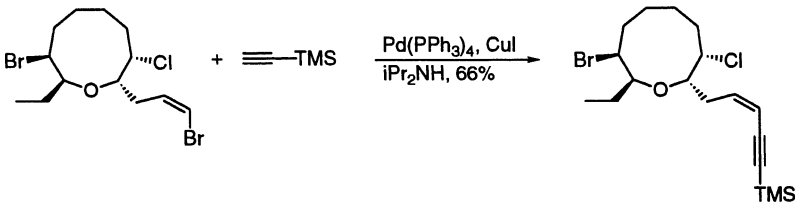

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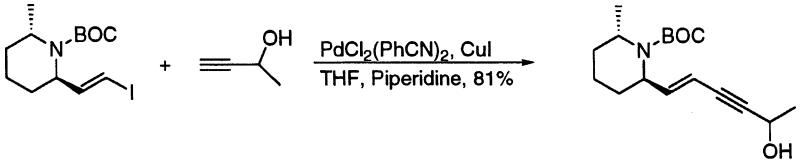

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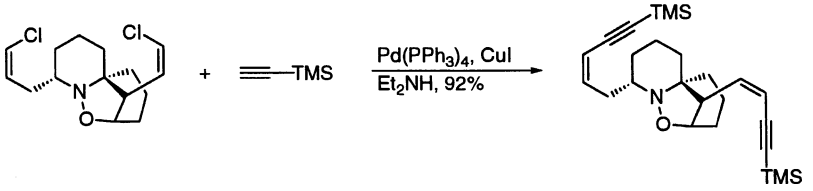

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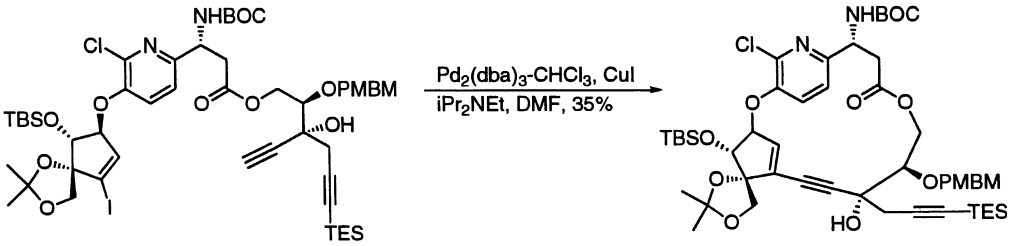
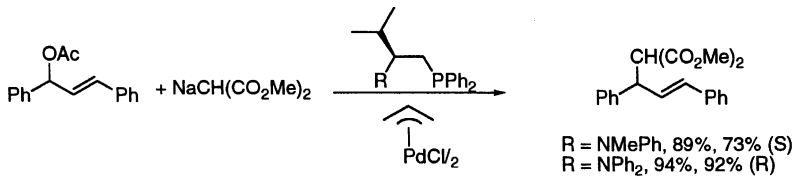


(94)

2.1.5. Alkylation of allyl, propargyl, and allenyl systems

Palladium was predominately employed as the catalyst of choice for allylic alkylation reactions [687–700]. A number of catalyst systems were reported, including a guanidinium phosphine palladium complex supported on glass beads [390]. Allylic substitution in water, using a number of nucleophiles and catalyzed by amphiphilic resin-supported palladium–phosphine complexes, were developed [701,702]. Ionic liquids were used as solvents for allylic alkylations [703]. A mechanistic study of additions to isolated η^3-allyl palladium complexes was reported [704]. Titanated stabilized nucleophiles and nitroalkanes [705] were used in allylic alkylation [706]. β-Fluoropropenoates were used as nucleophiles under neutral condition using molecular sieves [707]. Reaction of enamines with allylic benzotriazoles stereoselectively produced γ,δ-unsaturated ketones [708].

Asymmetric allylic alkylation of, primarily, 1,3-diphenyl-allylacetate continued to receive substantial attention. A variety of new bidentate ligands such as P,N- [339,709–722], N,N- [723–728], P,S/Se- [729–733], N,O- [344], P,P [734–737], and N,S- [340,738] were examined. Platinum [739] and molybdenum [740] also catalyzed enantioselective allylic alkylations. Palladium catalysts confined in microporous silica [741] and palladium supported on reverse-phase silica beads were successfully used [391]. Palladium catalyzed the asymmetric allylation of ketone enolates [742,743]. A substantial enhancement of enantioselectivity was observed using zinc enolates as the nucleophile [744,745]. Monodentate ligands were also shown to afford alkylation products in high enantiomeric excess in allylic alkylation reactions [746,747]. A dramatic change in enantioselectivity was observed upon small changes in the optically active ligand (Eq. (95)) [748,749]. Asymmetric cyclopropanations of ketene silyl acetal with allylic acetates were reported (Eq. (96)) [750,751].



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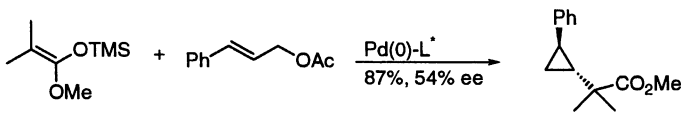
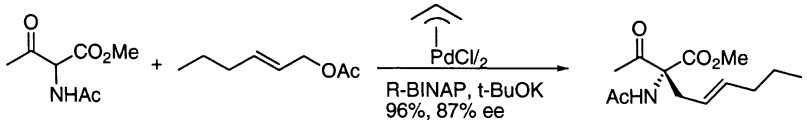


(96)

Palladium catalyzed the asymmetric alkylation using prochiral α-acetamido-β-ketoesters (Eq. (97)) [752], azalactones [753], ketone enolates [754], and stabilized phosphonates [755] as nucleophiles. Dynamic kinetic asymmetric transformations [756] were used to prepare one enantiomer from a racemic mixture (Eq. (98)) [757]. Dynamic kinetic resolution of acyclic allylic acetates using palladium and a lipase was developed [758]. Asymmetric desymmetrization of *cis*-2,5-dibenzyloxy-2,5-dihydrofuran was used in nucleoside synthesis [759]. Chiral recognition was used as a tool for control of alkene geometry in a transition metal-catalyzed allylic alkylation [760]. Tricyclohexylphosphine palladium complex-catalyzed allylic alkylation at the more substituted position [761]. Double asymmetric alkylation of 1,4-dichloro-2-butene gave optically active vinyl cyclopropanes (ee ≤ 32%) [762]. Palladium-catalyzed allylic alkylation using vinylogous sulfonates (Eq. (99)) [763,764] was reported.



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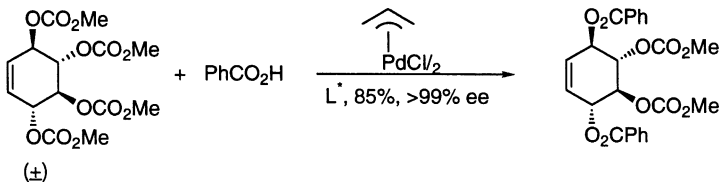

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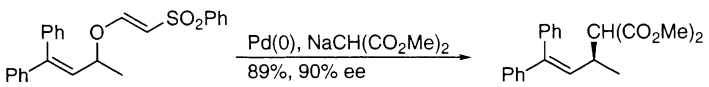
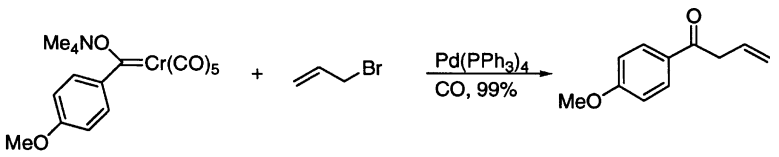


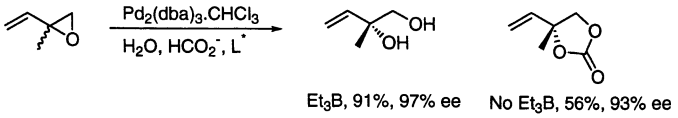
(99)

Palladium catalyzed the coupling reaction of acylchromate complexes and allylic bromides (Eq. (100)) [765]. Functionalized allyl silanes were prepared by regioselective allylic alkylation of β-silylsubstituted η^3-allyl palladium intermediates [766,767]. Palladium catalyzed the stereoselective intramolecular cyclization of allylic benzamides to oxazolines [768]. An allylic nitrogroup was readily displaced by carbon and nitrogen nucleophiles in the presence of a palladium catalyst [769].



(100)

Inorganic carbonates were used as nucleophiles for the asymmetric synthesis of vinylglycidols. Adding triethyl boron changed the product from a cyclic carbonate to a diol (Eq. (101)) [770]. Palladium catalyzed the reaction of vinyloxetanes with isocyanates forming oxazines (Eq. (102)) [771]. Reaction of carbodiimides in place of isocyanates produced the corresponding imine.



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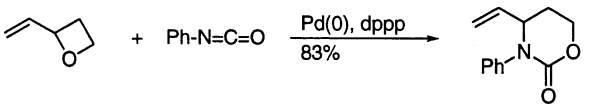
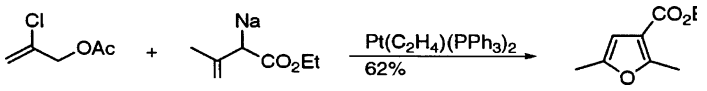


(102)

Transition metals other than palladium also catalyzed allylic alkylation reactions. Iridium catalyzed allylic substitution with retention of stereochemistry. Preferential alkylation at the most substituted site was observed [772]. Enantioselective iridium-catalyzed reactions were described [773]. Ruthenium catalyzed the allylic substitution of cyclic allylic carbonates with stabilized nucleophiles and amines [774]. Alkylation at the more substituted carbon was obtained using molybdenum hexacarbonyl as the catalyst [459]. Regio- and enantioselective molybdenum-catalyzed allylations were described [775]. Molybdenum and tungsten catalyzed the allylic alkylation using enolates [776]. A Lewis acid type mechanism was suggested. Platinum-catalyzed reactions of 2-chloroallyl acetates to give furans (Eq. (103)) [777]. Molybdenum catalyzed the allylation of electron rich aromatic and heteroaromatic substrates. The reaction was *para* -selective for anisol [778]. Nickel catalyzed the addition of aryl Grignards to furanoid and pyranoid glycals. The product formed depended on the ring size (Eqs. (104) and (105)) [779].



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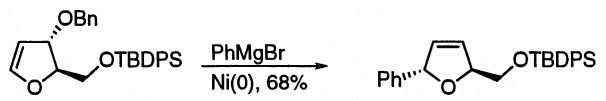

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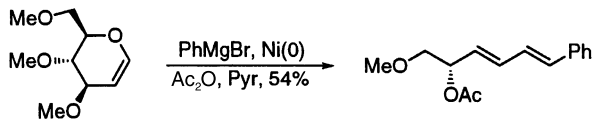


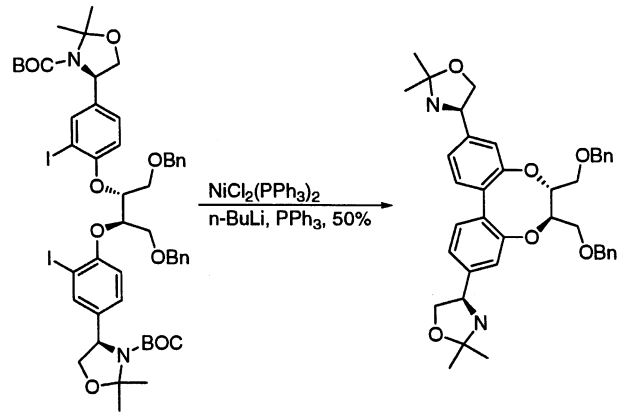
(105)

A number of allylic protecting groups were used in organic synthesis; groups readily removed using, primarily, palladium catalysts. Amines were protected as *N*-allyloxycarbonyl groups, [140,166,232,780–790] or as allylic amines [791–795]. In addition to a variety of palladium compounds, Wilkinson's catalyst was used to deprotect allylic amines [170,796]. Carboxylic acids were protected as allyl esters, [797–823], and alcohols as allyl ethers [643,824–826]. Phosphates were protected as allyl esters [800,827–830]. Palladium catalyzed the tandem deprotection-coupling of *N*-allyloxycarbonyl amino acids by treatment with triphenyl silane followed by an acid chloride [831]. Thiols were protected with allyloxycarbonylaminomethyl group ($RSCH_2NHCO_2$allyl) [832,833]. Arylmethyl esters were used as protecting groups for carboxylic, carbonic, and carbamic acids [834].

2.1.6. Coupling reactions

Copper or manganese catalyzed the homocoupling of a variety of organotin reagents [18,835]. Palladium catalyzed the homocoupling of an arylmercury chloride [836]. Homocoupling of aryl iodides to symmetrical biaryls using

palladium on carbon and an aqueous solvent, in air, was reported [268]. Palladium on carbon catalyzed the homocoupling of aryl chlorides in aqueous media [837,838]. Nickel and palladium catalyzed the homocoupling of aryl and heteroaromatic halides to symmetrical biaryls [839–842]. Nickel catalyzed the cross-coupling of organostannanes with iodonium salts, in the presence of carbon monoxide, forming unsymmetrically substituted ketones [843]. Nickel mediated the intramolecular coupling of aryl iodides (Eq. (106)) [844]. Oxovanadium(V) compounds mediated the oxidation of alkenylzirconocenes to dienes. (Eq. (107)) [845]. Enynes and diynes were also formed in related oxidation reactions.



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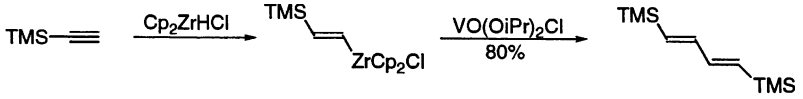
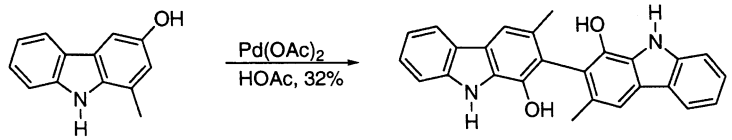


(107)

Palladium catalyzed the coupling of triarylboranes [846] and aryl boronic acids [847] to symmetrical biaryls. Homocoupling of an alkenylstannane affording a 1,3-diene [848] and of tetraselenafulvalene stannane [849] were reported. Compounds lacking a halide or a triflate were coupled via carbon–hydrogen bond activation. Examples of synthetic applications can be seen in Eq. (108) [850] and Eq. (109) [851].



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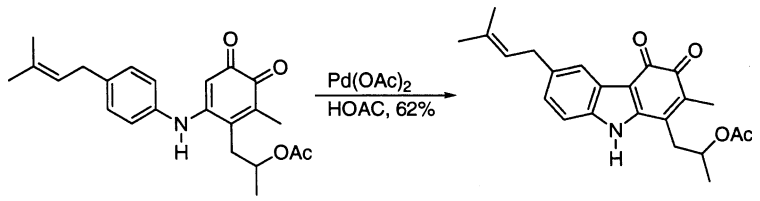
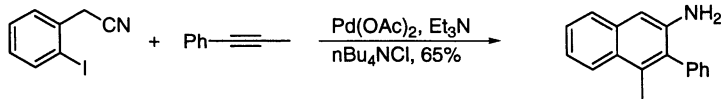


(109)

A variety of complex polycyclic molecules were obtained by reactions usually initiated by an oxidative addition to an aryl or a vinyl halide [852–863]. The product isolated depended on the sequence of events that follow for example, alkene and alkyne insertions, carbonylations, and terminations. Common termination reactions were carbonylation, β-hydride elimination, and reactions with nucleophiles. These cascade type reactions are unfortunately not readily categorized within this review and are therefore only briefly discussed. Palladium catalyzed the annulation of 2-iodobenzonitriles with alkynes forming 2-aminonaphthalenes (Eq. (110)) [864]. Palladium catalyzed the coupling of 2-bromoarylketones with alkynes affording indenols (Eq. (111)) [865,866].



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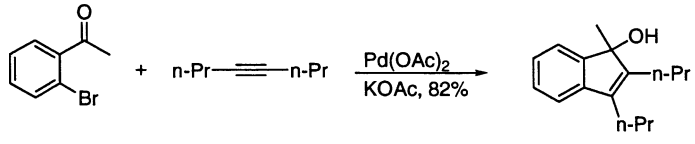
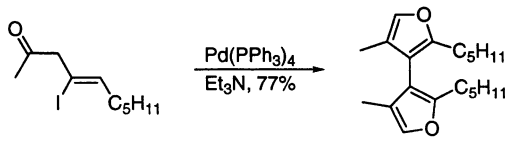


(111)

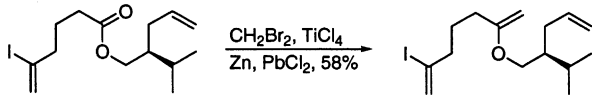
Palladium catalyzed the cycloisomerization dimerization of 3-iodo-3-alken-1-ones (Eq. (112)) [867,868]. Ruthenium catalyzed *ortho*-vinylations of aromatic ketones with alkynes [869]. Rhodium catalyzed *ortho*-alkylations of aromatic ketones with alkenes [870].



(112)

2.1.7. Alkylations of carbonyl compounds

Methylenation of ester carbonyls employing $TiCl_2$–TMEDA–$CH_2(ZnI)_2$ affording vinyl enol ethers was reported [871]. Reaction of *cis*-cinnamaldehyde with $CH_2(ZnI)_2$ in the presence of TMSCl gave a diene [218]. A combination of CH_2Br_2–Zn–$TiCl_4$ in the presence of a catalytic amount of $PbCl_2$ was used in synthesis [872] (Eq. (113)) [61]. Reaction of CH_2I_2–Zn–$TiCl_4$ was used in a similar fashion (Eq. (114)) [873]. Tebbe's reagent, $Cp_2Ti{=}CH_2$, methylenated bicyclic ketones [874] and was employed in total synthesis (Eq. (115)) [875]; (Eq. (116)) [876].



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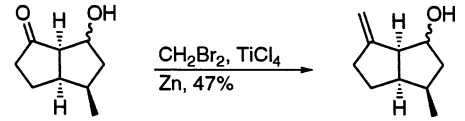

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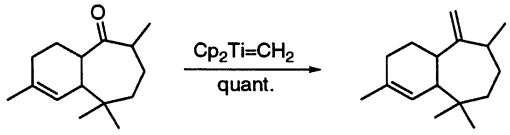

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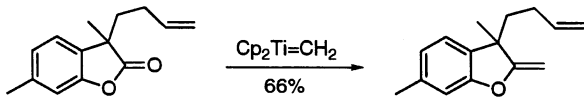
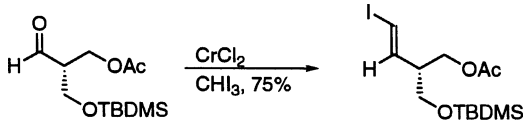


(116)

Vinyl iodides and bromides were prepared by the chromium(II) mediated Takai olefination [71,895]. Applications of the reaction in synthesis [148,156,167,363,877,878] can be seen in equations Eq. (117) [879] and Eq. (118) [152], respectively. Interesting alternatives to the haloforms were $Bu_3SnCHBr_2$ and Bu_3SnCHI_2, affording vinyl stannanes (Eq. (119)) [61]. Chromium dichloride mediated the α-chlorovinylation of aldehydes (Eq. (120)) [880].



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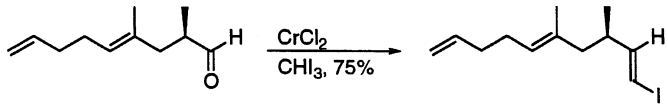

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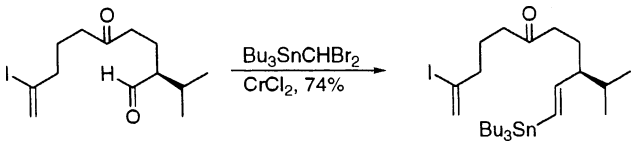

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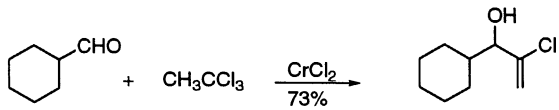
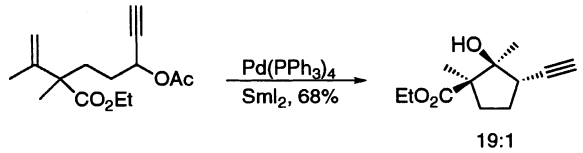


(120)

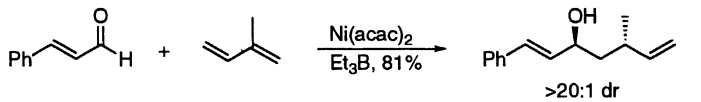
Palladium complexes catalyzed the diasteroselective alkylation of aldehydes by chiral propargylic mesylates in the presence of Et_2Zn [881,882], indium and bismuth salts [883]. In related reactions, palladium catalyzed allylations of aldehydes with allylic benzoates and ethers [884,885] in the presence of Et_3B, and allylic carbonates, esters, and mesylates in the presence of Et_2Zn [886–889]. Intramolecular palladium catalyzed allylation reactions using a

catalytic amount of SmI_2 (Eq. (121)) [890] and Et_2Zn or indium [891] were reported. Palladium catalyzed the alkylation of aromatic aldehydes with stannylcarborane [892].



(121)

A highly regio- and stereo-selective nickel-catalyzed addition of dienes to aldehydes was reported (Eq. (122)) [893]. Palladium catalyzed the asymmetric acylation of conjugated enones using acylzirconium reagents (Eq. (123)) [894]. A cycloaddition forming a tetrahydrofuran was described (Eq. (124)) [895].



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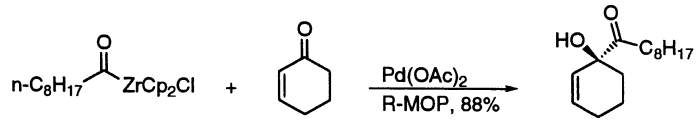

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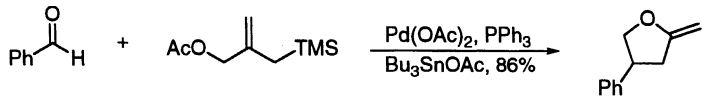
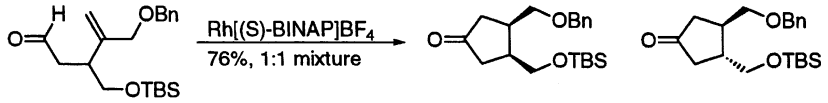


(124)

Rhodium catalyzed the enantioselective intramolecular alkylation of γ,δ-enal to form a cyclopentanone (Eq. (125)) [497]. Wilkinson's catalyst mediated intramolecular carbon–carbon bond formation (Eq. (126)) [896].



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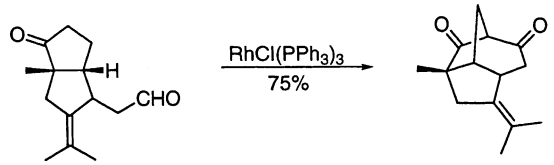
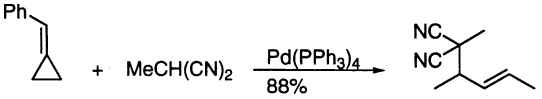


(126)

2.1.8. Carbon–hydrogen bond insertions

Palladium catalyzed the addition of carbon pronucleophiles to methylene cyclopropanes (Eq. (127)) [897]. The regiochemistry of the addition depended on the alkene substitutent. Palladium-catalyzed annulation reactions of 1,2-dibromobenzene and benzylketones, phenols, and α,β-unsaturated carbonyl compounds to form benzofurans, dibenzofurans (Eq. (128)), and polycyclic compounds, respectively [898].



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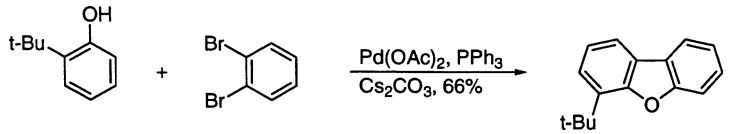
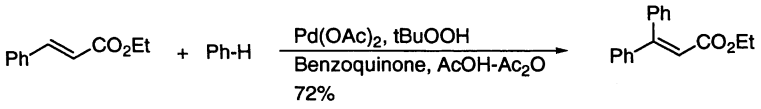


(128)

Palladium-catalyzed multiple arylation of phenols [899] and cross-coupling of arenes with activated alkenes in the presence of *tert*-butyl hydrogen peroxide (Eq. (129)) [900]. Rhodium catalyzed the insertion of alkynes, alkenes, and allenes into the carbon–hydrogen bond of 2-hydroxybenzaldehyde [901]. Palladium catalyzed the aromatic carbon–hydrogen bond activation followed by an asymmetric Heck type reaction [902]. Iridium catalyzed the regioselective insertion of internal alkynes into the *peri*-position of naphthols [903]. Ruthenium catalyzed the insertion of triethoxyvinylsilane into aromatic carbon–hydrogen bonds of aromatic imidates [904] and nitriles [905]. Rhodium

catalyzed the insertion of 1,5-hexadienes into the alkene carbon–hydrogen bond of vinyl pyridines [906]. Palladium catalyzed the insertion of carbon monoxide and urea into aldehydes forming substituted hydantoins (Eq. (130)) [907].



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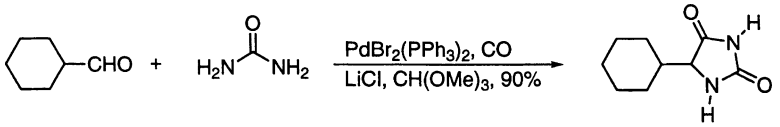
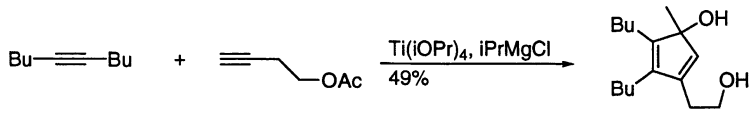


(130)

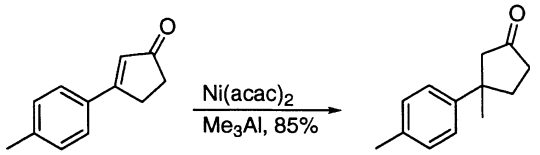
Titanium mediated the coupling of internal alkynes with alkynyl esters to form cyclopentadienols (Eq. (131)) [908].



(131)

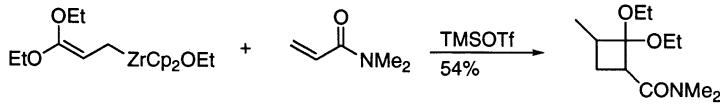
2.2. Conjugate addition

Conjugate addition reactions of 3-methoxy-2-propen-yl copper [909] and trimethylsilyl copper [910] were reported. Nickel catalyzed the conjugate addition of trimethylaluminum (Eq. (132)) [911].



(132)

Ruthenium catalyzed the Michael addition of stabilized carbonucleophiles and terminal alkynes to α,β-unsaturated ketones [912]. Rhodium catalyzed the asymmetric Michael addition of arylboronic acids transferring an aryl group to α,β-unsaturated esters [913,914]. Nickel dibromide catalyzed Michael type addition of aryl halides to α,β-unsaturated carbonyl compounds under electrochemical conditions [915]. Nickel catalyzed the four-component coupling of α,β-unsaturated cyclic ketones with an alkyne, alkynylzincs, and trimethylsilylchloride forming yne-ene-ones [916]. γ,γ-Dialkoxyallylzirconium species were allowed to react with acrylamide (Eq. (133)) [917] and α,β-unsaturated-*N*-acyloxazolidinones [918] affording cyclobutanes and cyclopropanes, respectively. Copper(I) and palladium catalyzed a sequential Michael addition-cycloisomerization (Eq. (134)) [919].



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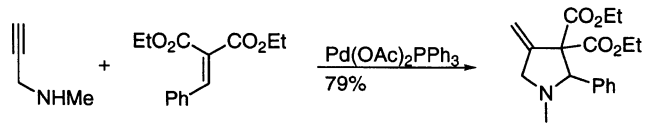
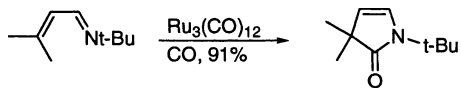


(134)

2.3. Acylation reactions excluding most hydroformylations

2.3.1. Carbonylations of alkenes

Ruthenium catalyzed the carbonylative [4 + 1] cycloaddition of enimines forming γ-lactams (Eq. (135)) [920]. Related rhodium-catalyzed cycloadditions of vinylallenes were reported (Eq. (136)) [921,922]. Nickel mediated the carbonylative cycloaddition of dienals to bicyclic compounds (Eq. (137)) [923].



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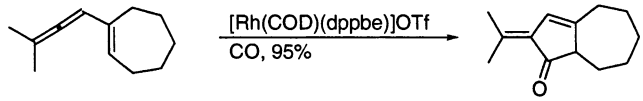

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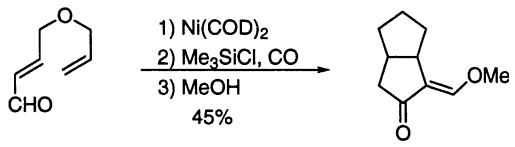
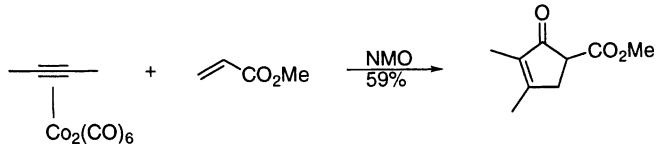


(137)

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

Metal mediated [2 + 2 + 1] cycloadditions forming cyclopentenones continued to be extensively studied, in particular the cobalt mediated, Pauson–Khand reaction [924–931]. Catalyst systems for this reaction, including $Co_2(CO)_6$-alkyne complexes [932], $CoBr_2$/Zn in toluene/benzene [933], $Co_2(CO)_8$ together with amines [934], were developed. Molecular sieves were shown to substantially improve the yields of the Pauson–Khand reaction [935]. Addition of alkyl methylthiolate promoted both intra- and inter-molecular reactions [936]. A polymer bound, recyclable, morpholine *N*-oxide was used as a promoter [937]. One of the carbon monoxide ligands of alkyne–dicobalt hexacarbonyl complexes was replaced with amines, phosphines, or a tethered sulfide [938,939]. It was shown that cobalt–alkyne complexes having a (*R*)-BINAP ligand cannot be used to induce chirality in the Pauson–Khand reaction [940].

Reactions of electron-deficient alkenes were reported (Eq. (138)) [941]. Vinyl esters were used as ethylene equivalents (Eq. (139)) [942]. Dimethylacetylenedicarboxylate cobalt complexes participated in the Pauson–Khand reaction [943].



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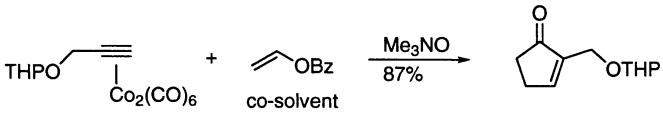
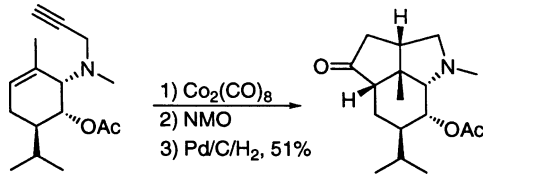


(139)

A synthetically interesting Pauson–Khand reaction is seen in Eq. (140) [944]. Asymmetric inductions were reported in intramolecular Pauson–Khand reactions of optically active enynes derived from allylglycine (Eq. (141)) [945], sugars [946–948] and diols [949]. Inter- and intramolecular cycloadditions of optically active vinyl- or alkynyl sulfones were described (Eq. (142)) [950,951]. A tandem aminocarbonylation-Pauson–Khand reaction was developed (Eq. (143)) [952].



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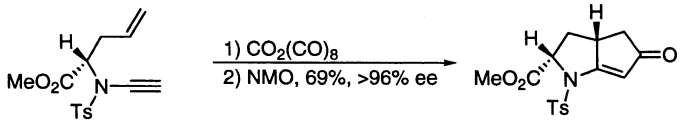

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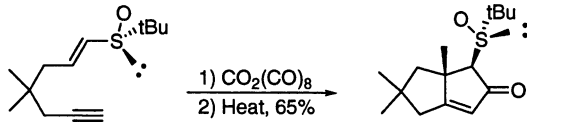
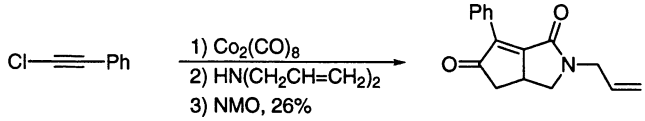
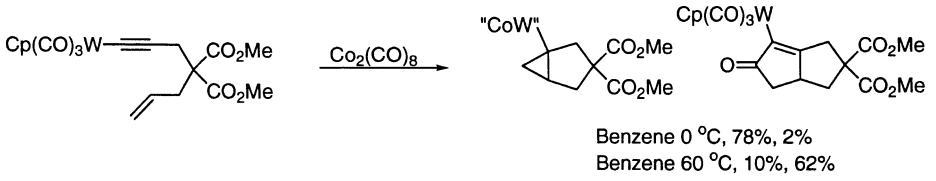


(142)



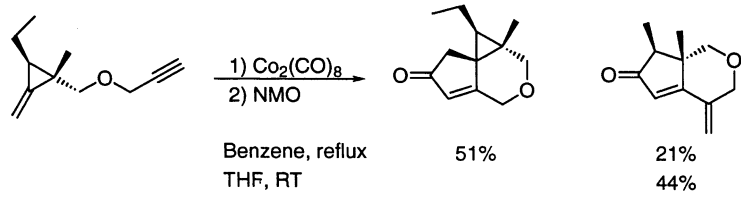
(143)

A novel entry into Pauson–Khand type products was reported. Reaction of alkynyl–tungsten complexes with dicobalt octacarbonyl gave either cyclopropanated products or cyclopentenones (Eq. (144)) [953]. The cyclopropanated product was shown to be a precursor to the cyclopentenone.



(144)

An unusual rearrangement was observed upon reaction of a methylenecyclopropane tethered alkyne (Eq. (145)) [954]. An allenic, molybdenum hexacarbonyl mediated, Pauson–Khand type reaction was used as a key step in a total synthesis (Eq. (146)) [955]. Titanocenes catalyzed intramolecular Pauson–Khand type [2 + 2 + 1] cycloadditions. Both racemic and enantioselective reactions were reported (Eq. (147)) [956–958].



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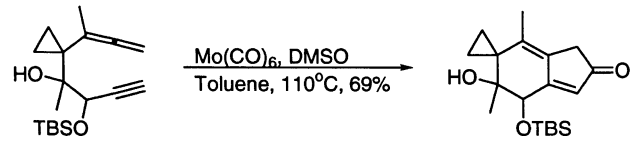

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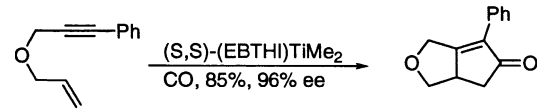
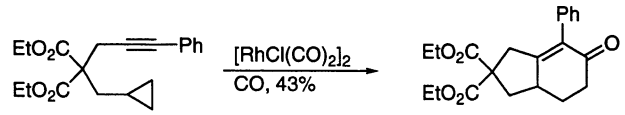
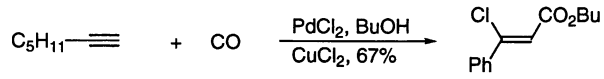


(147)

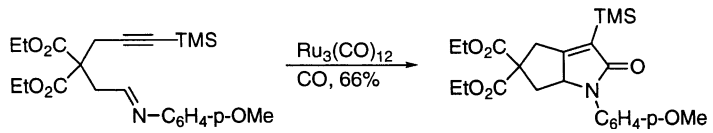
In the presence of carbon monoxide, $[RhCl(CO)_2]_2$ catalyzed the intramolecular transformation of 4-pentynyl cyclopropanes to bicyclo[4.3.0]nonenones via cleavage of the cyclopropane ring (Eq. (148)) [959]. Rhodium catalyzed the carbonylation of arylalkynes in the presence of alcohols to form indanones [960]. Palladium catalyzed the chloro-carbonylation of terminal alkynes (Eq. (149)) [961]. Ruthenium catalyzed the intramolecular reaction of yne-imines with carbon monoxide, leading to bicyclic lactams (Imine–Pauson–Khand) (Eq. (150)) [962].



(148)



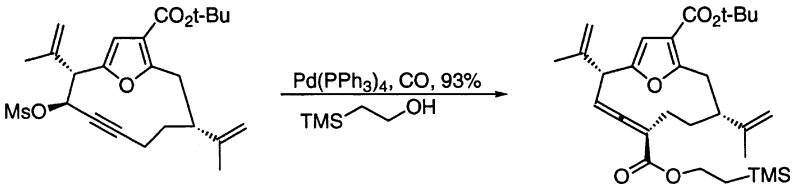
(149)



(150)

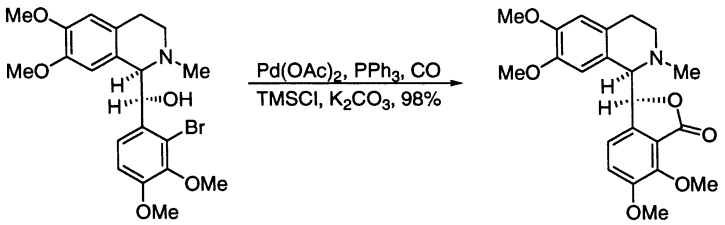
2.3.3. Carbonylation of halides and triflates

Palladium(0) complexes catalyzed the carbonylation of a variety of substrates to form carboxylic acids and derivatives. Esters and amides were prepared by carbonylation of halides and triflates. Alkoxycarbonylation in water catalyzed by an amphiphilic resin-supported palladium catalyst was developed [963]. The use of polymer supported alcohols (and amines) was reported [964]. Vinyl halides and triflates [21,62,147,965–969], aryl halides and triflates [171,472,970,971], β-carboline-4-triflate [180], chloropyridines [972–974], 2-chloropyrimidines and 2-chloromethylpyrimidine [975], 1,2-difluoro-1-iodoalkenes [976], 2-fluoro-1-alkenyl-1-iodonium salts [977], aryl halides [978,979], aryl diazo compounds [409], and 4-iodoquinoline [434] were carbonylated, forming esters or amides. A propargylic mesylate was alkoxycarbonylated, forming an allenylester (Eq. (151)) [891].



(151)

Formations of lactones by carbonylation of an aryl iodide followed by intramolecular addition of an alcohol in supercritical carbon monoxide were reported [980]. Palladium-catalyzed lactone and lactam formation was utilized in organic synthesis (Eq. (152)) [981]; [982]; [224]; (Eq. (153)) [983]. Palladium-catalyzed carbonylation of aryl iodides followed by intramolecular trapping with a tethered urea [984].



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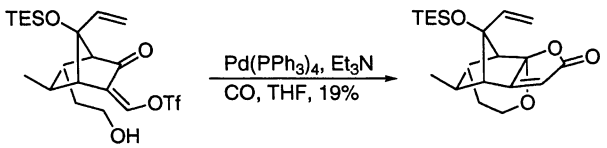
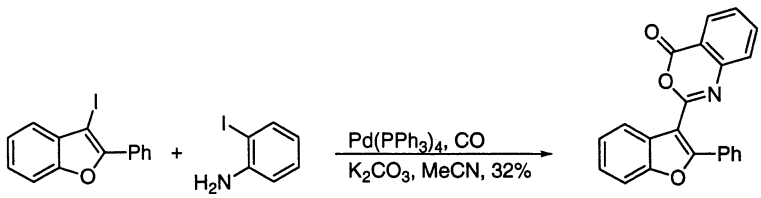


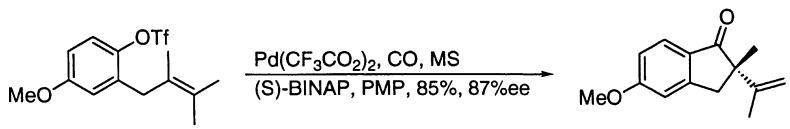
(153)

Amides were prepared by palladium-catalyzed carbonylation of vinyl iodide [455], allylic carbonates [985] bromopyridines [986], and benzyl and aryl halides [987] in the presence of an amine. Carbonylation of vinyl iodides in the presence of hydrazines gave hydrazides [988]. Palladium(0) complexes were used to carbonylate aryl halides and triflates, in the presence of water, to form carboxylic acids [989–991]. A vinylbromide was also carbonylated affording an α,β-unsaturated carboxylic acid [992]. Double carbonylation of iodopyridines gave α-keto amides and esters [993]. An interesting double carbonylation of 3-iodobenzofuran was reported (Eq. (154)) [70]. Palladium catalyzed the carbonylation of iodonium salts with chloroform under basic conditions to form carboxylic acids [994].



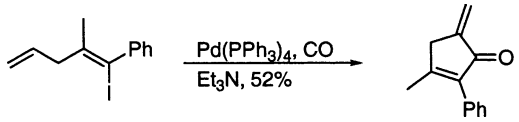
(154)

Palladium-catalyzed carbonylation of iodonium salts, in the presence of potassium aryltrifluoroborates, afforded unsymmetrically substituted diarylketones [995]. Nickel catalyzed carbonylative cross-coupling of organostannanes with iodonium salts forming unsymmetrically substituted ketones [996]. Aryl and heteroaryl boronic acids [997] and $NaBPh_4$ [998] were coupled with acid chlorides, using palladium catalysts, to form unsymmetrically substituted ketones. Palladium catalyzed the enantioselective carbonylation cyclization of aryl and alkenyl triflates (Eq. (155)) [999].

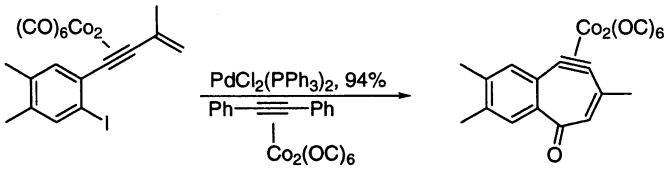


(155)

α-Methylenecyclopentenones were prepared by carbonylation of 1-iodo-1,4-dienes (Eq. (156)) [58]. An interesting carbonylation followed by alkene insertion of a cobalt alkyne complex was reported (Eq. (157)) [1000]. The carbonyl carbon was found to be derived from an added alkyne dicobalt hexacarbonyl complex.



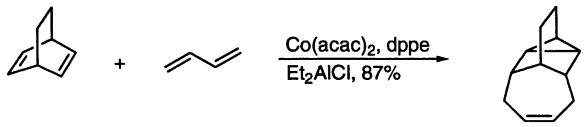
(156)



(157)

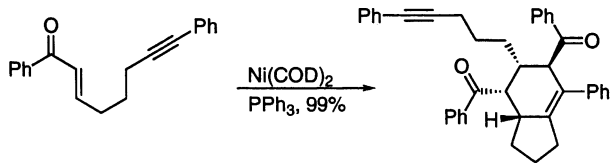
2.4. Oligomerization (including cyclotrimerization/cyclobenzannulation/cycloaddition)

Cyclopentadiene cobalt(I) complexes mediated or catalyzed the [2 + 2 + 2] cyclobenzannulation of diyne-ynes forming aromatic compounds [1001,1002]. Palladium also catalyzed this reaction [1003]. Cobalt catalyzed the [4 + 2 + 2] cycloaddition of bicyclo[2.2.2]octadienes with butadiene (Eq. (158)) [1004].



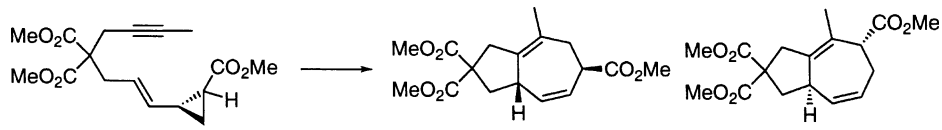
(158)

The diastereoselectivity of the cobalt mediated [2 + 2 + 2] cycloaddition of substituted linear enediynes was improved by introduction of an electron withdrawing group [1005,1006]. A nickel and aluminum catalytic system was developed for intermolecular selective cycloaromatization of enones with alkynes [1007]. Titanium complexes supported on a calixarene ligand catalyzed highly regioselective alkyne cyclotrimerizations [1008]. Alkyne cyclotrimerization forming benzenes was catalyzed by dicobalt octacarbonyl [557,1009,1010], Grubbs' metathesis catalyst [1011], nickel [539,1012], and rhodium [1013]. A tandem, dicobalt octacarbonyl-catalyzed, [2 + 2 + 1]–[2 + 2 + 2] cycloaddition reaction of diynes was reported [1014]. Nickel catalyzed [2 + 2 + 2] cycloadditions (Eq. (159)) [1015].



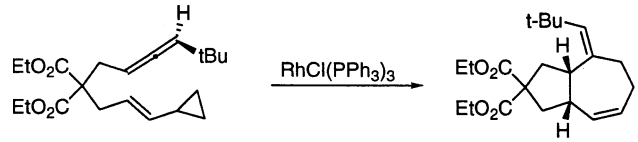
(159)

Intramolecular [5 + 2] cycloaddition of alkyne-vinylcyclopropanes was catalyzed by rhodium. The regio- and stereoselectivity were studied [1016]. The regiochemistry of the cycloaddition was controlled by the catalyst used (Eq. (160)) [1017]. Rhodium also catalyzed the closely related [5 + 2] cycloaddition of allenes-vinylcyclopropanes (Eq. (161)) [1018]. A complete transfer of chirality was observed. Free hydroxy groups are tolerated under the reaction conditions (Eq. (162)) [1019].



(160)

$Rh(PPh_3)_3OTf$, 81%, 20:1
$[Rh(CO)_2Cl]_2$, 93%, 1:11



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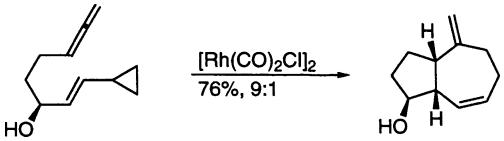
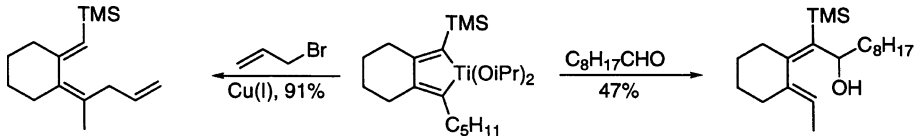


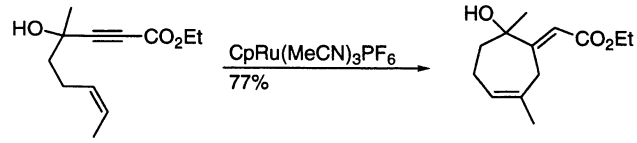
(162)

Palladium catalyzed the cocyclization of arynes, with alkynes selectively forming phenanthrenes and naphthalenes [1020,1021]. Related cyclotrimerizations of arynes were reported [1022]. Bicyclic titanacycles, prepared in situ, reacted with aldehydes and allylic halides to form polyfunctionalized cyclic compounds (Eq. (163)) [1023].



(163)

Palladium-catalyzed intramolecular enyne cycloisomerization reactions continued to be developed [1024–1027]. Ruthenium catalyzed cycloisomerization of 1,6-enynes initiated by carbon–hydrogen bond activation (Eq. (164)) [1028]. A new ruthenium catalyst proved to be effective in intermolecular enyne coupling reactions [896]. Ruthenium catalyzed coupling of alkenes with alkynes in the presence of a chloride source to give *E*-vinyl chlorides [1029]. Ruthenium catalyzed the intermolecular allene–alkene coupling forming 1,3-dienes (Eq. (165)) [1030]. Allenes having a tethered alcohol underwent cycloetherification (Eq. (166)) [1031]. Nickel catalyzed the tandem intramolecular Heck type reaction-cyanation of 2-bromo-1,6-dienes, forming five-membered rings containing a cyanomethyl and a methylene group [1032].



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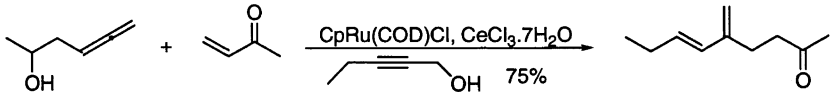

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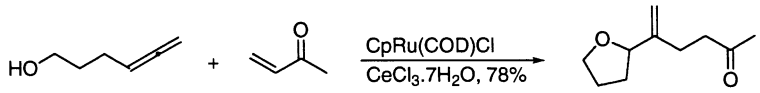
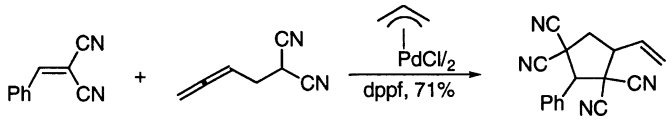
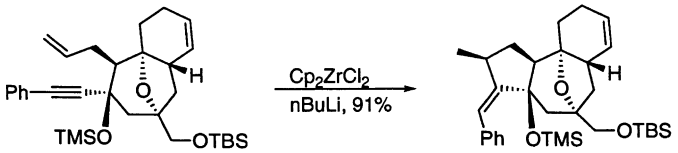


(166)

Palladium catalyzed a formal [3 + 2] cycloaddition via hydrocarbonation of allenes (Eq. (167)) [1033]. Zirconocene mediated enyne cyclization was used in synthesis (Eq. (168)) [464].

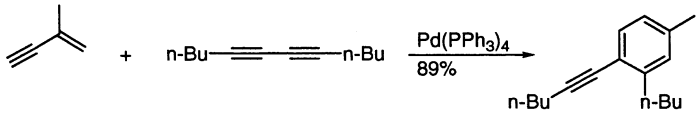


(167)



(168)

Furans were prepared from 4-yne-2-ene-1-ols using a ruthenium catalyst [1034], or by cycloisomerization of γ-yne-ones using a palladium catalyst [913]. Palladium catalyzed [4 + 2] cross-benzannulation reactions of conjugated enynes with dienes and triynes (Eq. (169)) [1035]. Related reactions of enynes were developed [1036].



(169)

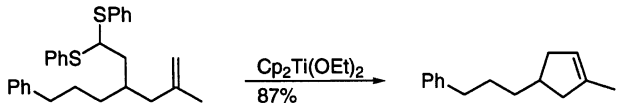
Evidence for a concerted mechanism in a palladium trimethylmethylenemethane cycloaddition was presented [1037].

2.5. Rearrangements

2.5.1. Metathesis

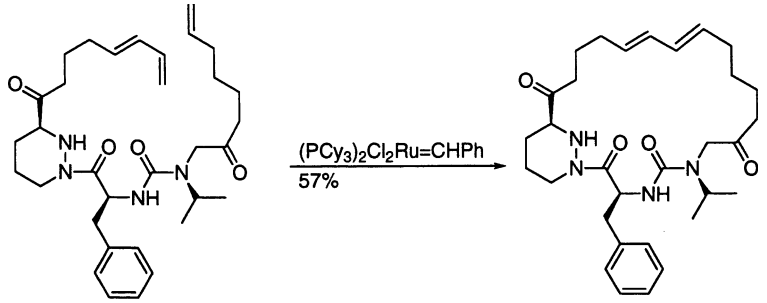
A substantial number of reports on ring-closing metathesis reactions using Grubbs' type ruthenium catalysts were published. The thermal stability and decomposition pathways of several ruthenium catalysts were studied [1038]. Addition of tris(hydroxymethyl)phosphine to the crude reaction mixture simplified the removal of the catalyst [1039]. Allylic hydroxy groups exerted a large activating effect on the annulation [1040]. New air and water tolerant ruthenium complexes were developed, some of which can be recycled [1041,1042]. Ruthenium catalysts having an imidazolinylidene carbene ligand were developed [1043–1049]. Ruthenium allenylidene complexes effectively cata-

lyzed the ring-closing metathesis of dienes and dienynes [1050]. Polymer supported, recyclable catalysts were reported [1051,1052]. Ring-closing metathesis based linkers were used in solid-phase synthesis [1053,1054]. Ring-closing metathesis was conveniently achieved by heating and irradiating of the diene substrate in the presence of a catalytic amount of *para*-cymene ruthenium dichloride dimer and tricyclohexylphosphine [1055]. Schrock's molybdenum catalyst was also employed in ring-closing metathesis reactions forming carbohydrates [872,1056] and cyclitols [1057]. Chiral molybdenum binol complexes catalyzed the enantioselective ring-closing metathesis [1058]. An interesting metathesis reaction of unsaturated thioacetals employing titanocene was developed (Eq. (170)) [1059].



(170)

Medium- and large-sized oxacycles [1060–1063], medium- and large-sized carbocycles [1064], oxepanes [1065,1066], catenanes [1067], silyloxycycloheptene [1068], cyclic silaketals [1069], cyclic peptides [1070], cyclic phosphinates [1071], cyclic phophine oxides [1072], spirocycles [689,857,1073,1074], cyclitols [1075,1076], five to seven-membered nitrogen heterocycles [1077–1080], bicyclic lactams [1081–1083], macrocyclic organometallic compounds [1084,1085], five- and six-membered oxygen heterocycles [1086–1091], cyclic phosphonamides [1092], cyclic sulfonamides [1093], cyclic disilanes [1094], were all prepared using the ring-closing metathesis methodology. Ring-closing metathesis saw substantial use in organic synthesis [44,54,61,162,325,476,969,1095–1124]. Some more complex examples are depicted in Eq. (171) [1125], Eq. (172) [150], Eq. (173) [1126], Eq. (174) [1127], Eq. (175) [1128], Eq. (176) [1129], Eq. (177) [479].



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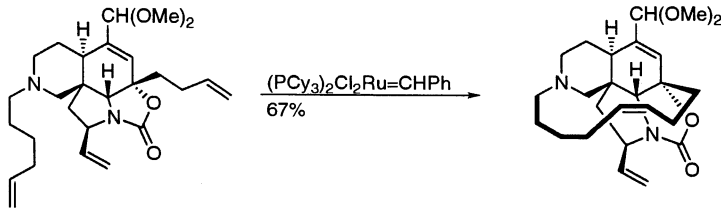

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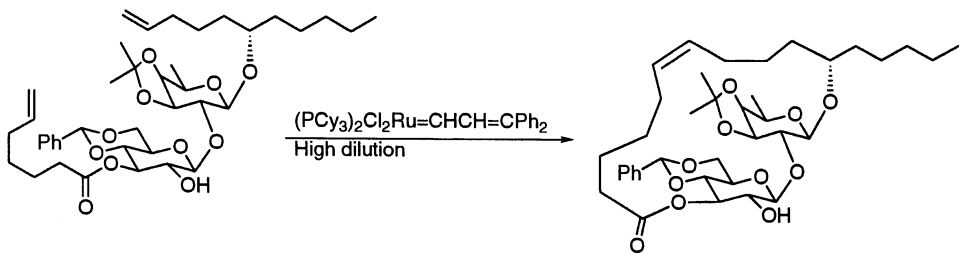
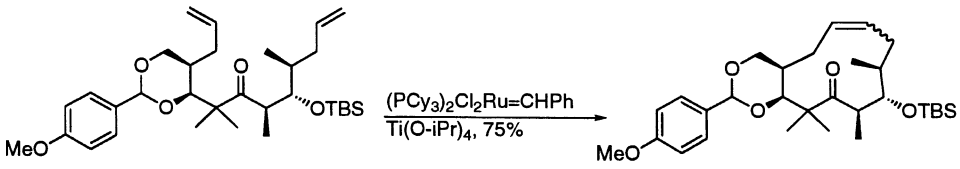


(173)



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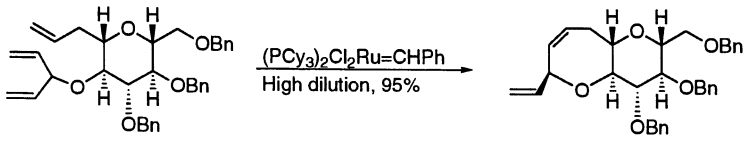

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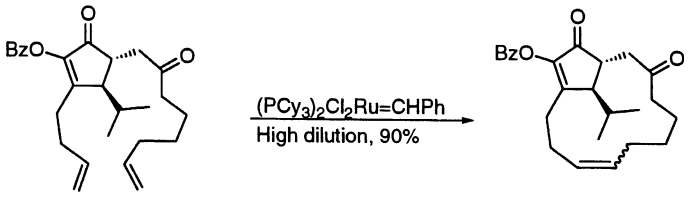

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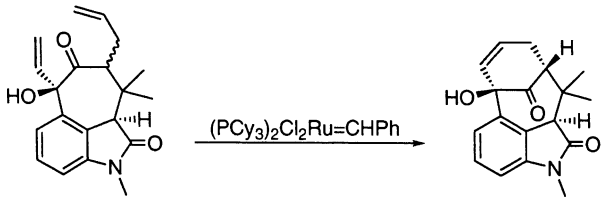
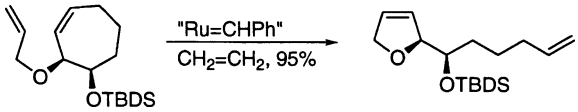
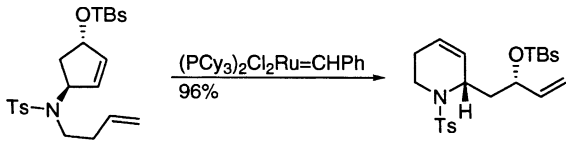


(177)

Ring-opening ring-closing methodologies were developed [1130]. A couple of examples are depicted in Eq. (178) [1131] and Eq. (179) [1132]. Both ruthenium and molybdenum complexes were successfully used. Ring-opening cross-coupling metathesis of 2-substituted 7-oxabicyclonorbornene gave, stereoselectively, trisubstituted tetrahydrofurans [1133]. Cyclooctadiene, catalyzed by $MoCl_5/SiO_2$–$SnMe_4$ [1134], or tricyclic alkenes using Grubbs' catalyst [1135] were reacted with ethene in a similar fashion. An asymmetric ring-opening cross-coupling metathesis was published [1136].

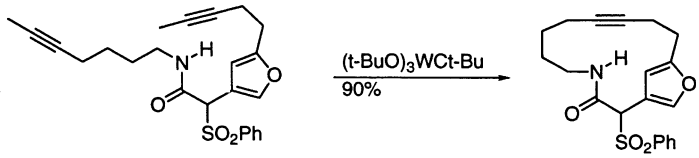


(178)



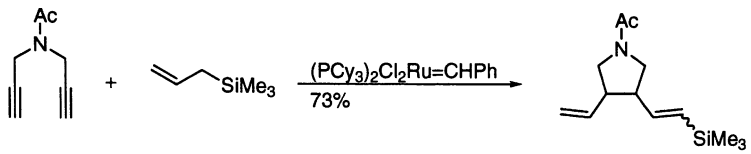
(179)

Tungsten carbynes, molybdenum hexacarbonyl in the presence of 4-chlorophenol, and molybdenum complexes ($Mo[Nt\text{-}Bu)(Ar)]_3$) catalyzed ring-closing alkyne–alkyne metathesis (Eq. (180)) [698,1137]. Molybdenum hexacarbonyl in the presence of 4-chlorophenol catalyzed the intermolecular alkyne metathesis of arylalkylethynes to diarylethynes [549].



(180)

Alkene–alkene cross-coupling metathesis did not enjoy the same amount of attention as ring-closing metathesis. Ruthenium catalyzed a tandem diyne cycloisomerization cross-coupling metathesis process (Eq. (181)) [1138]. Grubbs' catalyst was used in cross-coupling metathesis of terminal alkenes with acrolein acetals (Eq. (182)) [1139], allyl carbohydrates [1140,1141], and dimerizations of vinyl carbohydrates [1142] and long chained hydrocarbons [1143].



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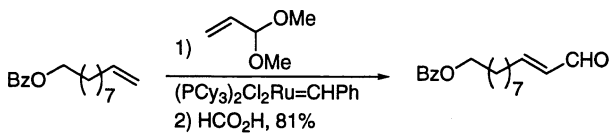
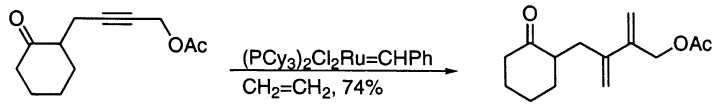


(182)

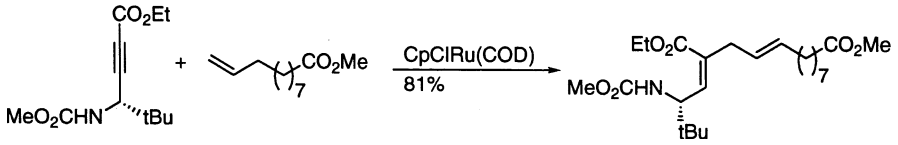
Intramolecular enyne metathesis was used to prepare a 2-vinylbutenolide [1144]. Grubbs' catalyst promoted intermolecular enyne cross-coupling metathesis [1145–1147]. 1,3-Dienes were prepared from alkynes and ethene (Eq. (183)) [1148].



(183)

2.5.2. Alkene isomerization including cycloisomerization

Enyne coupling reactions continued to be developed. Ruthenium catalyzed intermolecular enyne coupling, forming γ-amino-α,β-unsaturated esters (Eq. (184)) [1149]. Vinyl methylene cyclopentanes were obtained from titanocene-catalyzed enyne-cycloisomerizations [1150]. Tungsten pentacarbonyl THF complex catalyzed the cycloisomerization of aromatic enynes (Eq. (185)) [1151]. Aromatic double bonds was utilized as the ene component. Palladium catalyzed [4 + 4] cycloadditions of allene-enes to form cyclooctadienes and cyclohexenes (Eq. (186)) [1152].



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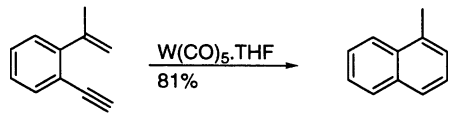

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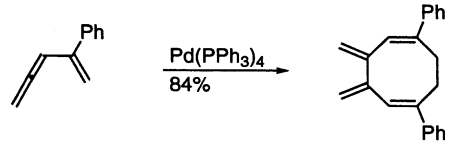
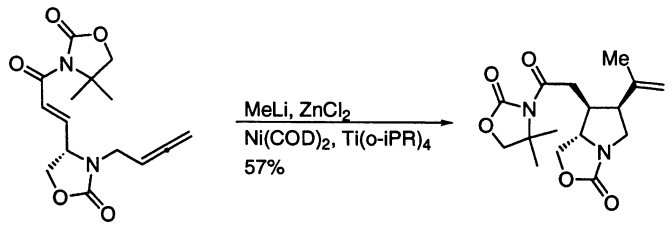


(186)

Ruthenium catalyzed cycloisomerization of 1,6-dienes to *exo*-methylenecyclopentanes [1153]. Nickel catalyzed intramolecular allene–alkene and alkyne–alkene cyclization (Eq. (187)) [1154]. Nickel catalyzed cyclizations of 1,6-dienes to form pentalenes in the presence of diethylzinc and zinc dichloride (Eq. (188)) [1155]. Triethylsilane promoted the palladium-catalyzed cycloisomerization of 1,6-dienes to cyclopentenes [1156].



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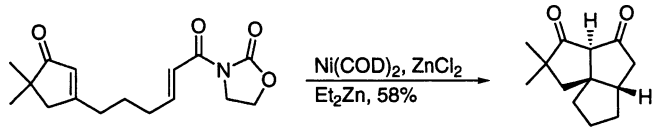
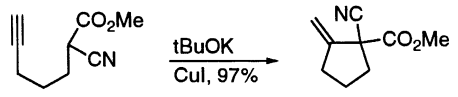


(188)

Iridium(I) catalyzed the double bond isomerization of allyl silyl ethers to silyl enol ethers [1157]. Rhodium and palladium complexes catalyzed alkene isomerizations [138,1158]. Rhodium catalyzed the isomerization of allylic alkoxides to enolates [1159]. Copper mediated the intramolecular carbocupration of alkynes bearing a stabilized nucleophile (Eq. (189)) [1160]. Cobalt(I) catalyzed cycloisomerizations of ε-acetylenic β-keto esters affording methylene cyclopentanes (Eq. (190)) [928].



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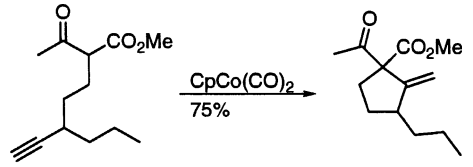
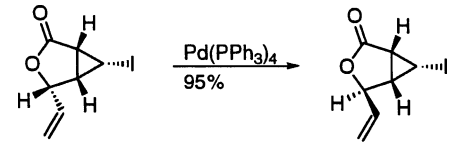


(190)

2.5.3. Rearrangements of allylic and propargylic compounds

Palladium-catalyzed allylic transposition of acetates to give the more highly substituted alkene [186,1075,1161]. Palladium-catalyzed rearrangements of allylic silanols to allylic silanes [1162] and of allylic trichloroacetimidates to allylic trichloroacetamides [1112]. High enantioselectivity was observed upon rearrangement of allylic imidates to allylic amides using palladium catalysts [1163,1164]. A related enantioselective rearrangement of *O*-allylic thiocarbamates to *S*-allylic thiocarbamates was reported [1165]. *Cis–trans* isomerizations of vinylic lactones (Eq. (191)) [1166] and vinyl-aziridides [1167,1168] were catalyzed by palladium complexes. Fischer carbene complexes were rearranged to β,γ-unsaturated ketones in the presence of a palladium catalyst (Eq. (192)) [1169].



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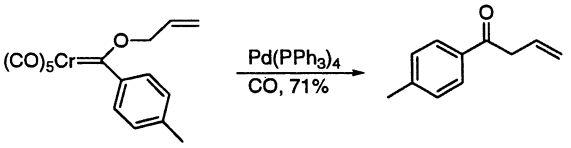
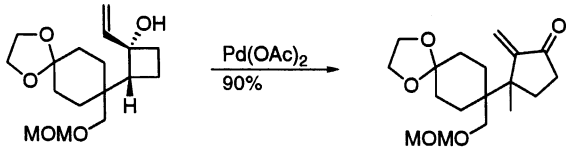


(192)

2.5.4. Skeletal and miscellaneous rearrangements

Palladium complexes catalyzed asymmetric Claisen rearrangements of allyl imidates [1170], Cope rearrangements of 1,5-dienes [1171], and asymmetric vinylcyclopropane-cyclopentene rearrangements [1172]. An iridium complex-catalyzed Cope rearrangement was developed [1173]. Platinum(II) and palladium(II) complexes catalyzed asymmetric hetero Diels–Alder reactions [1174] and 1,3-dipolar cycloaddition of imine-*N*-oxide with unsaturated amides [1175]. Cobalt (III)-salen catalyzed the asymmetric [2,3]sigmatropic rearrangement of allyl aryl sulfides [1176]. Palladium catalyzed a vinyl oxazolidinone to vinyloxazoline rearrangement [1103].

Palladium-catalyzed ring expansion of 1-vinylcyclobutan-1-ols to α-methylene cyclopentanones [1177,1178] (Eq. (193)) [1179]. A related ring expansion leading to cyclopentanones is shown in Eq. (194) [1180]. Palladium-catalyzed arylation, followed by ring expansion of hydroxy-allenylindanones, gave 4-oxa-α-tetralones [1181]. Palladium catalyzed a ring-opening ring-closing reaction of dihydropyranes (Eq. (195)) [1182].



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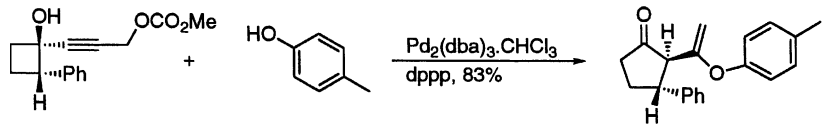

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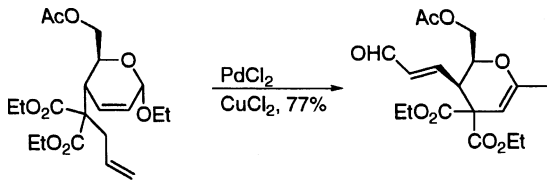


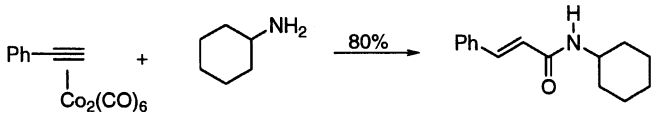
(195)

3. Functional group preparation

3.1. Halides, amides, nitriles, azides, imines

Grubbs' metathesis catalyst was used for Kharasch type addition of chloroform to alkenes, affording chlorinated hydrocarbons [1183]. Palladium catalyzed the cyanation of aryl and heteroaryl iodides and bromides with copper cyanide [1184] and zinc dicyanide [538,1185]. Palladium catalyzed the cyanation of carbohydrate derived allylic acetates, alcohols, and carbonates [1024,1186]. Reaction of hexacarbonyldicobalt alkyne complexes with an excess of amine produced α,β-unsaturated amides (Eq. (196)) [1187]. Palladium catalyzed the reaction of methylvinyl ketone

and ethylpropiolate in the presence of lithium chloride to give a vinyl chloride with defined stereochemistry (Eq. (197)) [971].



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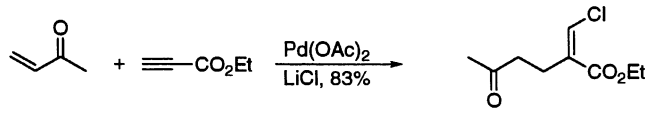


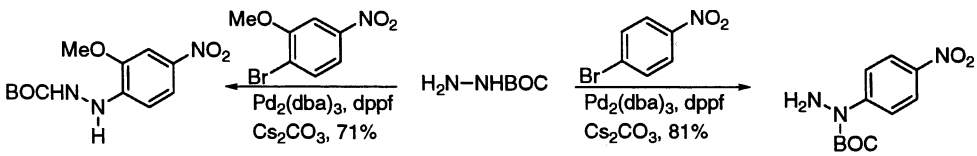
(197)

3.2. Amines, alcohols

A large variety of aromatic halides were aminated using palladium based catalyst systems [850,1188–1193]. Low catalyst loading (0.5% $Pd(OAc)_2$ was achieved using Xantphos as the ligand [1194]. Palladium-catalyzed amination of aromatic chlorides was reported in a number of cases [257,1195–1197]. Nickel 2,2′-bipyridine complex-catalyzed couplings of aryl chlorides with secondary amines and piperazines were described [1198]. Aminations of aryl bromides catalyzed by palladium particles immobilized onto a metal oxide support or in NaY Zeolite were developed [1199].

Unsymmetrical alkyldiarylamines were prepared by sequential reactions of a primary amine with two different aryl bromides. Two different catalyst systems were used [1200]. Palladium catalyzed the arylation of aminoferrocenes [1201], aza-crown ethers [1202], aza-cyclophanes [1203], benzophenone imine [1204], benzophenone hydrazone [1205], lactams [1206], chiral 1,2-diaminoethanes [1207], 3-aza-1,5-diaminopentane [1208], diamines (monoamination) [1209], amino group of nucleotides [1210,1211], and amino functionalized sugars [1212].

The regioselectivity of the arylation of *N*-BOC protected hydrazine depended on the substitution pattern of the aryl halides (Eq. (198)) [1213]. Sulfoximines were used in place of amines (Eq. (199)) [1214]. Heteroaryl imidoyl chloride were used in amination reactions (Eq. (200)) [1215].



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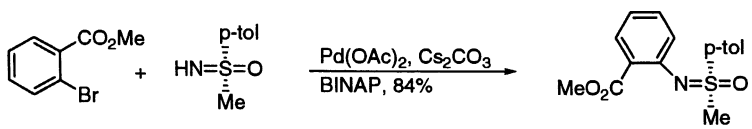

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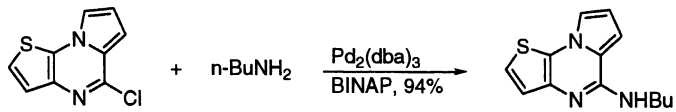
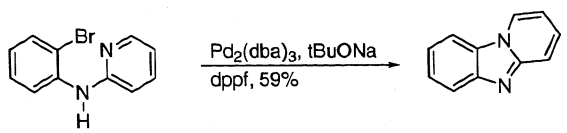


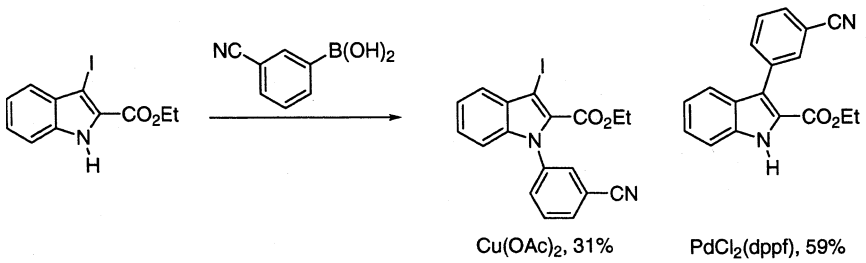
(200)

Intramolecular reaction of aromatic bromides with tethered secondary amides and carbamates gave five- to seven-membered rings [1216]. Intramolecular amination using the pyridine nitrogen as the nucleophile was reported (Eq. (201)) [163].



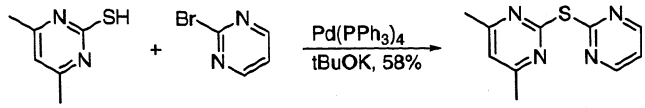
(201)

Transition metals other than palladium also catalyzed amination reactions [1217]. Copper(II) salts catalyzed the coupling of aryl halides with imidazoles [1218], and of arylamines and imidazoles using triarylbismuth compounds [1219,1220]. Copper(II) also catalyzed amination of aryl boronic acids [1221,1222]. Depending on the catalyst, either an amination or a Suzuki type coupling was achieved (Eq. (202)) [333].



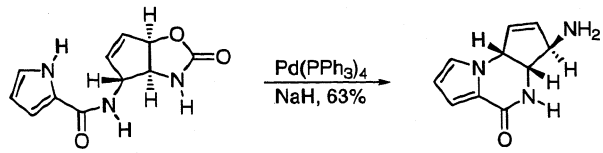
(202)

The closely related formation of ethers continued to be developed. Palladium catalyzed the carbon oxygen coupling forming diaryl and aryl *tert*-butyl ethers [1223–1225]. Copper salts also catalyzed the coupling of aryl halides with aryl alcohols [202,353]. A similar palladium-catalyzed thioether synthesis was reported (Eq. (203)) [1226].



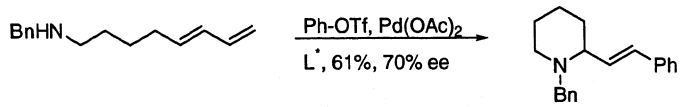
(203)

Palladium catalyzed a variety of aminations of allylic substrates via η^3-allyl intermediates [1227,1228]. A number of asymmetric allylic aminations were published [1229–1236]. Rhodium catalyzed the enantiospecific allylation of sulfonamides [754]. Rhodium catalyzed amination of allylic carbonates with retention of regio- and stereochemistry [1237]. Allylic alcohols were aminated with retention of double bond geometry using an iridium catalyst [1238]. Palladium also catalyzed the allylic amination of sulfonamides [1239]. An interesting example of the palladium-catalyzed allylic amination in synthesis is seen in Eq. (204) [1240]. An amino-palladation elimination sequence was also reported.



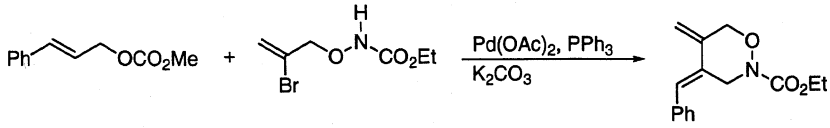
(204)

Polymer bound amines [1241,1242], nucleotides [1243–1245], and amino acids [1079] were used as nucleophiles in allylic aminations. Palladium catalyzed the intramolecular amination of allylic benzotriazoles giving 2-vinylpyrrolidines and piperidines [1246]. An enantioselective domino Heck-allylic amination was used to prepare piperidines and pyrrolidines (Eq. (205)) [1247].



(205)

β-Methylenepyrrolidines were prepared by intramolecular amination of allylic esters attached to a polymer support [1248]. Related diastereoselective allylic aminations furnished α-vinyl substituted nitrogen heterocycles [1249]. Palladium catalyzed a domino allylic amination Heck reaction (Eq. (206)) [1250]. A reversed sequence, i.e. Heck reaction followed by allylic amination, was used to prepare azetidines and tetrahydropyridines (Eq. (207)) [1251]. Mixtures of products were usually obtained.



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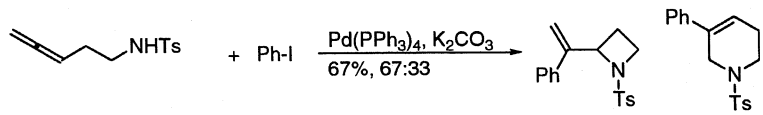
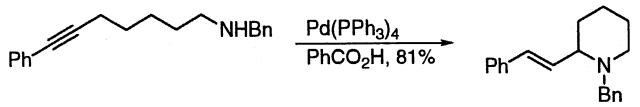


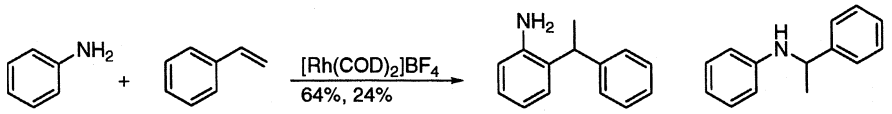
(207)

Palladium catalyzed the allylic amination of anilines using allylic alcohols in the presence of titanium tetraisopropoxide [1252–1254]. Palladium-catalyzed amination of internal alkynes to form allylic amines (Eq. (208)) [1255].



(208)

Cationic rhodium complexes catalyzed amination and alkylation of aromatic amines with styrenes (Eq. (209)) [1256]. Ruthenium catalyzed both alkylation of aromatic amines with styrenes and hydroamination of alkynes (Eq. (210)) [1257]. Organolanthanide complexes catalyzed the intramolecular hydroaminations of aminoallenes [1258] and alkenes (Eq. (211)) [1259]. Ruthenium complexes catalyzed allylic amination of unactivated olefins by nitroarenes [1260].



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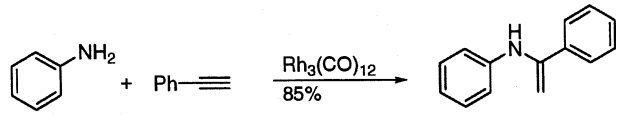

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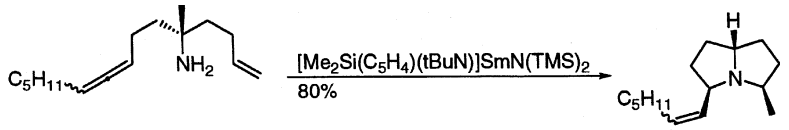
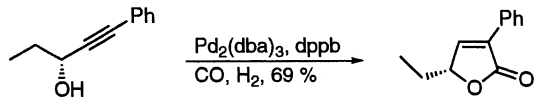


(211)

3.3. Ethers, esters, lactones, acids

Lactones were prepared by palladium-catalyzed hydrocarbonylation of propargylic alcohol (Eq. (212)) [239], by ruthenium-catalyzed [2 + 2 + 1] cycloaddition of ketones, alkenes, and carbon monoxide (Eq. (213)) [1261], and by palladium-catalyzed carbonylative cyclization of allylphenols [1262]. Palladium catalyzed the carbonylation of terminal alkynes, affording maleic anhydrides and lactones [1263,1264]. Formation of iron η^3-allyl complex followed by oxidative ring formation was used in synthesis (Eq. (214)) [149]. α-Methylenelactones were formed upon palladium-catalyzed reaction of tributyltin acetylene and tributyltin β-iodocarboxylate (Eq. (215)) [1265]. α-Methylenelactones and α-methylenelactams were formed by palladium(II)-catalyzed intramolecular cyclizations of alkyne acids and amides [683,1266].



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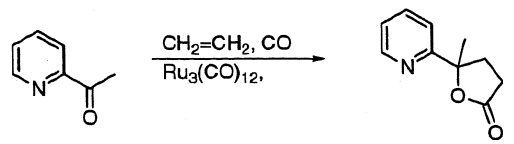
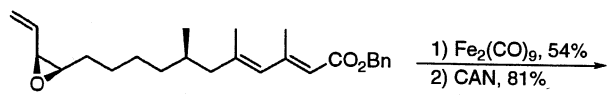
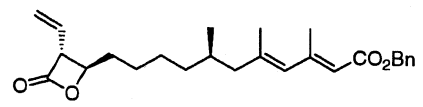


(213)





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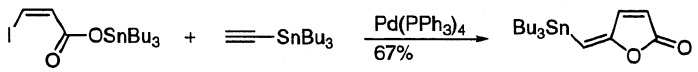
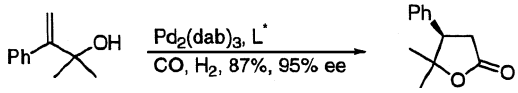


(215)

Palladium catalyzed the enantioselective carbonylation of allylic alcohols forming chiral lactones (Eq. (216)) [1267]. Regioselective synthesis of 3-substituted furan-2-(5*H*)-ones by palladium-catalyzed reductive carbonylation of terminal alkynes were reported (Eq. (217)) [1268]. Palladium catalyzed the formation of cyclic carbonates from allenols, carbon dioxide, and aryl iodides (Eq. (218)) [1269]. An interesting palladium-catalyzed 'hydroxy-palladation–carbonylation–lactonization' sequence was developed (Eq. (219)) [1270].



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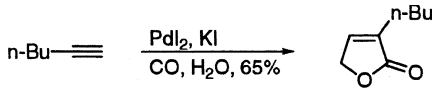
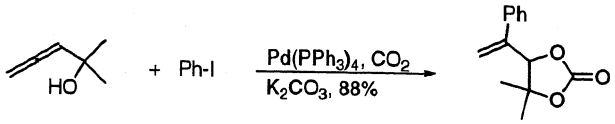
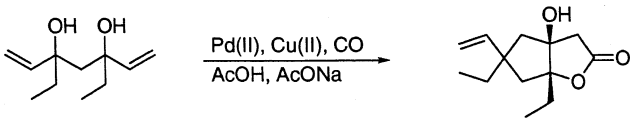


(217)

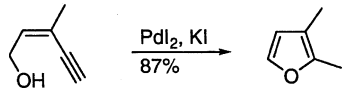


(218)



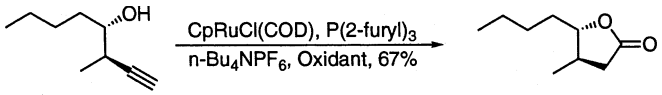
(219)

Palladium catalyzed the cycloisomerization of (*Z*)-2-en-4-yn-1-ols to give furans (Eq. (220)) [1271]. When the reaction was performed under a carbon monoxide–air atmosphere (100 atm total pressure), a furyl acetic acid ester was formed [1272].



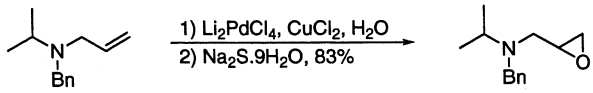
(220)

Ruthenium catalyzed the addition of carboxylic acids to terminal isolated diynes forming enol esters. The regioselectivity of the addition depended on the catalyst used [1273]. In a similar fashion, oxidative dimerization of alkynes in the presence of a carboxylic acids furnished dienolesters [1274]. Ruthenium catalyzed the reaction of formic acid with propargyl alcohol to give β-oxopropyl formate [1275]. Ruthenium catalyzed cycloisomerization–oxidation of homopropargylic alcohols to form γ-butyrolactones (Eq. (221)) [1276].



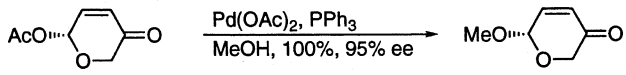
(221)

Vanadyl acetylacetonate-catalyzed epoxidations of allylic alcohols using peroxides continued to be a valuable tool in organic synthesis [138,513,848,1025,1087,1277–1279]. A number of enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *tert*-butyl hydrogen peroxide and an optically active tartrate ester [91,128,132,149,824,1063,1280–1284] were reported. A novel ruthenium porphyrin complex catalyzed the asymmetric epoxidation of alkenes using 2,6-dichloropyridine *N*-oxide as the oxidant [1285]. Homoallylic alcohols were epoxidized employing either vanadyl acetylacetonate [1286] or molybdenum hexacarbonyl as the catalyst [1287]. Palladium-catalyzed hydroxy-chlorination, followed by cyclization, was used to prepare allylic amine epoxide (Eq. (222)) [1288].



(222)

Palladium catalyzed the alkoxylation of an allylic chiral pyrrolinone [1289]. Palladium catalyzed the decarbonylative coupling of diallylcarbonates to diallyl ethers [948]. Palladium-catalyzed allylations of alcohols were reported [1290]. Optically active allylic acetates were stereospecifically substituted with alcohols (Eq. (223)) [1291]. Palladium catalyzed an intramolecular carbonation of alkoxyallenes (Eq. (224)) [1292].



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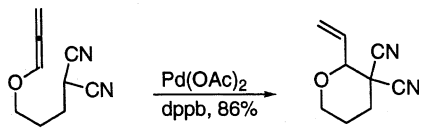
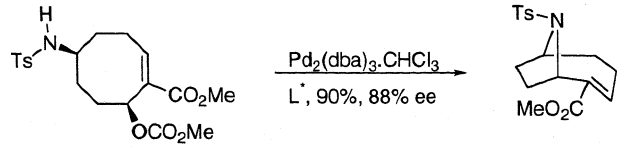


(224)

3.4. Heterocycles

Palladium-catalyzed inter- and intramolecular aminations were used to prepare 7-azabicyclo[2.2.1]heptane [1293] and 9-aza-bicyclo[4.2.1]nonane ring systems (Eq. (225)) [970]. Spirocyclization was also reported (Eq. (226)) [449].



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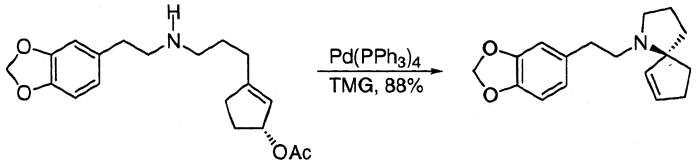
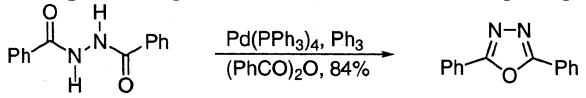


(226)

Palladium catalyzed the cyclization of *N*,*N*′-diacylhydrazines to 1,3,4-oxadiazoles (Eq. (227)) [1294]. Palladium catalyzed the carbonylative heteroannulation of 2-iodothiophenols with allenes and carbon monoxide, forming thiochroman-4-ones (Eq. (228)) [1295]. Related carbonylation of 2-iodophenol in the presence of carbodiimides or isocyanates afforded benzo[*e*]-1,3-oxazin-4-ones derivatives [1296]. Palladium-catalyzed carbonylation of 2-iodoanilines in the presence of acid chlorides, producing 4*H*-3,1-benzoxazin-4-ones [1297].



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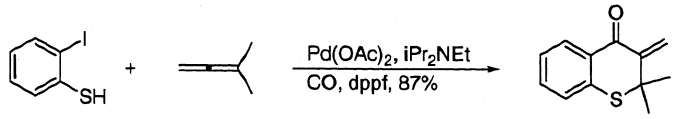
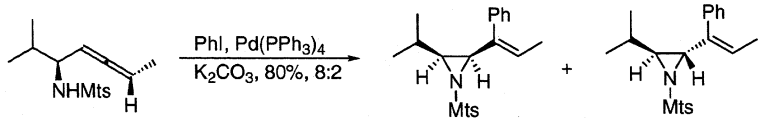


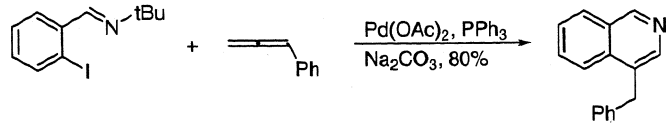
(228)

Insertion of allenes into complexes obtained from oxidative addition of palladium(0) to aryl and vinyl halides, and iodonium salts followed by an intramolecular nucleophilic termination were used to prepare a number of different heterocyclic compounds [47,1298–1300]. Palladium catalyzed the formation of vinyl aziridines from aminoallenes (Eq. (229)) [1301]. A similar reaction forming epoxides was reported [1302]. Vinyl aziridines were also obtained by intramolecular palladium-catalyzed amination of allyl carbonates [1167,1303,1304]. Palladium and nickel catalyzed ring expansion of diazacyclopropanes to give diazetidine-2,4-diones [1305].



(229)

Isoquinolines and pyridines were prepared from imino tethered vinyl and aryl halides and allenes (Eq. (230)) [1306]. A related reaction using alkynes in place of allenes was reported (Eq. (231)) [1307]. Palladium catalyzed the asymmetric hetero- and carboannulation of allenes using functionalized aryl and vinyl iodides. Nucleophilic substituents included tosylamides, alcohols, carboxylic acids, and stabilized carbanions (Eq. (232)) [1308].



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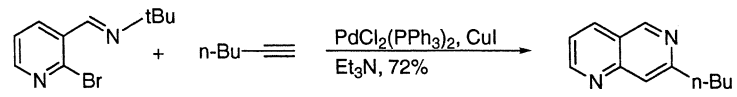

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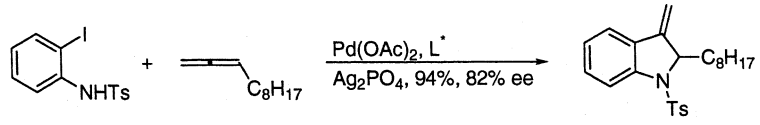
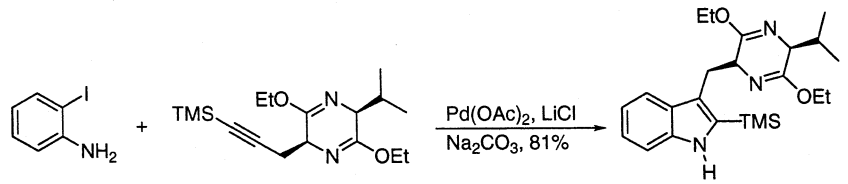


(232)

Palladium catalyzed the intramolcular amino-palladation of 2-(1-alkynyl)anilines affording indoles [1309]. Intramolecular amino-palladation of 2-allyl-azaanilines gave azaindoles [1310]. Related amino-palladations forming α-methylenepyrrolidines were reported [1311]. Indoles were prepared by reaction of TMS-substituted alkynes and 2-iodo-1-amino substituted aromatic compounds (Eq. (233)) [1312] [641,1313]. Palladium-catalyzed oxidative cyclization of *N*-carbamoylaminoalkynes, forming 2-pyrrolidinones (Eq. (234)) [1314]. Titanium and zirconium complexes mediated the formation of 2-(2-keto-1-alkylidene)pyrrolidines from 4-alkynylamines and acyl cyanides (Eq. (235)) [1315].



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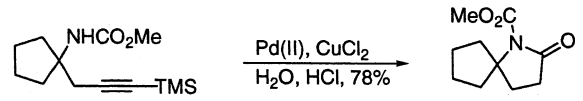

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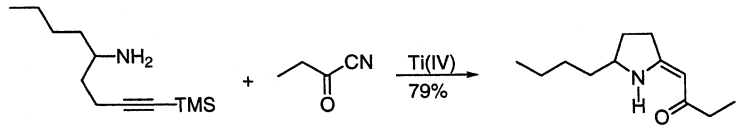
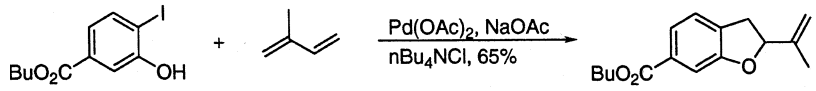


(235)

Palladium catalyzed the formation of dihydrobenzofuran from a 2-iodophenol and isoprene (Eq. (236)) [1316]. Palladium catalyzed the asymmetric intramolecular cyclization to form dihydrofurans (Eq. (237)) [1317]. Ruthenium catalyzed the carbon–carbon coupling of propargylic alcohols with allyl alcohol (Eq. (238)) [1318].



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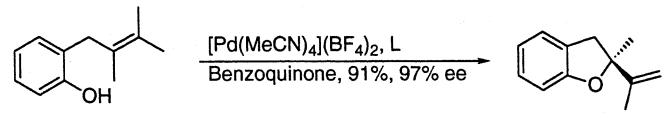
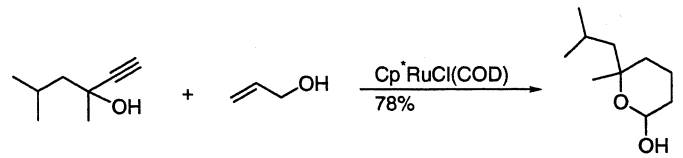
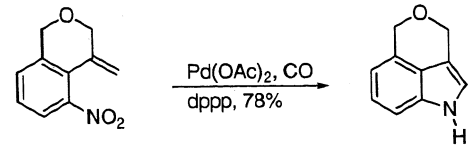


(237)



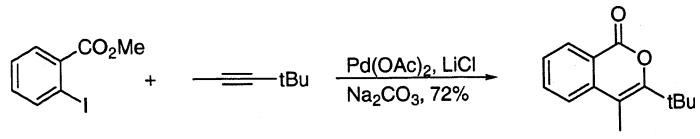
(238)

Palladium catalyzed the reductive *N*-heteroannulation of 2-nitrostyrenes to give indoles [1319]. 3,4-Fused indoles were prepared by a sequential Heck reaction–*N*-heteroannulation (Eq. (239)) [1320]. Selenium also catalyzed this reaction [1321]. The mechanism of the annulation was investigated. Initial reduction of the nitro group to an amine was ruled out as a major mechanistic pathway [1322].



(239)

Palladium catalyzed the reaction of 2-iodo-benzoic acid esters with internal alkynes, forming isocoumarines (Eq. (240)) [1323]. Similar reaction of 2-alkynylbenzoic acids to give isocoumarines or isoquinolinones was reported [1324]. Palladium catalyzed the reaction of 2-iodoaniline imines with terminal alkynes, producing isoindolo-[2,1-*a*]indoles (Eq. (241)) [1325].



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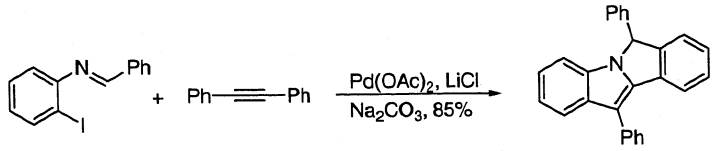
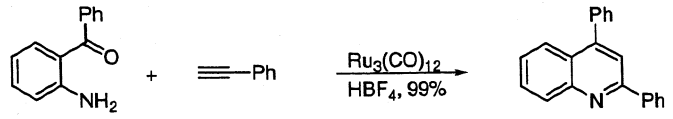


(241)

Ruthenium catalyzed the reaction of 2-aminobenzophenones with terminal alkynes, affording isoquinolines (Eq. (242)) [1326]. Ruthenium catalyzed the formation of 2-ethyl-3-methylquinolines from aniline and triallylamine (Eq. (243)) [1327].



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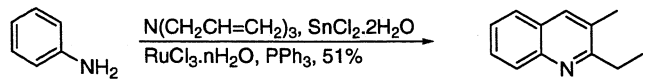
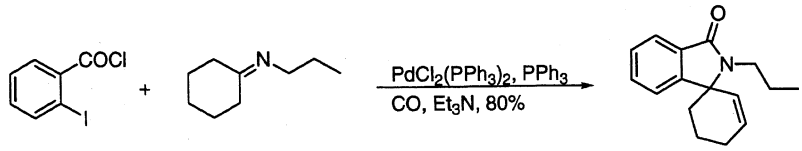


(243)

Palladium catalyzed the reaction of 2-iodo-benzoyl chloride with ketimines and carbon monoxide, forming spirocyclic lactams (Eq. (244)) [1328]. Reaction of 2-bromobenzaldehyde with phenols and carbon monoxide gave 3-alkoxy substituted phthalides (Eq. (245)) [1329]. Employing carboxylates as the nucleophile furnished 3-acyloxysubstituted phthalides [1330]. Palladium catalyzed the reaction of 2-iodobenzylalcohols with alkynes, producing 3-alkylidene-isobenzofurans [1331]. Palladium catalyzed the reaction of benzyl and allyl halides with imines and carbon monoxide, forming β-lactams in a regioselective fashion [1332]. Tungsten hexacarbonyl catalyzed the oxidative carbonylation of alkyldiamines, in the presence of iodine, to form cyclic ureas [1333]. Ruthenium catalyzed the heteroannulation of 3-amino-1-propanol to produce quinolines (Eq. (246)) [1334].



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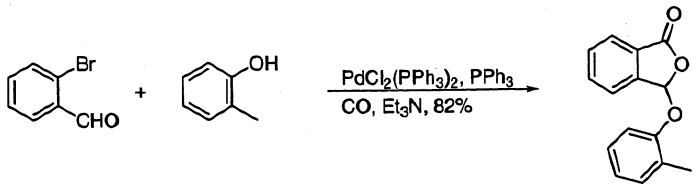

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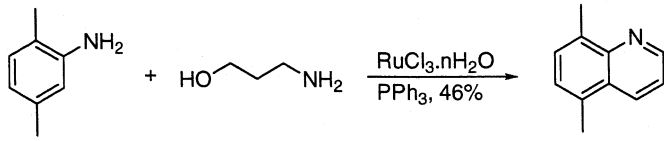
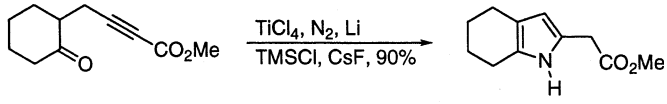


(246)

An unusual synthesis of tetrahydroindoles using titanium–nitrogen complexes derived from molecular nitrogen was developed (Eq. (247)) [1335]. Titanium mediated the cyclization of 1,6-dienes to form *syn*-3,4-disubstituted pyrrolidines (Eq. (248)) [1336]. Nickel catalyzed the stereoselective preparation of quinolizidine, pyrrolizidine, and indolizidine alkaloids by a hydrosilylation–alkylation sequence of ynals (Eq. (249)) [1337].



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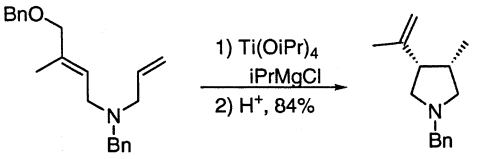

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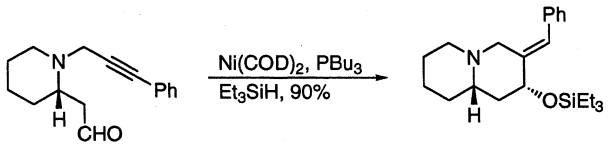
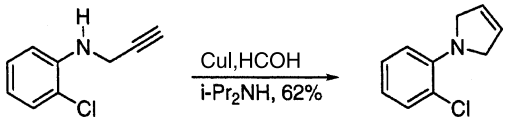


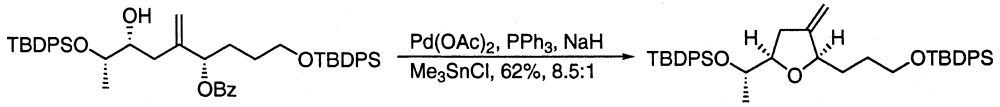
(249)

Copper(I) catalyzed the synthesis of *N*-aryl-2,5-dihydropyrroles (Eq. (250)) [1338]. Copper catalyzed the aziridination of styrene using hypervalent nitrene precursors bearing *N*-heterocyclic rings [1339] and the asymmetric aziridination of styrene using [(*para*-toluenesulfonyl)imino)]phenyliodinane [1340].



(250)

Palladium catalyzed the reaction of iodophenol with terminal alkynes, forming benzofurans [1341]. Intramolecular alkyne hydroxy-palladation–carbonylation of alkynes formed mixtures of substituted tetrahydrofurans [1342]. 3-Carboalkoxy substituted benzofurans were prepared by intramolecular hydroxy-palladation–carbonylation of 2-alkynyl-phenols [1343]. Palladium catalyzed the intramolecular hydroxy-palladation elimination to afford β-methylenetetrahydrofurans (Eq. (251)) [1344]. Palladium catalyzed the enantioselective intramolecular annulation of phenols with tethered allylic carbonates (Eq. (252)) [1345].



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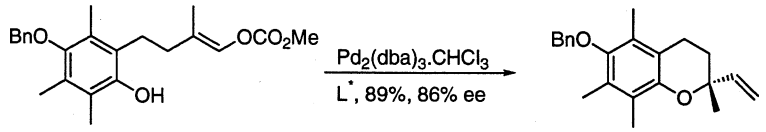
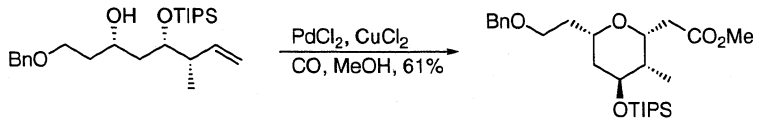


(252)

Palladium(II) catalyzed the intramolecular hydroxy-palladation–carbonylation of alkenes, forming stereodefined tetrahydropyrans (Eq. (253)) [1346]. Acetylenic ketones were isomerized to furans (Eq. (254)) [1347]. Palladium catalyzed the reaction of benzene-1,2-diols with propargylic carbonates, affording 2-alkylidene-1,4-benzodioxanes (Eq. (255)) [1348].



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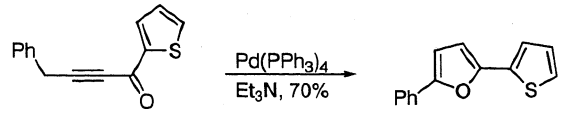

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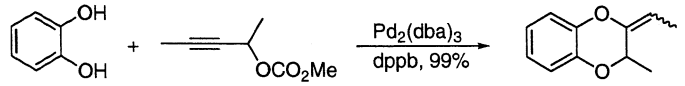
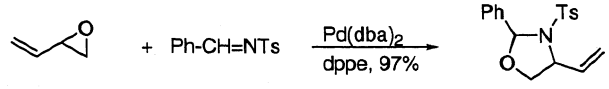


(255)

Palladium catalyzed the regioselective [3 + 2] cycloaddition of vinyloxiranes with imines (Eq. (256)) [1349]. Rhodium catalyzed the hydroformylation of 1,4-dienes in the presence of primary amines, leading to heterocyclic compounds [1350]. Thiophene diesters were prepared by annulation of sulfur containing 1,6-diynes (Eq. (257)) [116].



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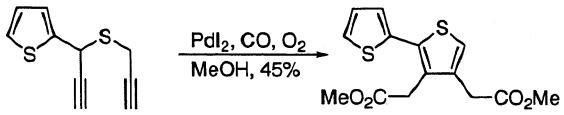
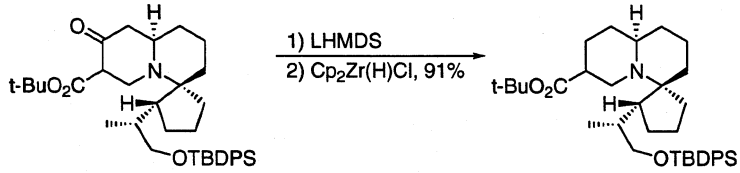


(257)

3.5. Alkenes, alkanes

A variety of functional groups were replaced by a hydrogen using palladium-catalyzed methodologies. Palladium(0) complexes, together with a hydride source such as Bu_3SnH, silanes, or ammonium formates catalyzed the reduction of vinyl triflates [72,1351–1356] to give alkenes. Similar reductions were reported for aromatic [1357,1358] and heterocyclic [76,346,1359–1361] triflates or halides. The borane dimethylamine complex was used to reduce aryl and vinyl triflates or nonaflates [1362]. Monodebromination of 1,1-dibromoalkenes was achieved using Bu_3SnH in the presence of a palladium catalyst forming a *cis*-substituted bromoalkene [210,684]. Esters of propargyl alcohols [1363,1364] and allylic alcohols [1154] were removed using ammonium formates and palladium catalysts.

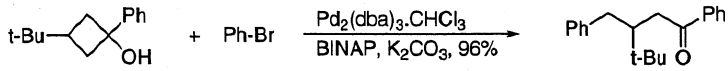
Allylic sulfones were readily displaced using lithium triethylborohydride [255,824,1365,1366] or superhydride [1367] in the presence of a palladium catalyst. Aromatic chlorides were reduced using Cp_2TiCl_2 and a Grignard reagent [1368]. Zirconocene-mediated removal of the keto group of a β-ketoester was reported (Eq. (258)) [365]. Methyl zirconation ($AlMe_3$, Cp_2ZrCl_2) of terminal alkyne, followed by lithiation and alkylation, gave regio- and stereodefined functionalized alkenes [1366]. Collman's reagent ($Na_2Fe(CO)_4$) mediated the formation of 1,1,2,2,-tetraarylethene from geminal dihalides [1369].



(258)

3.6. Ketones and aldehydes

The mechanism of the Saegusa oxidation of vinyl silylethers to α,β-unsaturated carbonyl compounds were studied [1370]. A number of synthetic applications of this reaction were published [138,1371–1377]. Wacker type oxidation, i.e. reaction of monosubstituted terminal alkenes with palladium(II) and water producing methyl ketones, was utilized in a number of syntheses [152,694,743,1378–1391]. The stereochemistry of the initial hydroxy-palladation was studied [1392]. The study suggested that water adds *syn* to palladium at low chloride concentration and *anti* at high concentration. Wacker type oxidation of 14β-allyl steroids surprisingly gave a 1:1 mixture of methyl ketone to aldehyde [1393]. Palladium catalyzed the addition of water to an internal alkyne, forming a ketone [912]. Palladium catalyzed the arylation of cyclobutanols to give ring opened products (Eq. (259)) [1394]. Palladium catalyzed the oxidative ring cleavage of cyclobutanols (Eq. (260)) [1395].



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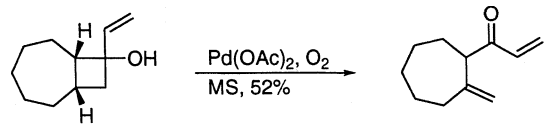
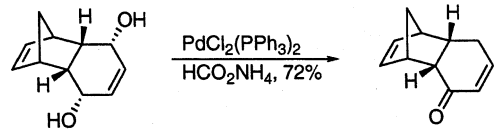


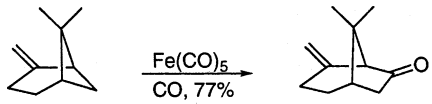
(260)

Oxidations of allylic alcohols to α,β-unsaturated ketones in the presence of a palladium catalyst were reported [1396,1397]. A 1,4-dihydroxy-2-ene was transformed into an α,β-unsaturated ketone (Eq. (261)) [1398].



(261)

Iron pentacarbonyl catalyzed the insertion of carbon monoxide into strained carbon–carbon bonds (Eq. (262)) [1119].

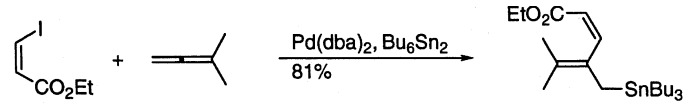


(262)

Palladium catalyzed the intermolecular arylation of enolizable ketones [1399,1400]. Intermolecular arylation of enolizable aldehydes and nitroalkanes were also reported [1401].

3.7. Organoboranes, silanes, germanes and stannanes

Allylic silanes and stannanes were prepared by palladium-catalyzed coupling of $Bu_3SnSiMe_3$ or hexaalkylditin, an allene, and an vinyl or aryl halide (Eq. (263)) [1402,1403]. Palladium catalyzed the insertion of alkynes into the carbon–silicon bonds of benzosilacylobutene forming benzosilacyclohexadienones [1404]. Rhodium catalyzed the extremely regioselective intramolecular silylformylation of bis(silylamino)alkynes (Eq. (264)) [1405]. Palladium catalyzed the dimerization–carbostannylation of alkynes [67].



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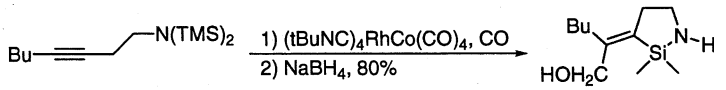
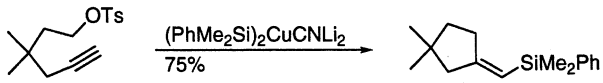


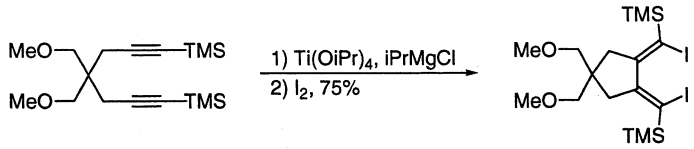
(264)

Palladium and platinum catalyzed the hydrochlorosilylation [1406,1407] and hydrosilylation [1408] of alkenes and alkynes. Palladium and rhodium catalyzed the asymmetric hydrotrichlorosilylation [1409] and hydrosilylation [171] of alkenes. Cobalt octacarbonyl catalyzed the oxidative hydrosilylation of ethyl acrylate to form β-silyl substituted acrylates [1410]. Acetylene dicobalt hexacarbonyl catalyzed the hydrosilylation of alkynes [1411]. Hydrosilylation of vinyl iodides produced vinyl silanes [1412]. Platinum catalyzed the intramolecular hydrosilylation of alkynes [1413]. Palladium catalyzed the reaction of dialkyl zinc reagents with terminal alkynes in the presence of trimethylsilyl iodide to give vinyl silanes [1414]. Silylmethylene substituted cyclopentanes were prepared using copper reagents (Eq. (265)) [1415].



(265)

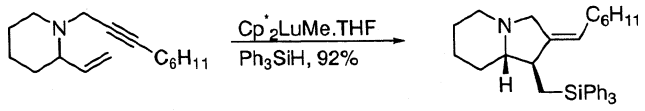
Hydrotitanation of silyl and stannyl alkynes followed by addition of an electrophile was developed [1416]. Hydrotitanation of TMS substituted 1,6-diynes, followed by addition of iodine produced a divinyl cyclopentane (Eq. (266)) [213].



(266)

In the presence of a chiral ligand, palladium catalyzed the enantioselective trichlororsilylation of terminal alkenes [1417]. Palladium catalyzed the hydrosilylation of 1,6-dienes forming *trans*-silylmethyl substituted cyclopentanes [1418–1420]. Palladium catalyzed a closely related hydrogermylation of functionalized 1,6-dienes [1421].

Related cyclizations hydrosilylation of functionalized 1,7-dienes forming silylmethyl substituted cyclohexanes were reported [1422]. Organolanthanide complexes catalyzed the cycloisomerization–silylation of nitrogen containing enynes (Eq. (267)) [1423]. Rhodium catalyzed the intramolecular silylcarbotricyclization of triynes (Eq. (268)) [1424]. Partial to complete desilylation was observed.



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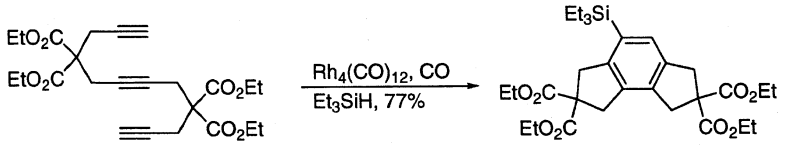
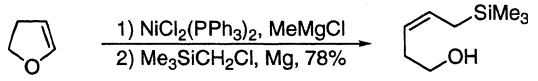


(268)

Nickel catalyzed the ring-opening methylsilylation of 2,3-dihydrofuran (Eq. (269)) [694]. Rhodium catalyzed the transformation of propargyl alcohol and derivatives to 2-silylmethyl-2-alkenals Eq. (270)) [1425].



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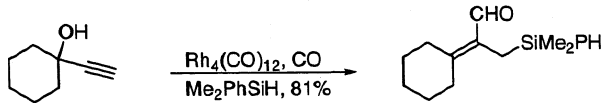
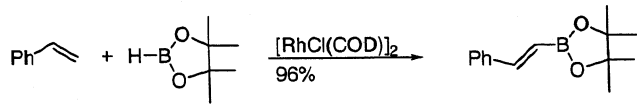


(270)

Palladium-catalyzed silaboration and stannylboration of allenes [1426,1427], nickel catalyzed 1,4-silaboration of 1,3-dienes [1428], and palladium and platinum complexes catalyzed silaboration of alkynes [1429]. Palladium catalyzed the addition of hexabutylditin to alkynes, forming 1,2-ditin-substituted alkenes [128]. Rhodium catalyzed the dehydrogenative coupling of vinylarenes with pinacolborane to give vinylboronates (Eq. (271)) [285].



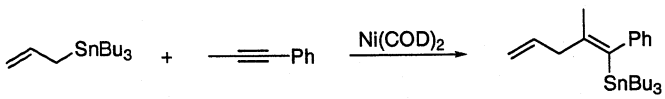
(271)

3.8. Organophosphorous and sulfur compounds

Palladium, rhodium, and platinum catalyzed the hydrothiolation of alkynes using a variety of transition metals [1430]. Rhodium preferentially afforded *anti*-Markovnikov addition and palladium Markovnikov addition. Palladium-catalyzed reactions of allylic substrates with potassium thioacetate offered a convenient entry into allylic thiols [1431]. Ruthenium catalyzed the allylation of thiols with retention of stereochemistry [1432]. Palladium catalyzed the reaction of an aryl triflate with potassium trimethylsilylsulfide [1433].

Aryl diethylphosphonates [979,1434] and diaryl alkylphosphinates [1435–1437] were prepared from aryl halides using palladium catalysts. Nickel catalyzed the reaction of vinyl halides with triethyl phosphite forming vinyl phosphonates [1438]. Nickel and palladium catalyzed the coupling of aryl triflates and halides with diphenylphosphine to form triarylphosphines [350,715,1439]. Triaryl phosphines were also prepared by nickel-catalyzed coupling of aryl halides and arylphosphine chlorides [1440]. Diaryl vinyl phosphines were obtained in a similar fashion [1441]. Aryl triflates coupled with diphenyl phosphine oxide to from triaryl phosphine oxides [171,1409,1442]. A cyclic phosphine oxide was arylated using a palladium catalyst [1443]. A variety of halides and triflates underwent palladium-catalyzed coupling to borane diaryl or alkyl aryl phosphine complexes [419,1444–1447]. Palladium catalyzed the thiophosphorylation of alkynes using phosphorothionate [1448].

Nickel-catalyzed carbostannylations of alkynes with allyl-, acyl, and alkynylstannanes affording, stereoselectively, trisubstituted vinylstannanes (Eq. (272)) [58] were reported.



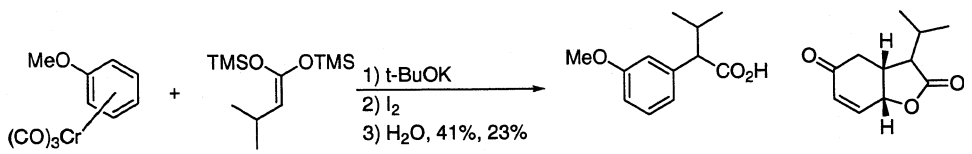
(272)

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

A number of reactions were published wherein metal complexes were allowed to react on one or more of the ligands without demetallation of the complex. Intramolecular Heck reactions of chromium tricarbonyl complexed aryl halides having a tethered alkene were reported [1449,1450]. Iodoferrocene underwent Heck reaction with vinylsilatrane [1451]. Sonogashira coupling of alkynes with chromium tricarbonyl complexed aryl halides [1452–1454] and of ferrocene iodide [1455,1456] were reported. Ferrocene [1457,1458] and cyclopentadiene–iron complex [1459] substituted alkynes also participated in the Sonagashira coupling. Appropriately substituted tungsten carbyne complexes were used in the Sonogashira reaction [1460,1461].

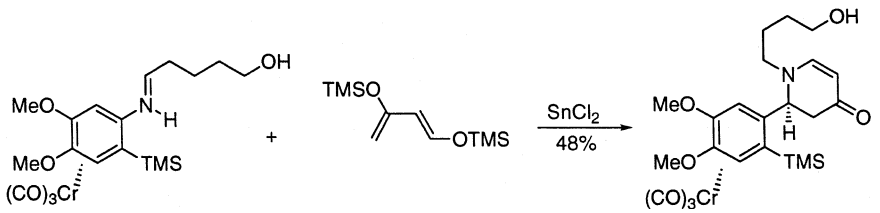
Reactions involving a transmetallation step were also used. Suzuki couplings of tricarbonyl chromium aryl chlorides [746,1462] and halo-ferrocenes [1463] were published. Stille type couplings were employed using a tin alkynyl substituted cyclopentadienylmanganese complex [1464], a tin substituted ferrocene [1465], a tin alkynyl substituted tungsten carbene [1466], and a tin bipyridyl substituted ruthenium complex [1467]. Trovalene [1468] and ferrocenylzinc chlorides [1417] complexes were used in palladium-catalyzed couplings.

Arene tricarbonyl chromium complexes continued to be extensively used as templates for organic reactions. For example, enantioselective ring deprotonation of the aromatic ring, followed by electrophilic addition [746,1469–1471], benzylic diastereoselective functionalization with electrophiles [1472–1474], asymmetric olefination of benzocyclobutenedione complex using phosphonates [1475], olefination reactions of either tethered phosphonates [1476,1477] or aldehydes [1478], diastereoselective nucleophilic additions to esters [1479] and aldehydes directly attached to the aromatic ring [1480], and nucleophilic aromatic substitution of fluorine [1481] were reported. *Endo*- or *exo*-selective Michael type reactions of a tethered enone were obtained, depending on the nucleophile [1482,1483]. A [3 + 2] cycloaddition to an attached imine was reported [1484]. Propargylic cations adjacent to a chromium tricarbonyl arene group underwent S_N1 reactions [1485]. Chromium tricarbonyl arene complexes were used as traceless linkers [1486]. Imination of attached chiral sulfoxides produced a chiral sulfonimidoyl moiety [1487]. Alkylation of arene complexes with TMS ketenes produced both single and double alkylation products (Eq. (273)) [1488].



(273)

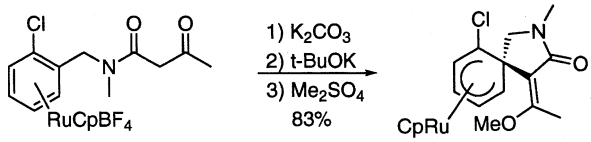
Complexation–chromatographic separation–decomplexation was used to prepare enantiomerically pure compounds [1489]. An application of chromium tricarbonyl complexes in organic synthesis can be seen in Eq. (274) [1490].



(274)

Isocyanates underwent cycloaddition to chromium tricarbonyl cycloheptatriene complexes forming bicyclic chromium diene complexes [1491]. Sequential reaction of chloroferrocene with an alcohol and an amine gave an aminosubstituted ferrocene [1492].

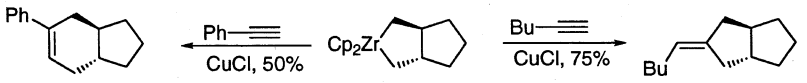
A cationic cyclopentadienyliron complexed aryl chloride underwent aromatic nucleophilic substitution with phenol [1493]. Intramolecular aromatic nucleophilic substitution of related ruthenium arene complexes were used in organic synthesis to construct cyclic peptides [1494]. Diastereoseolective alkylation of the benzylic position of cationic ruthenium arene complexes forming quartenary centers were reported [1495]. Nucleophilic intramolecular addition of stabilized enolates to ruthenium arene complexes surprisingly furnished ruthenium coordinated spirocycles (Eq. (275)) [1496].



(275)

Methoxymethyl substituted cyclobutadienyl iron tricarbonyl complexes were alkylated with silylenol ethers in the presence of a Lewis acid [1497].

Intramolecular [2 + 2 + 1] cycloaddition of aza-1,6-diynes using cyclopentadienyl cobalt dicarbonyl gave bicyclic cyclopentadienone cobalt complexes [1498]. Iron tricarbonyl cyclopentadienone complexes were formed in a related fashion [571]. Zirconacyclopentadienes reacted with carbon monoxide in the presence of butyl lithium, forming cyclopentenones [1499], with 1,1-dihalo-1-lithio species, forming cyclopentenes [1500], with sulfur dioxide, forming thiophene-1-oxides [1501], with alkynes in the presence of copper or nickel salts, forming aromatic compounds [1502], with acids, forming dienes, with NCS, forming vinyl chlorides, and with allyl chloride, forming allylated products [1503]. Addition of alkynes to zirconacycles furnished either bicyclo[4.4.0] or [4.3.0] ring systems (Eq. (276)) [1504]. Oxazirconacyclopentenes reacted with propynoates to give 2,5-dihydrofuran derivatives (Eq. (277)) [1505].



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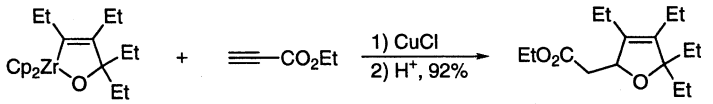
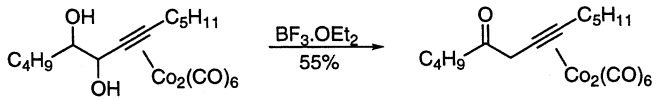


(277)

Reactions of alkyne hexacarbonyldicobalt complexes continued to be developed. Double propargylic alkylation of a cobalt complexed alkyne was reported [1506]. Cobalt alkyne complexes were dimerized [1507]. Propargylic alcohols were removed using triethylsilane [1508]. Reaction of complexed propargylic alcohols with DAST introduced a fluorine in the propargylic position [1509]. An interesting reduction–oxidation reaction can be seen in Eq. (278) [1510]. Cyclobutanes were formed from appropriately substituted enyne mesylates and trimethyl aluminum (Eq. (279)) [1511]. Cobalt complexed propargylic acetals are alkylated with enol ethers [1512]. Synthesis of polycyclic ethers using an intramolecular Nicholas reaction was reported [1513]. Trimethoxybenzene and other electron rich arenes were used as the nucleophile in Nicholas type reactions [1514]. Cobalt alkyne complexes served as protective groups for amino, hydroxy, and carboxy functionalities [1515]. Sodium methylthiolate was used to demetallate alkyne–cobalt complexes [1516].



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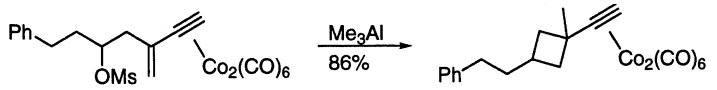
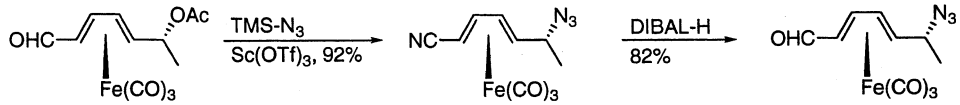
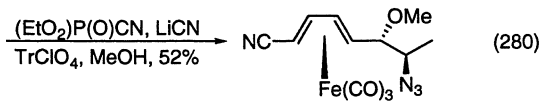


(279)

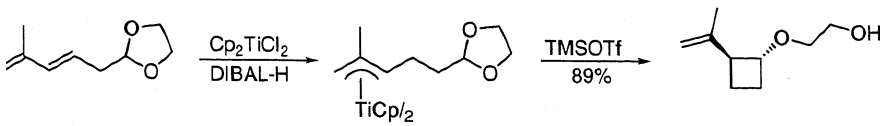
Lactones were prepared by intramolecular addition of carboxylates to iron tricarbonyl diene complexes [1517]. An asymmetric olefination of a carbonyl substituted iron diene complex was reported [1476]. Treatment of 1-methoxy-4-methyl-1,3-cyclohexadiene iron tricarbonyl with thallium tris(trifluoroacetate) produced the corresponding 4-methyl-2,4-cyclohexadienone complex [1518]. Asymmetric catalytic complexation of the iron tricarbonyl moiety to prochiral 1,3-dienes was achieved using a chiral camphor-derived azadiene catalyst [1519,1520]. Iron diene complexes were decomplexed by photolytic ligand exchange followed by air oxidation [1521]. Iron complexes of 1,3-dienes-4-ols having a tethered alkene underwent cationic cyclization related to reactions of alkyne cobalt complexes. The intermediately formed cation was captured by fluoride [1522]. A stepwise regio- and stereocontrolled functionalization of acyclic diene complexes was developed (Eq. (280)) [1523]. Lateral stereoselectivity upon dihydroxylation and Grignard reactions of functional groups adjacent to a η^3-allyl molybdenum moiety was also reported [1524]. Nucleophilic addition to molybdenum diene complexes gave a η^3-allyl molybdenum complex [1525]. A procedure for the preparation of cationic molybdenum diene complexes from the corresponding allylic precursor was reported [1526]. η^3-Pyranylmolybdenum π-complexes underwent enantiocontrolled [5 + 2] cycloaddition with alkenes to form oxabicyclo[3.2.1]octenes [1527]. Formation of a titanium η^3-allyl complex, followed by treatment with a Lewis acid, furnished stereospecifically a cyclobutane (Eq. (281)) [1528].



(280)



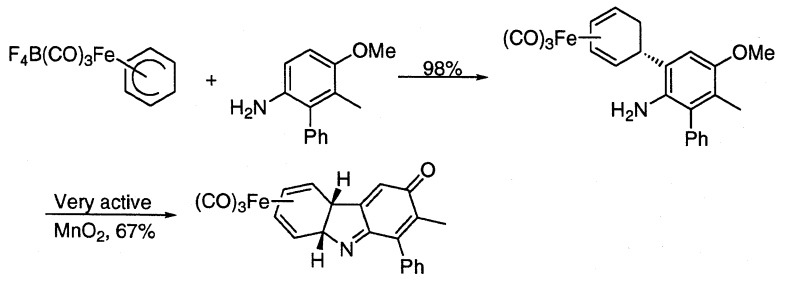
(280)



(281)

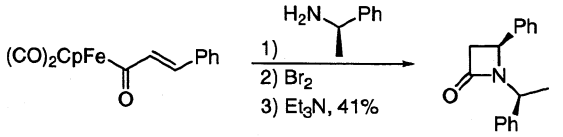
1,7-Induction of chirality using η^3-allyliron lactone and lactam complexes as chiral templates was observed in Mukayama type aldol condensations [1529]. Tin triflate mediated the stereoselective aldol condensations of tricarbonyl iron α-aminodienone complexes [1530]. Vinylogous iron dienyl complexes were determined to be intermediates in the reaction of cationic cyclohexadienyl iron complexes with anilines [1531]. Reactions of cyclic

η^5-iron complexes were used in the synthesis of carbazolle alkaloids (Eq. (282)) [1532,1533]. Anomalous [3 + 2 + 2] cycloadditions between η^3-allyl cobalt complexes and alkynes were reported. The allyl group did not remain intact in the product [1534].



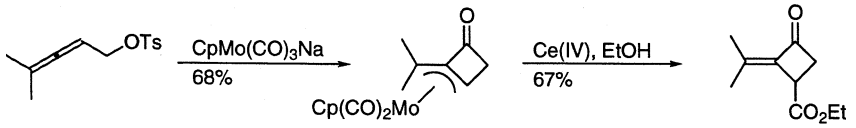
(282)

Asymmetric synthesis of β-lactams and pseudopeptides was realized via steroselective conjugate additions and cleavage of cyclopentadienyl iron dicarbonyl acylates (Eq. (283)) [793].



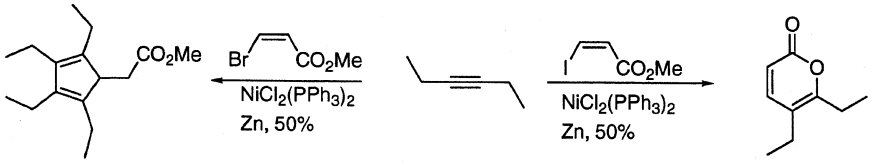
(283)

Cyclopentadienyl molybdenum dicarbonyl η^3-2-alkylidene cyclobutanonyl complexes were prepared from allenic electrophiles. Oxidative demetallation furnished 2-alkylidenecyclobutanones (Eq. (284)) [1535]. Synthesis of palladium [1536–1538], tungsten [1539–1541], iron [1542], and cobalt [1543] η^3-allyl complexes were described. Carbonylation of η^3-allylpalladium complexes was reported [1537].



(284)

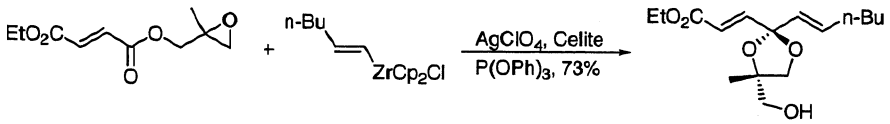
Transmetallation of a nickelacycle with organometallic reagents was studied [1544]. Halogen-dependent coupling reaction of alkynes with (*Z*)-3-halopropenoates, catalyzed by nickel, forming cyclopentadienes and pyrones were developed (Eq. (285)) [1545].



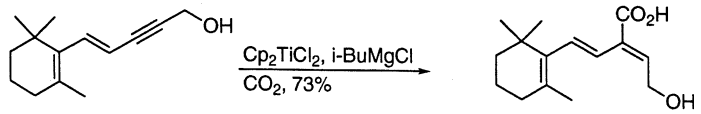
(285)

Palladium catalyzed the hydrostannantion of terminal alkynes using trialkyltin hydrides or hydride precursors to produce terminal *trans*-vinyl trialkylstannanes [135,143,153,154,244,1391,1546]. The trialkylstannyl moiety was regioselectively introduced on the carbon closest to the aromatic ring of aryl substituted internal alkynes [1547]. Tributyltin deuteride was used for deuterium labeling [1548]. Hydrostannation of bromoalkyne produced a terminal vinyltin reagent [130,167]. Palladium catalyzed the hydrostannation of 1,3-butadiene producing *cis*-2-butenylstannane [1549]. Regioselective hydrostannation of internal alkynes, followed by iodine cleavage, produced vinyl iodides [1550].

Hydrozirconation of alkynyl-stannanes, followed by protonolysis or reaction with arylsulfenyl chloride, produced a *trans*-vinyl stannane [14,291,1551] or a thiophenol substituted vinylstannane [13], respectively. Hydrozirconation of alkynes, followed by carbonylation and reaction with phenyl selenyl bromide, afforded α?,β-unsaturated selenoesters [1552]. Hydrozirconation of alkynes, followed by insertion of selenium and addition of iodonium salts to the intermediately formed vinylselenylzirconocene, afforded aryl vinyl selenides [1553]. Hydrozirconation, followed by copper(I) bromide mediated addition of a dialkylchlorophosphate, gave a vinylphosphonate [1554]. Hydrozirconation of alkynes [247], followed by addition of iodine [133,187] or sulfonyl chloride [1555] produced vinyl iodides and sulfones, respectively. Alkoxyvinylzirconocene, formed by hydrozirconation of a propargylic ether, underwent copper(I) mediated dimerization, forming a 1,4-dialkoxy-1,3-diene [1556]. An interesting silver(I)-catalyzed addition-rearrangement of vinylzirconocenes to epoxyesters was developed (Eq. (286)) [1557]. An α,β-unsaturated ester was isolated from hydrotitanation of an internal alkyne, followed by reaction with carbon dioxide (Eq. (287)) [1558].

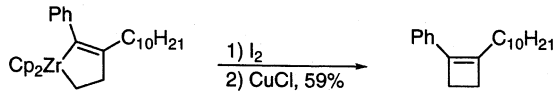


(286)



(287)

Reaction of, in situ prepared zirconacyclopentenes with iodine and copper chloride gave arylcyclobutanes (Eq. (288)) [1559]. Alkyl zirconation of terminal alkynes catalyzed by tetrakis(pentafluorophenyl)borate, followed by protonation, furnished 1,1-disubstituted alkenes [1560].



(288)

Reaction of terminal alkynes with trimethylaluminum and $ZrCp_2Cl_2$ [132] or Cp_2TiCl_2 [134] followed by iodine, afforded trisubstituted vinyl iodides. Methylzirconation using trimethyl aluminum and $ZrCp_2Cl_2$, followed by treatment with acetaldehyde, gave an allylic alcohol [1561]. The vinyl zirconium intermediate was transmetallated using methyl lithium [563,1562].

5. Reviews

The following reviews appeared in 1999:

- Transition metal-catalyzed addition reactions of H-heteroatom and inter-heteroatom bonds to carbon–carbon unsaturated linkages via oxidative additions (47 references) [1563].
- Element–element addition to alkynes catalyzed by the Group 10 metals (128 references) [1564].
- The palladium-catalyzed arylation and vinylation of alkenes-enantioselective fashion (100 references) [1565].
- Enantioselective Heck reactions using chiral P,N-ligands (40 references) [1566].
- Application of palladacycles in Heck type reactions (96 references) [1567].
- Heterocycles via cyclization of alkynes promoted by organopalladium reagents (68 references) [1568].
- Palladium-catalyzed cascade cyclization anion capture and molecular queues (75 references) [1569].
- Palladium in action: domino coupling and allylic substitution reactions for the efficient construction of complex organic molecules (140 references) [1570].
- Palladium-catalyzed annulation (55 references) [1571].
- Palladium-catalyzed amination of aryl halides and sulfonates (96 references) [1572].
- Recent advances in the cross-coupling reaction of organoboron derivatives with organic electrophiles (130 references) [1573].
- The palladium-iminophosphine catalyst for the reactions of organostannanes (40 references) [1574].
- Novel and selective substitution of ketones and other carbonyl compounds based on palladium-catalyzed coupling of α,β-unsaturated carbonyl derivatives containing α-halogen or α-metal groups (82 references) [1575].
- Catalytic asymmetric reactions via π-allyl palladium complexes coordinated with chiral monophosphine ligands (22 references) [1576].
- Enantioselective palladium-catalyzed allylic substitutions with asymmetric chiral ligands (63 references) [1577].
- Novel catalytic reactions involving π-allyl palladium and-nickel as the key intermediate: Umpolung and β-decarbopalladation of π-allylpalladium and -nickel-catalyzed homoallylation of carbonyl compounds with 1,3-dienes (56 references) [1578].
- Palladium-catalyzed enyne-yne [4 + 2] benzannulation as a new and general approach to polysubstituted benzenes (47 references) [1579].
- Mechanistic and kinetic studies of palladium catalytic systems (54 references) [1580].
- Palladium-catalyzed enantioselective rearrangement of allylic imidates to allylic amides (30 references) [1581].
- Recent developments of palladium(0)-catalyzed reactions in aqueous media (49 references) [1582].
- Metal vinylidenes in chemistry (57 references) [1583].
- Transition metals complexes in organic synthesis. Part 47. Organic synthesis via tricarbonyl(η^4-diene)iron complexes (40 references) [1584].
- Annulation reactions of chromium carbene complexes: scope, selectivity, and recent developments (40 references) [1585].

- Palladium-catalyzed pronucleophile addition to unactivated carbon–carbon multiple bonds (36 references) [1586].
- Synthesis of heterocycles using the intramolecular Heck reaction involving a formal *anti*-elimination process (33 references) [1587].
- The wonder of palladium catalysis: from carbon–carbon to metal–carbon bond formation. An opportunity of getting astonishment from reality (43 references) [1588].
- Homogenous catalysis in supercritical fluids (187 references) [1589].
- Planar chiral arene chromium(0) complexes: Potential ligands for asymmetric catalysis (35 references) [1590].
- Transition metal-catalyzed coupling reactions under C–H activation (64 references) [1591].
- Use of organomanganese reagents in organic synthesis (66 references) [1592].
- Reaction of acyl chlorides with organometallic reagents: A banquet table of metals for ketone synthesis (235 references) [1593].
- Chromium(0) promoted higher order cycloaddition reactions in organic synthesis (46 references) [1594].
- Application of stoichiometric organotransition metal complexes in organic synthesis (108 references) [1595].
- Asymmetric catalysis (382 references) [1596].
- The intramolecular Stille reaction (80 references) [1597].
- Catalytic applications of transition metals in organic synthesis (142 references) [1598].
- Olefin metathesis by molybdenum imido alkylidene catalysts (57 references) [1599].
- Recent advances in chromium(II)-and chromium(III)-mediated organic synthesis (351 references) [1600].
- Organoiron compounds for selective organic transformations (98 references) [1601].
- Cobalt carbonyl induced transformations of alkynyl- and propadienylcyclopropanes (40 references) [1602].
- Ruthenium-catalyzed coupling reactions of alkynes and alkenes (24 references) [1603].
- Palladium-catalyzed amination of aryl halides and sulfonates (96 references) [1604].
- Metathesis. The Schrock and Grubbs catalysts (nine references) [1605].
- Recent advances in chromium (II)- and chromium (III)-mediated organic synthesis (351 references) [1606].
- Zirconocene hydrochloride, 'Schwartz reagent' (12 references) [1607].
- Transition metal alkyne complexes: The Pauson–Khand reaction (66 references) [1608].
- Transition metal-catalyzed neoglycoconjugate synthesis (60 references) [1609].
- Venturing into catalysis based natural product synthesis (32 references) [1610].
- Towards efficient and wide-scope metal-catalyzed alkyl–alkyl cross-coupling reactions (21 references) [1611].
- Applications of (η^6-arene) chromium tricarbonyl complexes in organic synthesis: Nucleophilic aromatic substitution and lithiation (174 references) [1612].
- A comprehensive review of the applications of transition metal-catalyzed reactions to solid-phase synthesis (155 references) [1613].
- Synthesis of natural products via stereocontrolled palladium-catalyzed reaction (16 references) [1614].
- Chiral heterocycles as ligands in asymmetric catalysis (12 references) [1615].
- The directed *ortho*-metallation cross-coupling symbiosis in heteroaromatic chemistry synthesis (34 references) [1616].
- Recent advances in selective organic synthesis mediated by transition metal complexes (12 references) [1617].
- Palladium-catalyzed annulation (26 references) [1618].
- Selectivity and reactivity in asymmetric allylic alkylation (17 references) [1619].
- Mechanism and relevance of transition metal promoted coupling reactions (47 references) [1620].
- Palladium-promoted cyclization reactions in the synthesis of biologically active natural products (46 references) [1621].

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