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# Transition metals in organic synthesis: highlights for the year 2000

Björn C.G. Söderberg *

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

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**Contents**



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


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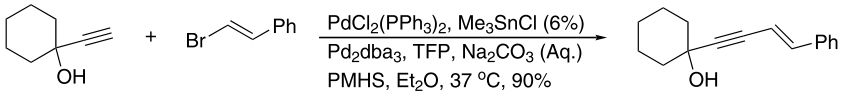
**Abstract**

A review with 1811 references about transition metal catalyzed or mediated reactions and functional group preparations is presented.


*Keywords:* Transition metals; Organic synthesis; Catalysis

## 1. General comments

This survey highlights the use of transition metals in organic synthesis for the year 2000. A fairly comprehensive literature coverage with extensive citations is presented. The field of organometallic chemistry continued to expand exponentially in 2000. Considering the large number of papers published, a complete coverage of all reported reactions employing transition metals is outside the scope of this review. A number of transition-metal catalyzed transformations are now standard reactions in the toolbox of most organic chemists. Thus, some of the more standard applications have not been included in this review. Reactions of unusual substrates, new reaction conditions, and new catalyst systems have been included. Also applications in total synthesis of more complex organic molecules have been reviewed. Only the more unusual or significant reactions were presented in equation form. Some emphasis was put on reactions used in total synthesis of biologically active compounds. Alkylations and most Michael type reactions of pre- or intermediately formed copper reagents have not been reviewed. The chemistry of metal carbon multiple bonds, e.g. reactions of Fischer and Schrock carbenes, are covered in a separate review although decompositions of diazo compounds and metathesis reactions were included herein. Most hydroformylations were not included apart from some reactions used in total synthesis.

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## 2. Carbon–carbon bond-forming reactions

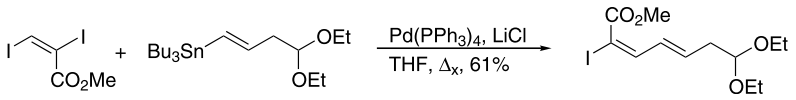
### 2.1. Alkylations

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

Palladium-catalyzed coupling reactions of organotin reagents with a large variety of electrophiles continue to be developed. This reaction is generally referred to as the Stille reaction, and this name will be used herein. The mechanism of the reaction of aryl triflates was examined, and some catalytic intermediates were observed [1]. A microwave assisted one-pot hydrostannylation–Stille coupling was described [2]. A Stille coupling catalytic in tin was developed (Eq. (1)) [3]. Palladium-catalyzed Stille type reactions using perfluoro-tagged palladium complexes in a fluorous biphasic system were reported [4]. A dendrimer supported palladium catalyst was used for the coupling reaction [5]. A stable and efficient oxime palladacycle catalyst was reported [6]. Palladium carbene complexes catalyzed the Stille coupling [7]. Coupling reactions of polymer-bound aryl halides [8,9] and polymer-bound tin reagents [10,11] were reported.

α-Halovinyl ethers [12] and vinyl phosphates were used as electrophiles in Stille couplings [13]. Carboxylic acid chlorides were used in place of halides forming unsymmetrical ketones [14–16]. Coupling of a vinyltin reagent with an acid chloride was used in the synthesis of 9-methoxystrobilurin A [17]. Ethyl chloroformate was coupled with a vinyltin reagent affording an α,β-unsaturated ester [18]. 3,4-Dibromo-2(5*H*)-furanone underwent regioselective coupling with arylstannanes to give 4-aryl-3-bromo-2(5*H*)-furanones [19]. The remaining bromide can be replaced in a second coupling reaction. Selective coupling of the terminal iodide of 1,2-diiodo-1-alkenes was achieved (Eq. (2)) [20]. A Stille coupling of a vinyl stannane with a 1,1-dibromo-1-alkene was used to prepare an ene–yne fragment of callipeltoside A (Eq. (3)) [21]. 3,5-Dichloro-2*H*-1,4-oxazin-2-one was selectively substituted in the 3-position (Eq. (4)) [22]. An aryl noflate was coupled with a vinyl tin reagent in the synthesis of nicandrenones (Eq. (5)) [23].

Stille type couplings of iodonium salts [24,25], 1-bromo-2-methoxy-3-aryloxy-2-propene [26], and a vinyltosylate [27] were described. Diarylmethanes were

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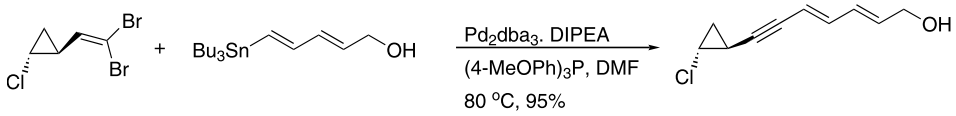


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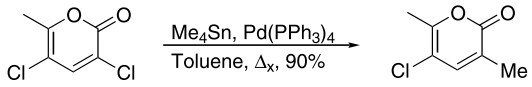


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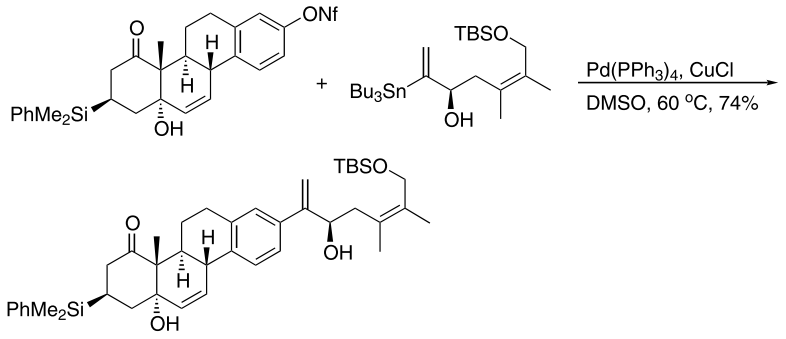


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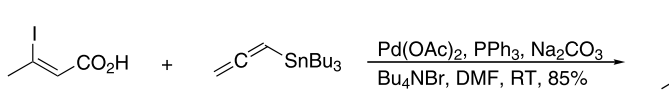
prepared by cross-coupling of aryl triflates with tetrabutylammonium difluorobenzylstannane [28]. Intramolecular coupling of two aryl halides in the presence of hexamethylditin was used in the synthesis of phenanthro[9,10-*d*]heterocycles [29,30]. Palladium catalyzed the formation of α-pyrones using β-iodo vinylic acids and allenyltin reagents (Eq. (6)) [31]. Reaction of an allyl stannane with a naphthoquinone–bromide was used in synthesis (Eq. (7)) [32]. Axially chiral diaryl compounds were prepared via coupling of an aryl stannane with an aryl bromide [33]. Cross-coupling of a vinyl triflate derived from a 1,3-dicarbonyl compound with tetramethyltin was reported [34].

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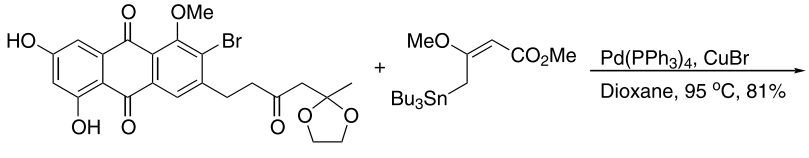


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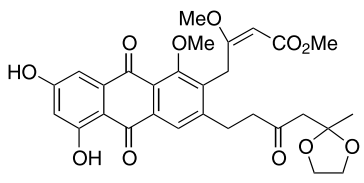
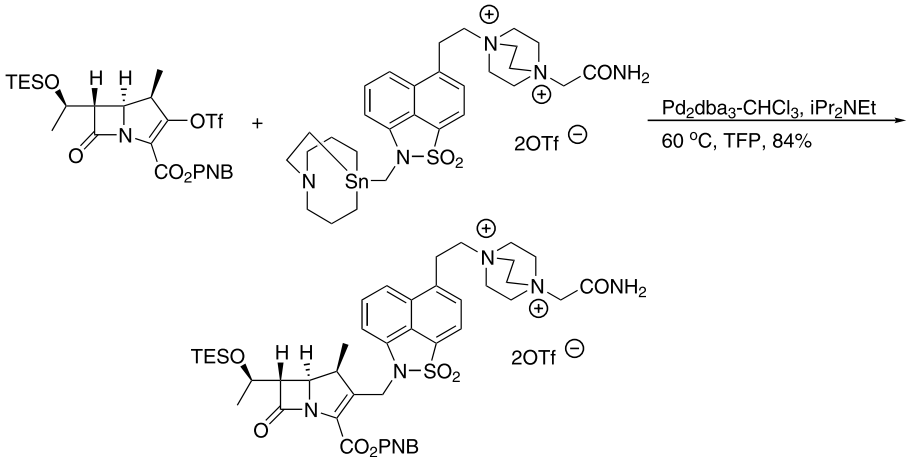


Tributylstannylcyclobutene [35], tributylstannyl (*Z*)-3-tributylstannyl-4-trimethylsilylbut-3-enoate [36], stannylcyclobutenediones [37], stannylcyclopropanes [38], and bis(tributylstannyl)ethyne [39] were used in cross-coupling reactions. Reaction of 2-trimethylsilyl-1-tributylstannylethene with an acid halide was used in the synthesis of 10,11-dihydroleukotriene B4 metabolite [40]. A stannatrane was employed for the introduction of a functionalized aminomethyl group (Eq. (8)) [41]. A double Stille coupling of 1,2-bis(tributylstannyl)ethyne with 3-iodo-6-chlorpyridine was reported [42]. 1,4-Bis(tributylstannyl)-1,3-butadiene was used in a synthesis of a suggested sponge metabolite [43], and 1,2-bis(trimethylstannyl)ethene was used to prepare dynemicin analogues [44].

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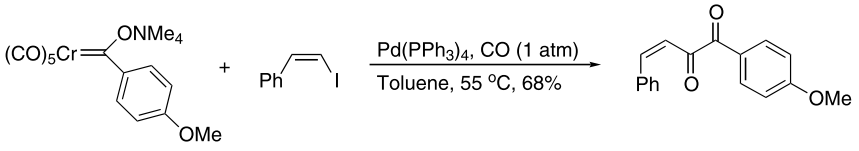
Palladium-catalyzed carbonylative cross-coupling of vinyl triflates with vinyl stannanes was shown to be promoted by addition of lithium chloride and copper iodide [45]. A palladium-catalyzed carbonylative coupling of allyl stannanes with allylic halides, in the presence of carbon dioxide (50 atm), to give allylic esters was described [46]. A carbonylative coupling of diaryl iodonium salts with organotin compounds was used to prepare $^{11}$C-labeled ketones [47]. Palladium catalyzed the coupling of acylchromates with vinyl and aryl halides in the presence of CO to give α-diketones (Eq. (9)) [48].

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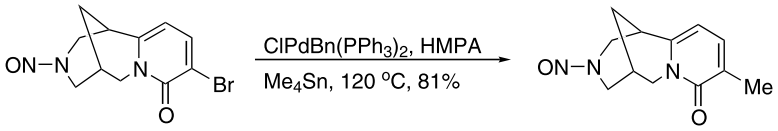
A large variety of heterocyclic halides and triflates were used as electrophiles in reactions with aryl- and vinylstannanes [49]. For example, reactions of derivatives of 4-bromomethylthiazole, in the synthesis of (−)-mycothiazole [50], 2-chloro- and 2-bromopyridines [42], 3-bromopyridine [51,52], 3-trifloxypyridine [53], 2-bromoquinoline, in the synthesis of nothapodytine B [54], 2-chloro- and 2-iodoquinoline [55,56], 5-bromomethyloxazole, in the synthesis of leucascandrolide A [57] and 9-(−)-halocytisine (Eq. (10)) [58], 3-iodoindole and 1-trifloxy-β-carboline, in the synthesis of pyridindolol K2 (Eq. (11)) [59], 2-iodo-5- or -7-azaindoles [60], 2-iodoindole [61,62], 7-aza-3-iodoindole [63], 1-bromoindolizine [64], 5-trifloxy-azepino[3,4-*b*]indole [65], 6-trifloxy-11*H*-indolo[3.2-*c*]quinoline [66], 7-bromotriazolo[4,3-b]pyridazine [67], iodo spirosulton nucleoside (Eq. (12)) [68], 8-iodocinnoline [69], 4-chloro and 8-iodoquinazoline [69], 2- and 4-bromothiazole [70,71], 8-iodoadenine [72], 4-trifloxyloxazole [73], 4,7-dibromo-

2,1,3-benzodithiazole [74], 8-bromoguanosine [75], 3-chloro or 3-iodopyridazine [76], 3-bromobenzo[*b*]selenophen and thiophen [77], and 5-bromopyrazole [78] were reported. Reaction of a pyridinyl triflate with tetramethyltin was used in the synthesis of costaclavin (Eq. (13)) [79].

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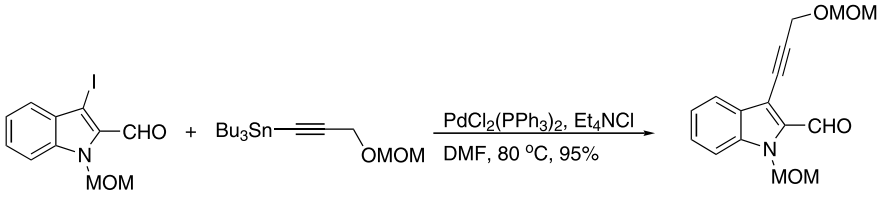


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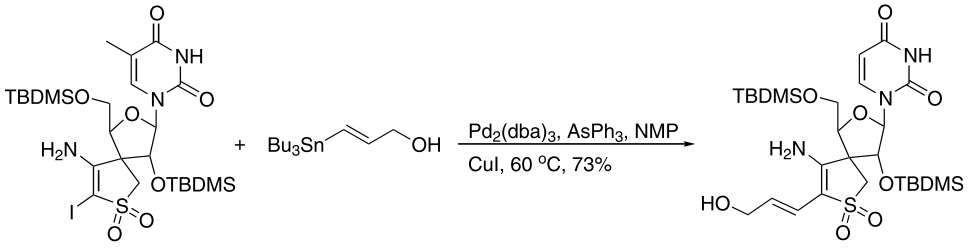


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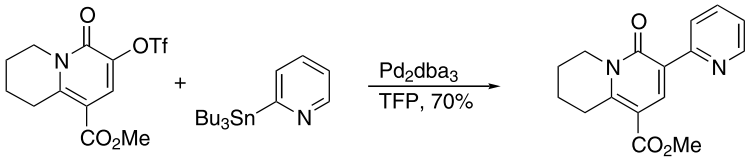
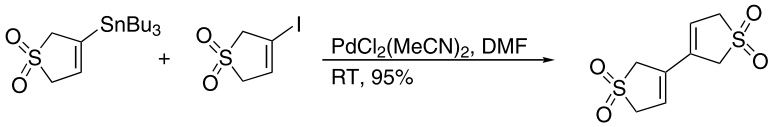



(13)

A variety of heterocyclic stannanes such as 2-pyridinyl [42,79–85], 4-pyridinyl [86], 9-(−)-cytisinyl [87], 2-benzothiazolyl [88], 3-sulfolen-3-yl (Eq. (14)) [89], 3-indolyl [90], 2-thienyl [91–97], 2-furyl [98,99], 3-benzofuryl [100], and phospholyl [101] were used in Stille type couplings with a variety of aromatic and heteroaromatic halides and triflates. Carbapenam-2-stannane was coupled with hydroxamoyl chlorides (Eq. (15)) [102]. In situ formation of a pyridinyltin reagent by reaction of a bromopyridine with hexabutylditin followed by Stille coupling with a second different bromopyridine was used in synthesis of cytisine [103].

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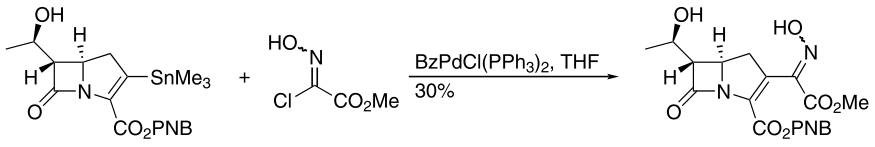


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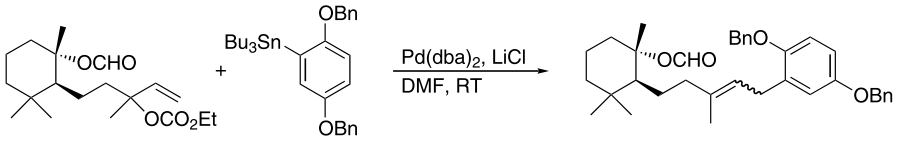
Reactions of vinyl and aryl halides and triflates [37,104–124], methyl iodide [125], allylic carbonates (Eq. (16)) [126], and 2,2-difluoro-1-iodo-enol derivatives [127] with tin reagents were proven useful in organic synthesis. Synthetic targets include, mesembrine [128], (+)-zaragozic acid C [129], aphidicolin and derivatives thereof [130,131], ocular age pigment A2-E [132], rubrolone aglycone [133], phthoxazolin A [134], sapinofuranone B [135], dihydroxerulin [136], (+)-maritimol [137], macrocyclic core of apoptoliddin [138], striatenic acid [139], polycitrin B [140], ecteinascidin and phthalascidin [141], crocacin A [142], salicylihalamide [143], (−)-asperpentyn [144], (+)-zearalenones [145], α- and β-zearalenol [146], diazonamide A [147], (−)-formylherbertenol [148], and a lasiodiplodin analogue [149]. Some examples of the Stille coupling in total synthesis of gibberlins (Eq. (17)) [150], (−)-reveromycin B (Eq. (18)) [151], (−)-amphidinolide (Eq. (19)) [152], torreyanic acid (Eq. (20)) [153,154], and (+)-hamabiwalactone B (Eq. (21)) [155] are shown below.

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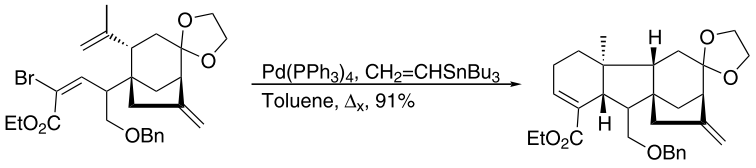


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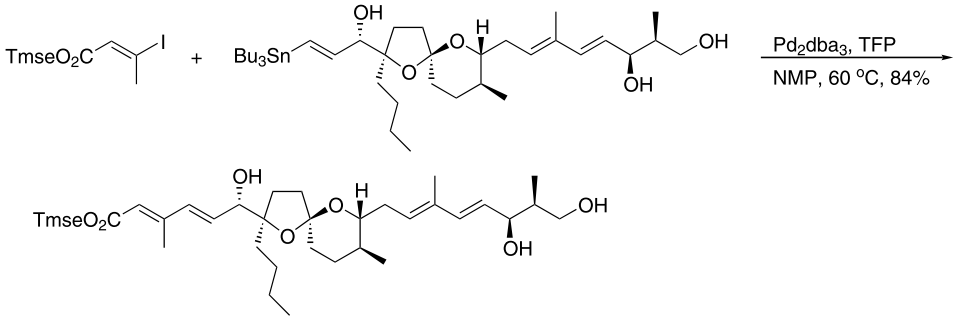


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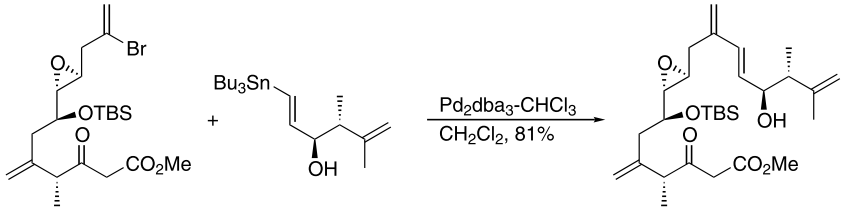


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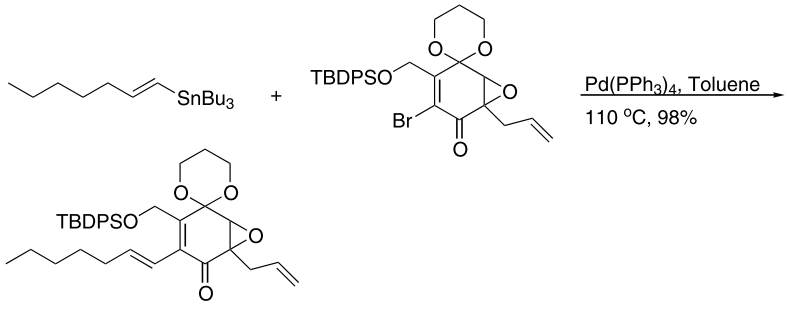


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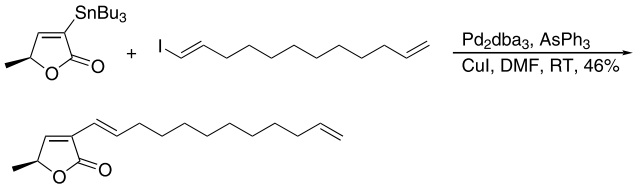


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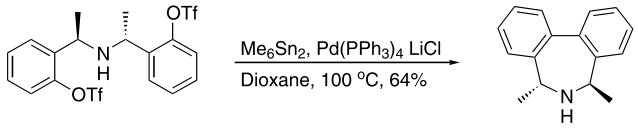
Intramolecular Stille type macrocyclization [156] was used in synthesis of pateamine fragment [157] and sanglifehrin A (Eq. (22)) [158], (+)-concanamycin F (Eq. (23)) [159], and (−)-pateamine (Eq. (24)) [160]. Intermolecular formation of a stannane using hexamethylditin followed by an intramolecular coupling with a tethered electrophile was used in diastereoselective synthesis of dibenz[*c*,*a*]azepines (Eq. (25)) [161], and hexaaryl[3]radialenes (Eq. (26)) [162].

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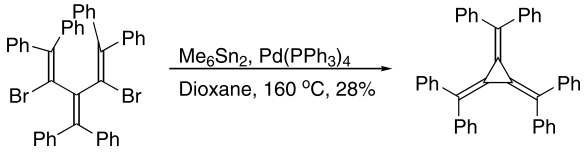


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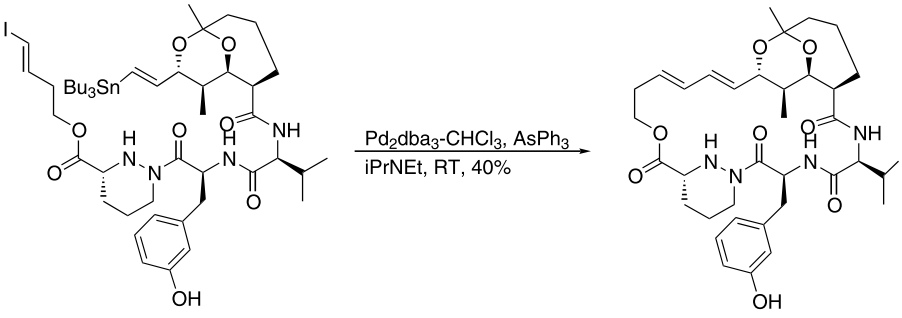
Nickel and palladium complex-catalyzed cross-couplings of Grignard reagents with halides and triflates were extensively used in organic synthesis [163–170]. Coupling reactions of aryl triflates [171], aryl halides [172], in the synthesis of (+)-diepoxin γ [173], vinyl triflates in the synthesis of (−)-taxol (Eq. (27)) [174], bromothiophens [175,176], and dibromopyridine [177]

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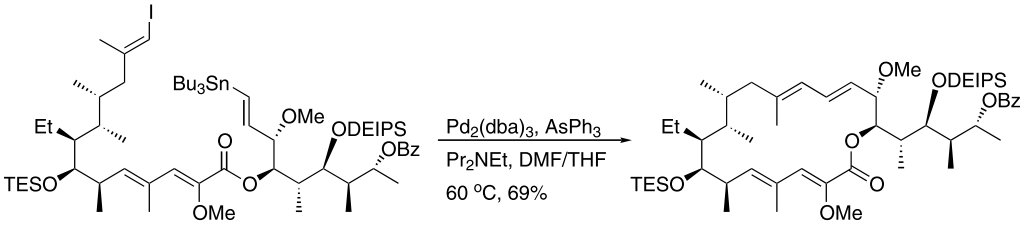


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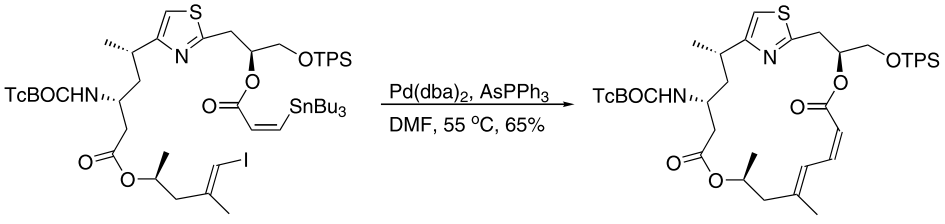
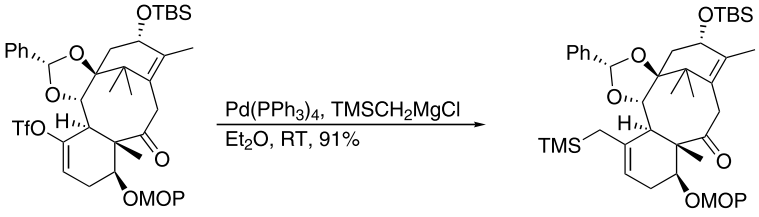



(24)

were described. A nickel-catalyzed cross-coupling of alkyl Grignards with bromothiophens without competing β-hydride elimination was reported [178]. A related palladium catalyzed coupling of aryl bromides with alkyl Grignards was described [179]. Nickel catalyzed the reaction of methylmagnesium bromide with an imidoyl chloride formed from 3-phenyl-3,4-dihydro-2*H*-isoquinoline (Eq. (28)) [180].

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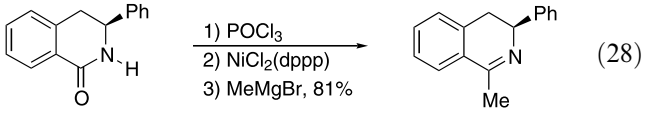


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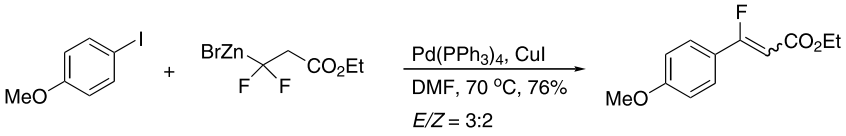
Couplings of halides and triflates with organozinc reagents, the Negishi type coupling, continued to grow as a viable alternative to tin and boron reagents [64,86,121,181–188]. Palladium-catalyzed cross-coupling of organozincs with aryl iodides in ionic liquids was reported [189]. Palladium/copper-cocatalyzed cross-coupling of the zinc reagent formed from ethyl 3-bromo-3,3-fluoropropanoate with aryl and alkenyl halides gave β-fluoro-α,β-unsaturated esters (Eq. (29)) [190]. 1,2-Difluorovinylzinc chloride was used as the nucleophile [191]. Nickel-catalyzed couplings of polymer-bound aryl iodides and alkyl zinc reagents were reported [192]. Both allenes and alkynes were formed by coupling of propargylic organozinc reagents with vinyl halides [193]. Benzylzinc halides were coupled with acryloyl chloride [194], alkenyl(phenyl)iodonium triflates [195], and methylthioheterocycles (Eq. (30)) [196]. A nickel-catalyzed cross-coupling of 5-aryloxy-1-phenyl-1*H*-tetrazoles or 3-aryloxy-1l$^6$,2-benzothiazole 1,1-dioxides with organic zinc reagents was developed (Eq. (31)) [197].

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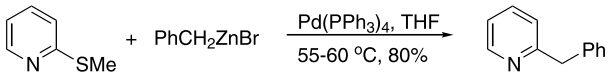


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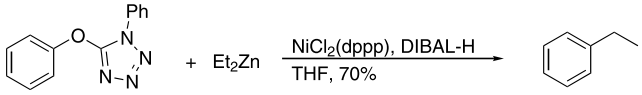
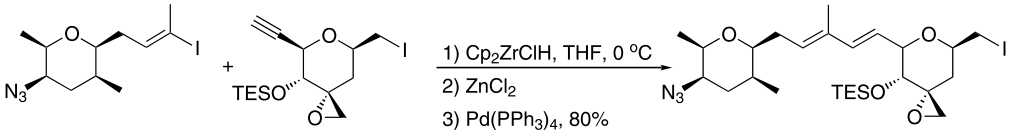



(31)

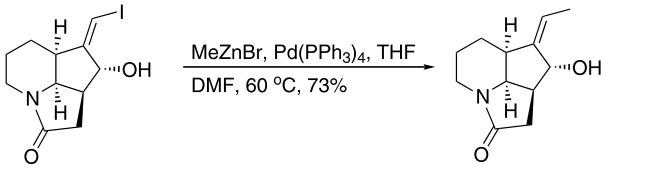
Arylzinc compounds were efficiently prepared from aryl halides using a cobalt catalyst via a sacrificial anode process [198]. Consecutive cross-couplings of an aryl dizinc reagent were described [199]. Alkylzinc reagents generated by zirconium-catalyzed ethylzincation of monosubstituted alkenes coupled with allyl, alkenyl, aryl, benzoyl halides, and vinyl triflates under palladium catalysis [200]. As expected, the bromine *trans* to an alkyl substituent of 1,1-dibromo-1-alkenes was exclusively coupled with vinylzinc reagents [201] and with alkynylzinc reagents [202]. Heterocyclic zinc reagents were used, including 2-and 3-pyridinyl [42,203], 4-pyrazolyl [204], tetrathiafulvalenyl [205], 2-thiophenyl [176,206,207], and dizincsiloles [208] zinc derivatives. 2,4-Dibromothiazole was exclusively substituted in the 2-position [71]. Hydrozirconation of terminal alkynes followed by transmetallation to zinc and coupling with vinyl iodides was reported [209]. This protocol was used in the synthesis of FR901464 (Eq. (32)) [210].



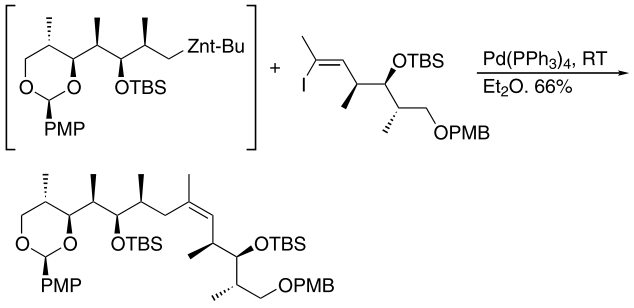

(32)

An important feature of the Negishi reaction is the cross-coupling of alkyl zinc reagents without competing β-hydride elimination. Alkyl zinc reagents were coupled with aryl halides [211,212], vinyl iodides and triflates [213,214], vinyl tosylates [215], acid chlorides [216,217], aryl triflates [218–220], and 7-bromotriazolo[4,3-*b*]pyridazine [67]. Alkylzinc reagents on a solid support were also used in coupling reactions [221].

Negishi type cross-coupling was used in the synthesis of xeruline [222], heliannuol [223], (−)-4a,5-dihydrostreptazolin (Eq. (33)) [224], methanophenazine [225], (+)-discodermolide (Eq. (34)) [226], (−)-dysiherbaine (Eq. (35)) [227], (−)-cholchicine [228], and (*S*)-methanophenazine [229].




(33)

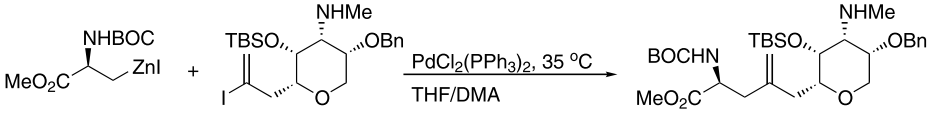



(34)

The Suzuki (or Suzuki–Miyaura) cross-coupling, i.e. palladium-catalyzed coupling of organoboronic acids and esters with halides and triflates, continue to see substantial use in organic chemistry [20,230,231]. A number of new ligands and catalyst systems were developed [232] for Suzuki reactions of aryl bromides at room temperature [233] of aryl chlorides [234–237], and of aryl and vinyl chlorides and triflates [238]. Reactions using heterocyclic carbenes [7,239,240], octaethyldiphosphaferrocene [241], and gluconamide-substituted triphenyl phosphine [242] as ligands for the palladium catalyst were reported. Both $PdCl_2(SEt_2)_2$ and $Pd(OAc)_2$ were used as catalyst precursors [243].

Coupling of aryl halides with organoboronic acids in aqueous solution catalyzed by palladium nanoparticles was reported [244]. Palladium nanoparticles were shown to be involved in phosphine-free Suzuki reactions [245]. Palladium and nickel together with an electron rich phosphine catalyzed Suzuki couplings of sterically hindered aryl bromides [246]. Coupling of aryl halides with phenylboronic acid under solvent free conditions was described [247]. A palladium-catalyzed Suzuki coupling reaction under thermomorphic conditions was described [248]. Palladium catalyzed reactions in ionic liquids at ambient temperature [249]. Thallium ethoxide promoted Suzuki couplings of a range of vinyl- and arylboronic acids [250]. Nickel on charcoal and a nickel triphenylphosphine complex catalyzed the coupling of aryl chlorides [251,252].

Polymer supported Suzuki reactions have attracted a fair amount of attention. A polymer supported palladium catalyst was used to couple chloropyridines with aryl boronic acids [253]. Polymer-bound aryl and heteroaryl halides were used together with aryl and heteroaryl boronic acids [9,254–257]. A Suzuki coupling was used to prepare biarylmethanes employing polymer supported benzylsulfonium salts (Eq. (36)) [258].

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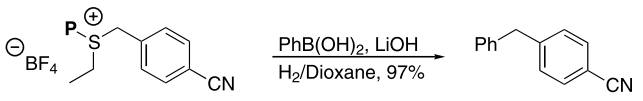
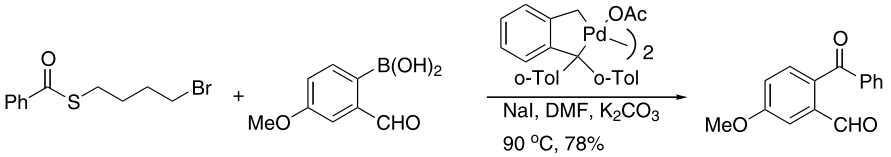



(36)

Thiols were protected with a 2-methoxyisobutyryl group for subsequent Suzuki couplings [259]. An interesting thiol-ester boronic acid cross-coupling forming ketones was described (Eq. (37)) [260,261]. Coupling of an aryl iodide with bis(pinacolyl)diboron forming an aryl boronic ester followed by an intramolecular coupling with a pendant aryl iodide was reported (Eq. (38)) [262]. Similar to the Stille reaction of 1,1-dibromo-2-nyl bromides were used in Suzuki reactions [267]. A 3,4-dibromofurane-2-one was selectively coupled in the 4-position using a vinylboronic ester [268]. Arylsubstituted pyrroles were prepared by a tandem Suzuki–dehydrogenation sequence [269]. Asymmetric Suzuki couplings were used to prepare chiral biaryls (Eq. (40)) [33,270,271]. Lactam derived *N*-BOC enol triflates were coupled with aryl and heteroaryl boron derivatives [272]. β-Chlorovinamidium salts cross-coupled with phenylboronic acid using a palladium-tri-*t*-butylphosphine catalyst (Eq. (41)) [273]. Sterically hindered aryl boronic esters were coupled with methyl 3-iodo-4-methoxybenzoate [274].

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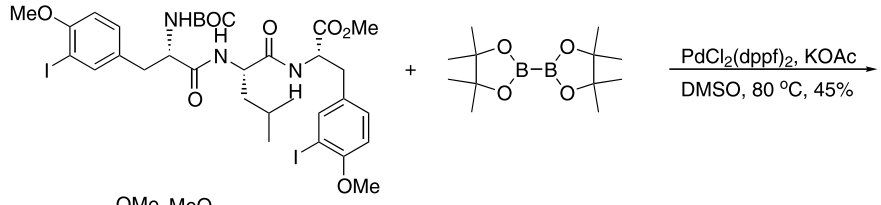
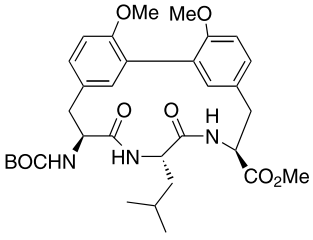
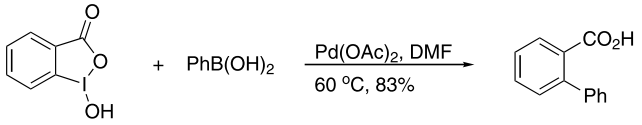



(38)

substituted alkenes produced z-alkenes upon coupling with boronic acids [263]. Minor amounts of product derived from disubstitution and substitution–elimination to an alkyne was observed [264].

Aryldiazonum salts [265], 1-hydroxy-1,2-benziodoxol-3(1*H*)-one (Eq. (39)) [266], and 2,2-difluoro-1-iodo-enol derivatives [127] were coupled with arylboronic acids and/or esters. β-Aminosubstituted β-trifluoromethylvi-

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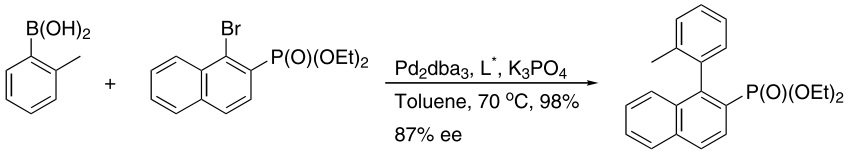


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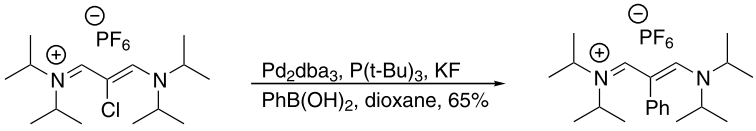
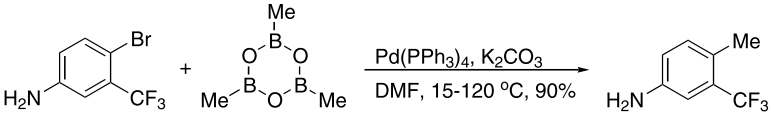



(41)

Suzuki coupling of cyclopropylboronic acids with acid halides [275], aryl triflates [276], vinyl bromides [277], vinyl triflates [278], and benzyl bromides [279] were described. In addition, 9-*B*-cyclopropyl-9-BBN was used [280]. Addition of silver oxide improved the yield of product upon reaction of cyclopropyl boronic acids esters with allyl bromides [281] and β-tetronic acid triflates [282].

Alkynylboronic ester "ate-complexes" generated in situ from acetylides and triisopropoxyborane were used in Suzuki couplings with aryl and vinyl bromides [283]. A methyl group was introduced using trimethylboroxine (Eq. (42)) [284]. Pyridineboronic ester "ate-complexes" were used in the synthesis of cytosine [103]. Nickel-catalyzed the cross-coupling of lithium vinylborates with vinylbromides was used in the synthesis of 10,11-dihydroleukotriene B4 metabolite (Eq. (43)) [40]. A nickel-catalyzed coupling of 8-acetoxyoctalenone with lithium arylborates was reported [285]. Palladium catalyzed the coupling of lithium alkynylborates with aryl and vinyl halides. This protocol was used to prepare terbinafine [286]. An interesting carbonylative coupling of a vinyl triflate or bromide with indolylborate was used in the synthesis of yuehchukene (Eq. (44)) [287,288] and ellipticine [289].

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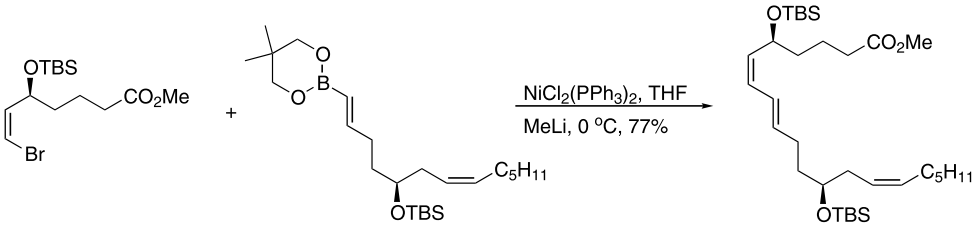
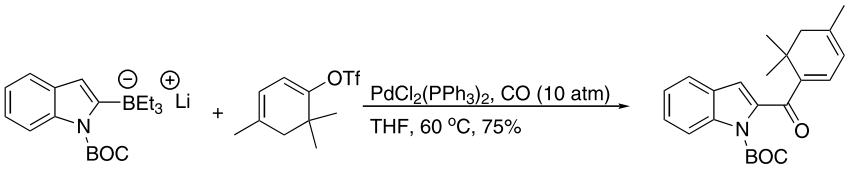


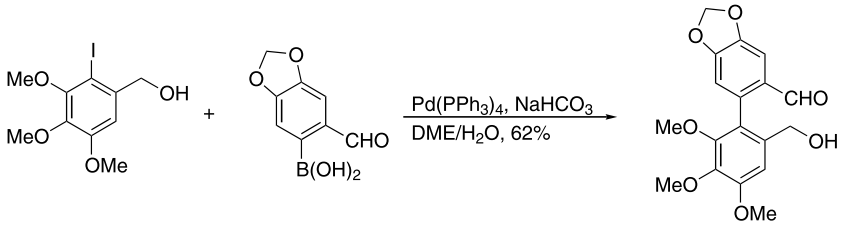

(43)




(44)

Aryl halides [120,123,124,290–300] and triflates [301,302], vinyl triflates and halides [303–305] were extensively used in Suzuki type coupling reaction of arylboronic acids and esters. Aryl–aryl cross-coupling reactions were used in synthesis of arnoamines A and B [306], antillatoxin [307], and (+)-diepoxin γ [173]. A plethora of total syntheses wherein vinylboronic acids or esters were coupled with a vinyl halides or triflate [104,164,263,308,309] were reported, for example orevactaene [310], and a fragment of diazonamide [311]. Some synthetic application toward (−)-steganone (Eq. (45)) [312], γ-lycorane (Eq. (46)) [313], and diazonamide A (Eq. (47)) [314,315] can be seen below. A nickel-

catalyzed Suzuki-type reaction was used to prepare koromycin (Eq. (48)) [316].

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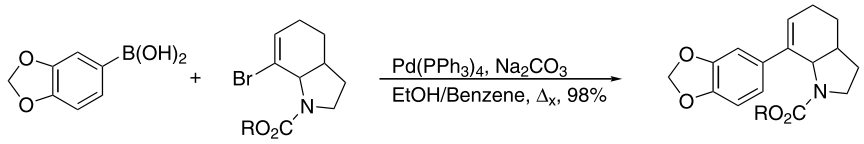


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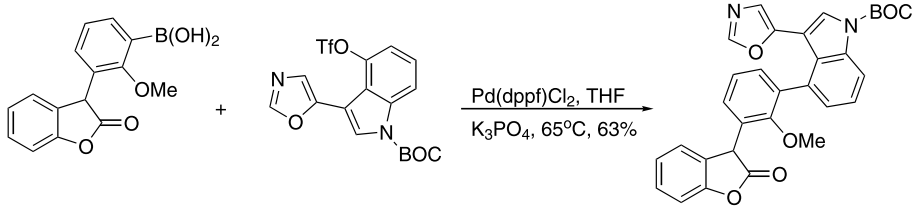
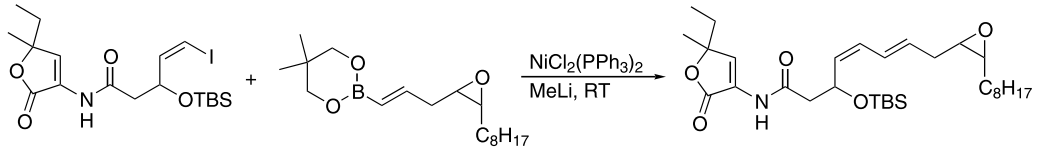



(47)



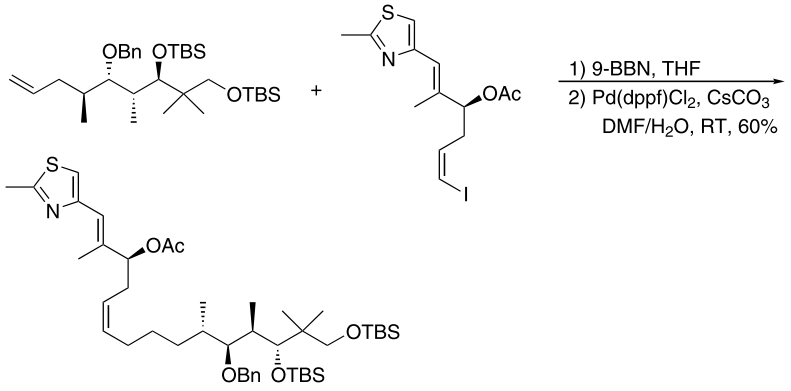

(48)

A large variety of functionalized heterocyclic halides [317] and triflates were employed in the Suzuki reaction [49,318]. For example, 2-iodo and bromoindole [62,319], in the synthesis of apicidin analogs [320], 3-iodo or 3-bromoindole [321,322], 4-bromoindole [323], 2,3-diiodoindole [324], 2-iodo-5- or -7-azaindoles [60], 3-iodoindazole [325], 1-bromoindolizine [64], 2-iodopyrrole [326], 4-chloroquinazoline [327], 2-chloroquinoxaline [328], halopyrroles [329,330], in the synthesis of B-norrhazinal [331], 7-iodoisatin [332], 6-trifloxy-11*H*-indolo[3.2-*c*]quinoline [66], polymer-supported 2-iodothiophen with 2-thiophen boronic esters [333,334], 2-iodothiophen [335,336], 3,5-dibromopyridine [172], 3-iodoimidazolo[1,2-*a*]pyridines [337], 5-bromo-9,10-phenanthroline [338], 5-trifloxy-azepino[3,4-*b*]indole [65], 4,7-dibromo-2,1,3-benzodithiazole [74], a polymer-bound 2-bromofuran [339], 3- and 4-halopyridines [176,340], 3-chloro or -iodopyridazine [76], 4-chloro- and 8-iodocinnoline [69], 2,6-diiodopyridine [341], 4-chloro- and 8-iodoquinazoline [69], 3-bromobenzo[*b*]selenophen and thiophen [77], 5-bromopyrazole [78], 5-bromo-2-pyrone [342] were all used in Suzuki type couplings. A sequential Suzuki coupling of 3- and 4-iodopyrrole was used in the synthesis of lukianol A [343]. Reaction of a 2,4-dibromothiophen with phenylboronic acid resulted in the exclusive coupling in the 2-position [344].

A variety of heteroaryl boronic acids [345] were used, such as thiophen boronic acid [99,346–350], 1-furyl [351], benzo-[*b*]-thiophen-2-boronic acid [328], benzothiophen-2-boronic acid [352], indole-3-boronic acid [90,353], and silole-2,5-diboronic acid [354].

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 [355]. Aryl halides and triflates [218,356,357], and vinyl triflates [358] were all coupled with alkylboronic acids. A Suzuki coupling of a cyclopropyl iodide was reported [359]. Palladium catalyzed the cross-coupling of trialkylboranes with acid

chlorides [360]. Cross-coupling of vinyl and heteroaryl iodide or bromides with alkyl boronic acid or esters was used in synthesis of, 12,13-desoxyepothilone F [361] and 15(*S*)-aza-12,13-desoxyepothilone B [362], haliclamines A and B [363], epothilone A (Eq. (49)) [364–366], ring system of phomactin D [367], carbacyclin [368], sansalvamide A [369], eleuthrobin (Eq. (50)) [370], and plaunotol [371]. An intramolecular Suzuki reaction was used to form a macrocycle [372]. A ciguatoxin fragment (Eq. (51)) [373,374], and a fragment of gambierol were prepared using vinyl phosphates [375].

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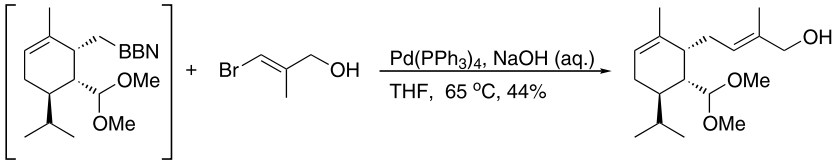


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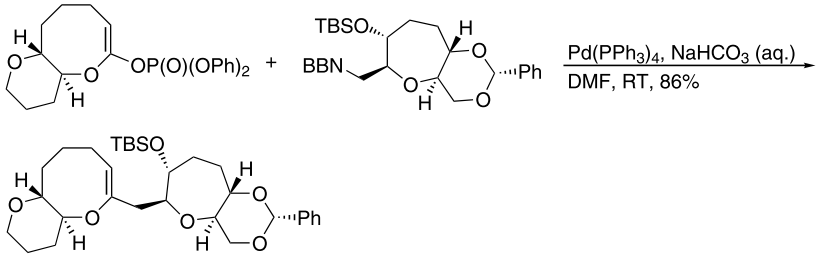
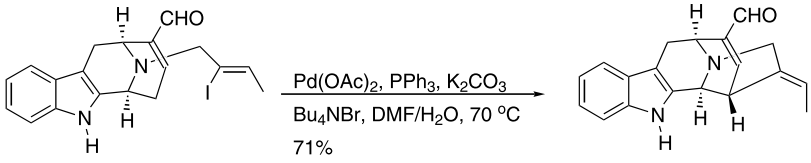



(51)

Carbon nucleophiles were also employed in alkylation reactions of halides and triflates. Palladium and nickel complexes catalyzed the alkylation of aryl halides with malonitrile [376,377]. Highly active and efficient palladium catalysts were developed for intermolecular arylations of enolizable ketones [378] and dimethylmalonate [379]. Intramolecular reactions were used to prepare substituted indanes [380]. An intramolecular enolate alkylation of a bromopyridine was used as an approach to the huperzine A skeleton [381]. An interesting coupling of an allylic position was observed in place of a Heck reaction (Eq. (52)) [382,383]. A modified substrate was used in the synthesis of (+)-vellosimine (Eq. (53)). Palladium-catalyzed intramolecular coupling of vinyl halides and ketone enolates to give bridged azabicyclic compounds was described (Eq. (54)) [384].

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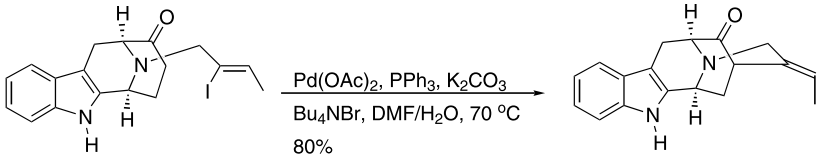


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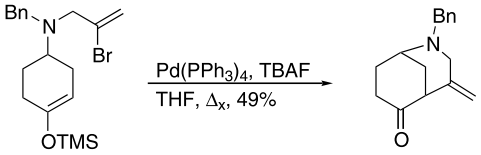
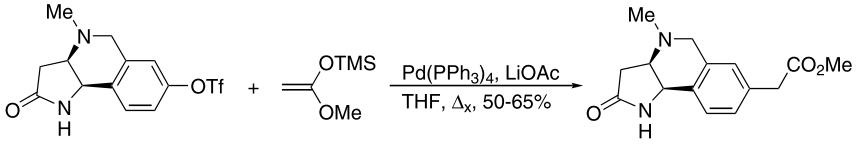



(54)

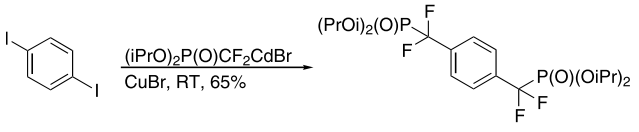
Reaction of a bromoporphyrin dimer with malonitrile gave a cumulenic porphyrin dimer [385]. Palladium catalyzed the reaction between an aryl triflate and methyl trimethylsilylketene acetal introducing an acetic acid ester functionality (Eq. (55)) [386]. A related protocol was used to introduce an acetic acid ester group in the synthesis of CP225,917 and CP263,114 [268]. The yield of bistrifluoromethylated products using a copper mediated reaction of polyfluorinated[2.2]paracyclophane diiodides with methyl 2-(fluorosulfonyl) difluoroacetate was vastly improved in the presence of palladium dichloride [387].




(55)

A number of silicon reagents were developed as alternatives to organotin compounds. Palladium catalyzed coupling reactions of alkenylsilanols and alkenylsilylhydrides [388,389], alkenylsilacyclobutanes and alkenylsilanols [390,391], vinylsilanes with arenediazonium salts [392], silanols [393], alkynylsilanes in the presence of sliver salts [394], silanediols and silanetriols with aryl iodides in the presence of silver(I) oxide [395], and arylflourosilanes with a polymer-bound aryliodide [396]. The reactions were usually performed in the presence of a fluoride source. Cross-couplings of aryl chlorides with phenyl- or vinyltrimethoxysilanes using a palladium–imidazolium chloride system were reported [397].

Some more unusual cross-coupling reactions employing organometallic reagents were developed. Palladium catalyzed coupling reactions of aryl iodides with benzylic manganese halide reagents [398], of dimethylaluminum and gallium reagents with electron deficient aryl chlorides [399], of tributylstannyl-2-trialkyl or 2-triphenylgermylethene [400], and of alkynylstibines with acid chlorides [401]. Nickel catalyzed the coupling of trimethylaluminum and aryl halides [402,403]. Copper catalyzed the cross-coupling of diisopropyl bromocadmium difluoromethylphosphonate (Eq. (56)) [404] or diethyl bromozinc difluoromethylphosphonate [45] with 1,4-diiodobenzene. Palladium catalyzed the coupling of dialkyl zinc and alkynyl zinc reagents with unsaturated organotellurium compounds [405,406]. Electrochemical reaction of a mixture of aryl and pyridyl halides using a sacrificial iron anode, in the presence of a nickel catalyst, produced 2-arylpyridines (Eq. (57)) [407].

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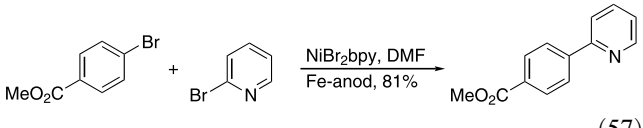
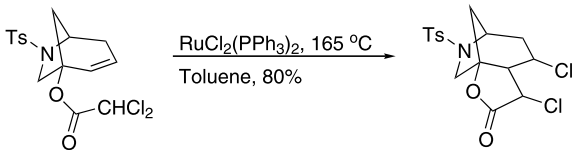



(57)

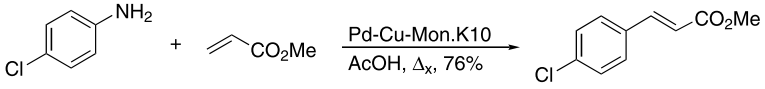
A zirconated vinyl sulfoxide was coupled with an alkynylphenyl iodonium salt [408]. Nickel catalyzed the coupling of vinylzirconocenes with α-bromo-α,α-difluoroacetic acid esters [409]. Vinyl zirconocenes prepared in situ by hydrozirconation of alkynes were used in cross-coupling reactions with vinyl halides [305], for example in the synthesis of xeruline [222]. A zirconocene dichloride-catalyzed methyl alumination of terminal alkynes followed by a palladium-catalyzed coupling with 4-triflloxyoxazole was described [73]. Zirconocene dichloride-catalyzed the methyl alumination of a terminal alkyne followed by exchange with methyl lithium and alkylation was used in the synthesis of a scyphostanin fragment [410]. Ruthenium catalyzed the intermolecular insertion reaction of α,α-dichloro or α-chloro-α-thiophenol esters with alkenes forming a five-membered ring (Eq. (58)) [411].




(58)

for example, a disulfonated phosphine for use in biphasic reactions [424], including a $Pd(dba)_2$ di-1-adamantyl-*n*-butylphosphine system for deactivated aryl chlorides [425], an oxime palladacycle [6], palladium carbene complexes [7,171,240,426], a sulfur-containing palladacycle [427], polymer-supported palladium-carbene complexes [428], pyrazole and benzothiazole palladacycles [429]. The formation of palladium nanoparticles was suggested as the active catalyst using cyclopalladated imine catalysts [430], and were shown to be involved in phosphine-free Heck reactions [245]. Palladium complexed to dendrimers on silica were used [431] and palladium adsorbed on solid support was used as the catalysts [432]. A number of new ligands or substrates for intra- and intermolecular asymmetric Heck reactions were developed [101,433–443]. An interesting palladium–copper–montmorillonite catalyzed coupling of methyl acrylate and aryl amines was reported (Eq. (59)) [444].

The use of non-standard organic solvents for the Heck reaction was described. A palladium-catalyzed Heck reaction under thermomorphic conditions was reported [445]. Catalyst systems and reactions in ionic liquids [446–448]-supercritical carbon dioxide [449], and aqueous solutions [450] were developed. Reactions of

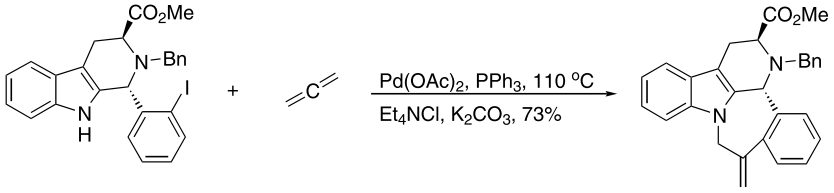



(59)

### 2.1.2. Alkylations of alkenes and allenes

The Heck reaction continued to be one of the most versatile methods for the alkylation of alkenes [99,293,412–420]. A model study of the early steps of the Heck reaction was reported [421]. A number of papers dealing with different aspects of the catalyst system and the reaction conditions appeared. Several highly efficient catalyst systems were reported [422,423], polymer-bound aryl halides were reported [9,258,451].

Heck type reaction of aryl and vinyl halides and triflates with allenes produced 1,3-dienes [452]. The formed $\eta^3$-allyl intermediate can be intercepted by a tethered nucleophile (Eq. (60)) [453]. Reaction of a vinyl boronic ester with a vinyl halide gave a mixture of Heck and Suzuki type products [134]. By proper selection of catalyst system, arylation of allyltrimethylsilane can be

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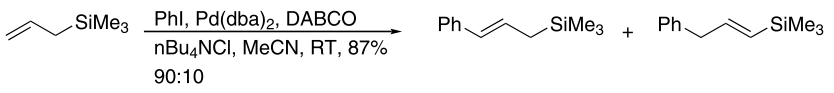
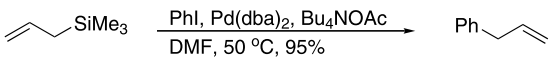


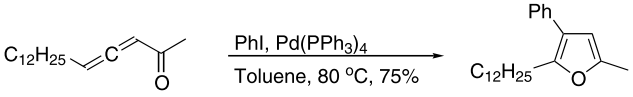

(61)

directed toward the formation of either (*E*)-1-aryl-3-trimethylsilyl-1-propene or 3-aryl-1-propene (Eq. (61), (62)) [454].




(62)

Polysubstituted furans were obtained from reaction of 1,2-diene-3-ones with aryl and vinyl halides (Eq. (63)) [455]. Methylene substituted cyclic carbonates (Eq. (64)) [456], and 3-sulfolene [457] were used in Heck reactions. Palladium catalyzed the arylation of α-methylene-γ-butyrolactones affording mainly exocylic elimination products using aryl iodides containing strongly electron-withdrawing groups. Electron-rich aryl iodides gave slightly more *endo* product (Eq. (65)) [458]. Heck reactions of vinyl silanes [459], and 2-vinylpyridine [460] were described. Thermal and microwave heated Heck reaction of vinyl triflates with butyl vinyl ether or 2-hydroxyethyl vinyl ether affording 2-alkoxy dienes or cyclic acetals was described (Eq. (66)) [461]. Reaction of aryl triflates with dialkylallylamines gave regioselectively arylation of the internal carbon (Eq. (67)) [462]. Double Heck reactions of 1,2-dibromo-1-cycloalkenes were reported [463].

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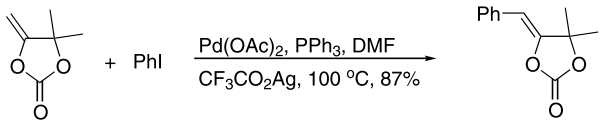


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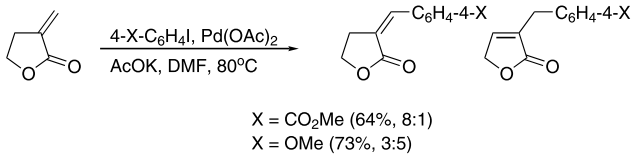


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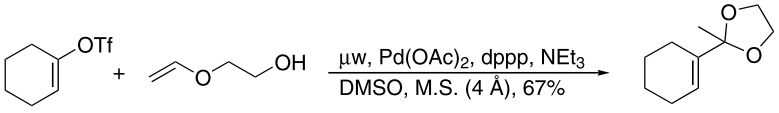


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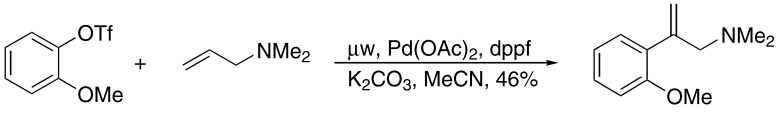
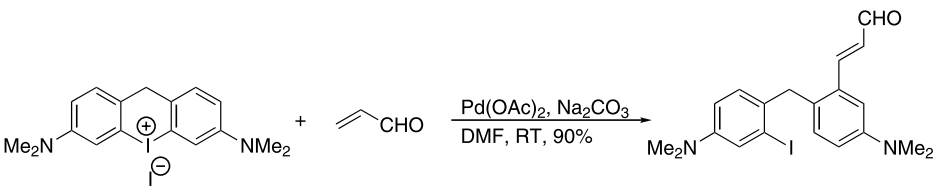



(67)

Aryl iodides reacted preferentially in the presence of a tethered vinyl bromide [26]. Diaryliodonium salts [464], 2-fluoro-1-iodo-1-alkenyliodonium salts [465], diazonium salts [466], and aryldiazonium salts and vinylphosphonates [467] were used in place of aryl halides. Heck reaction of iodine heterocycles were reported (Eq. (68)) [468]. Organosilanols and organotin compounds were used in place of aryl or alkenyl halides [393,469].

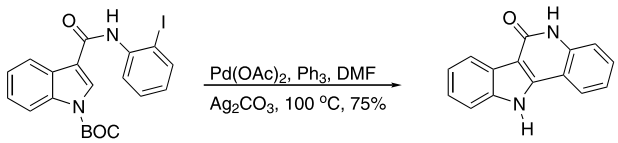
The intermolecular Heck reaction was used extensively in organic total synthesis [470–472]. For example, annulated nicotine analogues of anabasin [473], 9-methoxystrobilurin A [17], (+)-anatoxin a [474], (+)-ratjadone [475], and cryptophycin-24 (Eq. (69)) [476] were prepared. Enantioselective reaction of an optically active 4,5-dihydrofuran was used in the synthesis of analogues of deoxy-β-L-cytidine (Eq. (70)) [477]. Asymmetric reactions using 2,2-diethyl-2,3-dihydrofuran were




(68)

reported [478]. Heck reaction of a diazonium salts with enantiomerically pure 2,3-dihydropyrroles was used in the synthesis of (−)-codonopsinine (Eq. (71)) [479].

Aromatic systems were shown to undergo, at least formally, Heck type arylations [480–487]. This type of reaction was used to prepare Korupensamines A and B (Eq. (72)) [488], kinafluorenone and WS-5995 [489], and mastigophorones [490]. The pyrrole ring of indole also

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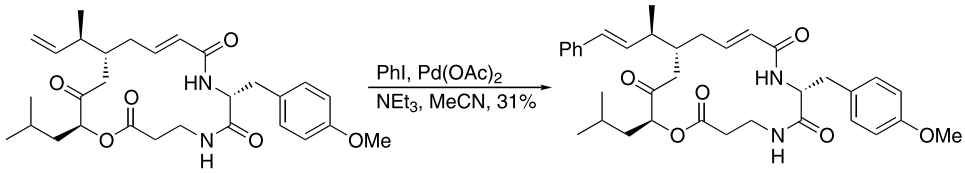
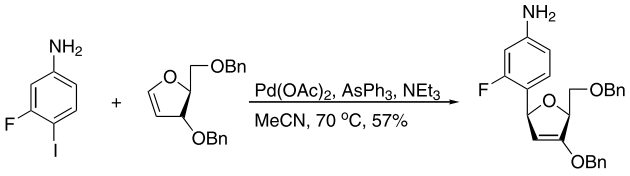



(69)

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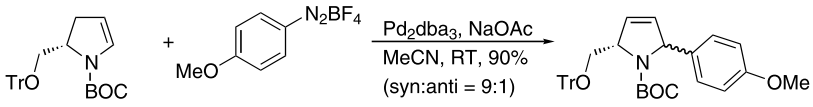
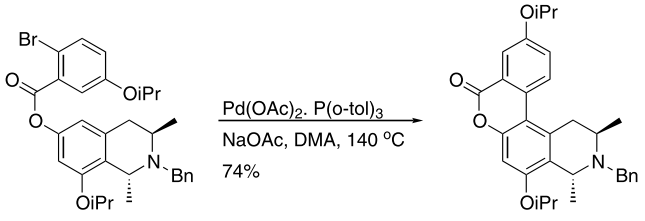



(71)

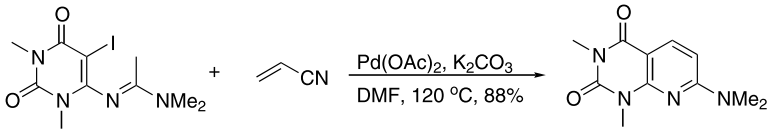
participated in an intramolecular Heck reaction (Eq. (73)) [66] as did the five-membered ring of azulene [491].




(72)

Halides and triflates of a variety of heterocyclic ring systems were used, such as 3-iodopyrrole [330,343], 5-iodopyrimidine [492], 2-iodoindole [62], 3-iodo-2-azaindole [493], 2-iodo-5- or -7-azaindoles [60]. Heck reaction of iodouracils having acetamidine moiety gave pyrido[2,3-*d*]pyrimidines (Eq. (74)) [494].

The intramolecular variation [495–501] of the Heck reaction continued to be used as the key step toward an array of natural products, for example a fragment of balanol [502], aspidospermidine [503], cytisine (Eq. (75)) [504], cephalotaxine [505], ent-gelsedine [506], D-homosteroids [507], vitamin D analogs [508], strychnine (Eq. (76)) [509,510], toward galanthamine ring system (Eq. (77)) [511], toward diazonamide A (Eq. (78)) [512], spirotryprostatin B [513], toward neocarzinostatin [514], corynantheidol and corynantheidine (Eq. (79)) [515], and herbertenediol [490].

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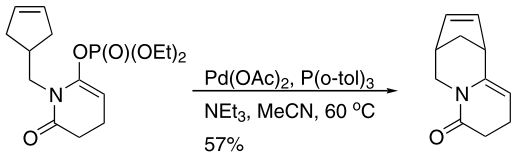


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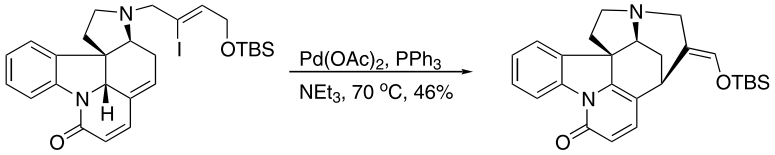


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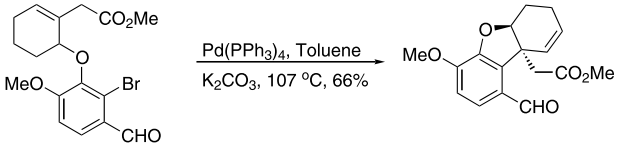


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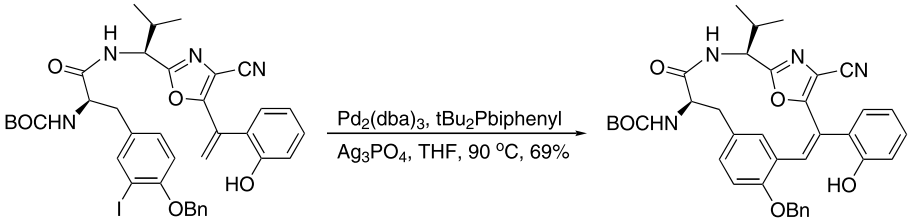


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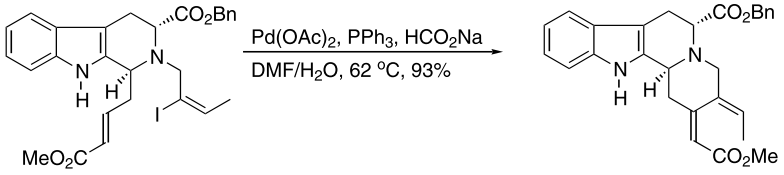
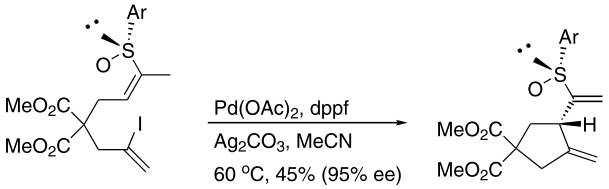



(79)

A chiral 2-(*N*,*N*-dimethylamino)phenylsulfinyl group was used as an efficient chiral auxiliary for intramolecular Heck reactions (Eq. (80)) [516]. Pyrroles were prepared by intramolecular Heck reaction of enamines (Eq. (81)) [517]. Intramolecular Heck reaction of a β-amino-α,β-unsaturated ketone was reported [518]. Double intramolecular alkene insertion was achieved using 1,1-dibromo-1-alkenes (Eq. (82)) [519]. Intramolecular Heck reaction followed by a Suzuki coupling of the intermediately formed σ-palladium species, was reported. Coordination of the σ-palladium intermediate to one of the oxygens of the tosyl group was suggested to suppress β-hydride elimination (Eq. (83)) [520]. Reaction of 2-bromobenzaldehydes with α,β-unsaturated aldehydes or ketones in the presence of a palladium catalyst furnished naphthalenes or naphthols (Eq. (84)) [521].

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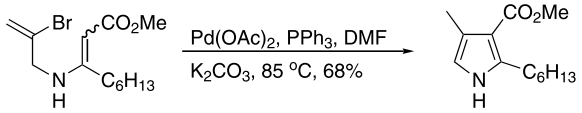
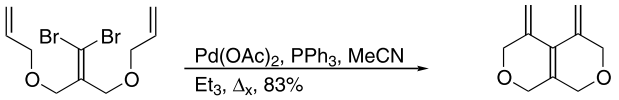



(81)

Heck reactions of alkenes and alkynes with halides and triflates to give reduced products were used to some

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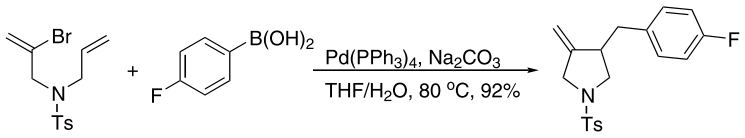


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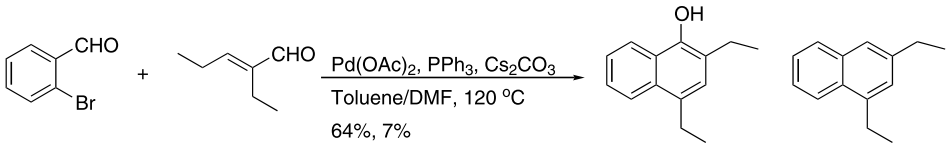
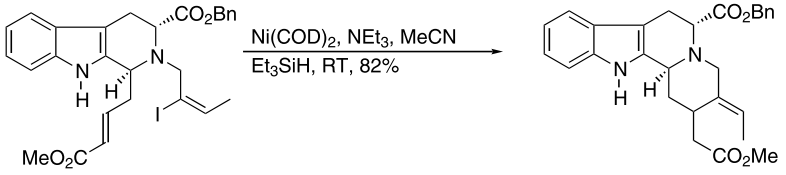



(84)

extent [522]. Reductive Heck reactions in supercritical carbon dioxide were reported [523]. An enantioselective reductive Heck reaction was described [524]. A nickel-mediated reductive Heck-type reaction was used in the synthesis of geissoschizine (Eq. (85)) [525]. Highly regioselective reductive Heck reactions using a rigid tetracyclic oxanorbornene (Eq. (86)) [526] and azabicyclo[2.2.0]hex-5-enes [527] were reported.

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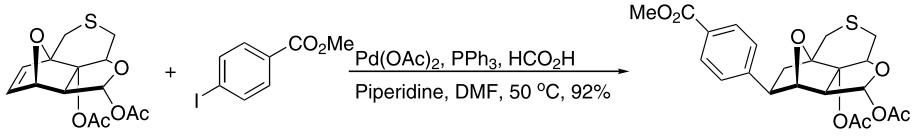
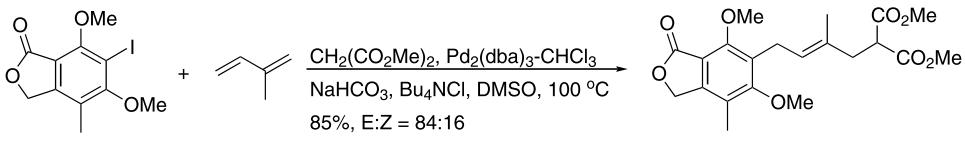



(86)

Multiple alkylations on aromatic rings were observed upon reaction of aromatic boronic esters with 2-norbornene in the presence of a rhodium catalyst [528]. Palladium catalyzed similar multiple arylations [529–531]. A three-component reaction between an aryl halide, isoprene and a stabilized nucleophile was used in the synthesis of mycophenolic acid (Eq. (87)) [532]. Multiple carbon–carbon bonds were formed upon reaction of 2-heteroatom substituted aryl iodides mediated by palladium (Eq. (88)) [533].

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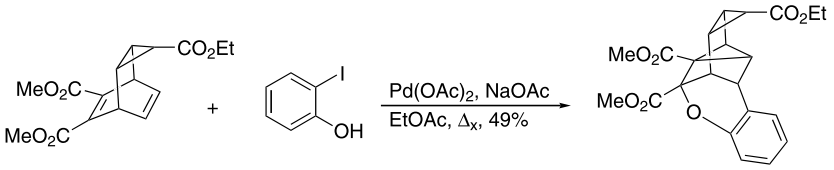
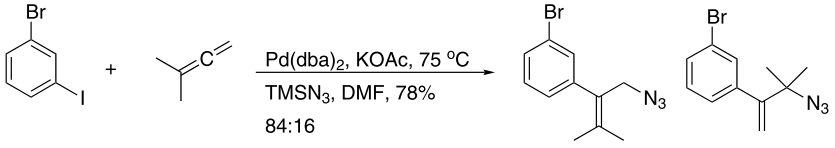



(88)

Palladium catalyzed the alkylation of aryl iodides with allenes in the presence of trimethylsilyl azide (Eq. (89)) [534]. A nickel-catalyzed reaction of ethene with styrenes producing allylic arenes was described (Eq. (90)) [535]. Palladium catalyzed the arylation of hydroxy methoxyallenylphthalans to give, after ring expansion, substituted 4-isochromanone derivatives (Eq. (91)) [536]. Palladium catalyzed cycloalkenation was used in the synthesis of (−)-methyl atis-16-en-19-oate (Eq. (92)) [537] and terpenoid ring systems [538].

was reported (Eq. (93)) [540]. A related expansion of thioacetals was also described [541]. A rhodium-catalyzed Wolff rearrangement followed by intramolecular [2+2]cycloaddition was reported [542]. The related thia-Wolff rearrangement was catalyzed by rhodium forming a phenylthioketene [543]. Copper catalyzed the formation of an allyl sulfonium ylide followed by a [2,3]sigmatropic rearrangement [544].

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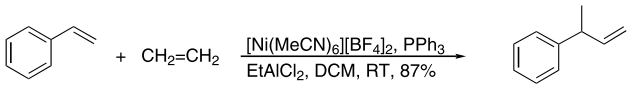


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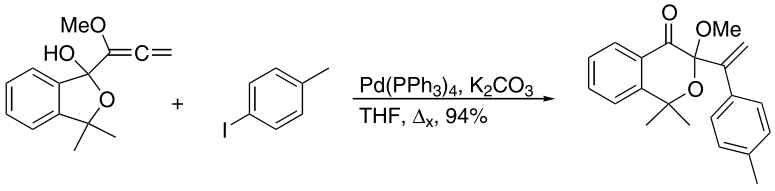


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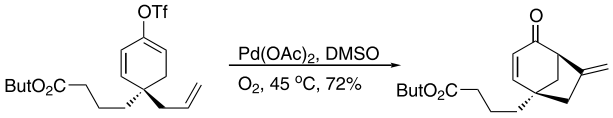
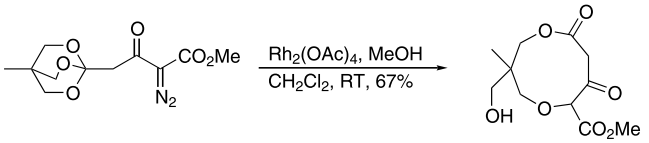



(92)

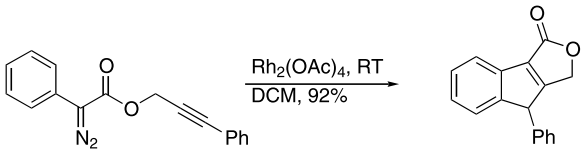
### 2.1.3. Metal-catalyzed diazo decomposition (including other cyclopropanations)

Ruthenium catalyzed the dimerization of α-diazocarbonyl compounds in high yields [539]. A number of rhodium-catalyzed reactions of resin-bound α-trimethylsilyl diazoketones were described [254]. Rhodium-catalyzed ring-enlargement of bicyclic *ortho*-esters via intramolecular formation of tricyclooxonium ylides

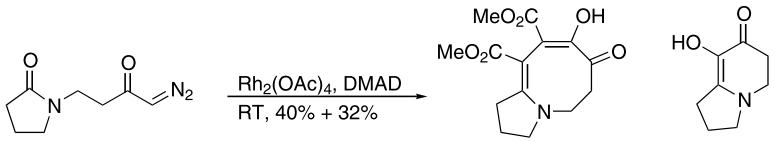



(93)

Rhodium catalyzed carbocyclization of α-diazocarbonyls with tethered unsaturations was reported (Eq. (94)) [545]. The reactivity order of intramolecular addition reactions was found to be propargyl > furfuryl > methallyl, allyl [546]. Rhodium catalyzed cyclizations of amido diazo carbonyl compounds with DMAD to form polyfunctionalized bicyclic products (Eq. (95)) [547].

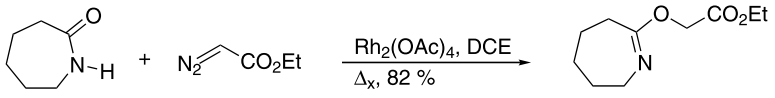



(94)




(95)

Ruthenium- and rhodium-catalyzed intermolecular insertions of α-diazo carbonyl derived carbenoids into oxygen–hydrogen bonds of alcohols were used in synthesis [548–550] of an ascomycin derivative [551], 3-alkoxyindoles [552], and C1–C15 subunit of halichondrin B [553]. Decomposition of vinyldiazoacetates in the presence of an alcohol and a molybdenum catalyst affords products derived from preferential insertion at the vinylogous position and not the carbenoid site [554]. Rhodium catalyzed the reaction of diazo esters with carbonyl compounds to form acetic esters of the corresponding enol (Eq. (96)) [555].

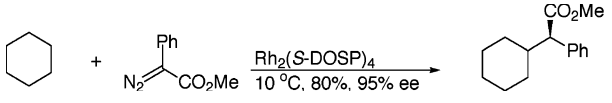



(96)

Intramolecular rhodium carbenoid nitrogen–hydrogen bond insertion was used to prepare a carbapenem [41], a dipeptide [556], α-aminoketones [557], a diazonamide A fragment [315,558] and a trialkylsilyl substituted α-amino acids [559].

The product ratio of intermolecular carbon–hydrogen bond insertion and cyclopropanation was shown to depend on the metal, ligands, and substituents on the diazo compound [560]. Reaction of diazocarbonyl compounds with cycloheptatriene in the presence of a rhodium catalyst gave either cyclopropanation or carbon–hydrogen bond insertion products, depending on the carbenoid structure [561]. Intermolecular insertions of ruthenium carbenoid species into aldehyde carbon–hydrogen bonds to give an unsymmetrical ketone [562], and into an α-carbon–hydrogen bond of tertraalkoxysilanes to give *syn*-aldol products [563] were reported.

Intramolecular rhodium-catalyzed carbon–hydrogen bond insertions were used in a number of synthetic applications forming five-membered rings [564–568]. For example, (−)-eburnamonine and (+)-*epi*-eburnamonine [569], (*R*)-carvone [570], (1*R*,7*R*,8*R*)-turneforcidine [571], grimaldone [572] α-substituted-γ-butyrolactones of nucleoside [573], and lactams [574,575] were prepared via insertion reactions. Asymmetric carbon–hydrogen (Eq. (97)) [576] and silicon–hydrogen [577,578] bond insertions were described. An iron-catalyzed Doyle–Kirmse reaction of allyl sulfides with trimethylsilyldiazomethane was reported (Eq. (98)) [579].

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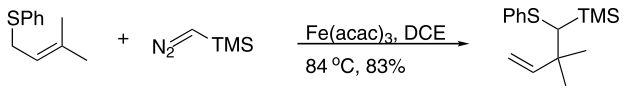
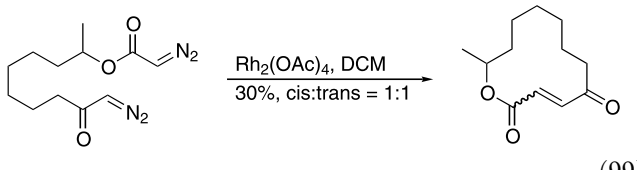



(98)

Metal-catalyzed macrocylization via alkene formation by decomposition of bisdiazo compounds was used in synthesis of patulolides A and B (Eq. (99)) [580]. A pyridine fused furan was formed upon rhodium-catalyzed decomposition of a diazoester (Eq. (100)) [581]. Double addition of a diazo compound to alkynes produced conjugated dienes [582]. A metal-catalyzed ylide formation cycloaddition cascade was reported [583]. Intramolecular reactions of ruthenium vinyl carbenoids with dienes affords complex cyclic structures (Eq. (101)) [584]. The basic core of CP-263,1114 was prepared using a related rhodium catalyzed intramolecular [3+4]cycloaddition [585].

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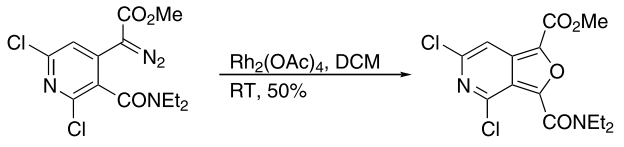


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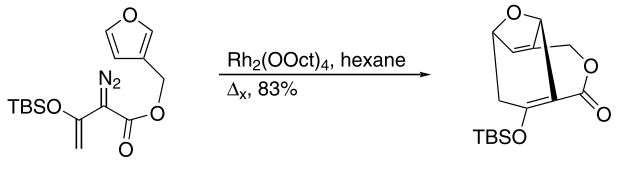
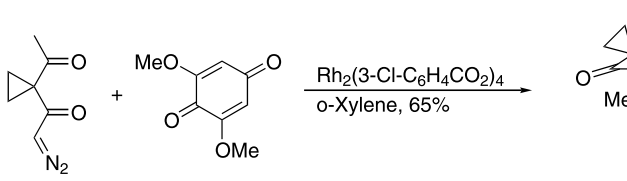



(101)

Rhodium–carbenoids participated in a variety of cycloaddition reactions. A computational study of the reaction pathway of push–pull carbonyl and ammonium ylides was reported [586]. An enantioselective intermolecular 1,3-dipolar cycloaddition was reported [587]. 2-Silylated oxazoles were prepared by intermolecular cycloaddition of diazocarbenoids with nitriles [588]. Diazocarbonyl ylide derived from 2-diazo-1*H*-1,3-phenalenedione was cyclized with, for example, nitriles, isocyanates, and ketones to afford oxazoles, oxazolones, and 1,3-dioxazoles derivatives, respectively [589]. Dipolar cycloadditions of rhodium generated carbonyl ylides with *p*-quinones (Eq. (102)) [590], with arylidenetetralones [591], and with dimethyl acetylene dicarboxylate and *N*-phenylmaleimide [592] were developed.

A cascade sequence leading to polycyclic compounds was reported [593]. Reaction of α,α′-diazocarbonyl compounds with vinyl butyl ether gave dihydrofurans [594]. Cycloaddition of *t*-butylphospha acetylene to carbonyl ylide derived from α-diazo-β-dicarbonyl compounds gave 1,3-oxaphospholes (Eq. (103)) [595]. Rhodium-catalyzed carbonyl ylide formation followed by cycloaddition was used as an approach to zaragozic acid and squalestatin skeletons (Eq. (104)) [596]. A rhodium-catalyzed diazodecomposition–cycloaddition was used in the preparation of nicotine and anabasine analogs (Eq. (105)) [597]. Rhodium catalyzed the decomposition of 2-diazo-1-oxiranyl-1-ethanone adding the resulting carbenoid to benzene, furan, and propionitrile to give an epoxyketone substituted cycloheptatriene, a *trans*,*trans*-dienal, and an oxazole, respectively (Eqs. (106), (107)) [598].

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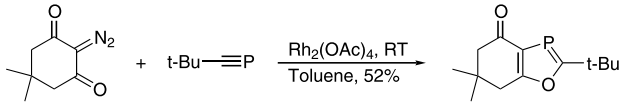


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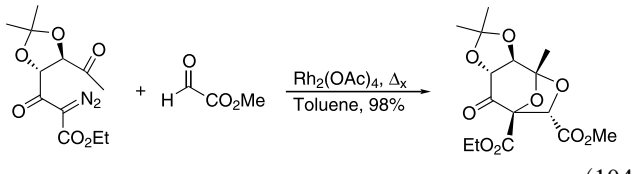


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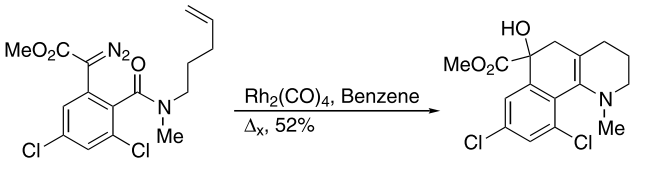


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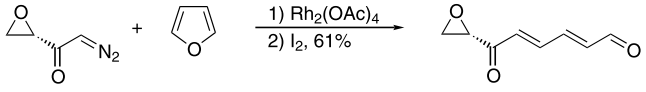


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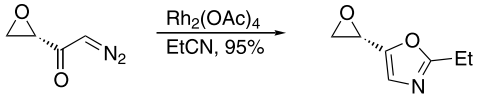
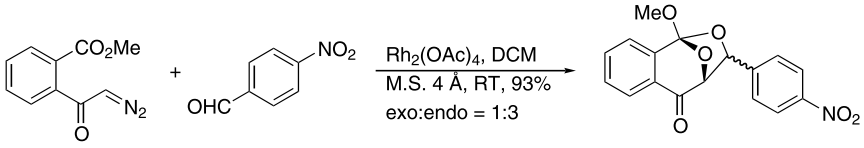



(107)

Rhodium-catalyzed decomposition of cyclic 2-diazo-1,3-dicarbonyl compounds in the presence of diketene produced fused furans [599]. Formation of bicyclic *ortho* esters from decomposition of *o*-(alkoxycarbonyl)-α-diazophenone in the presence of benzaldehydes was also described (Eq. (108)) [600]. An unusual [1,4]migration was observed using carbohydrate-derived α-diazo-β-keto-esters (Eq. (109)) [601].

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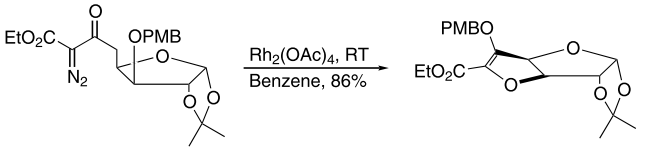
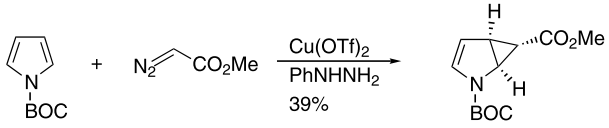



(109)

Cyclopropanations of alkenes via transition metal-catalyzed decomposition of α-diazocarbonyl compounds continued to be developed [602–611]. The chemoselectivity of rhodium carbenoids for cyclopropanation was examined. Donor/acceptor substituted carbenoids were found to be more selective [612]. A cyclopropanation using copper complexes on silica gel was described [613]. Palladium catalyzed the cyclopropanation of alkenes using diazomethane [614,615].

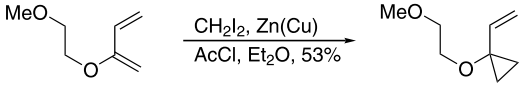
Enantioselective cyclopropanations [616,617], using chiral iron carbene complexes [618], copper salts [619–629], a cobalt–salen complex [630], ruthenium catalysts [631,632], and rhodium catalysts [633–636] were reported. Enantioselective cyclopropanations in supercritical fluoroform, using a rhodium catalyst, was developed [637].

Rhodium catalyzed the cylopropanation of an exocyclic double bond forming a spirocyclic cyclopropane [638]. 2,3-Dibromo-1-propene [639], and *N*-Boc protected pyrrole (Eq. (110)) [640] were cyclopropanated via a metal carbenoid. A rhodium-catalyzed intermolecular cyclopropanation forming 1-alkynylcyclopropane-1-carboxylates was described [641]. Intramolecular cyclopropanation of diazoacetates with allylic groups tethered by ethylene glycol units forming macrocyclic lactones [642], and of a benzofuran [643] were reported.




(110)

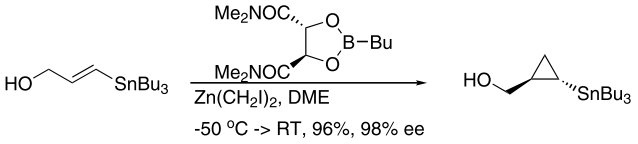
Cyclopropanation of alkenes using diethylzinc and diiodomethane or chloroiodomethane were employed in organic synthesis by a number of groups [359,644–647], for example, in the synthesis of halicholactone [648], (*S*)-(−)-β-cuparenone and (*S*)-(−)-cuparene [649], and the C29–C44 unit of spongistatin 1 [650]. Cyclopropanation of alkenes using various zinc, aluminum, and samarium carbenoids was investigated [651]. Cyclopropanation of the more electron rich double bond using diiodomethane and zinc copper in the presence of acetyl chloride was reported (Eq. (111)) [652].



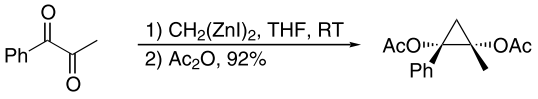

(111)

Asymmetric cyclopropanations of allylic alcohols using $ZnEt_2$ and $CH_2I_2$ were reported [653]. A tin functionalized cyclopropane was prepared in a related fashion (Eq. (112)) [38]. Iodomethylzinc phenoxides were shown to cyclopropanate alkenes in high yields [654]. An isolable and storable haloalkylzinc reagent for

cyclopropanations has been developed [655]. Cyclopropanation of 1,2-diketones using bis(iodozincio)methane was reported (Eq. (113)) [656].

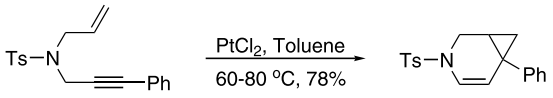



(112)




(113)

Titanium also mediated the intermolecular coupling of vinylsilanes with esters, forming silacyclopropanols [657]. Related cyclopropanations of α-hydroxyesters, 3-buten-1-ol esters [658,659], and diethylacetal [660] were reported. Platinum catalyzed an interesting intramolecular cyclopropanation starting from 1,6-enynes (Eq. (114)) [661].

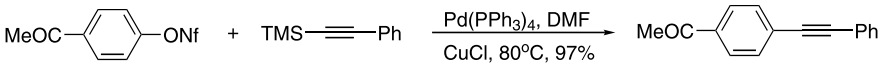



(114)

### 2.1.4. Alkylation of alkynes and arynes

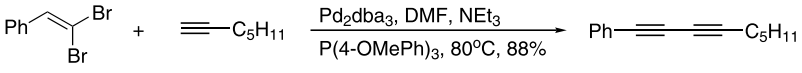
The palladium-catalyzed coupling between terminal alkynes and aryl and vinyl halides or triflates, usually called the Sonogashira reaction, was one of the more frequently utilized carbon–carbon coupling reaction [12,21,104,188,305,509,662–669].

Coupling reactions of polymer-bound halides [9,10] were reported. A palladium/copper system catalyzed the coupling of substituted alkynes with aryl triflates, nonaflates, and chlorides, and alkynyl chlorides (Eq. (115)) [670]. Sonogashira reactions in the absence of a copper cocatalyst [671] using silver(I) oxide, tetrabutylammonium fluoride, or tetrabutylammonium hydroxide as the promoter were described [672]. A stable and efficient oxime palladacycle catalyst was reported [6]. A solventless, microwave-enhanced, Sonogashira coupling on alumina was reported [673]. Heterocyclic carbene–palladium complexes were efficient catalysts for the coupling [240,674]. Coupling of aryl bromides at room temperature was achieved using a $Pd(PhCN)_2Cl_2$–P(*t*-Bu)$_3$ catalyst system [675]. A palladium catalyzed coupling under thermomorphic conditions was described [248].




(115)

α-Halovinyl ethers and vinyl triflates derived from 1,3-dicarbonyl compounds [676,677], alkynyl bromides [678], iodo- and bromoazulenes [679–681], and (*Z*)-(2-ethoxy-1-iodoethyl)diphenylphosphine oxide [682] were used in Sonogashira couplings. Aryl iodides were reacted preferentially in the presence of a vinyl bromide [26], and aryl dialkyltriazenes were coupled with alkynes [683]. Both symmetric and unsymmetric 1,3-diynes were prepared from 1,1-dibromo-1-alkenes (Eq. (116)) [684]. Benzoheteroazepines were prepared by a one-pot Sonogashira coupling of aryl halides with propargylic alcohols, followed by an isomerization and cyclocondensation (Eq. (117)) [685]. Coupling of propargylic substrates with terminal alkynes gave 1,2-dien-4-ynes (Eq. (118)) [686]. An unusual cleavage of a carbon–sulfur bond, followed by *S*-arylation was observed (Eq. (119)) [687].

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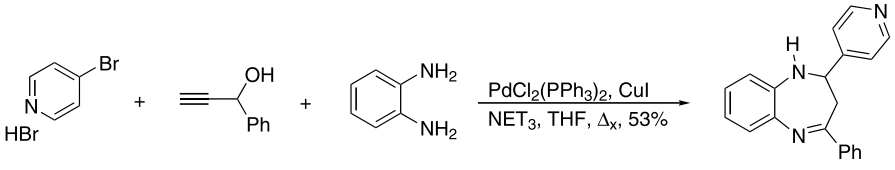


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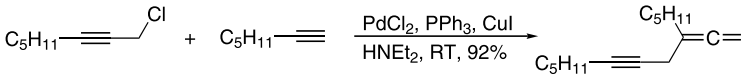


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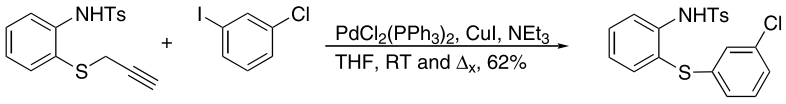
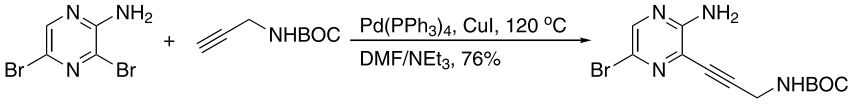


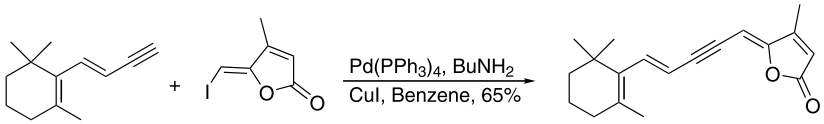

(119)

(119). Heterocyclic substrates were shown to readily participate in Sonogashira type couplings. Reactions of 4,7-dibromocarbazole [688], 2- and 3-iodopyrroles [343,677,689], halothiophens [690–694], 2-iodofuran [695], 2-chloroquinoline, in the synthesis of the ABCD ring system of camptothecin [696] and mappicine and nothapodtine B [697], 5-iododeoxyuridine [698], 5-bromothymidine [699], 5-iodouridine [700–702], 2-bromopurine [703], 8-iodoadenine [72], 8-bromo-2′-deoxyadenosine [704], 4-iodo-isoquinoline-1-one [705,706], 9-trifloxyacridine [707], bromo and iodopyridines [42,203,708–710], in the synthesis of anabasin [473], 3-iodopyridazin [711], 5-bromopyrazole [78], 7-bromotriazolo[4,3-*b*]pyridazine [67], 2-bromo-pyrazine [712], 2,6-dibromopyridazine (Eq. (120)) [90], bromo-1,10-phenanthrolines [713], 2-and 5-halopyrimidines [714,715], 4-chloropyrimidine [716], 5-iodopyrimidine [717], iodouracils [718], 4-iodoimidazoles in the synthesis of oroidin [719], 4-iodooxzoles [588], bromothiazoles [720], 2-iodoindole [62], 3-iodo-7-azaindole [721], 3-chloro-2-quinoxalinones [722], 2-halotellurophens and -selenophens [723], and 3-bromoisoquinoline [724] were reported.




(120)

The Sonogashira reaction continued to be extensively used in organic syntheses [725–727]. Examples of synthetic applications include dihydroxerulin [136], (*S*)-coriolic acid [728], vibsanol [729], lutenone and luteone hydrate [730], γ-alkylidene butenolides (Eq. (121)) [731], ciguatoxin [732–734], cyclomenol A [735], (+)-muconin (Eq. (122)) [736], ningalin B [737], (+)-prelaureatin [738], 1α,25-dihydroxyvitamin $D_3$ analogs [739], xeruline [222], (−)-cholchicine [228], (+)-hamabiwalactone [740], and muricatetrocin C (Eq. (123))

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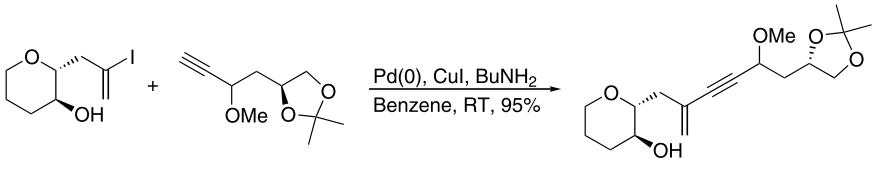


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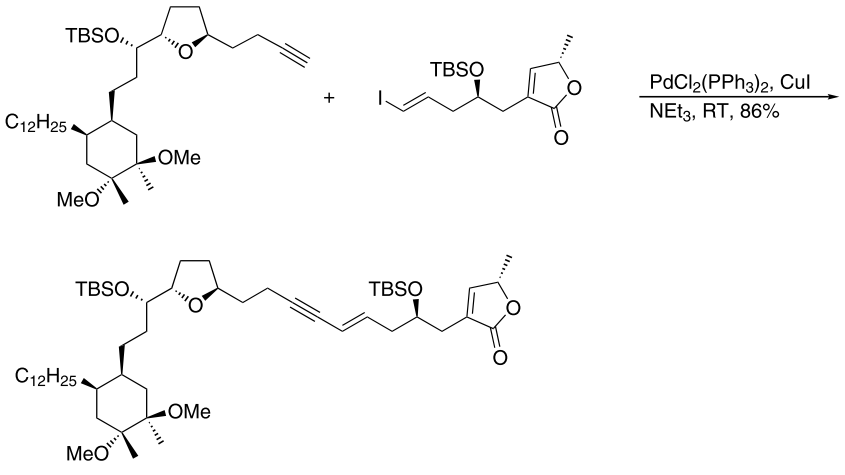
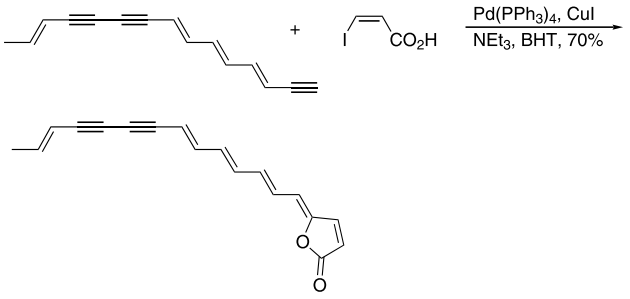



(123)

[741].

Sonogashira coupling of 3-iodopropenoic acid results in cross-coupling and intramolecular lactonization. This sequence was used in the synthesis of xeruline (Eq. (124)) [222]. Sonogashira coupling of aryl halides with propargylic alcohols in the presence of amidinium salts afforded substituted pyrimidines (Eq. (125)) [742]. Coupling of phenylacetylene with 2-bromo-5-nitrothiophene in the presence of diethylamine resulted in the formation of an enamine derived from the initial coupling product (Eq. (126)) [743].

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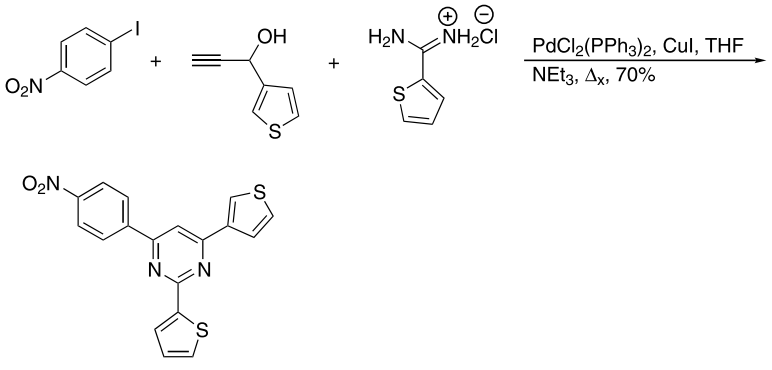


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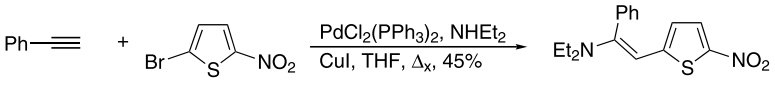
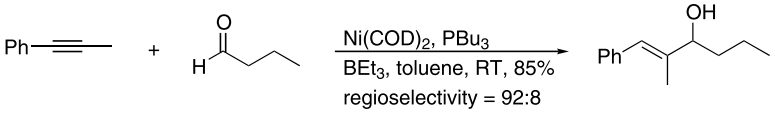



(126)

Nickel catalyzed the intramolecular reductive coupling of alkynes with aldehydes (Eq. (127)) [744]. Reaction of diethylzirconozene with alkynes and chloroformates gave a metallated intermediate that was cleaved using a variety of electrophiles (Eq. (128)) [745]. Copper mediated the coupling of aryl selenylbromides with polymer-bound terminal alkynes [746]. Cyclization of a vinylbromide tethered alkyne was used in the synthesis of (+)-strepazolin (Eq. (129)) [747]. A palladium-catalyzed diarylation of 1,4-diarylbutadiynes affording 1,1,4,4-tetraarylsubstituted-1,2,3-trienes was described [748].



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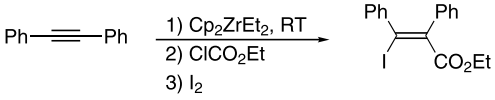

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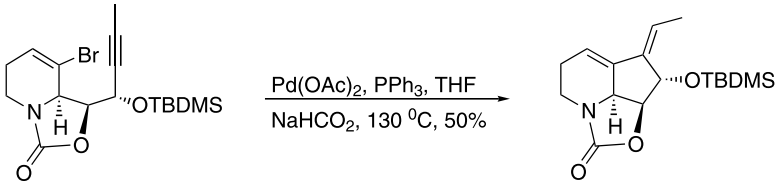
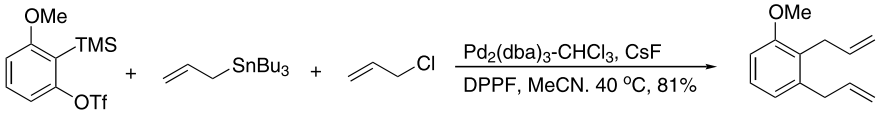


(129)

Palladium catalyzed the coupling of arynes with bis-$\eta^3$-allyl palladium complexes to give diallylated products (Eq. (130)) [749]. Reaction of benzynes with allylic chlorides produced phenanthrenes (Eq. (131)) [750]. A related reaction of benzynes with alkynes gave indenes or phenanthrenes depending on the solvent used [751].



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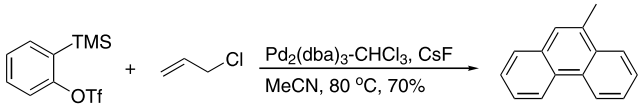
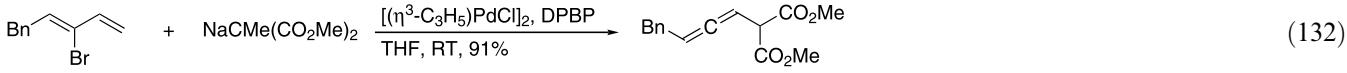


(131)

*2.1.5. Alkylation of allyl, propargyl, and allenyl systems*

Palladium continues to dominate as the catalyst of choice for allylic alkylation reactions [104,752]. Allylic substitution in water, using a number of nucleophiles, catalyzed by a colloidal dispersion system created by Triton X-100 was reported [753]. An enol acetate was used as pronucleophile activated by tributyltin methoxide [754]. A sulfonylbutenolide [755], bis(trimethylsilyl)ketene acetal [756], methyl *N*-benzylideneglycinate [757], and $Et_3GeCH(ZnBr)_2$ introducing a $Et_3GeCHZnBr$ group [758] were used as nucleophiles.

Palladium-catalyzed reaction of 2-bromo-1,3-butadienes with stabilized nucleophiles, and heteronucleophiles produced allenes (Eq. (132)) [201]. Reaction of 2-chlororallyl acetate with a mixture of sodium dimethylmalonate and sodium phenoxide in the presence of a platinum catalyst gave alkylation with the carbon nucleophile on the central atom and the phenoxide on the terminal atom (Eq. (133)) [759]. Platinum-catalyzed reaction of 3-chloro-1,3-diene monoepoxides with stabilized carbon nucleophiles produced furan derivatives (Eq. (134)) [760]. Triethylborane promoted the palladium-catalyzed allylation of allylic alcohols (Eq. (135)) [761].



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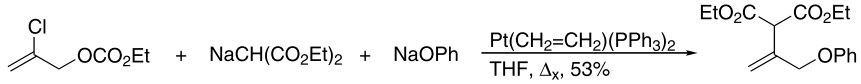

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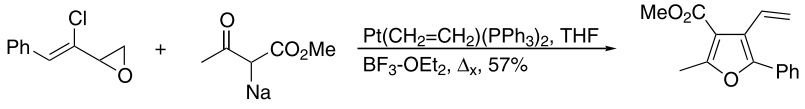

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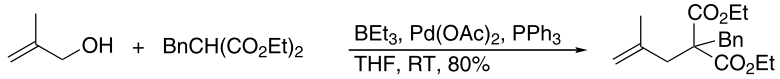
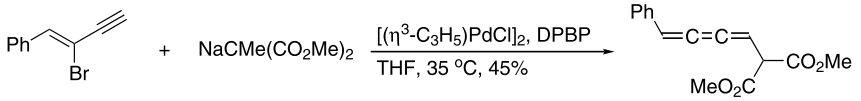


(135)

1-Tosyloxy-1-vinylcyclopropanes [762], and C-2-acetoxymethylglycals [763] were used as substrates. Cyclopropanes were formed from 1,4-dichlorobut-2-ene [764]. Palladium catalyzed the formation of butatrienes from 2-bromo-1-buten-3-yne and a nucleophile (Eq. (136)) [202]. Allylic alkylations of vinyl epoxides [765,766], and 3,3-dimethylcyclopropenylmethyl carbonates were reported [767].

Palladium-catalyzed asymmetric allylic alkylation of, primarily, 1,3-diphenyl-allylacetate continued to receive substantial attention [101,443,627,768–783]. Libraries of β-turn palladium phosphine complexes were screened [784]. A polymer-supported heterogenous catalysis was described [785]. Molybdenum [786] and rhodium [787] also catalyzed enantioselective allylic alkylations. Nickel catalyzed asymmetric allylic alkylation using Grignard reagents [788]. A microwave accelerated asymmetric allylic alkylation was reported [789–791]. A palladium-catalyzed asymmetric allylic alkylation in aqueous micelles was described [792].

A palladium catalyzed desymmetrization leading to chiral organozinc compounds was described (Eq. (137)) [793]. 1,2-Diketones were used as nucleophiles in asymmetric synthesis of cycloalkenones (Eq. (138)) [303]. α-Acetoxysulfones were used as chiral aldehyde equivalents [794]. Enantioselective formation of quartenary centers on β-ketoesters were reported [795].



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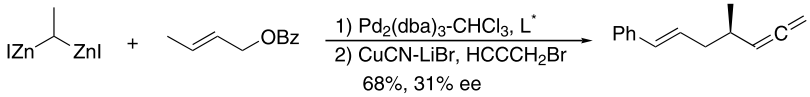

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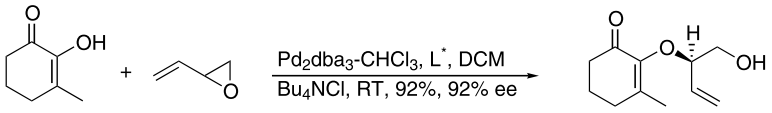
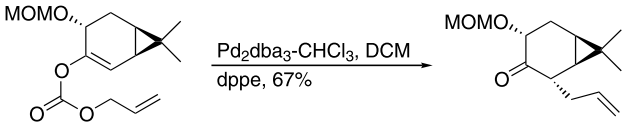



(138)

Asymmetric alkylation of cycloalkendiol acetates was described [796]. Palladium catalyzed diastereoselective allylations using optically active allylic benzoates having a fixed stereogenic center [797]. Asymmetric allylic alkylation was used in the synthesis of (−)-wine lactone [798]. An optically active 1,2,3,6-tetrahydro-2-pyrazinone derivative was employed as the nucleophile together with allylic carbonates [799].

Palladium catalyzed asymmetric alkylation of barbituric acid derivatives [800], ketone enolates [801], ketone enolate derived from an allylic carbonate (Eq. (139)) [802], nitroalkanes [803,804], and of imino esters [805]. Dimethyl pivaloyloxymalonate was used as the nucleophile in a diastereoselective alkylation of an optically active allylic lactone in a synthesis of (−)-juvabione (Eq. (140)) [806]. A double allylic alkylation using a chiral β-ketoester was used in the synthesis of (+)-huperzine A [807].

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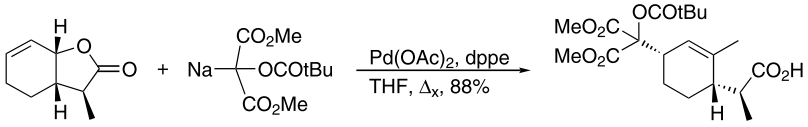
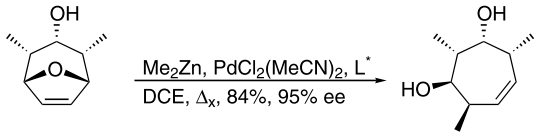



(140)

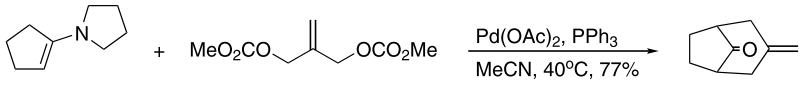
Dynamic kinetic resolution of cyclic allylic acetates using palladium and Chirazyme L-2 was developed [808]. Kinetic resolution of racemic allylic carbonates with sulfur nucleophiles was reported [809]. Intramolecular diastereoselective alkylations were reported [810]. A palladium-catalyzed enantioselective alkylative ring opening of oxabicyclic alkenes, using dialkyl zinc reagents, was described (Eq. (141)) [811,812]. Alcohols and amines can also be used as the nucleophile [813].




(141)

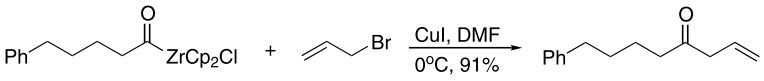
α,α′-Allylation of enamines with allylic diacetates or carbonates produced bicyclic ketones (Eq. (142)) [814]. A palladium-catalyzed stereoselective intramolecular cyclization of allylic benzamides to oxazolines was used in the synthesis of (+)-preussin [815].

In addition to palladium, a number of other transition-metals catalyzed allylic alkylation reactions. Enantioselective copper-catalyzed reactions using Grignard reagents were described. Preferential $S_N2'$alkylation was observed [816]. Rhodium catalyzed the reaction of allylic carbonates with trimethylsilyl-enolates. Low regioselectivity was observed [817]. A platinum-catalyzed asymmetric allylic alkylation affording branched products was reported [818]. γ,γ-Dialkoxyallylzirconium species were reacted in the presence of a copper catalyst with allylic bromides, phosphates, and acetates, and with acid halides to give γ,ε-unsaturated esters and 4-oxoesters, respectively [819]. A copper-catalyzed cross-coupling of acylzirconocenes with allylic or propargylic




(142)

halides was developed (Eq. (143)) [820]. A rhodium-catalyzed hydroallylation of α,β-unsaturated compounds was developed (Eq. (144)) [821]. Regioselective allylic alkylation of dienyl and enynyl acetates catalyzed by an iridium complex was described (Eq. (145)) [822]. Nickel catalyzed the reaction of allylic substrates with 1-alkynyl(trialkoxy)borates [823], and with zinc borates [824]. A nickel catalyzed asymmetric reaction of allylic compounds with arylboronic acids was reported [825].



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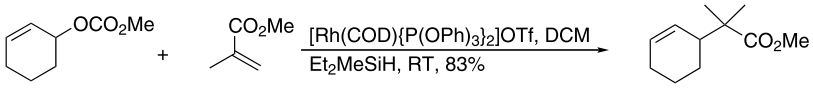

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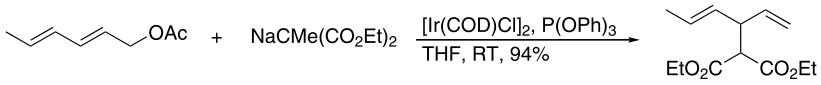
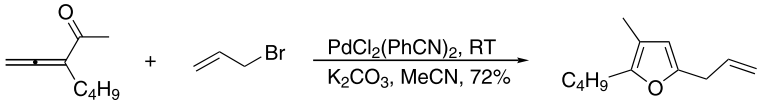


(145)

Benzylic nucleophilic substitution was reported using quinoline and isoquinoline based substrates [826]. Palladium catalyzed the cyclization of allyl bromides with 1,2-dienylketones to afford polysubstituted furans (Eq. (146)) [827]. Palladium-catalyzed cyclization reaction of polymer-supported aryl iodides with 1,2-allenyl carboxylic acids to give butenolides was reported [828]. A palladium-catalyzed selective tandem intramolecular Heck reaction followed by an intermolecular nucleophilic substitution of 2-(2′,3′-dienyl)malonates was reported forming vinyl functionalized cyclopropanes (Eq. (147)) [829]. Palladium catalyzed the coupling of propargylic mesylates, having a fluoroalkyl group, with organozinc reagents to give fluorine containing trisubstituted allenes [830]. Palladium catalyzed an asymmetric cycloaddition of vinylcyclopropanes with acrylonitrile (Eq. (148)) [831].



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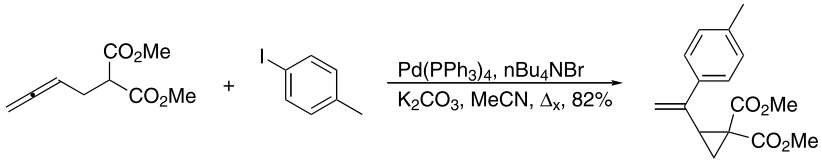

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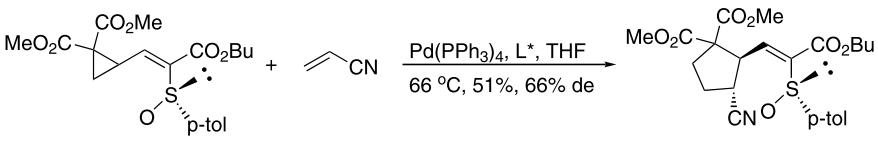

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*2.1.6. Coupling reactions*

An electrochemical cross-coupling of functionalized aryl halides with 2-chloropyrimidine and 2-chloropyrazine was catalyzed by a nickel 2,2-bipyridine complex [832]. A nickel-catalyzed electrochemical coupling of vinyl halides with activated alkyl halides and heteroaryl halides was reported [833].

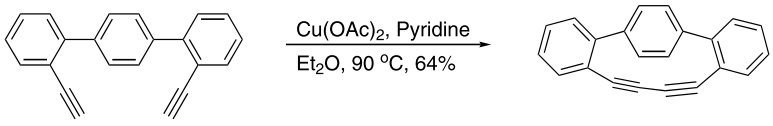
The mechanism of the palladium-catalyzed coupling of aryl halides to biaryls in water in the presence of zinc was examined [834]. Water was found to be one of the reagents in the reaction forming hydrogen gas and zinc oxide. The hydrogen gas, in turn, reduced palladium(II) to palladium(0) regenerating the catalyst.

Copper salts catalyzed the homo- [835–838] and cross-coupling of alkynes [670], homo-coupling of bromothiophen derivatives [92], homo-coupling of alkenyl and arylfluorosilanes [839], cross-coupling of hindered aryl chlorides [840], and iodides [841]. Cobalt catalyzed the homo-coupling of a benzylic bromide [842]. Palladium catalyzed [6] the cross-coupling of arylbromides with aryl iodides [843], homo-coupling of alkynes [7], and the homo-coupling of 3-iodo-3-sulfolene [89]. Nickel catalyzed the homo-coupling [844] of 2-chloropyrazine [845], of bromopyridines [846–848], and of 3-halopropenoates [849]. A $NiCl_2/CrCl_2$/Mn-mediated homo-coupling of aryl halides was reported [850].

Intramolecular coupling of alkynes was used to prepare cyclic diynes and polyynes (Eq. (149)) [678,692,851–854]. Palladium catalyzed the intramolecular coupling of a vinyl bromide with an aryl iodide to form a dihydrobenzopyrane [26]. A nickel-mediated intramolecular couplings of aryl halides was used to prepare 7-membered biaryl lactones [855]. Palladium catalyzed reductive and oxidative coupling of benzene and chlorobenzene to form biphenyls [856]. A nickel catalyst was used to achieve an intramolecular coupling of two benzyl chlorides in the synthesis of marchantin quinone (Eq. (150)) [857]. Palladium catalyzed the coupling of α-chloroglycine residues to give dimers having a carbon–carbon double bond [858].

A number of organometallic reagents underwent carbon–carbon coupling reactions. Copper mediated the intramolecular coupling of aryl stannanes [187,859,860] and the intermolecular coupling of a vinyl tin reagent [25]. Palladium catalyzed the intermolecular homocoupling of aryl- and alkynylzinc reagents [861], and of tetrathiafulvalene–tin reagents [205]. Oxovanadium(V) compounds mediated the selective cross-coupling of triorgaozincates [862].

Palladium catalyzed the dimerization of aminoynes to afford 1,3-diamino-1-yne-3-enes (Eq. (151)) [863]. An iridium-catalyzed dimerization of terminal alkynes to enynes or 1,2,3-butadienes was reported (Eq. (152)) [864]. Ruthenium vinylidene complexes also catalyzed this reaction [865]. A related reaction of a sterically constrained trialkyne was reported to give an enyne. In addition, the remaining alkyne coupled with a phosphine ligand [866]. 5,7-Dimethylbenzofuran was oxidatively coupled with dimethylgluconate, a reaction mediated by palladium (Eq. (153)) [867].

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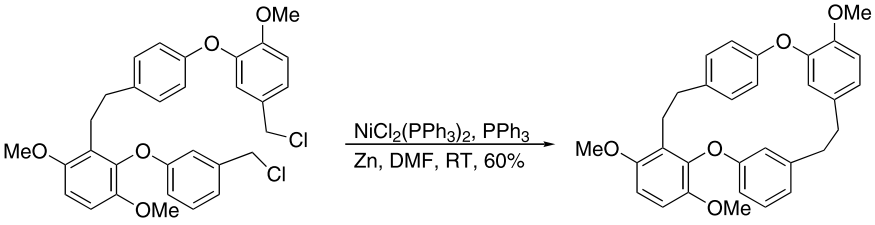


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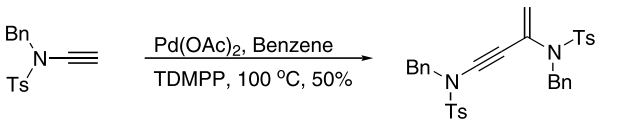


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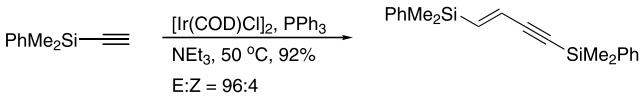


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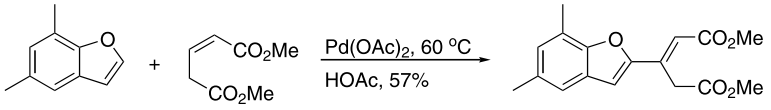
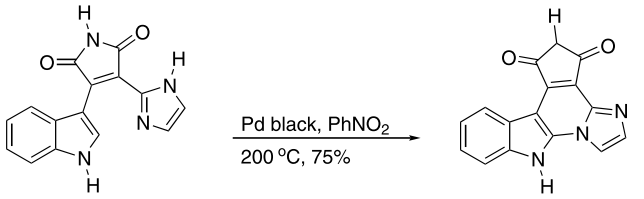
Palladium catalyzed an oxidative intramolecular C–N




(153)

coupling via a carbon–hydrogen bond insertion to form a variety of indole alkaloids (Eq. (154)) [868]. An excess of palladium dichloride was used in an oxidative coupling toward KT5823 indolocarbazole aglycone (Eq. (155)) [869]. Palladium mediated the oxidative coupling to give indolo[2,3-*c*]carbazole [870] and clausenamine A [871].

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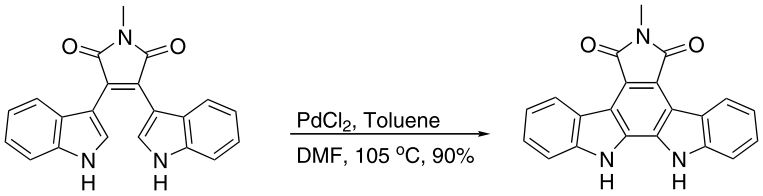


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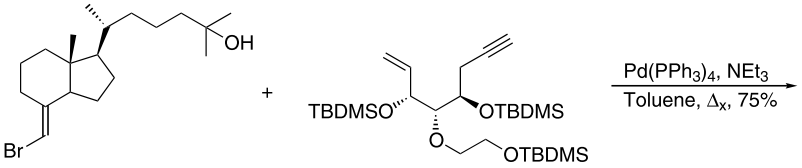


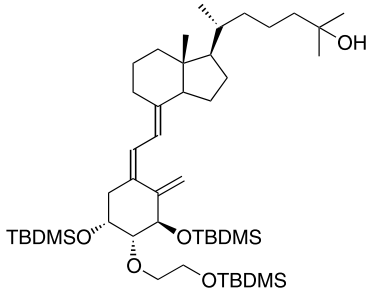


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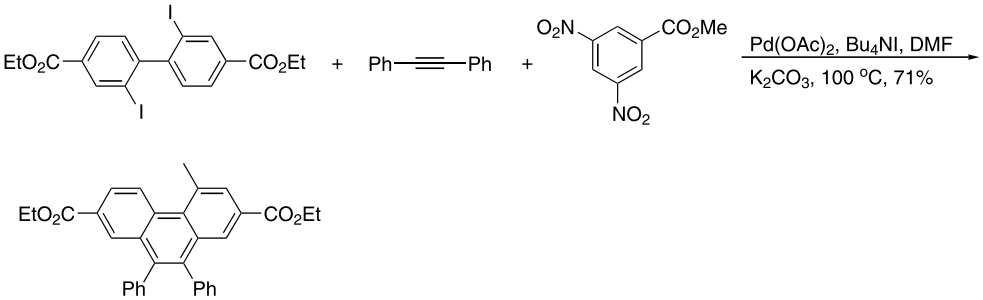
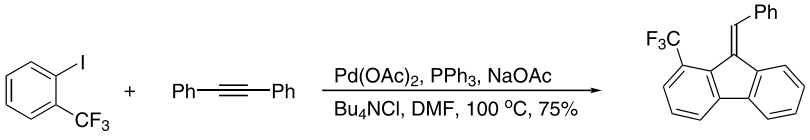



(157)

A palladium-catalyzed coupling–cyclization was used in the synthesis of 2α-(γ-hydroxyalkyl)-1α,25-dihydroxyvitamin $D_3$ (Eq. (156)) [872]. Palladium catalyzed the annulation of 2,2′-diiodobiphenyls with internal alkynes forming phenanthrenes (Eq. (157)) [873] and the reaction of aryl and heteroaryl iodides with diphenylethyne to give 9-alkylidene-9*H*-fluorenes (Eq. (158)) [874]. A titanium-mediated dihydrodimerization of 2,5-dihydrofurans, 2,5-dihydropyrroles, and 2.5-dihydrothiophen was reported (Eq. (159)) [875].

Ketone to methylene transformation was also achieved using $(t\text{-BuCp})_2Ti(Me)_2$ [893]. Related reaction of in situ prepared bis(2-methyl-1-propenyl)titanocene with a ketone producing an allene was used in the synthesis of (+)-aphanamol I (Eq. (160)) [894]. A vinyltitanocene carbene was used to form trimethylsilyl substituted 1,3-dienes (Eq. (161)) [895].

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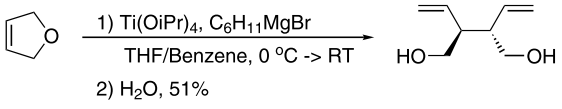


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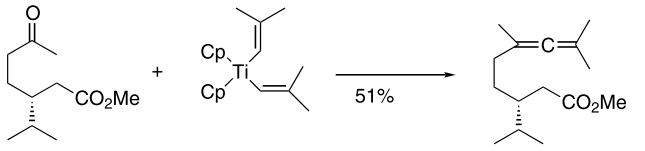


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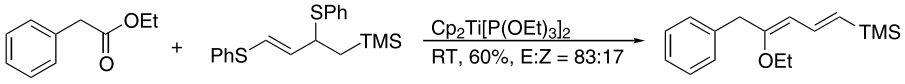



(161)

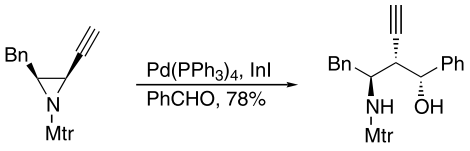
### 2.1.7. *Alkylations of carbonyl compounds*

Methylenation of ketones employing $TiCl_4$–$CH_2I_2$–Zn–$PbI_2$ was reported [876]. Reaction of $CH_2Br_2$–Zn–$TiCl_4$ was used to prepare (*R*)-carvone [877], and reaction of $CH_2I_2$–Zn–$TiCl_4$ was used in the synthesis of 6-epi-sarsolide A [878]. Tebbe's reagent, $Cp_2TiCH_2ClAlMe_3$, was used to methylenate ketones [879–881], esters [882–884], and α- and β-lactams [885], and was employed in total synthesis [323,886] of, for example, conduritol [887], (1*S*,3*S*,7*R*)-3-methyl-α-himachalene [888], (−)-hennoxole A [163], eleuthrobin [370], and (+)-ratjadone [475].

Petasis reagent, $Cp_2TiMe_2$, was used to methylenate [889,890] a formate ester forming a vinyl ether in the synthesis of C(16),C(18)-bis-epi-cytochalasin D [891], and β-lactams to form methylene substituted azetidines [892].

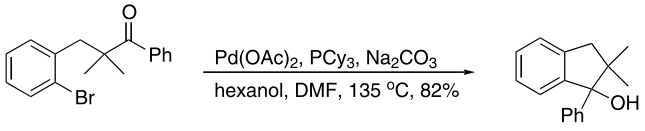
Palladium complexes catalyzed the diastereoselective alkylation of aldehydes by chiral propargylic mesylates in the presence of $Et_2Zn$ [896,897], or indium iodide [898]. In related reactions, palladium catalyzed allylations of aldehydes with allylic substrates in the presence of indium iodide [899] or triethylboron [900]. The $\eta^3$-allylic intermediate can be prepared from an allene and an aryl iodide. Both inter- and intermolecular reactions were reported [901–903]. Diastereo- and regioselective reactions of $\eta^3$-allyl molybdenum complexes having a remote coordinating heteroatom were described [904]. Allylation of aldehydes using molybdenum $\eta^3$-allyl complexes was reported [905]. Stereo- and regioselective allylation of aldehydes via a palladium-catalyzed in situ hydrostannylation of allenes in the presence of $SnCl_2$ was reported [906]. Chiral 2-ethynylaziridines were stereoselectively transformed into 1,3-aminoalcohols

bearing three chiral centers by a palladium-catalyzed indium-mediated umpolung (Eq. (162)) [907]. Intramolecular, palladium-catalyzed allylation of carbohydrates resulting in a ring contraction using a catalytic amount of $SmI_2$ [908] were reported.

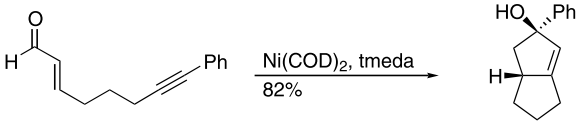



(162)

Palladium catalyzed the intramolecular alkylation of arylbromides having a tethered ketone (Eq. (163)) [909]. A nickel-mediated intramolecular reaction of alkynylenones or enals to give bicyclic systems was developed. In the presence of oxygen, a cyclopropanated product was obtained (Eq. (164), (165)) [910]. Intramolecular nickel catalyzed reductive cyclization of alkyne-5-ones was used in the synthesis of pumilotoxins (Eq. (166)) [911]. Multiple stereogenic centers were obtained upon addition of aldehydes, ketones, and chiral imines to an enyne–titanium complex (Eq. (167)) [912].




(163)

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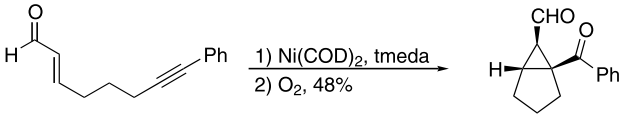


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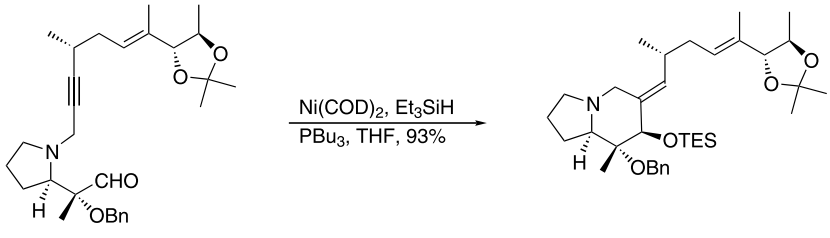
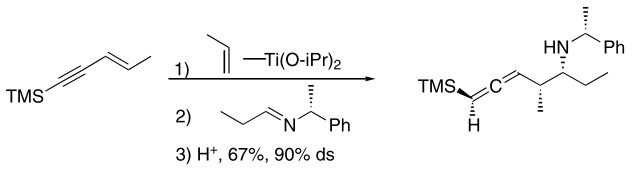
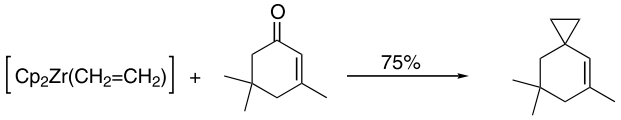



(166)




(167)

An interesting titanium-mediated synthesis of vinylcyclopropanes from α,β-unsaturated ketones was developed (Eq. (168)) [913]. Titanium complexes mediated the intramolecular cyclization of 1-yne-5- or 6-ones to give 4- and 5-membered cycloalkanols (Eq. (169)) [914].

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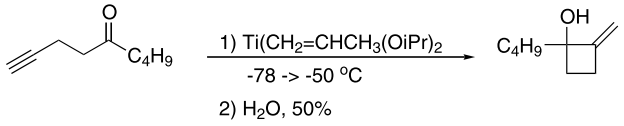
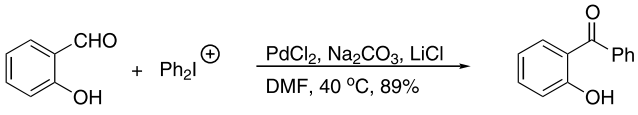



(169)

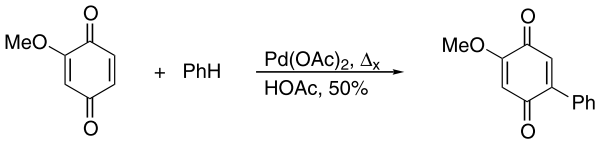
### 2.1.8. Carbon–hydrogen bond insertions

Palladium catalyzed the coupling of 2-hydroxybenzaldehydes with hypervalent iodonium salts resulting in an insertion into the carbon–hydrogen bond of the aldehyde (Eq. (170)) [915]. Palladium mediated the oxidative coupling of electron-rich arenes with 2-methoxy-1,4-benzoquinone to give methoxyarylbenzoquinones (Eq. (171)) [916]. Palladium catalyzed a regiospecific diarylation of benzanilides using aryl triflates or bromides (Eq. (172)) [917]. An interesting coupling of aryl iodides with 6- or 7-bromo-2-alkenoic esters was described (Eq. (173)) [918]. A palladium-mediated annulation was used in the synthesis of paraherquamide A (Eq. (174)) [919].

Rhodium catalyzed the insertion of a tethered vinylcyclopropane into the carbon–hydrogen bond of aldehydes to form cyclic compounds [920]. Palladium catalyzed the aromatic carbon–hydrogen bond activa-




(170)

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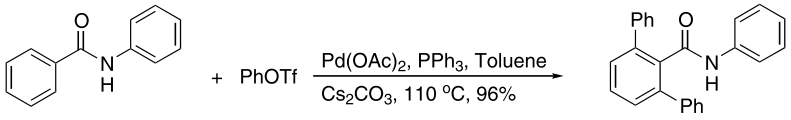


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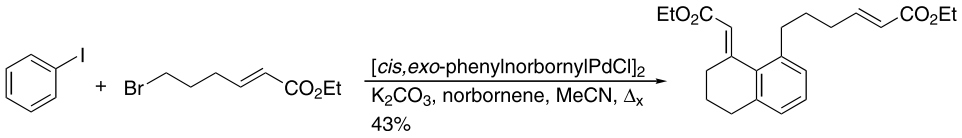
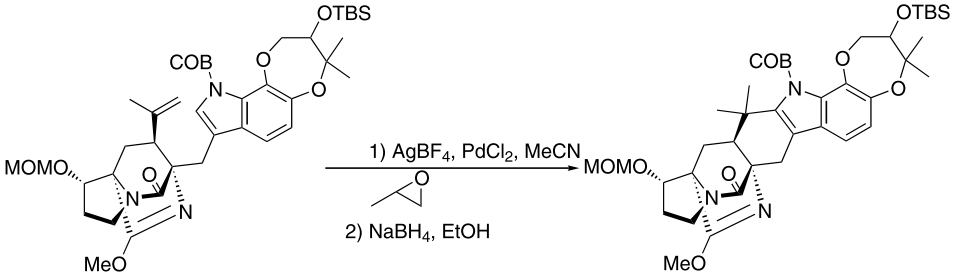
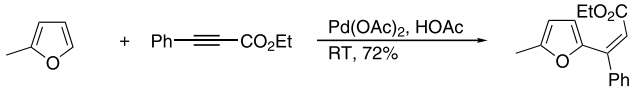



(173)




(174)

tion of indoles, pyrroles, and furan followed by addition to alkynes (Eq. (175)) [921]. A palladium-catalyzed carbon–hydrogen bond activation of the 4-position of thiazole and imidazole, followed by coupling with polymer-bound aryl iodide, was reported (Eq. (176)) [922]. In the presence of CuI, the coupling occurs in the 2-position (Eq. (177)).

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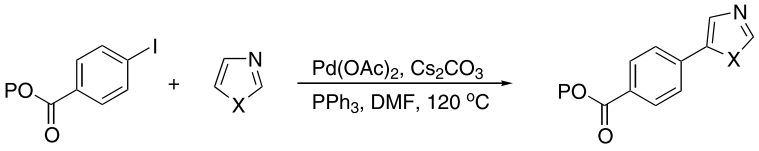
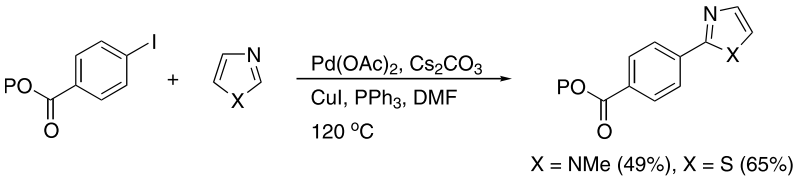



X = NMe (67%), X = S (54%)

(176)

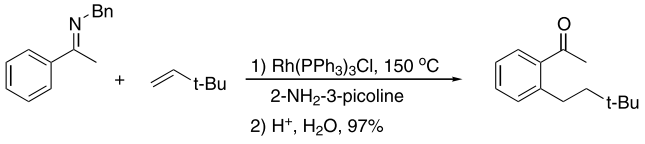



(177)

*ortho*-Alkylations of aromatic amines using Wilkinson's catalyst (Eq. (178)) [923] and of hydroquinones using a rhodium catalyst [924] were reported. Amidation of saturated carbon–hydrogen bonds catalyzed by electron-deficient ruthenium or magnesium porphyrins was developed [925]. A hydrofurylation of alkylidenecyclopropanes catalyzed by palladium was reported (Eq. (179)) [926]. Ruthenium catalyzed the carbonylation followed by alkene insertion into carbon–hydrogen bonds of *N*-heterocycles as a method for acylation (Eq. (180)) [927]. A rhodium-catalyzed asymmetric cyclization of 4-pentenals to cyclopentanones introducing two chiral centers was reported (Eq. (181)) [928]. Rhodium catalyzed asymmetric carbon–hydrogen bond insertions [929], and insertion of alkenes into carbon–hydrogen bonds of aldehydes to afford unsymmetrical ketones (Eq. (182)) [930].

### 2.2. Conjugate addition

A nickel catalyzed electrochemical driven conjugate addition of vinyl halides was developed [833]. Rhodium complexes catalyzed Michael addition of arylboronic acids [167]. Additions to cinnamaldehyde were accelerated using tri(*t*-butyl)phosphine [931]. Asymmetric conjugate addition of arylboronic esters [932] and nitroalkenes [933] to α,β-unsaturated ester catalyzed by rhodium(I)-(*S*)-BINAP were reported. A palladium-

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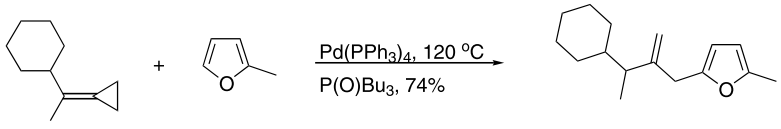


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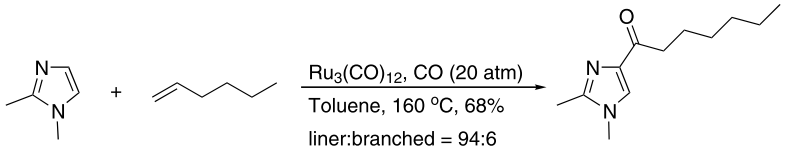


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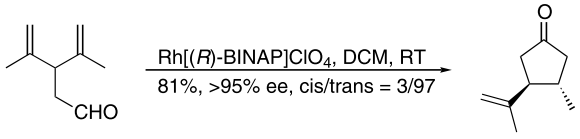
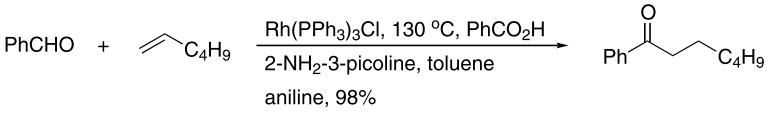



(181)



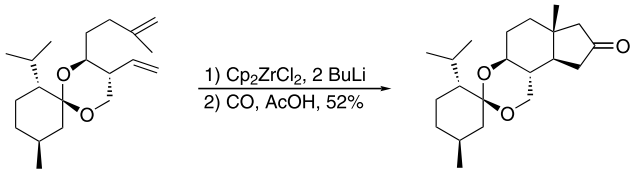

(182)

catalyzed Michael-type addition of aryltin compounds was described [934].

### 2.3. Acylation reactions excluding most hydroformylations

#### 2.3.1. Carbonylations of alkenes and arenes

A 1,6-diene was cyclozirconated and cyclized to form a cyclopentanone which was used in a synthesis of (−)-androst-4-ene-3,16-dione (Eq. (183)) [935]. Cyclotitanation to give vinyl cyclopentanes was used in the synthesis of (−)-α-kainic acid (Eq. (184)) [936]. An iron-mediated carbonylation of alkenyl cyclopropanes leading to enantiomerically pure cyclohexenones was developed (Eq. (185)) [937].

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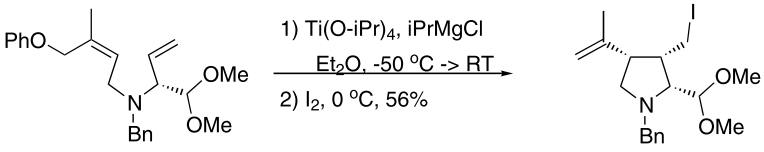


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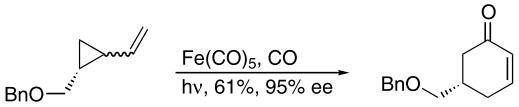



(185)

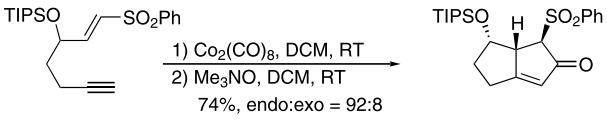
#### 2.3.2. Carbonylations of alkynes (including the Pauson–Khand reaction)

Metal-mediated [2+2+1] cycloadditions forming cyclopentenones continued to be extensively studied, in particular the cobalt-mediated, Pauson–Khand reaction [485,938–940]. Stereochemical and mechanistic features of the Pauson–Khand reaction were studied [941].

Catalyst systems for the Pauson–Khand reaction, including $Co_2(CO)_8$ together with cyclohexyl amine [942], dodecacarbonyl tetracobalt together with cyclohexyl amine [943], cobalt on charcoal under 20 atm of carbon monoxide [944], cobalt on mesoporous silica under 20 atm of carbon monoxide [945], and phosphane sulfide octacarbonyldicobalt under an atmospheric pressure of carbon monoxide [946] were developed. Polymer-supported cobalt carbonyl complexes were used as catalysts [947]. A molecular sieve-promoted Pauson–Khand reaction was used to prepare tricyclic aromatic compounds [948]. A high loading polymer-bound, recyclable, morpholine *N*-oxide was used as a promoter [949]. A polymer-bound alkyl methyl sulfide [950], and high intensity ultrasound [951] were used as promoters. Catalytic intermolecular reactions in supercritical ethane were reported [952].

Intermolecular Pauson-Khand reactions of terminal ynamine–cobalt complexes smoothly proceeded at −35 °C. Chiral alkyneamines gave high diastereoselectivities [953] as did phenylpropiolic amides derived from (*S*)-methionine [954]. An enantiospecific Pauson–Khand reaction was reported using optically pure heterobimetallic alkyne complexes [955]. Asymmetric Pauson–Khand reactions using cobalt complexes were reported [956–959]. Brucine *N*-oxide promoted asymmetric Pauson–Khand reactions [960,961]. A rhodium–BINAP complex was used in the asymmetric Pauson–Khand reaction [962]. Pauson–Khand reactions of optically active 6,7-bis(*t*-butyldimethylsilyloxy)non-1-en-8-ynes were developed [963].

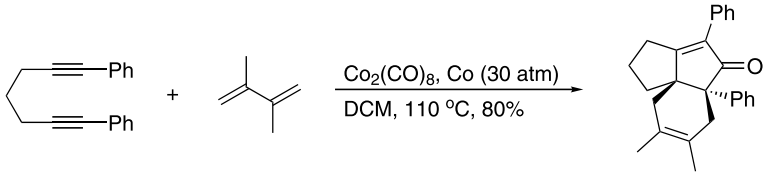
Methylthio-substituted alkynes were shown to afford a higher yield and stereoselectivity compared to unsubstituted alkynes in intramolecular Pauson–Khand reactions [964]. 1-Phenylsulfonyl substituted 1-ene-6-ynes gave predominantly products derived from *endo* cyclization (Eq. (186)) [965]. An intermolecular Pauson–Khand reaction was used in synthesis of asteriscanolide [966].




(186)

A dicobalt octacarbonyl-catalyzed tandem cycloaddition between diynes and dienes under carbon monoxide

was reported (Eq. (187)) [967]. A cobalt-catalyzed double carbonylative [2+2+1] or [2+2+2] cycloaddition was described (Eq. (188)) [968]. Tandem Pauson–Khand reactions forming tetracycles were reported [969]. An alkyne tethered cyclopropene gave the expected product in low yield (Eq. (189)) [767].

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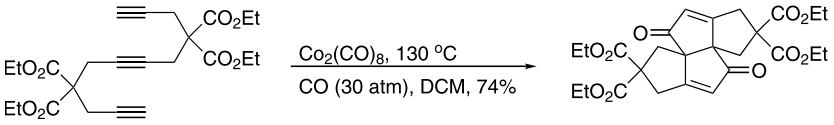
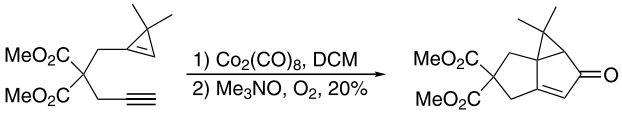


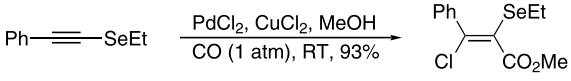

(188)




(189)

An allenic, molybdenum hexacarbonyl-mediated, Pauson–Khand type reaction was used to prepare bicyclo[4.3.0]alkanedienones [970] and was employed in the synthesis of hydroxymethylacylfulvalene [971]. Titanium alkoxide mediated [972], rhodium catalyzed [973], and molybdenum mediated [974] Pauson–Khand type reactions were described. Ruthenium catalyzed cyclopentenone formation from reaction of allylic carbonates with alkenes in the presence of carbon monoxide (Eq. (190)) [975]. An enantioselective iridium-catalyzed Pauson–Khand reaction was developed [976].

Palladium-catalyzed chlorocarbonylation of alkynyl selenides was reported (Eq. (191)) [977]. A palladium-catalyzed carbonylation of propargylic acetates forming γ-acetoxy-β-methoxy-α,β-unsaturated esters was reported (Eq. (192)) [978]. Iron carbonyl species reacted with terminal alkynes to give either 1,4-benzoquinones or 1,2-cyclobutendiones (Eq. (193), (194)) [979,980]. Carbonylation of a 2-butyne-1,4-diol furnished a 3,4-dihydro-1,5-furanedione [981].

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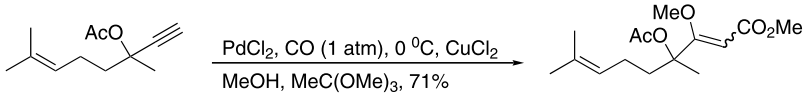


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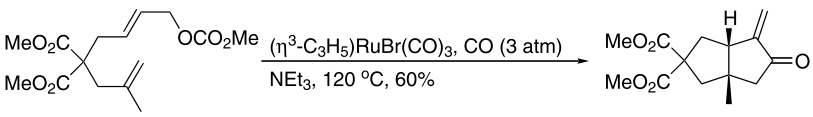


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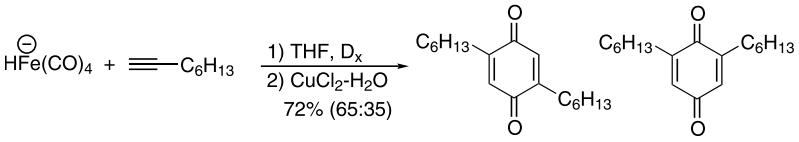


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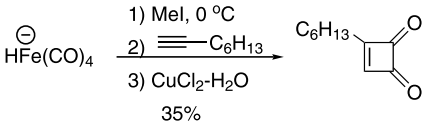
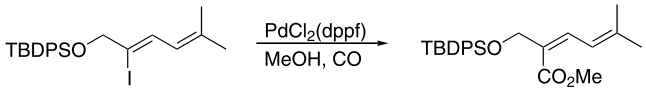


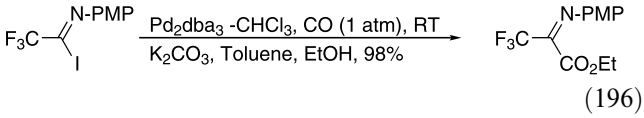

(194)

### 2.3.3. Carbonylation of halides, triflates, and phosphonates

Palladium complexes catalyzed the carbonylation of a variety of substrates to form carboxylic acids and derivatives. Esters, amides, and hydroxamic acid derivatives [982] were prepared by carbonylation of halides and triflates. Vinyl halides [983] and triflates [984], in the synthesis of coronafacic acid [985], ent-gelsedine [506], roseophilin [986], allocyathin $B_3$ [987], enolphosphates [988], and (−)-spirotryprostatin B (Eq. (195)) [989], aryl halides, in the synthesis of analogs of calcitrol [990], bromopyridines [991], in the synthesis of (*S*)-acromelobic acid [992], 4-bromobenzopyrans [993], trifluoroacetimidyl iodides (Eq. (196)) [994], and α-halovinyl ethers [12] were carbonylated, forming esters or amides. Carbonylation of an alkynyl substituted cyclic carbonate produced an allenic ester (Eq. (197)) [995]. Carbonylation of iodoindoles in the presence of tributyltin hydride gave the corresponding aldehyde [42].




(195)

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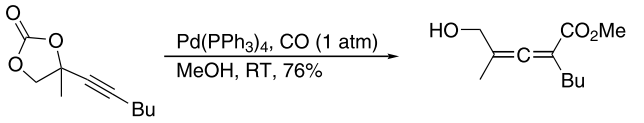
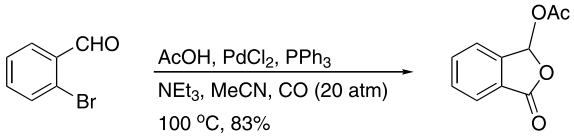


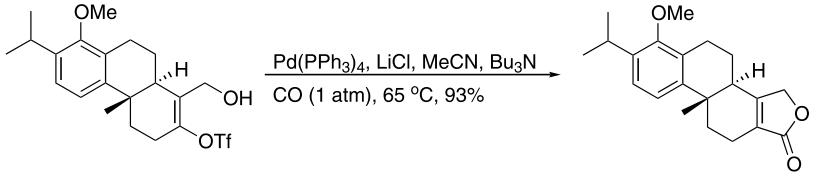

(197)

Formation of lactones by carbonylation of 2-bromobenzaldehyde in the presence of a carboxylic acid was reported (Eq. (198)) [996]. Palladium-catalyzed carbonylation–lactone formation [997] was utilized in organic synthesis (Eq. (199)) [998] of (+)-hamabiwalactone [740]. Carbonylation of aryl bromides having a tethered amino group affords lactams [999]. Rhodium catalyzed the carbonylation of benzyl halides in the presence of potassium iodide and formic acid to give phenylacetic acids.

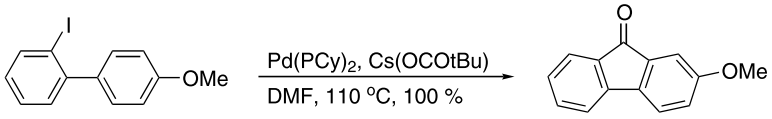



(198)

Palladium catalyzed the formation of fluorene-9-ones by carbonylation of 2-halobiphenyls (Eq. (200)) [1000]. Palladium-catalyzed carbonylation of a vinyl iodide in the presence of sodium tetraphenylborate gave a vinyl phenyl ketone [1001]. An interesting synthesis of cyclo-2-buten-1-one starting from a vinyltriflate was reported (Eq. (201)) [1002].




(199)

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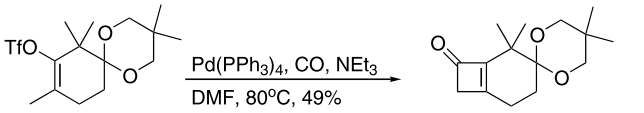
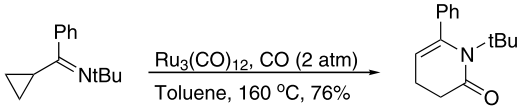



(201)

### 2.3.4. Miscellaneous carbonylations

3,4-Dihydro-2(1*H*)pyridinones and pyrroles were obtained by carbonylative [5+1] cycloaddition of cyclopropyl imines catalyzed by ruthenium carbonyl complexes (Eq. (202)) [1003]. Ruthenium catalyzed the carbonylation of allenyl alcohols to afford γ- and δ-lactones (Eq. (203)) [1004]. Palladium catalyzed carbonylation of 4-yn-1-ols to give 2*E*-[(methoxycarbonyl)methylene]tetrahydrofurans [1005]. Rhodium-catalyzed hydroformylation of alkylmercurials was reported [1006]. Rhodium catalyzed the cyclization of α-dicarbonyl compounds, alkenes and carbon monoxide (Eq. (204)) [1007]. Rhodium catalyzed the carbonylation of $sp^3$ carbon–hydrogen bond adjacent to nitrogen in amines (Eq. (205)) [1008].

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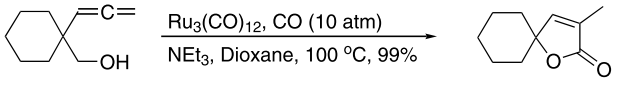


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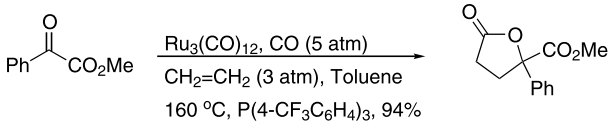
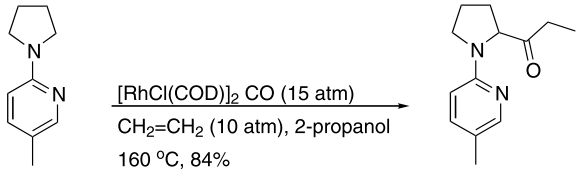
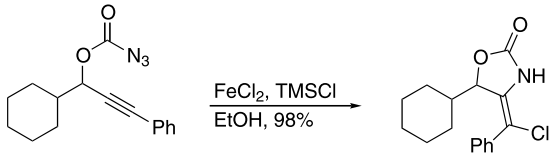



(204)




(205)

Palladium catalyzed the oxidative carbonylation of 2-amino-1-alkanols to give 2-oxazolidinones [1009]. Iron-catalyzed decomposition of propargyloxycarbonyl azides to afford oxazolidinones was described (Eq. (206)) [1010]. Palladium-catalyzed carbonylation of a 2-iodo-*N*-allylaniline produced acetic acid ester substituted quinolinones by double carbonylation [1011]. Diiron nonacarbonyl catalyzed the carbonylative dimerization of 2-bromobenzyl bromide [487]. A rhodium-catalyzed carbonylative carbotricylization of enediynes was developed (Eq. (207)) [1012]. Palladium catalyzed the decarboxylative carbonylation of 5-vinylazolidin-2-ones (Eq. (208)) [1013].

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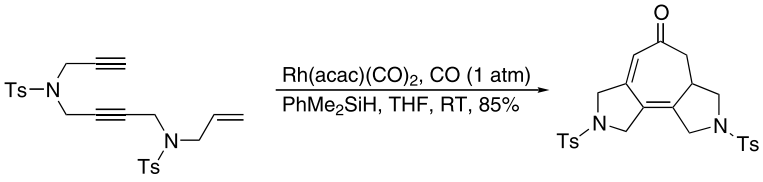
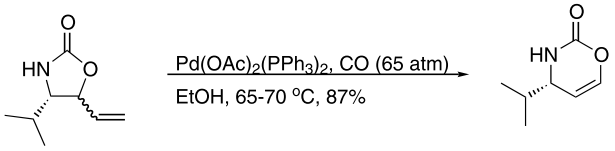



(207)

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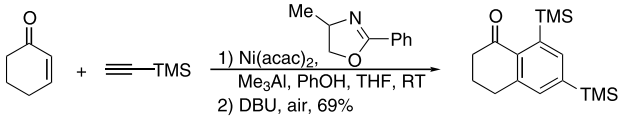


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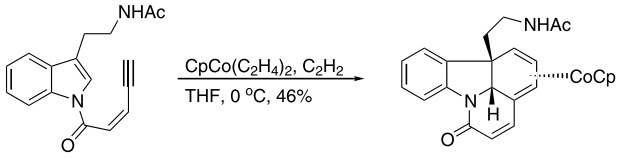
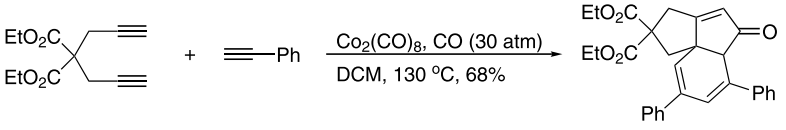



(210)

### 2.4. Oligomerization (including cyclotrimerization/cyclobenzannulation/cycloaddition)

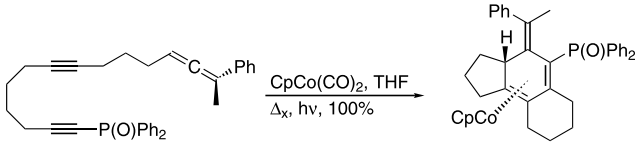
Nickel catalyzed the [2+2]annulation of electron deficient allenes to give cyclobutanes regioselectively [1014] and of alkynes with activated cyclic alkenes [1015]. Palladium catalyzed the [2+2]cycloaddition of ketenes with aldehydes [1016] of allylic acetates and norbornene [1017], and of ketenes with cyclopentadiene [1018]. Ruthenium catalyzed [2+2]cycloaddition between norbornenes and ethyl 3-phenylpropynoate [1019]. Palladium catalyzed an asymmetric [4+2]cycloaddition of vinylallenes with 1,3-dienes [1020].

A molybdenum mediated [2+2+2]cyclobenzannulation was reported [1021]. Iron-catalyzed cyclotrimerization of alkynes was reported [1022]. Ruthenium, Wilkinson's, and Grubb's catalysts were used for highly regio- and stereoselective [2+2+2]cyclobenzannulation of 1,6-diynes with alkynes [480,1023,1024]. Wilkinson's catalyst was used in a related reaction to give, for example, constrained peptides [1025,1026]. A nickel/aluminum catalyst system was used for the cyclotrimerization of enones with alkynes (Eq. (209)) [1027]. Cyclotrimerization of nitrogen containing 1,6-diynes with dimethyl acetylendicarboxylate using Wilkinson's catalyst produced an isoindoline [1028]. Cobalt-mediated [2+2+2]cyclobenzannulation was used in a formal synthesis of strychnine (Eq. (210)) [509], of morphinoids [1029], and of "molecular asterisks" [1030]. Nickel catalyzed a stereoselective [2+2+2]cycloaddition of oxa- and azabenzonorbornadienes with alkynes [1031]. Alkyne–nitrile cyclotrimerization to give pyridines in aqueous solution employing a water soluble cobalt catalyst was reported [1032].




(211)

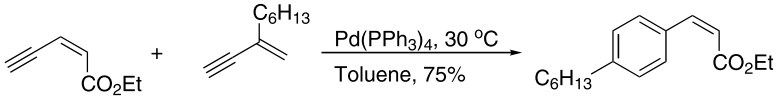
Dicobalt octacarbonyl-catalyzed tandem [2+2+1] and [2+2+2]cycloaddition of diynes with two phenylacetylenes under carbon monoxide was described (Eq. (211)) [1033]. Total chirality transfer in cobalt mediated intramolecular diyne–allene cycloaddition was reported (Eq. (212)) [1034]. Palladium catalyzed the intramolecular cyclotrimerization of a triyne. A triyne–palladium complex was isolated upon reaction with a stoichiometric amount of palladium [1035]. Titanium complexes supported on a calixarene ligand catalyzed highly regioselective alkyne cyclotrimerizations [1036].




(212)

Palladium catalyzed homo-benzannulation of electron-deficient enynes was reported [1037]. A related reaction was used to prepare metacyclophanes via an intramolecular benzannulation [1038]. Palladium catalyzed cross-benzannulation of conjugated enynes forming functionalized benzenes (Eq. (213)) [1039]. A platinum–enyne complex was isolated and shown to afford functionalized benzenes upon thermolysis in the presence of an additional molecule of the uncomplexed enyne [1040]. Performing the same reaction in the presence of a nickel or palladium catalyst gave bicy-

clo[4.2.0]octa-1,3,7-trienes (Eq. (214)) [1041]. Palladium-catalyzed cyclotrimerizations of phenylethenes and alkynes to afford fulvenes were reported (Eq. (215)) [1042,1043].

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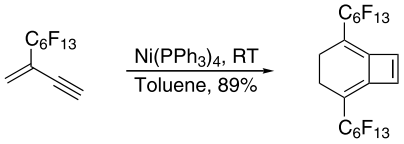
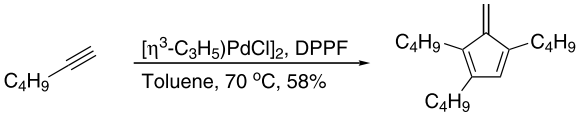


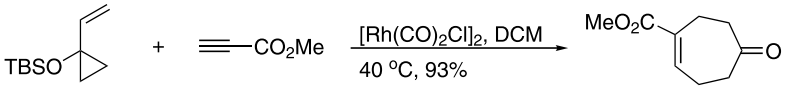

(214)




(215)

Rhodium catalyzed an intermolecular [5+2]cycloaddition of alkynes with 1-alkoxy-1-vinylcyclopropane to afford substituted cycloheptenones (Eq. (216)) [1044]. Ruthenium catalyzed the intramolecular [5+2]cycloaddition of alkyne tethered vinylcyclopropanes (Eq. (217)) [1045]. Intramolecular [5+2]cycloaddition of allenes with vinylcyclopropanes was used in the synthesis of (+)-aphanamol I (Eq. (218)) [894]. The influence of the substituents on the cyclopropyl group on the regioselectivity of the intramolecular reaction was studied [752]. A new rhodium catalyst system was developed for intramolecular [4+2]- and [5+2]-cycloadditions [1046]. An interesting rhodium-catalyzed [6+2]cycloaddition affording eight-membered rings was developed (Eq. (219)) [1047].

Palladium catalyzed the cocyclization of arynes with dimethyl acetylendicarboxylate forming polycyclic aromatic hydrocarbons [1048,1049]. Allyl tosylates were benzannulated with two equivalents of an alkyne to give polysubstituted benzenes (Eq. (220)) [1050]. Palladium catalyzed the formation of 9,10-phenanthryne followed by trimerization [1051].

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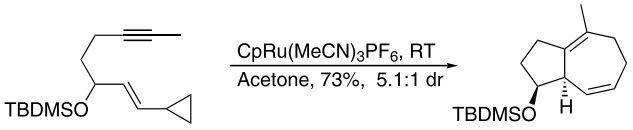


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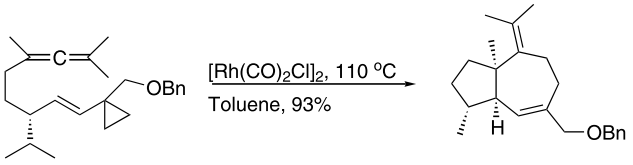
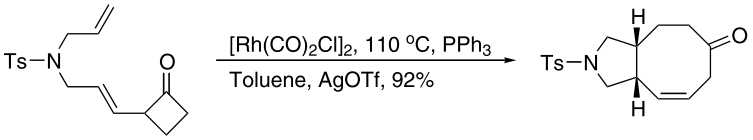
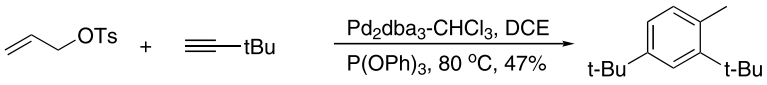



(218)

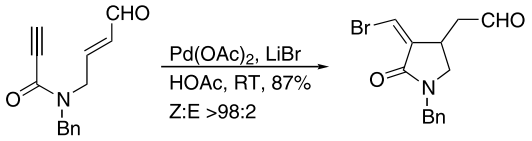



(219)




(220)

Transition-metal-catalyzed intramolecular 1,6- and 1,7-enyne cycloisomerization reactions continued to be developed [1052,1053]. Incorporation of a bromide was observed using lithium bromide as a co-reactant (Eq. (221)) [1054]. Ruthenium catalyzed the cycloisomerization of 4-oxa-1,6-enynes (allyl propargyl ethers) in the presence of acetic acid or ethanol to give 3,4-dialkylidenetetrahydrofuranes [1055]. A number of transition-metal complexes catalyzed the intramolecular 1,6-enyne cyclization of allylic stannanes and silanes [1056].

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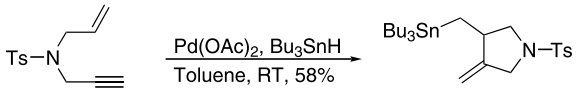
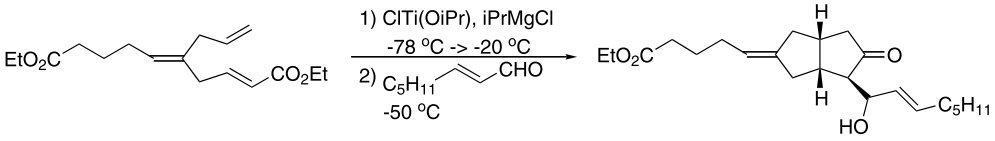


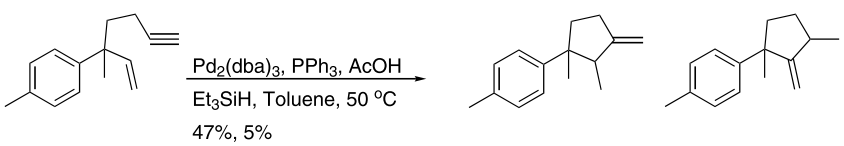

(222)




(223)

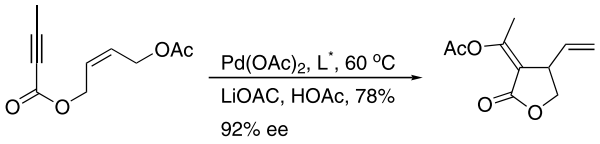
Ruthenium catalyzed the cycloisomerization of 1,2-di- and trisubstituted 1,6- and 1,7-enynes [1057]. Treatment of 1,6-enynes with tri(*n*-butyl)tin hydride in the presence of a palladium catalyst afforded *exo*-methylenecyclopentanes having a tri(*n*-butyl)stannylmethyl group (Eq. (222)) [1058]. A titanium-mediated cyclization of 1,6-dienes, followed by reaction with an aldehyde, was used in the synthesis of carbacyclin (Eq. (223)) [368]. Titanium mediated the cyclization of 1,6-bisacetylenic amides and esters forming lactams and lactones, respectively, having two adjacent exocyclic double bonds [1059].

A highly enantioselective rhodium-catalyzed 1,6-enyne cycloisomerization was reported [1060]. Excellent transfer of chirality was observed upon titanium-mediated cyclization of optically active 1,6-enynes [1061]. Asymmetric cyclization of 1,6-dienes having a tethered alcohol was reported [1062]. A tetrahydrofuran soluble zirconocene dihydride complex was used in reductive 1,7-diene and 1,6-enyne annulations [1063]. A reductive palladium-catalyzed 1,6-enyne annulation was used in the synthesis of laurene (Eq. (224)) [1064]. Palladium catalyzed cyclization of 1,6-enynes having an allylic acetate to give optically active γ-butyrolactones (Eq. (225)) [1065]. Ruthenium-catalyzed cycloaddition

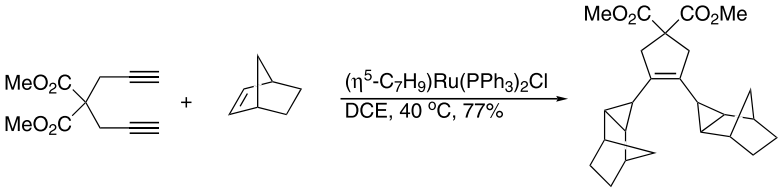



(224)

of 1,6-diynes with alkenes affords polycyclic products via a ruthenacyclopentadiene (Eq. (226)) [1066].

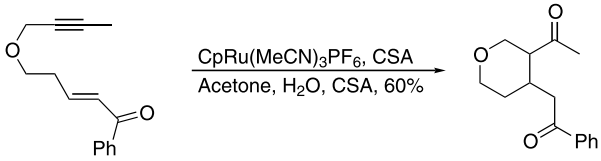



(225)




(226)

Ruthenium catalyzed cyclizations of 1,6- and 1,7-enynes to form diketones or pyranes depending, on the reaction conditions (Eq. (227)) [1067]. Platinum catalyzed alkoxy and hydroxycyclization of 1,6-enynes (Eq. (228), (229)) [1068]. Treatment of enynes with tributylmanganate(II) gave alkylidenecyclopentanes or hexanes depending on the starting material [1069].

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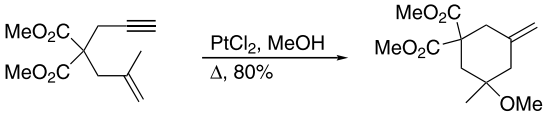


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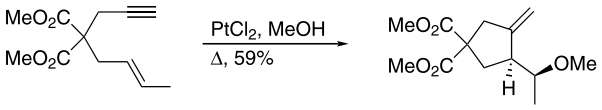
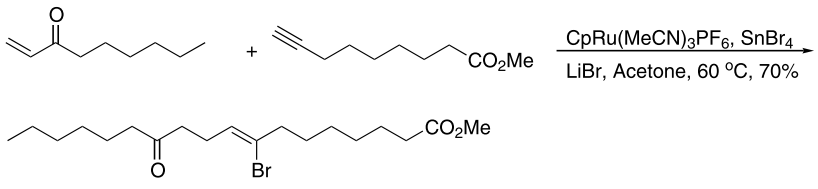



(229)

A ruthenium-catalyzed three-component reaction between an HBr equivalent, an alkene, and an alkyne was used to prepare 5-bromo-4-alkenone (Eq. (230)) [1070]. This methodology was extended to a four-component coupling affording polyfunctionalized compounds (Eq. (231)) [1071]. Ruthenium-catalyzed reaction of silylalkynes with terminal alkenes proceeded with complete control of the regioselectivity to give geometrically defined vinylsilanes (Eq. (232)) [1072].

Palladium catalyzed [4+2]cross-benzannulation reactions of conjugated alkoxy substituted enynes with diynes to form aryl ethers (Eq. (233)) [1073]. In a similar fashion, polysubsubstituted anilines were prepared from aminoenynes and diynes [863].

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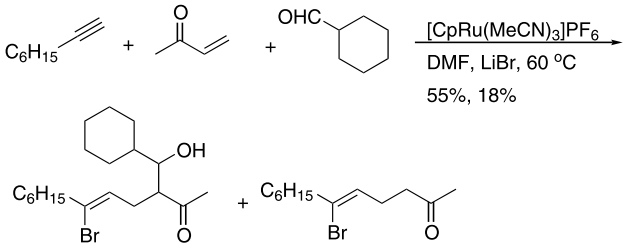


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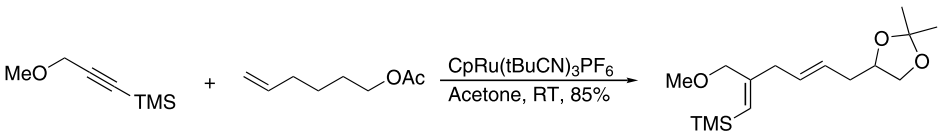


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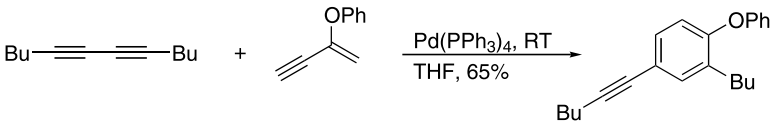
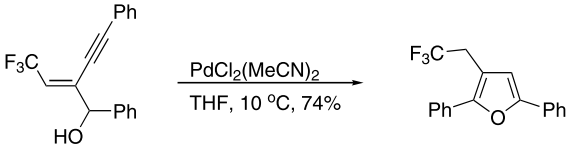



(233)

Tungsten catalyzed cycloisomerization of 5-hydroxy-1-alkynes to give dihydropyranes [1074]. Palladium catalyzed the cycloisomerization of *Z*-2-alkynyl-3-trifluoromethyl allylic alcohols forming 3-trifluoroethylfuranes (Eq. (234)) [1075]. Palladium catalyzed the cycloisomerization of (*Z*)-2-en-4-yne-1-thiols to give thiophens (Eq. (235)) [1076]. Cationic ruthenium complexes catalyzed the cycloisomerization of (*Z*)-2-ene-4-yne-1-ols to furans and 3- or 4-yne carboxylic acids to α-alkylidene lactones [1077]. In contrast, palladium-catalyzed cycloisomerization of (*Z*)-3-trifluoromethyl-4-yne-2-ene-1-ols gave 4-trifluoromethyl-2*H*-pyrans (Eq. (236)) [1078].

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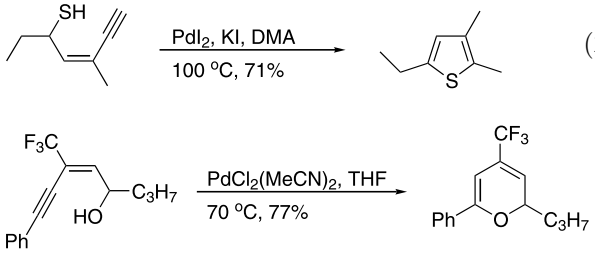
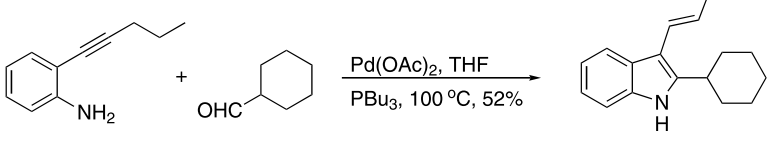



(235)

(236)

Palladium catalyzed cycloisomerization of imines with a tethered alkyne to give vinylindoles (Eq. (237)) [1079]. Titanium-mediated intermolecular reaction of alkynes with allylic or propargylic esters, followed by trapping of the resulting vinyltitanium intermediate with water, iodine, or aldehydes, gave substituted 1,4-dienes and 1,2,4-trienes (Eq. (238)) [1080]. Macrocyclization of terminal diynes using diruthenium complexes to form (*Z*)-1-en-3-ynes was reported [1081]. A ruthenium carbene complex catalyzed the coupling of two alkynes

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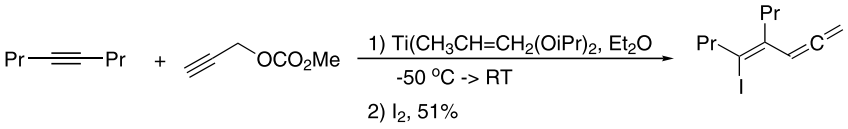



(238)

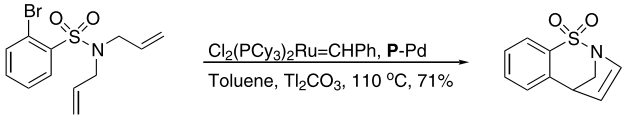
to form 1-ene-3-ynes [1082].

## 2.5. Rearrangements

### 2.5.1. Metathesis

A substantial number of reports on ring-closing metathesis reactions using Grubbs' type ruthenium catalysts were published. Addition of 1.5 equivalents of lead tetraacetate (relative to the catalyst) effectively removed colored impurities from the reaction mixture [1083]. IR-thermography was used to assess the initial rate of initiation of ring-closing metathesis [1084]. New ruthenium catalysts were developed [1085–1092]. Diastereoselective ring-closing metathesis forming dihydropyranes was reported. The selectivity was induced by a chiral group on the tether [1093]. A diastereoselective double ring closing metathesis was described [1094].

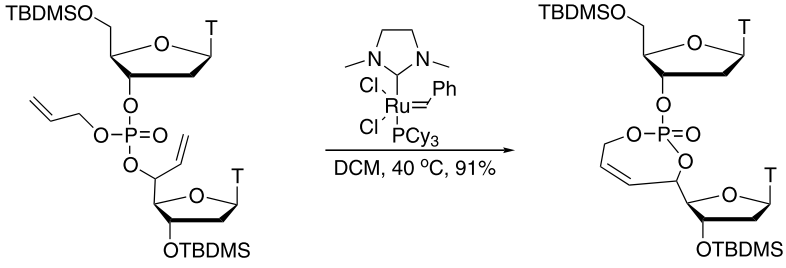
Dendrimer supported ruthenium catalysts were reported [1095,1096]. Polymer supported, recyclable catalysts were reported [1097–1100]. Ring-closing metathesis was used in solid phase synthesis [10,1101–1103]. Cyclic amino acids were prepared using polymer-supported substrates [1104]. A cascade ring closing metathesis intramolecular Heck reaction was achieved using a fluorous biphasic system or a polymer-supported palladium catalyst (Eq. (239)) [1105].




(239)

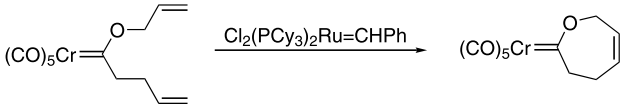
The *E*/*Z* ratio of the macrocylic lactone formed by alkene metathesis was high regardless of the initial alkene stereochemistry. A kinetic study demonstrated that the high *E*/*Z* ratio is due to secondary metathesis reactions isomerizing the product to the thermodynamic ratio [1106]. Remote substituents controlled the alkene stereochemistry of a macrolactonization [302]. Schrock's molybdenum catalyst was also employed in ring-closing metathesis reactions forming disaccharide glycals from enolether–alkene cyclization [1107], and in synthesis of glycosphingolipids [1108].

A large variety of ring systems were prepared via ring-closing metathesis. Oxa- and carbacycles fused carbohydrates [1109,1110], fused carbohydrates having two ether linkages [1111,1112], deoxyaza sugars [1113], cyclic phosphonamides [1114], a variety of phosphorous containing heterocycles [1115], chromenes [1116], 6- and 7-membered α-amino phosphonates [1117], benzazepine, benzazocine, and benzannulated 7-, 8-, 9-, and 15-membered sulfonamide heterocycles [1118], an endocyclic 7-membered sulfonamide [1119], 5-8-membered nitrogen heterocycles [496,1120–1125], medium-sized bridged ring systems [984], macrocyclic lactams [1126], macrocyclic ethers [1127], macrocyclic amines [1128], *trans*-fused oxepanes [1129], difluorinated dihydropyranes [1130], cyclic ethers [1131–1133], seven-membered ethers [1134], medium-ring ethers [1135,1136], a macrocyclic taxiod [1137], macrocyclic bisphosphine metal complexes [1138], spirocyclic ethers [1139–1142], spirocyclic lactones [1143–1145], spirocyclic compounds [1146,1147], spirocyclic dipeptides [1148], 5–8 membered cycloalkenes [802,1149–1160], 9-oxabicyclo[4.2.1]nonenes [1161], bicyclo-[3.3.1]nonenes and -[4.3.1]decenes [1162], bicyclo[4.1.0]oct-2-enes [1163], bicyclic lactams [1164], tricyclo[7.4.1.0$^{1,5}$]tetradecan-14-



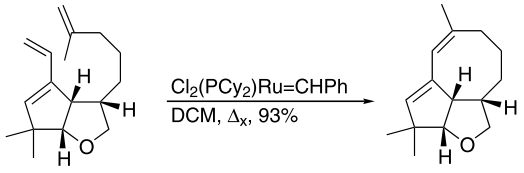

(240)

ones [1165], macrocycles [372], oxepanes [1166], azabicyclo[n.5] systems [1167], butenolide fused medium-ring ethers [1168], cyclic peptides [1169,1170], amino acid derived cyclic phophonamides [1171], conformationally restricted dinucleotides (Eq. (240)) [1172], cyclic ureas [1173], cyclic phosphanes [1174], phosphorous heterocycles [1175], azaboracycloalkenes [1176], bicyclic lactams [1177], bicyclic sulfones [1178], 6-membered lactones [1179], silyloxacycloheptenes [1180], silyldioxacycloheptenes [1181,1182], cyclic sulfamides [1183], and cyclic Fischer type carbenes (Eq. (241)) [1184], were all prepared using ring-closing metathesis methodology.




(241)

Ring-closing metathesis saw substantial use in organic total synthesis [1185–1188]. Synthetic applications toward (−)-shikimic acid [1189], (+)-FR900482 [1190], salicylihalamide [143], goniothalmin, hexadecanolide, massoia lactone [1191], (+)-boronolide [1192], (+)-malyngolide [1193], (+)-asteriscanolide (Eq. (242)) [1194], laulimalide fragment [1195], civetone (Eq. (243)) [1196], (*R*)-muscone [1197], 1-desoxyhypnophilin [1198], (−)-balanol [1199], a substructure of solanoeclepin A [1200], a fragment of laulimalide (Eq. (244)) [1201,1202], (*S*)-zearalenone [145], brevetoxin-B fragment [1203], lasiodiplodin analogue [149], conduritol [887], (+)-australine (Eq. (245)) [1204], halicholactone [648], (−)-croalbinecine [1205], (+)-madindoline A and (−)-madindoline B [1206], (+)-aspicilin (Eq. (246)) [1207,1208], hemibrevetoxin B ring system [1209], roseophilin [986], (+)-prelaureatin and (+)-laurallen [738], (−)-4a,5-dihydrostreptazolin [224], radicicol dimethyl ether [1210], (+)-febrifugine [1211], (−)-griseoviridin [1212], asteriscanolide [966], salicylihaamide (Eq. (247)) [1213], lactam analogues of epothilones [1214],

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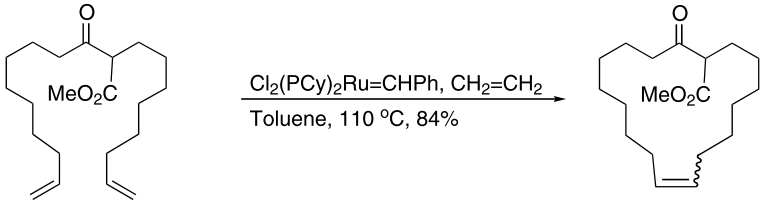


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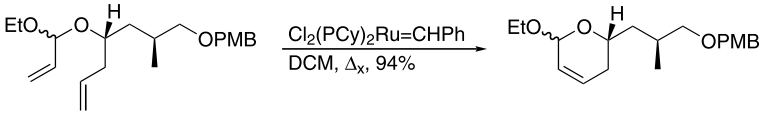


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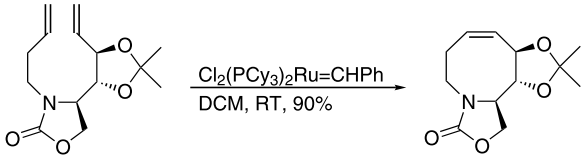


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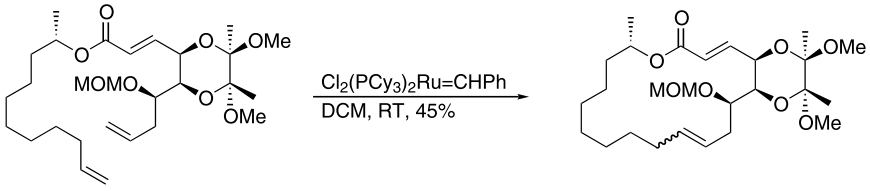


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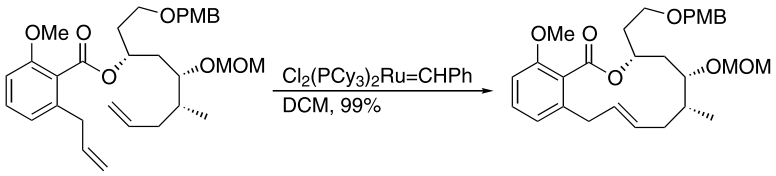
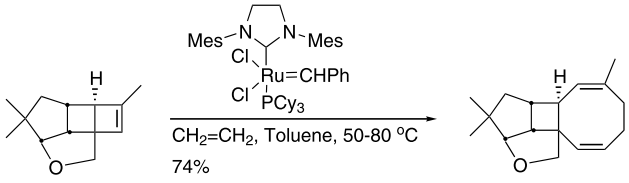



(247)

(−)-teubrevin G [1215], and ciguatoxin fragments [1216,1217] were reported.

Water-soluble ruthenium complexes for ring-opening metathesis were described [1218]. Regiochemical aspects of ring-opening cross-coupling metathesis were examined [1219]. Selenium containing ruthenium carbene complexes were used for ring-opening cross-coupling metathesis of norbornene derivatives [1220]. Ring-opening ring-closing methodology was used to prepare azasugars [1221], (+)-asteriscanolide (Eq. (248)) [1222], tetraponerines [1223], indolizidine alkaloids [1224], 2,6-dioxabicyclo[4.3.0]nonenes [1225], and α,α′-disubstituted piperidines [1226]. An asymmetric ring-opening ring-closing metathesis was described (Eq. (249)) [1227].

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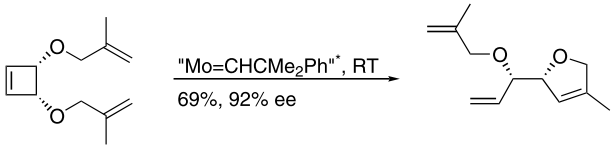
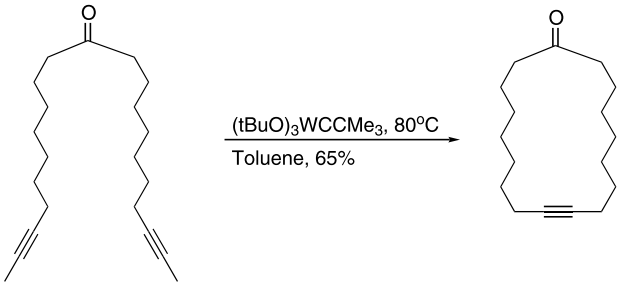



(249)

Tungsten carbyne ($(t\text{-BuO})_3W{\equiv}CCMe_3$) or molybdenum hexacarbonyl [1228] catalyzed the ring-closing alkyne–alkyne metathesis. Alkyne–alkyne metathesis was used in the synthesis of the motuporamine C [1229], of civetone (Eq. (250)) [1230], and of protaglandins [1231]. Alkyne–alkyne ring-closing metathesis was used to prepare the 26-membered ring of sophorolipid lactone [1232] and prostaglandin $E_2$-1,15-lactone [1233] using $Mo[N(t\text{-Bu})(Ar)_3]_3$ as the catalyst.

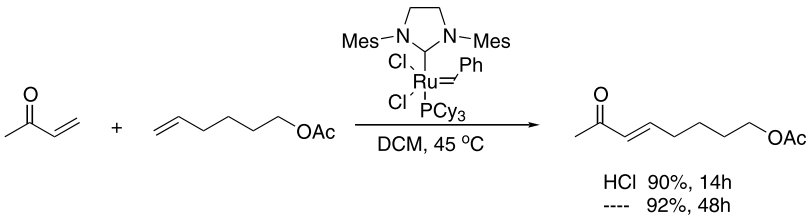
The intermolecular cross-coupling metathesis did not enjoy the same amount of attention as ring-closing metathesis [1030,1234]. Cross-metathesis of vinylsilanes




(250)

with styrene using a ruthenium catalyst was developed [1235]. Cross-coupling metathesis of polymer-bound allyl alcohol was described [1236]. Cross-coupling metathesis of α,β-unsaturated carbonyl compounds with alkenes using an in situ generated ruthenium catalyst was described. A dramatic reduction in reaction time was observed in ethereal HCl (Eq. (251)) [1237]. Intermolecular alkyne–alkyne cross metathesis using molybdenum hexacarbonyl in the presence of 4-chlorophenol was reported [1238]. Allene [1239] and alkene [1240,1241] cross-metathesis was reported using Grubb's catalyst. Grubbs' catalyst was used in cross-coupling metathesis of terminal alkynes with ethene under moderate pressure to give 1,3-dienes [1242], of terminal alkynes with allylic ethers forming 1,3-dienes [1243], and of ethene with alkynes having an N or O functionality in the propargylic position [1244]. Dimerizations, with loss of ethene, of cyclodextrin bound allyl ethers [1245], dimerization of carbohydrate-bound vinyl and allylbenzenes [1246], vinylcyclopropanes [1247], and preparation of calcitrol dimers [1248] were described.

A Schrock type complex was used in cross metathesis of allyl ethers with styrenes [1108]. An interesting combination of intramolecular and intermolecular metathesis in the synthesis of (−)-cylindrocyclophanes A is seen in Eq. (252) [1249]. Intermolecular cross-metathesis

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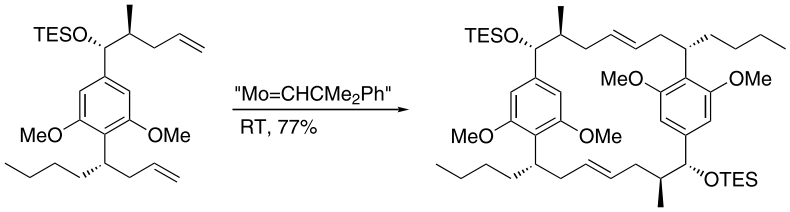


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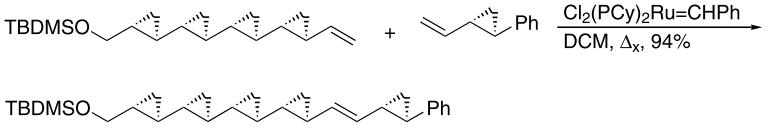
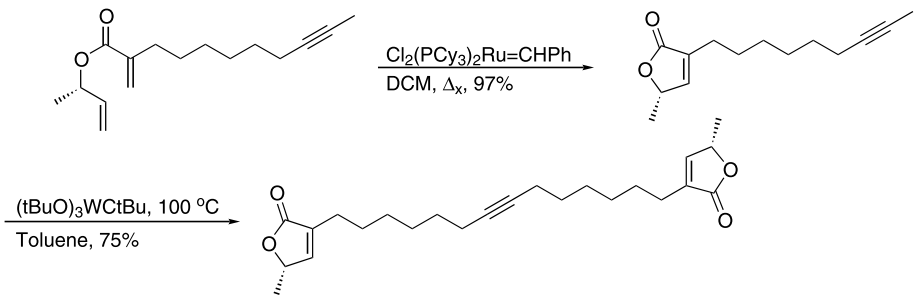



(253)

was used in the synthesis of FR-900848 (Eq. (253)) [1250].

A ruthenium dihydroimidazole carbene-catalyzed intermolecular enyne cross-coupling metathesis was reported [1251]. Intramolecular ene–yne metathesis [1252] was used in synthesis (Eq. (254)) [1253] of eight-membered rings (Eq. (255)) [1254], of cyclic 1,3-dienylboronic esters (Eq. (256)) [1255], and of pipicolic acid derivatives [1256]. Platinum also catalyzed intramolecular enyne metathesis (Eq. (257)) [661] and this was

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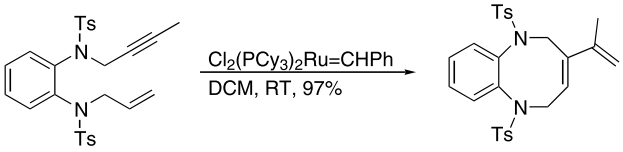


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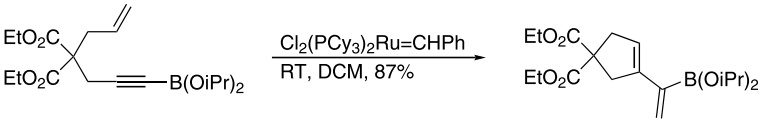
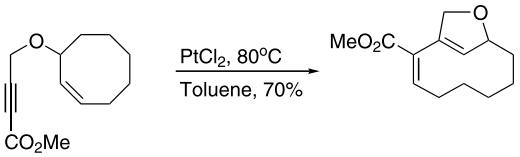



(256)

employed in the synthesis of roseophilin (Eq. (258)) [1257].

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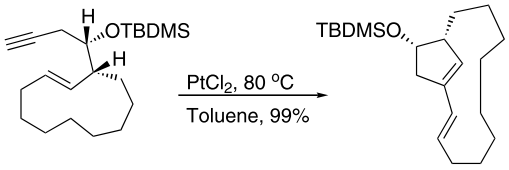
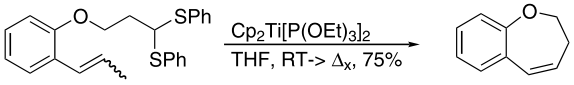



(258)

An interesting titanocene promoted metathesis of unsaturated thioacetals to afford nitrogen containing heterocycles [1258], and to give carbocyclic compounds and oxygen and sulfur heterocycles was developed (Eq. (259)) [1259]. Intermolecular reaction of thioacetals with alkane nitriles in the presence of a titanocene promoter gave unsymmetrically substituted ketones (Eq. (260)) [1260]. An in situ formed vinyltitanocene carbene was reacted with esters to give dienol ethers (Eq. (261)) [1261]. Titanocene-promoted metathesis reaction of ω,ω-bis(phenylthio)alkyl esters produced ω-hydroxyketones [1262]. Polymer supported esters were converted to enol ethers by a related intermolecular reaction [1263].

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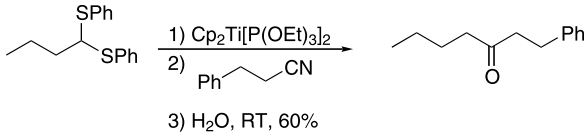
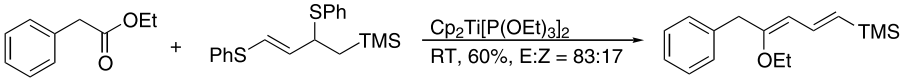



(260)

### 2.5.2. Alkene isomerization (including cycloisomerization)

Organoyttrium-catalyzed sequential cyclization/silylation reactions afforded five- and six-membered rings with a methyl trimethylsilylmethyl groups (Eq. (262)) [1264]. A titanium catalyzed regio- and stereoselective cycloisomerization of 1,6-dienes to *exo*-methylenecyclopentanes was reported [1265]. An arene ruthenium complex also catalyzed this reaction [1085]. Using a sterically more encumbered catalyst gave predominantly methylenecyclohexanes [1266]. A palladium-catalyzed enantioselective diene cyclization/silylation to form cyclopentanes, having a *cis* methyl–trimethylsilylmethyl configuration, using triethylsilan was reported [1267]. Low e.e.s were obtained upon reductive cycloisomerization of 1,6-dienes to cyclopentanes catalyzed by palladium-bisoxazoline complexes in the presence of silane [1268,1269]. An asymmetric 1,6-diene cycloisomerization in the presence of 1-*t*-butyl-3,3-dimethyl-1,1-diphenyldisiloxane and $NaB(3,5\text{-}C_6H_3(CF_3)_2)_4$ forming silylated cyclopentanes was described [1270]. Dicobalt hexacarbonyl mediated cyclopentene formation of 1,6-enynes. In addition to the cyclopentene, the corresponding Pauson–Khand and alkene isomerization products were observed (Eq. (263)) [1271]. The carbapenam skeleton was prepared via a ruthenium-catalyzed 1,6-enyne cycloisomerization (Eq. (264)) [1272].

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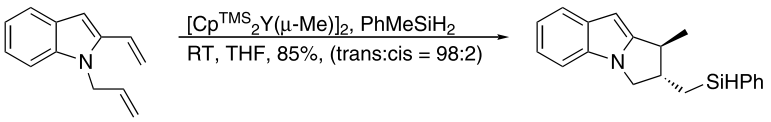
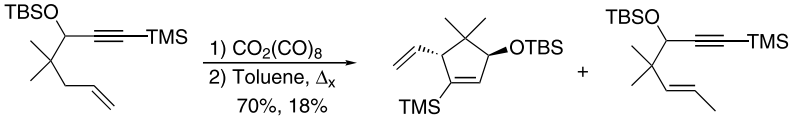
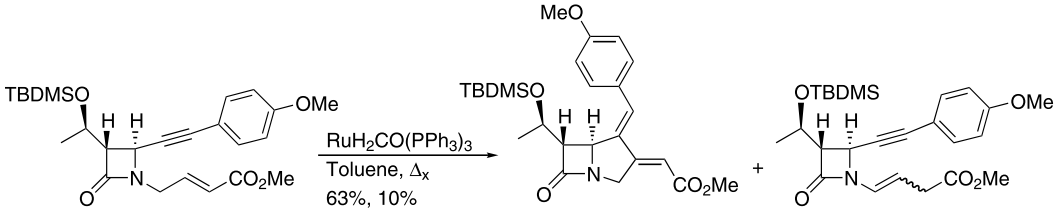



(262)

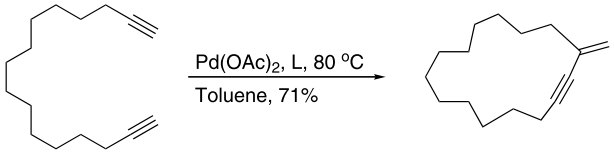



(263)




(264)

A ruthenium-catalyzed deconjugation of α,β-unsaturated esters to a remote functionality was described [1273]. Grubb's metathesis catalyst [1274] and Wilkinson's catalyst [1275] were used to isomerize *O*-allyl glycosides to 1-propen-1-yl glycosides. Palladium complexes with phosphinooxazoline ligands catalyzed the coupling of alkynes to form enynes (Eq. (265)) [1276]. Ruthenium and platinum catalyzed cycloisomerizations of ω-aryl-1-alkynes (Eq. (266)) [1277].

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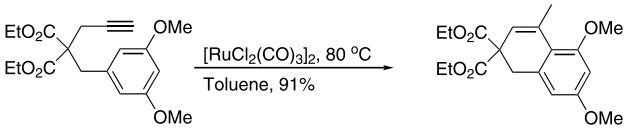
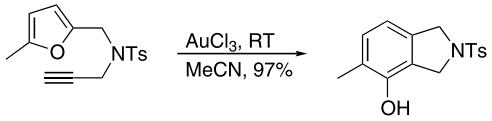



(266)

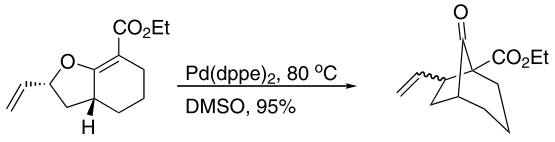
Gold-catalyzed cycloisomerizations of 1,2-diene-3-ones, α,β-unsaturated ketones, 1-yne-4-ones to form furans were reported [1278]. A related cycloisomerization of 1-yne-4-ones having a tethered alkyne gave substituted arenes (Eq. (267)) [1279]. Group VI metal carbonyls promoted cycloisomerization of homopropargylic thiols to dihydrothiophens and annulated thiophens [1280] and 1-yne-5-ols to tetrahydropyrans [1281].




(267)

### 2.5.3. Rearrangements of allylic and propargylic compounds

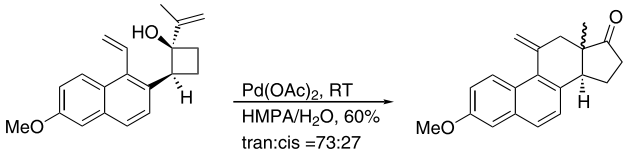
Palladium catalyzed rearrangements of 2-alkylidene-5-vinyltetrahydrofuranes to afford bicyclo[3.2.1]octane-8-ones (Eq. (268)) [1282]. Palladium-catalyzed allylic transposition was used in the synthesis of carbacyclin [368].




(268)

### 2.5.4. Skeletal and miscellaneous rearrangements

Iridium catalyzed the rearrangement of allyl homoallyl ethers to γ,δ-unsaturated aldehydes [1283]. Zirconocene promotes the retro-Brook rearrangement of 3-trimethylsilyloxy-2-aza-1,3-dienes [1284,1285]. Palladium and nickel complexes catalyzed thio-Claisen rearrangements of chiral bicyclic thiolactams [1286], and palladium catalyzed oxy-Cope rearrangements [1287]. Palladium diacetate catalyzed ring expansion of 1-vinylcyclobutan-1-ols to α-methylene cyclopentanones [1288] was used to prepare 4-deoxyverrucarol [1289] and (+)-equilenin (Eq. (269)) [1290].




(269)

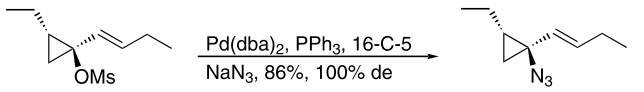
## 3. Functional group preparation

### 3.1. Halides, amides, nitriles, azides, imines, ureas, amidines

Vinyl iodides and bromides were prepared by the chromium(II) mediated Takai olefination [38,158,725,1291]. Hydroalumination followed by iodonolysis was used to prepare a vinyl iodide [1292]. Arylsulfonyl chlorides were transformed into aryl halides using $ZnI_2$, LiCl, Ti(O-*i*Pr)$_4$ and a palladium catalyst [1293].

Palladium catalyzed the cyanation of aryl, heteroaryl, and vinyl bromides with zinc dicyanide under microwave irradiation [1294], of aryl triflates with zinc dicyanide [218], of 2-iodoindole with KCN [62], of aryl nonaflates with zinc dicyanide [1295], and of aryl

chlorides with zinc dicyanide [38]. Nickel also catalyzed cyanation of aryl triflates [1296]. A regio- and enantioselective synthesis of D-α-amino nitriles, amides, and acids catalyzed by titanium was developed (Eq. (270)) [1297]. Palladium catalyzed the ring cleavage of cyclobutanone oximes to give nitriles (Eq. (271)) [1298]. Nickel catalyzed an asymmetric hydrocyanation of alkenes [1299]. Palladium catalyzed the cyanation of α,β-unsaturated ketones using trimethysilylcyanide and acidic workup [1300]. A three-component palladium-catalyzed cyano allylation of activated alkenes using an alkene, an allylic chloride, and trimethysilyl cyanide was reported (Eq. (272)) [1301]. Alkene hydrozirconation followed by reaction of *t*-BuNC afforded an alkyl nitrile was used in the synthesis of (*S*)-zearalenone [145]. Propargylic carbonates were transformed to cyanoallenes using trimethylsilyl cyanide in the presence of a palladium catalyst. Dicyanated products can be obtained using an excess of trimethylsilyl cyanide (Eq. (273)) [1302].

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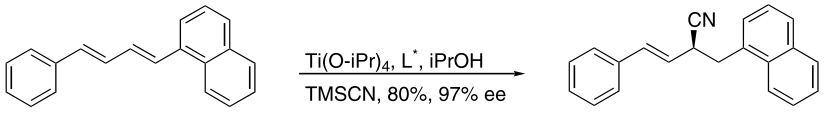
Reaction of allyl esters in an alcohol in the presence of a palladium catalyst, diphenylphosphoryl azide, and sodium azide directly gave urethanes [1305]. Amidation of allylic and benzylic hydrocarbons with primary amides or tosylates was catalyzed by ruthenium complexes (Eq. (276)) [1306]. The mechanism of the palladium-catalyzed formation of α-methylenelactams by intramolecular cyclizations of alkynyl amides was examined [1307]. A palladium-catalyzed allylic azidation was used in the synthesis of lactam analogues of epothilones (Eq. (277)) [1214]. Tungsten, chromium, and molybdenum hexacarbonyl catalyzed the oxidative carbonylation of amines, in the presence of iodine, to form ureas [1308].

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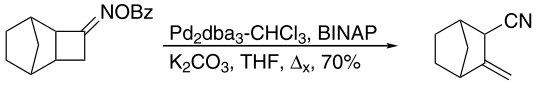


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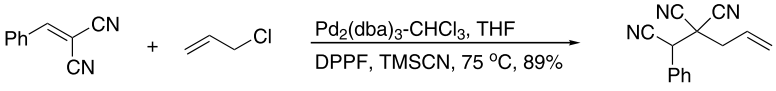


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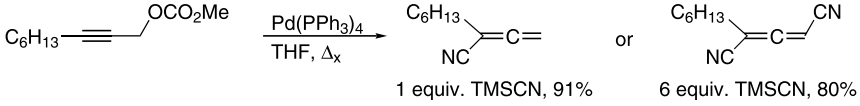
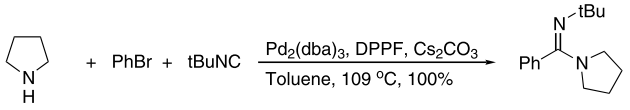



(273)

Reaction of isocyanides with an arylhalide and an amine produced amidines (Eq. (274)) [1303]. A palladium-catalyzed azidation of 1-vinyl-1-mesylates was used in the synthesis of coronamic acid (Eq. (275)) [1304].

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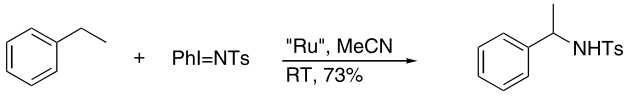


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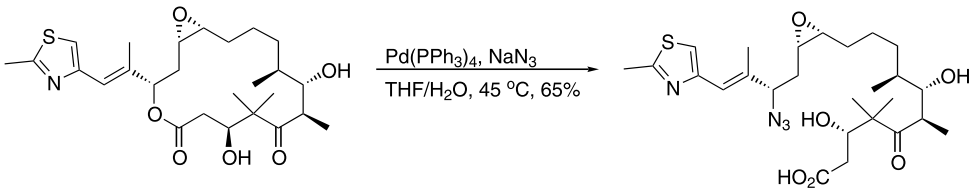



(277)

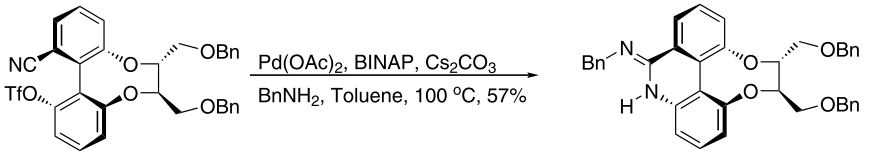
### 3.2. Amines, alcohols

A large variety of aromatic chlorides, bromides, and triflates were aminated using palladium-based catalyst systems [167,674,1309–1321]. Amination of aryl bromides using low catalyst loading (0.05% $Pd(OAc)_2$) was achieved using BINAP as the ligand [1322]. Sodium alkoxides containing β-hydrogens were used as bases in amination reactions [1323].

Amination of an aryltriflate having an adjacent carboxylic acid functionality surprisingly gave a cyclic amidine (Eq. (278)) [218]. Palladium catalyzed the amidation of aryl halides and triflates using amides as the nucleophile [1324]. Amination of aryl halides with vinylogous amides was reported (Eq. (279)) [518]. Unsymmetrical triarylamines were prepared by a one-pot double amination using two aryl halides with substantially different reactivity. Triarylamines containing a heterocyclic group required a one-pot, two-step procedure [1325].

Palladium catalyzed the *N*-arylation of indoles [1326], of 3-iodoindazole [325], of benzophenone imine [1327], of phenoxazines [1328], of pyrrole, indole, and carbazole [1329], of an amino group of nucleotides and other arylamines with a nucleotide bromides [1330], of an aminonucleoside with a bromonucleoside [1331], of amines with 2-bromo-deoxyinosine [1332] and of an amino sugar with 6-chloro-9-SEM-purine toward spicamycin amino nucleoside (Eq. (280)) [1333]. A palladium- and nickel-catalyzed *N*-arylation of sulfoximines was reported [1334,1335].

Palladium catalyzed amination of 2-trifloxytropone [1336], 4-chloro-3(2*H*)-pyridazinones [1337], dibromothiophens [1338], 2-bromopyridine [1339], of 2-amino-6-chloropurine with cyclopropyl amine [1149], and of electron-deficient halothiophens [1340]. Palladium-catalyzed amination was used in synthesis of isoaaptamine [1341], *N*-aryl-aza-crown ethers [1342], (−)-fumiquinazoline A, B, and I (Eq. (281)) [1343], and indoles (Eq. (282)) [1344]. Intramolecular amination of *N*-aryl-*N*-(2-bromobenzyl)hydrazines produced 2-aryl-2*H*-indazoles

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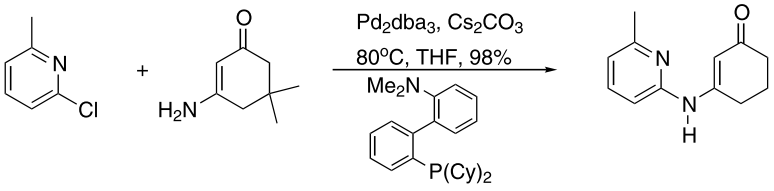


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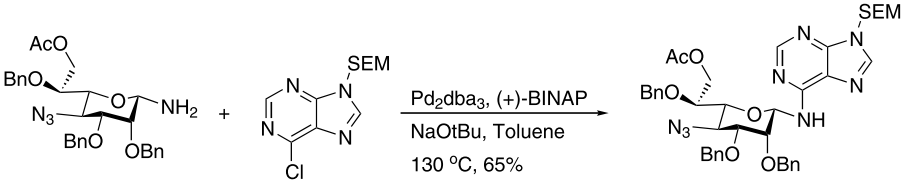
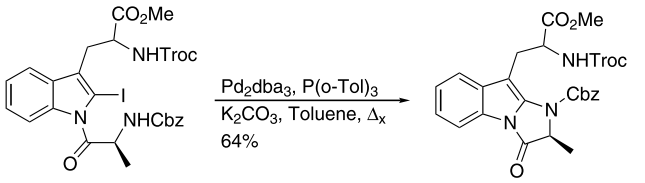



(280)

(Eq. (283)) [1345,1346]. 1-Aminoindoles were prepared by intramolecular cyclization of 2-chloroarylacetaldehyde *N*,*N*-dimethylhydrazones (Eq. (284)) [1347]. Phenazines were prepared by an inter- and intramolecular amination sequence [1348]. A 1-amino-4*H*-quinoline was also prepared using the same conditions. An interesting synthesis of the morphine skeleton was reported (Eq. (285)) [1349].

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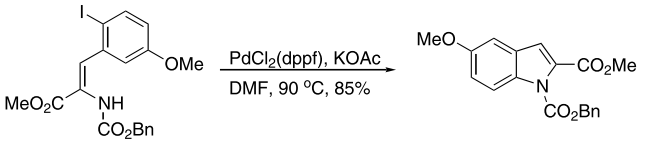


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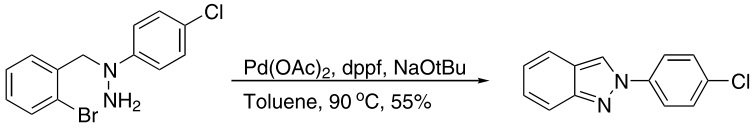


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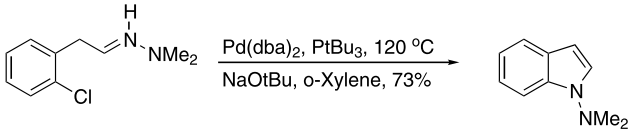


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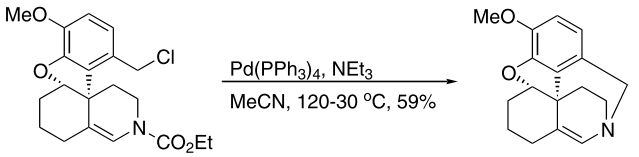


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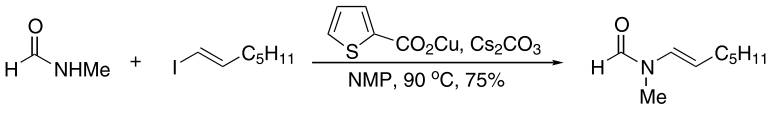
Transition metals other than palladium also catalyzed amination reactions. Couplings of aryl amines with aryl iodides using copper–bronze or copper were reported [88,1350]. Copper catalyzed the coupling of sodium imidazolates with aryl iodides [1351], of amides with vinyliodides (Eq. (286)) [1352], the arylation of *N*-aryl-*N*-phenylaminophthalimides with triphenylbismuth (Eq. (287)) [1353], and amination using aryl trimethylsiloxane [1354]. Copper complexes promoted amination of aryl boronic acids and aryl stannanes [1355,1356], of aryl lead and bismuth compounds [1357,1358], and iodonium salts with aromatic and aliphatic amines, azoles, and amides [1359]. A copper-catalyzed synthesis of cyclic sulfonamides via intramolecular aziridination of iminoiodanes was developed (Eq. (288)) [1360].

Nickel on charcoal catalyzed the amination of aromatic chlorides at 130 °C in the presence of lithium *t*-butoxide and bisdiphenylphosphinoferrocene [1361]. Nickel 2,2′-bipyridine complex-catalyzed couplings of arylchlorides with secondary amines were described [1362,1363]. Copper catalyzed the aminohalogenation of electron-deficient alkenes using 2-nitrobenzenesulfonamide *N*,*N*-dichloride as the nitrogen and halogen source (Eq.

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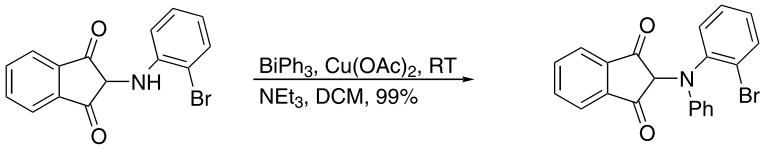


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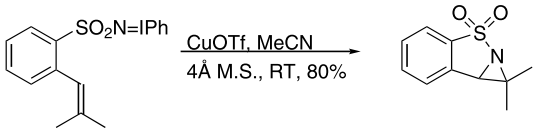


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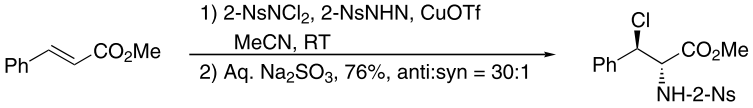
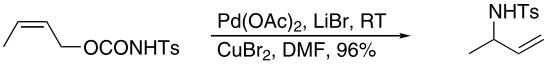



(289)

(289)) [1364].

Palladium catalyzed a variety of aminations of allylic substrates via $\eta^3$-allyl intermediates [973,1221,1365,1366]. A polymer-bound allylic benzoate was used as the electrophile [1367]. *N*-Allylbenzotriazoles were used as substrates in allylic amination reactions of amines and sulfonamides [1368]. 2-Amino-6-chloropurine [1149], 2,6-dichloropurine [1367], cytosine [1369], phthalimide [1370], and azetidines were used as *N*-nucleophiles. Palladium-catalyzed amination of a vinyl carbonate with 2-amino-6-chloropurine was used in the synthesis of famciclovir (Eq. (290)) [1371].

pared by an intramolecular amination (Eq. (292)) [1376].

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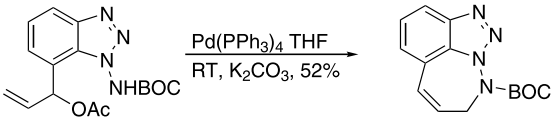
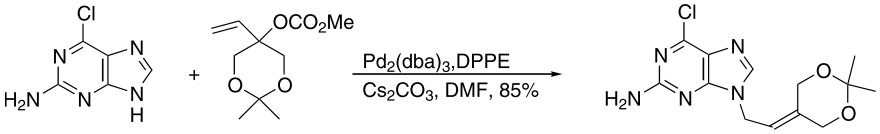



(292)

A number of palladium-catalyzed asymmetric allylic aminations were published using optically active ligands [1224,1377,1378]. Optically active allylic carbonates were aminated with high retention of enantiomeric

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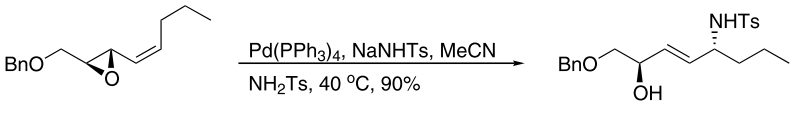
A regio-and stereoselective formation of *N*-tosyl allylic amines starting from allylic alcohols and tosyl isocyanate was reported (Eq. (291)) [1372]. Palladium catalyzed the allylic amination of anilines using allylic alcohols in the presence of titanium tetraisopropoxide [1373]. 1-Tosyloxy-1-vinylcyclopropane was aminated using anilides as the nucleophile [1374]. Intramolecular reactions of *N*-allylbenzotriazoles furnished 1,2-disubstituted pyrroles [1375]. 1,2-Benzodiazepines were pre-

purity [776]. Palladium-catalyzed dynamic kinetic resolution of butadiene monoepoxide was used to prepare the amino acid vigabatrin and the aminoalcohol ethambutol [419]. Aminations of an optically active vinyl epoxide were used in the synthesis of (+)-pseudoconhydrine and epi-pseudoconhydrine (Eq. (293)) [1379].

Palladium-catalyzed amination of optically active allylic cyclopentene carbonate was used in carbovir and aristeromycin synthesis [1380]. A number of carbanucleosides were prepared using enantioselective allylic

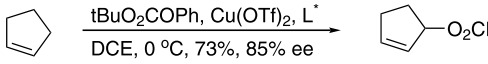



(293)

amination of *cis*-3,5-dibenzyloxycyclopent-2-ene [1381]. Asymmetric allylic amination was used in the synthesis of tetraponerines [1223]. Moderate desymmetrization of cyclic allylic biscarbamates was reported using palladium and a chiral ligand [1312].

Palladium catalyzed the amination of alkoxyallenes. The regiochemistry of the addition depended on the alkoxy group (Eq. (294), (295)) [1123]. Aminodiphenylmethane was used as an ammonia equivalent for the dimethyltitanocene-catalyzed hydroamination of alkynes (Eq. (296)) [1382]. Cationic rhodium complexes catalyzed the intramolecular hydroamination of alkynes [1383]. A number of additional Group 7–12 served as catalysts for this reaction [1384]. A palladium-catalyzed hydroamination of vinylarenes using aminobenzenes was developed (Eq. (297)) [1385]. Transition metal catalyzed intramolecular hydroamination of vinyl ethers was described [1386]. Amination of alkenes with nitro aromatic compounds using an iron catalyst was reported [1387].

Vanadium catalyzed the oxidation of aryl allylic selenides to give allylic alcohols via a [2,3]sigmatropic rearrangement (Eq. (298)) [1388]. An asymmetric allylic oxidation of cycloalkenes using a chiral copper complex to give allylic alcohols was developed (Eq. (299)) [1389]. A related copper-catalyzed oxidation furnishing optically enriched esters was also reported [1390]. Nickel catalyzed the regio-and stereoselective coupling of trimethylboron, isoprene, and an aldehyde to give functionalized 3-hexen-1-ols (Eq. (300)) [1391]. Zirconocene-catalyzed enantioselective methylalumination of terminal alkenes was greatly accelerated in the presence of water forming a primary alcohol (Eq. (301)) [1392].

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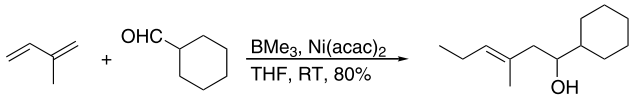


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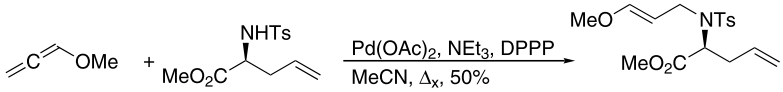


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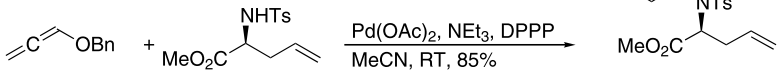


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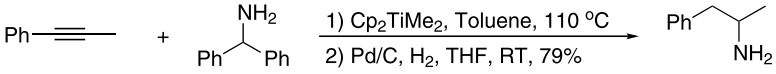


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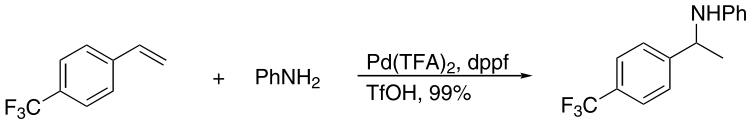


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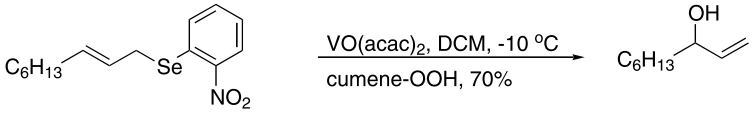


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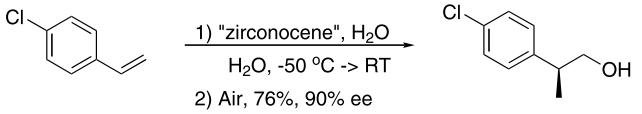
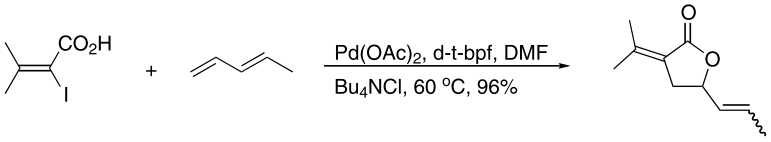



(301)

### 3.3. Ethers, esters, lactones, acids

α-Alkylidene-γ-butyrolactones were formed from palladium catalyzed reaction of 1,3-dienes and α-iodo or α-bromoacrylic acids (Eq. (302)) [1393]. A few examples of formation of the corresponding six-membered lactone from 1,4-dienes were also described. A rhodium catalyzed formation of lactones via carbon–carbon bond cleavage of cyclobutanones having an 2-hydroxybenzene substituent was reported (Eq. (303)) [1394].

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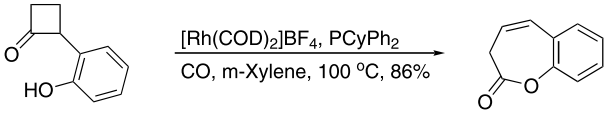
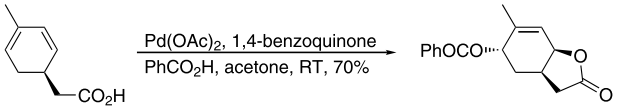


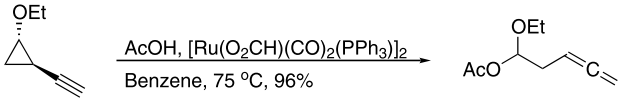

(303)

A palladium-catalyzed *cis*-oxylactonization was utilized in the synthesis of paeonilactione A (Eq. (304)) [1395]. Ketopyranose subunits of polyketides were obtained using a similar sequence [1396]. A zwitterionic rhodium complex catalyzed the chemo- and regioselective formation of unsaturated lactones by carbonylation of α-ketoalkynes (Eq. (305)) [1397].



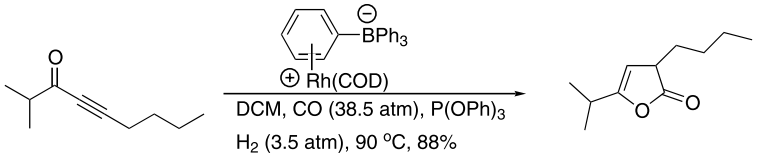

(304)

Ruthenium catalyzed the addition of carboxylic acids to 1-ethoxy-2-ethenylcyclopropane to afford allenes and enol esters (Eq. (306)) [1398]. A related addition of benzoic acid to 2-propyn-1-ols was also described [1399]. Palladium catalyzed the addition of acetic acid to cyclopentadiene monoepoxide to give *cis*-4-acetoxy-2-cyclopenten-1-ol [1400]. A copper-catalyzed asymmetric allylic oxidation of cycloalkenes forming allylic esters was reported [1401]. Palladium-catalyzed acetoxylation of cyclic allylic phosphonates, using isopentyl nitrite and oxygen as the oxidants, was described [1402]. Optically active γ-fluoroalkylated allylic esters were prepared by a palladium catalyzed nucleophilic substitution of fluorinated allylic mesylates [1403]. Baylis–Hillman type adducts were deracemized using palladium catalysis (Eq. (307)) [1404]. Palladium catalyzed the regio- and stereoselective reductive ring-opening of diene–ester derived monoepoxides, using the dimethylamine–borane complex to give δ-hydroxy-α,β-unsaturated esters [1405].




(306)

Vanadyl acetylacetonate-catalyzed epoxidations of allylic alcohols using peroxides continued to be a valuable tool in organic synthesis [568,854,1406]. For example, aphidicolin [130], (+)-gelsemine [1407], ginkolide [1408], carbanucleosides [1381], and (−)-bakkenolides [1409] were prepared using this methodology. Metallocene-catalyzed diastereoselective epoxidation of allylic alcohols was described [1410]. Vanadium com-

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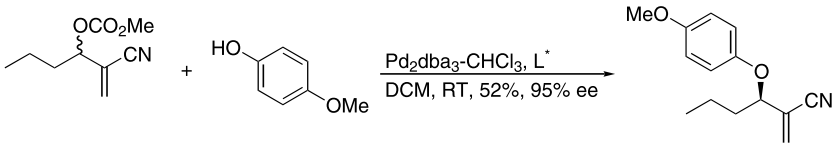
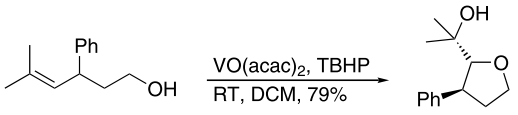



(307)

plexes of *N*-hydroxy-[2.2]paracyclophane-4-carboxylic amides [1411], 1,1′-binaphthyl substituted hydroxamic acids [1412], and α-amino acid based hydroxamic acids [1413] catalyzed asymmetric epoxidation of allylic alcohols. Vanadium-catalysts were developed for stereoselective synthesis of functionalized disubstituted tetrahydrofurans (Eq. (308)) [1414]. Epoxidation of alkenes in the presence of a tethered alcohol resulted in the formation of tetrahydrofurans (Eq. (309)) [1415].

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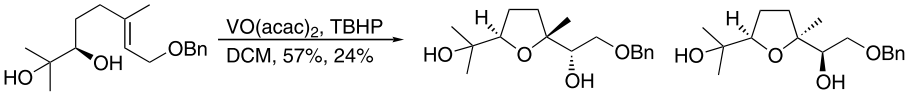
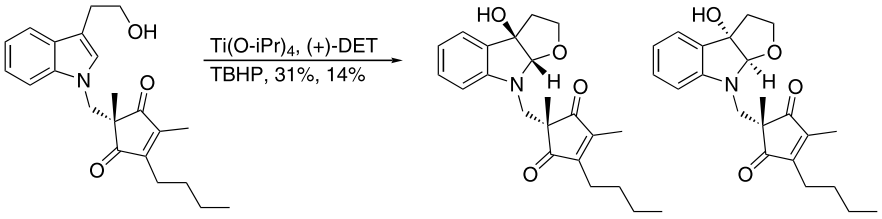



(309)

Enantioselective epoxidations of allylic alcohols using titanium tetraisopropoxide together with *t*-butyl hydrogen peroxide and an optically active tartrate ester continue to be used in organic synthesis. For example, this reaction was used in synthetic approaches to epothilones A and B [1416,1417], 12,13-desoxyepothilone B [1418], sphingofungin [1419], (−)-dysiherbaine [1420], (−)-androst-4-ene-3,16-dione [935], (+)-australine [1204], deoxyaza sugars [1113], radicicol dimethyl ether [1210], (+)-eurylene and (+)-15-deacetyleurylen [1421]. Interesting examples include the synthesis of (+)-madindoline A and (−)-madindoline B (Eq. (310)) [1206]. A recyclable polymeric titanium(IV)glycolate epoxidation catalyst was developed [1422]. Ruthenium complexes having a chiral tetradentate ligand catalyzed asymmetric epoxidation of alkenes using hydrogen peroxide [1423].

A palladium-catalyzed asymmetric allylic addition of an alkoxide was used in the synthesis of (−)-malyngolide [1424], (−)-galanthamine (Eq. (311)) [1425]. Bisallyloxyarenes were prepared by palladium-catalyzed allylic alkoxylation using dihydroxybenzenes as the nucleophile [1426]. Palladium-catalyzed allylic substitution using phenoxide as the nucleophile was described [1427]. A rhodium-catalyzed asymmetric ring opening of oxabicyclic alkanes with phenols was reported [1428]. Rhodium catalyzed the diastereo- and regioselective ring opening of vinyl epoxide with alcohols and amines forming *trans*-1,2-amino alcohols or alkoxy alcohols (Eq. (312)) [1429].

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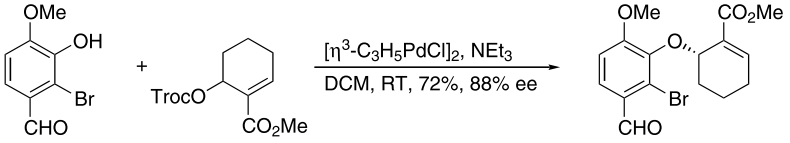


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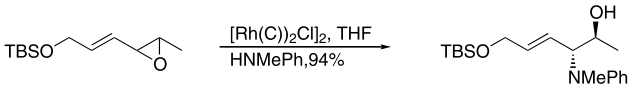
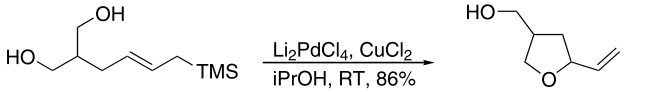



(312)

Palladium catalyzed the carbon–oxygen coupling forming diaryl and aryl *t*-butyl ethers from aryl halides [1430]. A palladium-catalyzed intramolecular reaction forming oxygen heterocycles was developed [1431].

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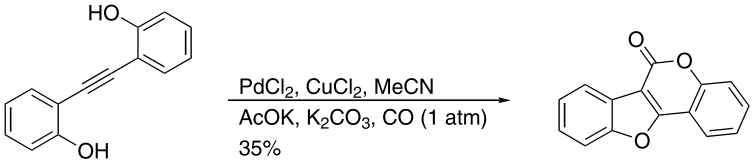


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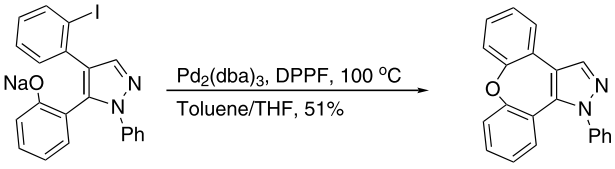


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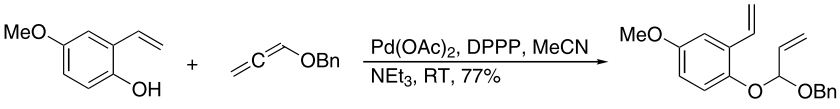
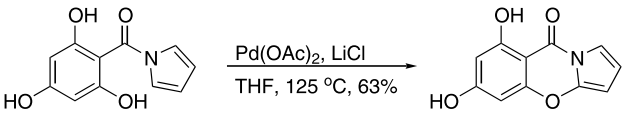



(317)

Copper catalyzed the coupling of aryl halides with aryl thiols to give biarylthioethers [1432]. Copper-mediated aryl alkyl ether formation was used in the synthesis of (+)-diepoxin γ [173]. Copper catalyzed the intramolecular etherification of 2-(2-chlorophenyl)-1-ethanols [1433]. Palladium mediated an intramolecular coupling of 2-hydroxybenzoylpyrroles (Eq. (313)) [1434]. An intramolecular annulation of allylsilyl alcohols forming 2-vinyltetrahydrofuranes was described (Eq. (314)) [1435]. Palladium-catalyzed "hydroxy–palladation–carbonylation–lactonization" sequence was used in natural product synthesis (Eq. (315)) [1436,1437]. An intramolecular palladium-catalyzed diaryl ether formation was used to prepare dibenzo[4,5-*d*]pyrazoles (Eq. (316)) [1438]. Palladium-catalyzed addition of phenols to benzyloxyallene furnished vinylacetals (Eq. (317)) [1116].

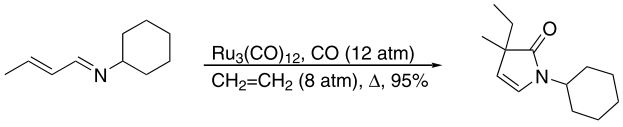



(313)

### 3.4. Heterocycles

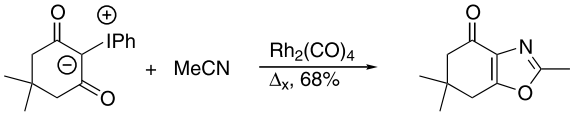
A variety of complex polycyclic molecules was obtained by reactions usually initiated by an oxidative addition to an aryl or a vinyl halide [1439–1442]. The product isolated depended on the sequence of events that follow, for example, alkene and alkyne insertions, carbonylations, and terminations. These cascade-type reactions are unfortunately not readily categorized within this review and are therefore only briefly discussed. Insertion of allenes into acyl complexes obtained from oxidative addition of palladium to aryl halides in the presence of carbon monoxide, followed by an intramolecular nucleophilic termination was used to prepare a number of different heterocyclic compounds [1443]. Palladium catalyzed the carbonylation of 2-iodoanilines in the presence of isocyanates, carbodiimides, ketenimines to give 2,4-(1*H*,3*H*)-quinazolinediones, 4(3*H*)-quinazolinones, and 2-alkyl-4(3*H*)-quinazolinones, respectively [1444]. A ruthenium-catalyzed synthesis of dihydropyrrol-2-one derivatives using α,β-unsaturated imines, ethene, and carbon monoxide was reported (Eq. (318)) [1445]. Coumarins were pre-

pared by palladium-catalyzed carbonylative reaction of internal alkynes with 2-iodophenols [1446]. Carbonylation of 2-iodophenol or 2-iodoaniline in the presence of an allene and a palladium catalyst gave chroman-4-ones and quinol-4-ones, respectively [1447]. A palladium-catalyzed carbonylative annulation of iodophenol acetates with alkynes to form flavones was reported [1448]. A palladium–thiourea complex catalyzed the carbonylative annulation of 2-hydroxyaryl acetylenes to give 2-substituted benzofuran-3-carboxylic esters [1449].



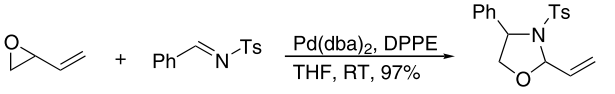

(318)

Palladium catalyzed the cyclization of carbamoyl chlorides onto tethered alkenes or alkynes followed by reaction with tin or boron reagents (Eq. (319)) [1450]. Related reactions of alkyne tethered chloroformates produced α-methylene-γ-butyrolactones [1451]. Palladium catalyzed the annulation of 2-iodobenzamides with alkynes forming 3-alkylideneisoindolin-1-ones [1452]. Palladium catalyzed the reaction of aryl halides or a nitrile, respectively (Eq. (320)) [1454]. Palladium catalyzed the reaction of iodophenol with terminal alkynes, forming benzofurans [291].




(320)

Palladium catalyzed a room temperature ring-opening-cyclization of 2-vinyl aziridines with isocyanates, carbodiimides, and isothiocyanates to give imidazolidinones, imidazolidinimines, and imidazolidinthiones, respectively [1455]. Cycloaddition of vinyl epoxides with imines in the presence of a palladium catalyst furnished 1,3-oxazolidines (Eq. (321)) [1456]. Palladium catalyzed the reaction between allylic acetates, nitriles, and trimethylsilyl azide to give tetrazoles (Eq. (322)) [1457]. Palladium catalyzed the formation of oxazolidinones from tosyl isocyanate and vinyl epoxides [1458]. Amines reacted with *Z*-2-buten-1,4-diol dimethylcarbonate in the presence of a palladium catalyst to give oxazolidones (Eq. (323)) [1459].

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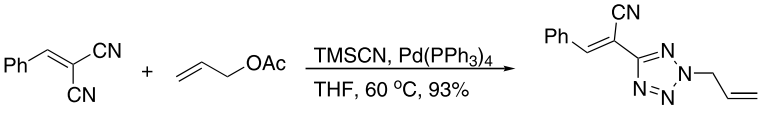


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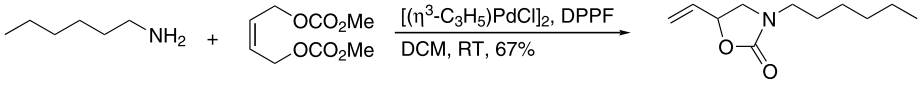
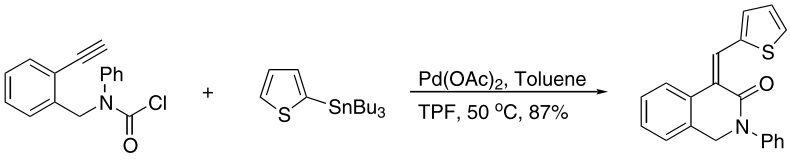



(323)

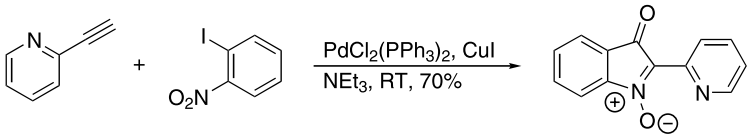
with 2-(1-alkynyl)benzoic acids to give isobenzofuran-1(3*H*)-ones and furan-2(5*H*)-ones [1453]. Furans and oxazoles were prepared by a rhodium-catalyzed decomposition of iodonium ylids in the presence of an alkyne

A palladium-catalyzed annulation of 2-nitrophenylalkynes leading to 2-aryl-3*H*-indole-3-one *N*-oxides was reported (Eq. (324)) [1460]. Reaction of formamidine or acetamidine substituted iodouracils with alkynes




(319)

in the presence of a palladium catalyst furnished pyrido[2,3-*d*]pyrimidines (Eq. (325)) [1461]. Isoquinolines and/or isoindoles were prepared by the reaction of aryl iodides with 2-alkynylbenzonitriles (Eq. (326)) [1462]. Palladium catalyzed the reaction of 2-iodobenzoylchloride with ketone imines to form isoindoline-1-ones (Eq. (327)) [1463].

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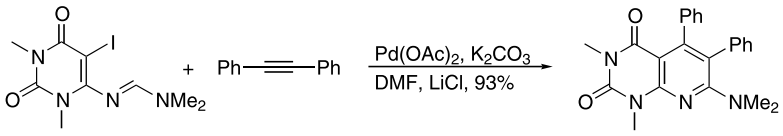


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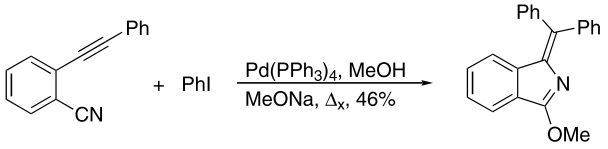


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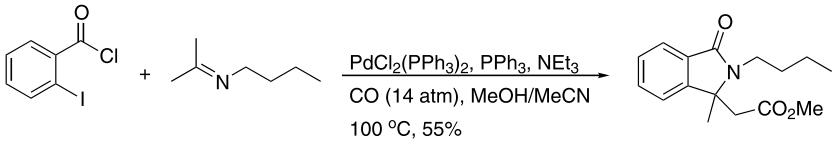
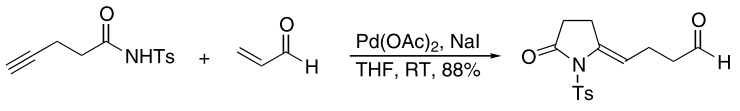



(327)

Sequential palladium-catalyzed Sonogashira coupling followed by intramolecular amino-palladation of 2-iodoanilines gave indoles [1464,1465]. A palladium-catalyzed tandem aminopalladation of alkynes followed by a reductive Heck reaction was reported to give oxazolidinones, imidazoles, and lactams (Eq. (328)) [1466]. Intramolecular aminopalladation of *ortho*-alkynylsubstituted anilides in the presence of α-haloacetates or α-bromoketones afforded 2-alkoxycarbonyl- or 2-acylindoles, respectively [1467], and in the presence of benzyl bromide or ethyliodoacetate gave 2-substituted 3-alkylindoles (Eq. (329)) [1468]. Internal alkynes were reacted with 2-iodoarylamines to give 2,3-disubstituted indoles [1469]. α-Methylenepyrrolidinones were prepared by palladium-catalyzed intramolecular aminopalladation [689]. Aminopalladation was used in the synthesis of 1-deoxymannojirimycin (Eq. (330)) [1470] and, when followed by carbonylation–lactonization of the intermediately formed σ-palladium complex, homologues of 1-deoxynojirimycin and 1-deoxy-L-iodonojirimycin [1471,1472]. Palladium-catalyzed reaction of amine-tethered allenes in the presence of lithium bromide and copper acetate furnished 2-vinyl substituted pyrrolidines [1473].

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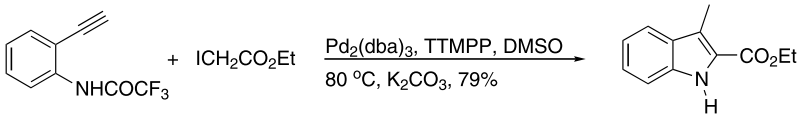


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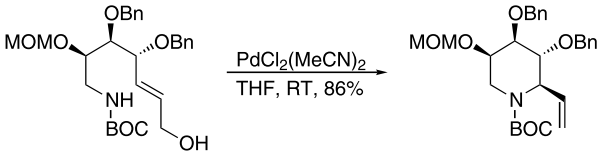
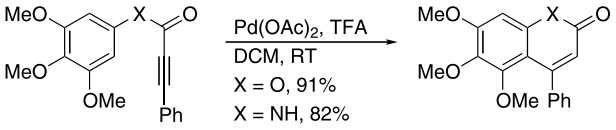



(330)

Palladium catalyzed the intramolecular hydroarylation of alkynes to form coumarins and quinolinones (Eq. (331)) [1474]. Both palladium and platinum catalyzed the intermolecular hydroarylation of electron-rich aromatic compounds [1475]. Ruthenium catalyzed the formation of 2,3-dialkyl substituted quinolines from anilines and trialkylamines [1476,1477]. Allylammonium chloride and anilines were used in a related reaction using aqueous solvent [1478]. Reaction of triethanol ammonium chloride with aryl amines in the presence of a ruthenium catalyst and a Lewis acid produced indoles (Eq. (332)) [1479].

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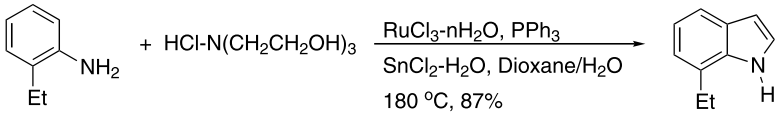
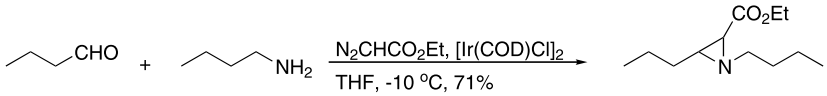



(332)

Copper catalyzed the aziridination of styrene and a number of asymmetric aziridination of alkenes using chiral ligands [1480–1482]. The mechanism of the copper-catalyzed aziridination was investigated [1483]. An unsaturated steroid was aziridinated using a hypervalent nitrene precursor [1484]. Conjugated dienes were monoaziridinated using a nitridomanganese complex [1485]. Iridium catalyzed the formation of aziridines in a three-component reaction of aliphatic aldehydes, aliphatic amines and ethyl diazoacetate (Eq. (333)) [1486].

Intramolecular alkene hydroxy-palladation carbonylation was used in the synthesis of leucascandrolide A (Eq. (334)) [1487]. Intramolecular alkylation of a phenol was used as a key step in the synthesis of aglaiastatin (Eq. (335)) [1488]. Chiral tetrahydrofuran and tetrahydropyran derivatives were prepared via a palladium-catalyzed intramolecular allylic hydroxylation (Eq. (336)) [1489]. Palladium-catalyzed intramolecular hydroxypalladation of alkenes was used in synthesis [1490]. Palladium-catalyzed double nucleophilic substitution reaction of allylic bisacetates to form 2-vinylmorpholines and 2-vinylpiperazines was described [1491]. Palladium catalyzed the reaction of benzene-1,2-diols with propargylic carbonates in the presence of a chiral diphosphine to give 2-alkylidene-1,4-benzodioxanes in moderate to high e.e. [1492]. Palladium catalyzed the reaction of 2,3-diene-1-ols with allylic halides to form substituted 2,5-dihydrofuranes (Eq. (337)) [1493]. A ruthenium-catalyzed oxidative cyclization of 1,6-dienes to give *trans*-2,6-bis(hydroxymethyl)tetrahydropyranyldiols was described (Eq. (338)) [1494].

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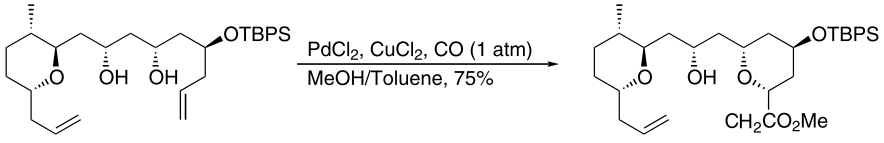


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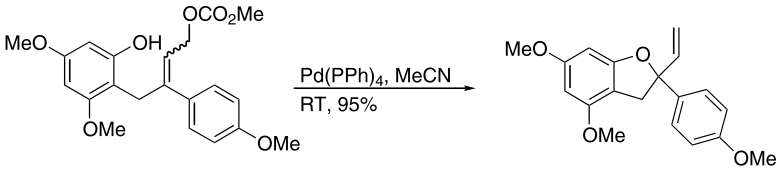


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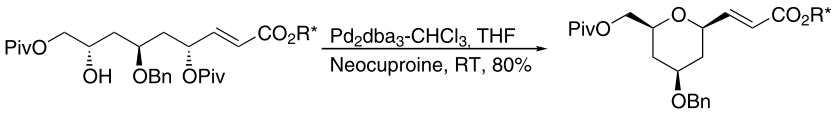


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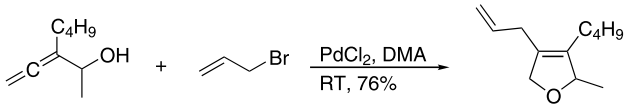


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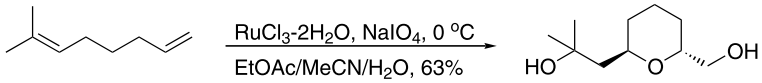
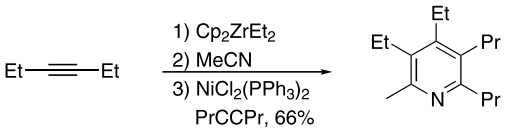


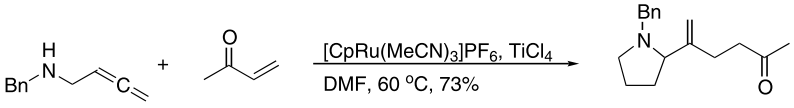

(338)

Substituted pyridines were prepared by nickel-catalyzed coupling of alkynes with azazirconacyclopentadienes prepared from an alkyne and a nitrile (Eq. (339)) [1495]. A synthesis of amides by incorporation of $N_2$ and CO using a titanium–nitrogen complex derived from molecular nitrogen in the presence of a palladium catalyst was developed [1496].




(339)

A ruthenium-catalyzed synthesis of pyrrolines and piperidines was developed (Eq. (340)) [1497]. Palladium catalyzed an interesting annulation of propargyl nucleophiles with α-sulfonyl-α,β-unsaturated ketones to give furo[3,4-*c*]heterocycles (Eq. (341)) [1498]. Palladium catalyzed a number of intramolecular additions to allenes to afford lactones, cyclic ethers, cyclic amines, and cyclic amides having a vinyl bromide substituent (Eq. (342)) [1499]. A one-pot, palladium-catalyzed alkyne–alkynoate coupling was used to prepare oxygen heterocycles (Eq. (343)) [1500].

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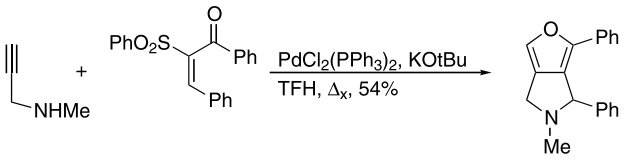


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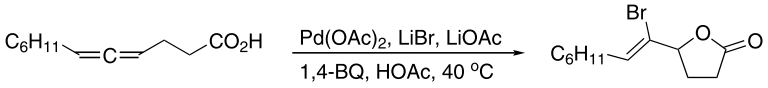


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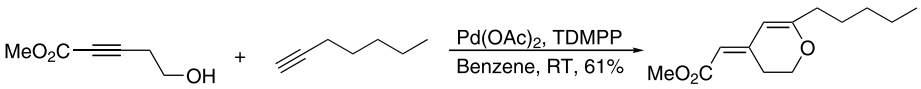
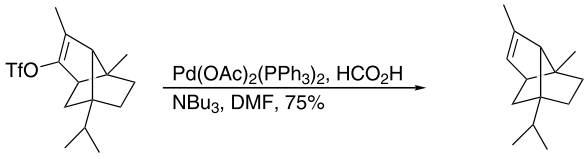



(343)

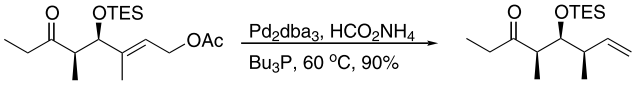
### 3.5. Alkenes, allenes, alkanes, arenes

A variety of functional groups were replaced by a hydrogen using palladium-catalyzed methodologies. Palladium(0) complexes, together with a hydride source, such as tributyltin hydride, silanes, or ammonium formats, catalyzed the reduction of vinyl triflates [303,1501], in the synthesis of allocyathin B3 [987] and a stink bug sesquiterpene (Eq. (344)) [1502], of vinyl phosphates [13] to give alkenes. A 2-pyridyl triflate [79] and an aryltriflate [1503] were also removed using similar methodologies. Palladium-catalyzed debromination of aryl bromides was observed upon attempted Heck cyclization [1504]. Monodebromination of 1,1-dibromoalkenes was achieved using tributyltin hydride in the presence of a palladium catalyst forming *cis*-substituted bromoalkenes [170]. Wilkinson's catalyst was used to remove an aldehyde functionality [1505].



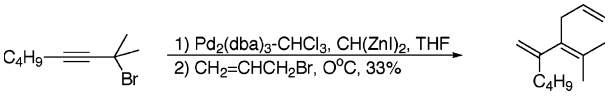

(344)

Diastereo- and enantioselective ammonium formate reduction of allylic esters and carbonates were reported (Eq. (345)) [1506,1507]. A stereoselective palladium-catalyzed hydrogenolysis of a vinyl epoxide to homoallylic alcohols was described [1508] and used in synthesis of a polyoxypeptin fragment [1509]. $\eta^3$-(1-Methylallyl)palladium(II) formates were prepared and studied as model intermediates in this reduction [1510]. Palladium catalyzed the regioselective dechlorination of allylic chlorides using ammonium formate [1511]. Palladium catalyzed the transformation of an allylic acetate to a diene [1512]. A related reaction of an allylic triflate was used in the synthesis of oidiolactone C [1513]. Esters of 4-hydroxymethylquinoline and 1-hydroxymethylnaphthalene were readily reduced to 4-methylquinoline and 1-methylnaphthalene using a palladium–formate system. The esters were suggested as novel protecting groups [1514].




(345)

Reaction of propargylic bromides with alkylzinc reagents in the presence of palladium, followed by addition of an alkyl halide or an aldehyde, gave 1,3-dienes and 1,2-dienes, respectively (Eq. (346)) [1515]. Reaction of α,β-epoxy vinyl triflates with tributyltin hydride in the presence of a palladium catalyst gave products derived from replacement of the triflate and an allene-ol from rearrangement. The rearranged product was also observed together with the corresponding ketone in the absence of tributyltin hydride (Eq. (347), (348)) [1516]. Nickel catalyzed a dihydroiodination of an alkyl iodide to form an alkene [928]. Chiral terminal allenes were prepared via a palladium-catalyzed reduction of mesylates of 2-bromo-2-en-ols using diethyl zinc (Eq. (349)) [1517].

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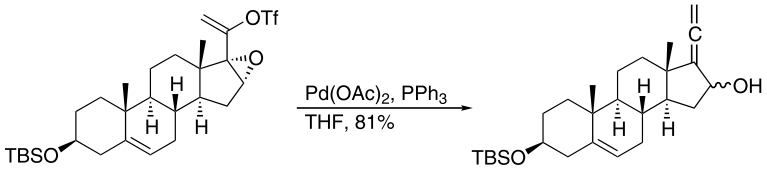


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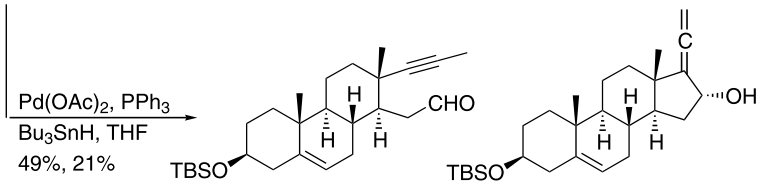


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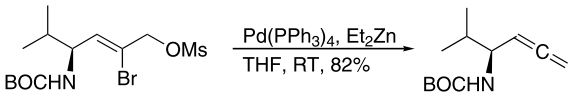
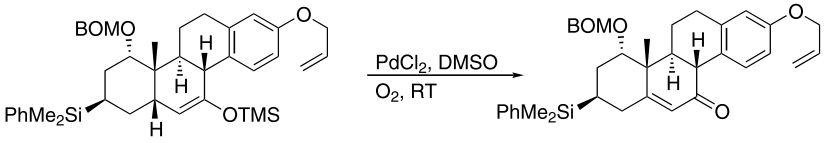



(349)

### 3.6. Ketones and aldehydes

A number of synthetic applications of the Saegusa reaction were published [1518,1519]. For example, synthesis of vitamin $D_3$ and calcitrol dimers [1248], steroids [1520,1521], an aphidicoline derivative [131], radermachol [1522], a segment of pamamycin 621A [1523], nicandrenones (Eq. (350)) [23], and dehydrorabelomycin (Eq. (351)) [1524] were described. An interesting benzylic oxidation was used in the synthesis of (+)-sambutoxin (Eq. (352)) [1525].

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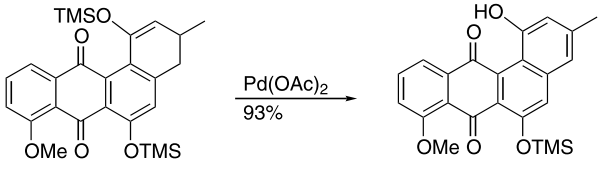


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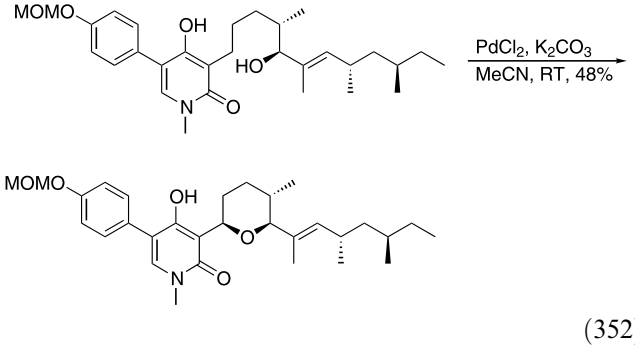
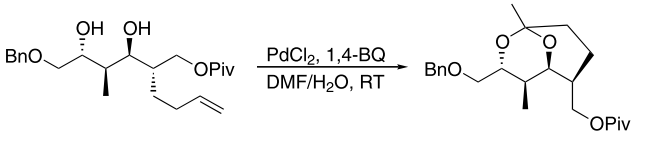



(352)

Wacker type oxidation, i.e. reaction of monosubstituted terminal alkenes with palladium(II) and water to afford methyl ketones, was utilized in a number of syntheses [886,928,1222,1526,1527]. Dysidiolide [1528], manzacidin A and C [1529], myxothiazols [1530], sanglifehrin A (Eq. (353)) [158], nakamurol A [1531] were prepared using a Wacker type reaction in one of the steps. Wacker oxidation in the presence of benzoquinone and perchloric acid was reported [1532]. Oxidation using molecular oxygen [1533] and an oxygen palladium diacetate/molybdovanadophosphate system [1534] were reported.

Addition of water to the terminal position was also observed in a few cases. Wacker type oxidation of 3-hydroxy-2-vinyl substituted tetrahydrofurans gave products derived from hydroxylation of the terminal alkene position forming a hemiacetal (Eq. (354)) [1535]. A vinyl-β-lactam was regioselectively transformed into an aldehyde [1536], and a vinyl substituted steroid gave a mixture of methyl ketone and an aldehyde [1537].

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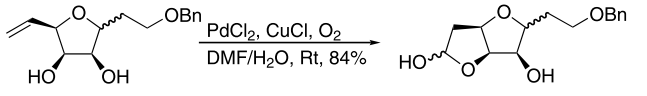
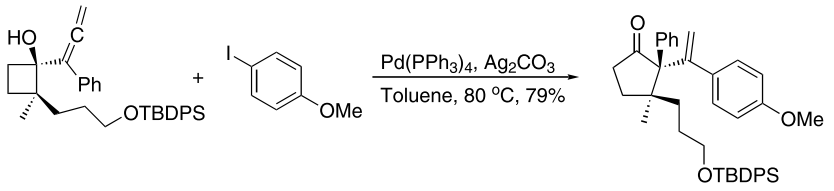



(354)

Palladium catalyzed the arylation of 1-allenecyclobutan-1-ols to give α-vinylsubstituted cyclopentanones (Eq. (355)) [1538]. Palladium catalyzed the arylation and vinylation of 1-alkynyl-1-cyclobutanols to give ring expanded α-methylenecyclopentanones [1539]. Palladium catalyzed the ring-opening of substituted cyclopropanols to give α,β-unsaturated ketones in good yields (Eq. (356)) [1540].

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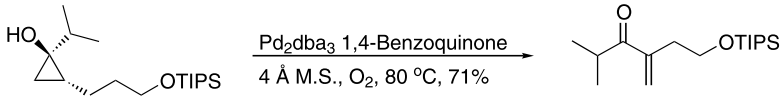
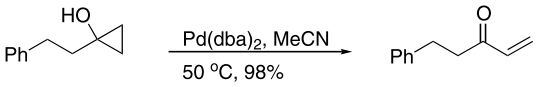



(356)

Ruthenium catalyzed the oxidation of alkanes to ketones using *t*-butylhydrogen peroxide or peracetic acid [1541]. Palladium catalyzed the ring-opening of cyclopropanols predominantly at the less substituted carbon–carbon bond to afford unsaturated ketones (Eq. (357)) [1542]. Asymmetric oxidation of an allylic alcohol to an α,β-unsaturated ketone in the presence of a ruthenium catalyst was used in the synthesis of (+)-tanikolide (Eq. (358)) [1543]. A palladium-catalyzed conversion of a chiral *cis*-1-hydroxy-2-acetoxy-2-ene to give an α,β-unsaturated ketone was used in the synthesis of (−)-shikimic acid [1544].

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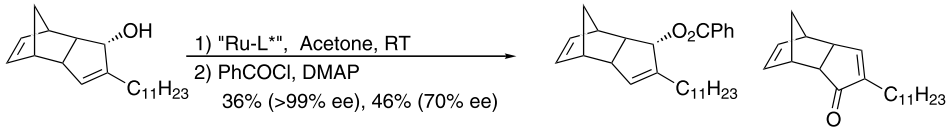
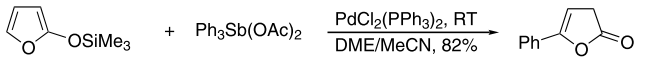



(358)

Arylation of silyloxy compounds with triarylantimony diacetates was reported (Eq. (359)) [1545]. Direct carbonylation of an aryl carbon–hydrogen bond using a ruthenium catalyst in the presence of ethene produced arylethylketones (Eq. (360)) [1546]. A ruthenium-catalyzed synthesis of bicyclic cyclopentenones using cyclobutendiones and norbornene was developed (Eq. (361)) [1547].

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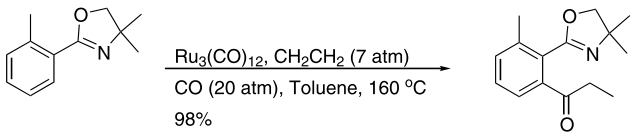
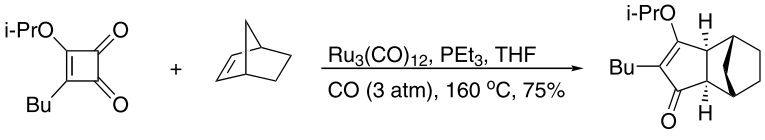



(360)

### 3.7. Organoboranes, silanes, germanes and stannanes

Cyclization of 1,6-diynes in the presence of a palladium catalyst and trimethylsilyltributyltin to give silylmethylene and stannylmethylene substituted cyclopentadienes was reported [1548]. A related reaction of bisallenes was described [1549]. Rhodium catalyzed



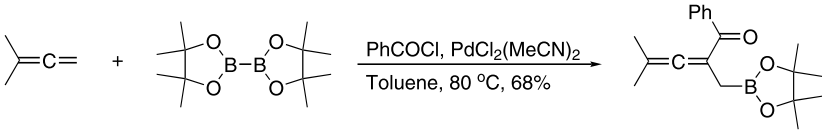

(361)

cyclization of a 1,6-diyne in the presence of silane to form silyl substituted cyclopentane derivatives [1550]. Palladium catalyzed the hydrosilylation of 1,6-dienes in the presence of pentamethyldisoloxane forming *trans*-substituted cyclopentanes [1551].

Ruthenium catalyzed the hydrosilylation of alkynes [1552], cobalt catalyzed hydrosilylation of oxygen containing alkenes [1553], platinum catalyzed hydrosilylation of alkenes [1554] and alkynes [1555], and rhodium catalyzed the intermolecular hydrosilylation of enamines [1556]. An intramolecular rhodium-catalyzed hydrosilylation was used in synthesis [889]. Platinum catalyzed the intramolecular hydrosilylation of homopropargylic silyloxy compounds to form silanes (Eq. (362)) [896]. Reaction of aryl bromides and iodides with hexamethyldisilane in the presence of a palladium catalyst gave trimethylsilyl substituted arenes (Eq. (363)) [1557,1558]. Ruthenium- (Eq. (364)) [1559] and iridium- [1560] catalyzed dehydrogenative silylation of terminal alkenes were described. Palladium catalyzed the asymmetric hydrotrichlorosilylation of alkenes [1561]. Titanocene-catalyzed carbosilylation of alkenes or dienes using alkyl halides and chlorosilanes was reported (Eq. (365)) [1562].

Palladium catalyzed the borylation of aryl halides and triflates using dialkoxyborane forming arylboronic esters [1563]. Palladium catalyzed the cross-coupling of aryl halides, triflates, and diazonium salts with dipinacolylborane or pinacoleborane producing aryl pinacolyl boronic esters [30,297,323,1564–1567]. Palladium-catalyzed synthesis of vinylboronic esters starting from vinyl iodides and triflates and pinacolborane was reported [1568–1570]. Allylic halides were transformed into allylic boronic esters in a similar fashion using pinacolborane [1571]. A one-pot boration-Suzuki coupling affording unsymmetrically substituted biphenyls was developed [1572].

A mechanistic study of the titanocene catalyzed hydroboration of alkenes and alkynes was reported [1573]. Platinum catalyzed the 1,2-diboration of alkynes, alkenes, and aldimines [1574]. Rhodium and iridium complexes catalyzed the *trans*-hydroboration of terminal alkynes forming (*Z*)-alkeneboron products [1575]. Rhodium-complexes catalyzed the asymmetric hydroboration of alkenes [1576] and a rhodium-catalyzed hydroboration in supercritical carbon dioxide was described [1577]. A three-component palladium catalyzed acylboration of allenes was developed (Eq. (366))

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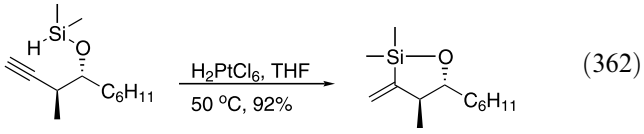


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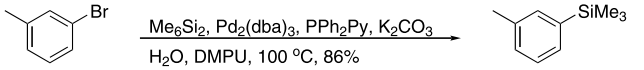


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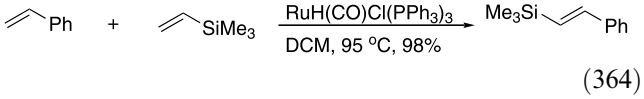


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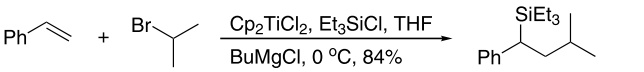
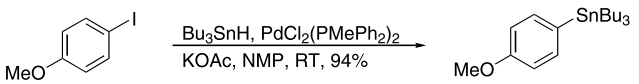



(365)

[1578]. Palladium and platinum catalyzed the silaboration of methylenecyclopropanes [1579], and palladium catalyzed a related reaction of 1,2-dienes [1580].

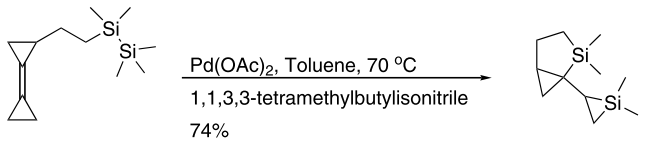
A palladium-catalyzed arylstannation of norbornene was reported [1581]. Palladium catalyzed the reaction of hexamethylditin with vinyl iodides or bromides [45,70], in the synthesis of (−)-mycothiazole [50] and disorazole $C_1$ [1582], of a 2-trifloxycarbapenam [102], and of aryl bromides [16,33,121,125,1583] to give organotin compounds for Stille type reactions. A palladium-catalyzed cross-coupling of tributyltin hydride with electron rich aryl iodides forming aryltin compounds was developed (Eq. (367)) [1584].




(367)

A palladium–iminophosphine catalyzed alkynylstannation of alkynes was developed (Eq. (368)) [1585]. A molybdenum-catalyzed hydrostannation of alkynes was

developed [14,1586]. Palladium-catalyzed hydrostannylation of terminal alkynes using tributyltin hydride forming a vinylstannane [159,1587] was used in total synthesis of sanglifehrin A [158], the macrocyclic core of apoptoliddin [138], orevactaene [310], (+)-thiazinotrienomycin E [114], and (−)-amphidinolide P [152]. Depending on the catalyst, rhodium versus nickel, both regioisomers of carbamoylstannation of terminal alkynes were obtained (Eq. (369)) [1588]. Rhodium catalyzed the hydrostannation of a propargylic alcohol positioning the tin on the internal carbon. This reaction was used in the synthesis of nicandrenones (Eq. (370)) [23].

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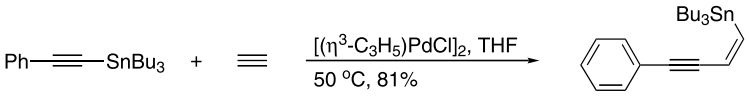
### 3.8. Organophosphorous, selenium, and sulfur compounds

Aryl diethylphosphonates [404,1595], and vinyl diethylphosphonates [1596] were prepared from aryl vinyl halides using palladium catalysts and diethylpho-

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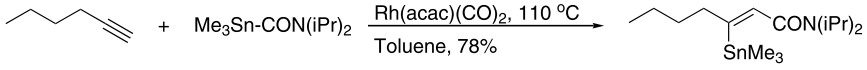


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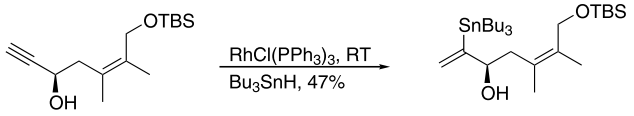


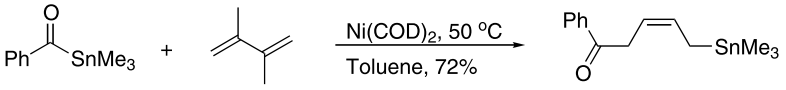

(370)

Palladium catalyzed the carbostannylations of norbornene and norbornadiene [1589]. Nickel catalyzed the acylstannation of 1,3-dienes (Eq. (371)) [1590]. Palladium catalyzed the allylstannation of alkynes using an α-methylallylstannane to give 1-stannyl substituted 1,4-dienes. High regioselectivity was observed in some cases [1591]. Palladium catalyzed the hydrostannation of a 7-oxabicyclo-2-heptene [18]. Chromium dichloride mediated coupling of $Bu_3SnCHI_2$ with an aldehyde gave a vinylstannane [142]. Palladium catalyzed the germyl stannation of alkynes to give 1-germyl-2-stannyl substituted alkenes [1592]. Palladium catalyzed the addition of disilanes, silylstannanes, and silyl cyanides across the double bond of bicyclopropylidene (Eq. (372)) [1593]. Both intra- and intermolecular reactions were reported. Regioselective silylstannylation of terminal alkynes was reported [36,1594].

sphite or trimethylsilyl diphenylphosphine [419]. Vinyl phosphonates were prepared from vinyl iodides and dialkylphosphites using a palladium catalyst [1597]. Palladium catalyzed the phosphorous–carbon coupling of hydroxymethylphosphines to give biarylmethylphosphine oxides and triarylphosphine oxides [1598]. Palladium catalyzed the formation of a mixed triaryl phosphine from triphenylphosphine and an aryl triflate or bromide [1599–1601].

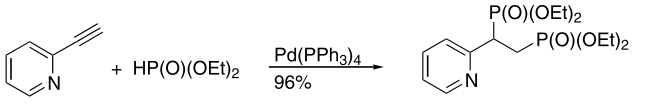
Palladium catalyzed the coupling of aryl halides with diphenylphosphine to form triarylphosphines [1602,1603]. Nickel catalyzed the coupling of aryl halides and triflates with diphenylphosphine [1604–1606] or chlorodiphenylphosphine [435,1607].

Palladium catalyzed the formation of alkynylphosphonates starting from 1,1-dibromoalkenes in the presence of methyloxirane [1608]. A palladium-catalyzed synthesis of bisphosphonates from terminal al-




(371)

kynes was described (Eq. (373)) [1609]. Palladium catalyzed the arylation of copper salt of bis(dimethylamino)phosphonyl-stabilized anions (Eq. (374)) [1610].

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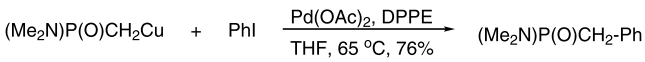
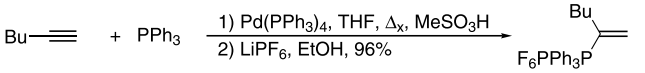



(374)

Addition of phosphines to alkynes in the presence of a palladium catalyst and methanesulfonic acid to afford vinylphosphonium salts was described (Eq. (375)) [1611]. A regio- and stereoselective palladium-catalyzed hydrophosphorylation of allenes forming allyl phosphonates was developed (Eq. (376)) [1612]. Palladium catalyzed hydrophosphorylation of alkenes [1613]. Phospholes were prepared using a zirconocene dichloride mediated reaction of 1,7-diynes followed by reaction of the cyclometallated compound with dibromophenylphosphine [1614].

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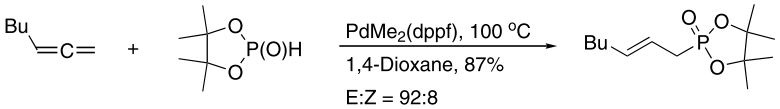
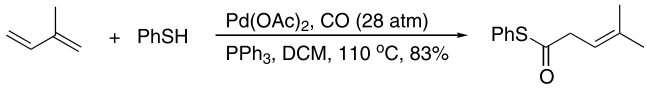



(376)

Nickel and palladium catalyzed the cross coupling of diselenides with iodoaryl compounds in the presence of a polymer-supported borohydride [1615]. Palladium catalyzed the reaction of tributyltin phenylselenide aryl iodides and triflates to give unsymmetrical diaryl selenides [1616].

Copper mediated the cross-coupling of aryl boronic acids with alkyl thiols to give sulfides [1617]. Palladium, in the presence of a bifunctional arene–chromium tricarbonyl complex, catalyzed an asymmetric allylic sulfonation of allylic carbonates [1618]. Palladium-catalyzed reaction of chiral allylic carbonates with trimethylsilyl methyl sulfide gave allylic thioethers [1619]. Palladium catalyzed a regioselective thiocarbonylation of conjugated dienes (Eq. (377)) [1620]. Tebbe's reagent was used to deoxygenate sulfoxides, *N*-oxides, and selenoxides (Eq. (378)) [1621]. 2-Methylpyridines were formed from *N*-pyridine oxides. Hydrozirconation of alkynes followed by addition of electrophiles was described. Using this methodology, dienes, thiophen oxides, or thiophens sulfurdioxide were obtained from reaction with sulfurdioxide, disulfurdichloride produced, respectively [1622],

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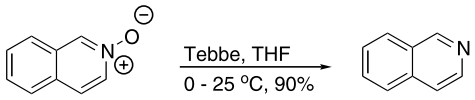



(378)

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

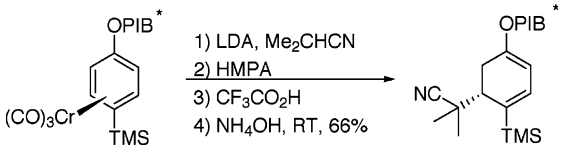
A number of transition-metal catalyzed reactions were published wherein metal complexes were allowed to react on one or more of the ligands without demetallation of the complex. A β-iodovinylferrocene underwent Heck reaction [1623]. Sonogashira type couplings of a σ-alkyne bonded iron complex [1624], of alkynes tethered to ferrocenes [1625–1627], of alkyne–arene chromium tricarbonyl complexes, [1628], and of ferrocene iodide [1629] were reported. Chromium tricarbonyl complexed chlororarenes did not furnish the expected Sonogashira coupling product when reacted with propargylic alcohols but gave the corresponding Heck product [1630].

Reactions involving a transmetallation step from boron or tin were also used. Suzuki couplings of tricarbonyl chromium aryl bromide [1631] were published. Stille type couplings were employed using trialkyltin substituted ferrocenes [1632–1635]. Stille cross-coupling of chromiumtricarbonyl complexed benzylhalides was used in the synthesis of (−)-steganone [312]. Stille and Suzuki type cross-couplings of flouroarenes

complexed to chromium tricarbonyl were reported [1636]. Palladium catalyzed the coupling of ferrocene zinc chloride with iodonaphthalene [1637].

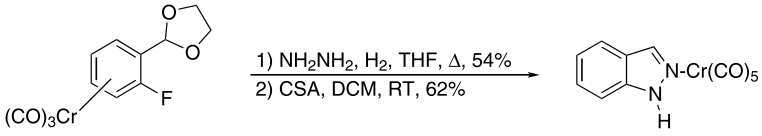
Arene tricarbonyl chromium complexes continued to be extensively used as templates for organic reactions [1638]. For example, as a traceless polymer-supported linker [1639], in selective functionalization of alkyl side chains [1640], in enantioselective ring lithiation of the aromatic ring, followed by addition of an electrophile [161,1641], in stereoselective allylation of aldimine side chain [1642], stereoselective [2+2]cycloaddition to aldimine side-chain [1643], in stereoselective Diels–Alder reaction of side-chain aldimine [1644], stereoselective reduction of the forming mitosanes [1645], diastereoselective nucleophilic additions to esters [1646], and aldehydes directly attached to the aromatic ring [1647,1648].

Enantiotopic lithiation of *N*-methyl-*N*-acyl-2,6-dimethylanilide complex was reported [1649,1650]. Regioselective deprotonation of fluoroarene chromium tricarbonyl complexes followed by alkylation was reported [1651]. Evidence for the formation of an trianion was presented [1652]. A high degree of 1,5-asymmetric induction was observed upon reaction of chiral alkoxybenzene chromium tricarbonyl complexes with nucleophiles (Eq. (379)) [1653]. Stereoselective Michael additions to an unsaturated side-chain were reported [1654]. The π-shielding interactions in asymmetric Diels–Alder reactions were probed [1655].




(379)

Propargylic anions adjacent to a chromium tricarbonyl arene group underwent nucleophilic substitution reactions with electrophiles to afford an alkyne or an allene depending on the electrophile [1656]. Propargylic cations adjacent to a chromium tricarbonyl arene group smoothly reacted with nucleophiles [1657]. Indazoles were prepared from arene chromium tricarbonyl complexes (Eq. (380)) [1658]. Axially chiral (−)-steganone and *O*,*O*′-dimethylkorupensamine A (Eq. (381)) [1659] and ent-Korupensamine B [1660] were prepared using chiral arene chromiumtricarbonyl complexes.

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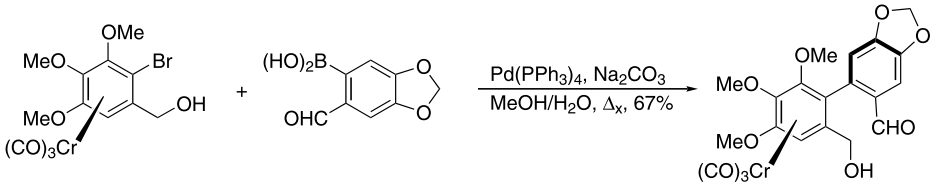
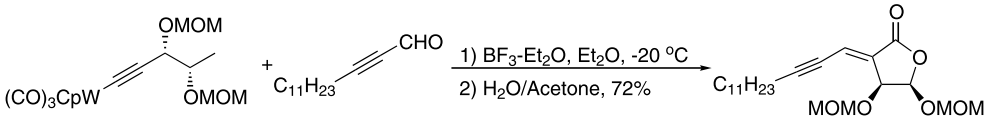



(381)

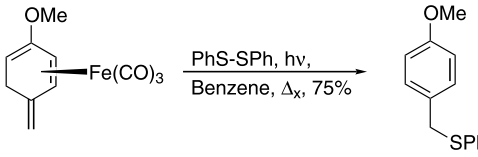
Tungsten–σ-alkyne complexes were used in the synthesis of (−)-epilitsenolide $C_2$ and (−)-isodihydromahubanolide B (Eq. (382)) [1661]. A resin based chromium catalyst was used for [6+2]cycloadditions of cycloheptatriene with alkenes [1662].

Complexation of one ring of naphthalene to $CpFe^+$ rendered the remaining ring easily reduced using palladium on carbon under mild conditions [1663]. Cyclopentadiene chloroarene iron complexes smoothly



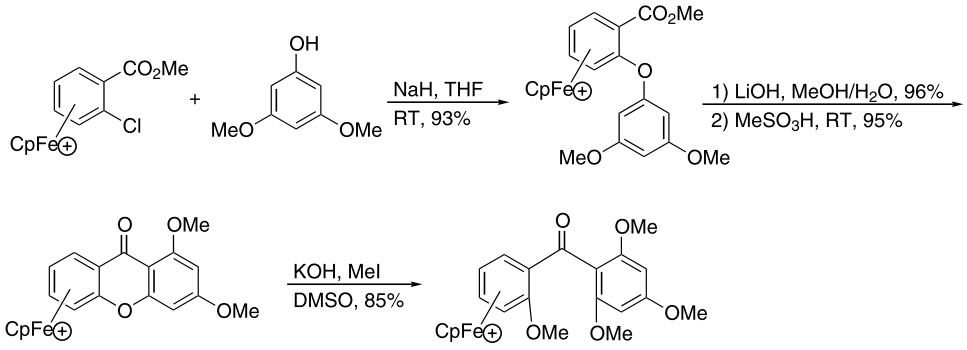

(382)

reacted with phenoxide, thiophenoxide, and selenophenoxide to give the corresponding ethers [1664]. Iron 5-methylenecyclohexa-1,3-diene complexes underwent regioselective radical addition to the exocyclic methylene group (Eq. (383)) [1665].



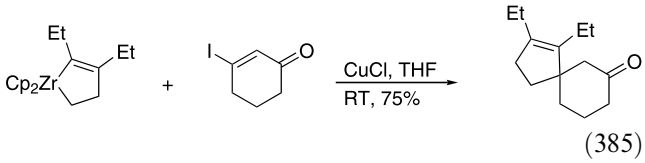

(383)

Intermolecular aminations [1666] and etherifications [1667,1668]. In the synthesis of ristocetin A fragment [1669], of ruthenium chloroarene complexes were reported. Intermolecular nucleophilic substitution of cationic iron chlorosubstituted arene complexes followed by nucleophilic ring opening to form functionalized benzophenones was developed (Eq. (384)) [1670].




(384)

Intramolecular [2+2+1]cycloaddition of aza-1,6-diynes using cyclopentadienyl cobalt dicarbonyl and iron pentacarbonyl gave bicyclic cyclopentadienone metal complexes [1671]. Zirconacyclopentadienes reacted with 3-iodopropenoates and 3-iodocycloenones in the presence of CuCl to give cyclopentadienes and spirocyclic compounds, respectively (Eq. (385)) [1672], with 2-iodobenzyl and benzoyl halides to afford benzocycloheptene derivatives [1673], with alkynyl bromides to give bisalkynylation products [1674], with aldehydes to give cyclopentadienes [1675], with alkynes to give aromatic compounds [1676,1677], and with dialkyltin dichlorides to give tincyclopentenes and tinpentanes [1678]. Bicyclo[4.3.0]systems were obtained upon reaction of γ,δ-unsaturated (cycloalkenyl)alkylzirconocenes with terminal alkynes [1679]. Insertion of lithiated epoxides into zirconacycles was reported (Eq. (386)) [1680].

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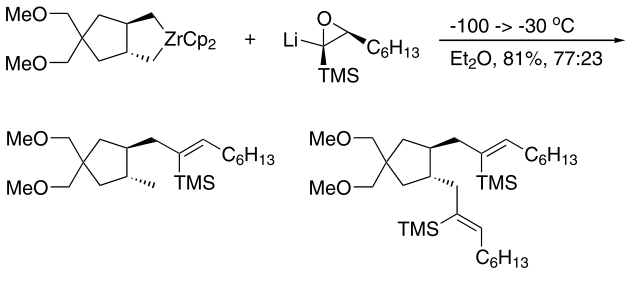


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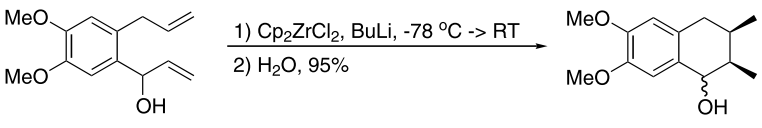
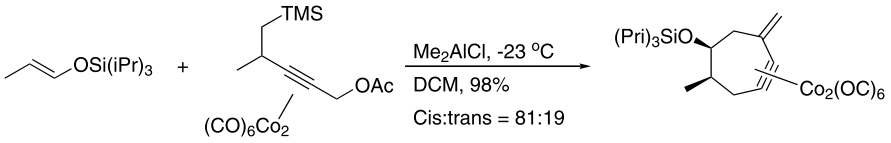
Hydrozirconation of a 1,7-diene followed by protonolysis of the intermediate zirconacycle to give a *cis*-1,2-



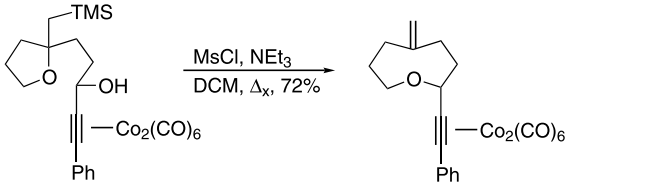

(387)

dimethylcyclohexane was used in the synthesis of galbulin and isogalbulin (Eq. (387)) [1681]. The regioselectivity of insertion of chloroallylides into a variety of unsymmetrical zirconacycles was examined. Results indicated that the carbeniod acted mostly as an electrophile [1682].




(388)

Complexation of cobalt carbonyl to an alkyne initiated an intramolecular Diels–Alder reaction [1683]. An interesting [5+2]cycloaddition of dicobalt–alkyne complexes with silyl enol ethers was described (Eq. (388)) [1684]. Synthesis of cyclic ethers using an intramolecular Nicholas reaction of 5,6-epoxy-7-octynol was reported [1685]. A related strategy was used in the synthesis of a ciguatoxin fragment [732–734,1686,1687] and oxocane and oxonane derivatives by ring expansion of tetrahydrofuran tethetered to a propargylic alcohol (Eq. (389)) [1688]. Radical cyclizations forming carbocyclic systems were developed [1689]. Depending on the amount of nucleophile, selective monosubstitution, substitution with two identical or two different nucleophiles of diyne tetracobalt complexes of hepta-2,5-diynyl-1,7-diol derivatives was described [1690]. A propargylic alcohol was removed by complexation to cobalt followed by treatment with boron trifluoride etherate [1691]. A Nicholas type reaction using an enolate derived from an oxazolidinone as the nucleophile was reported [677]. Cobalt alkyne complexes having a stereodefined benzyl group in the γ-position and an alcohol in the α-position gave upon treatment with boron trifluoride etherate a stereoisomerically pure α,γ-disubstituted product [1692]. Cobalt alkyne stabilized α-trifluoromethyl propargylic carbenium ions were formed and reacted with amines and enolates [1693]. Reactions of dicobalt hexacarbonyl coordinated enynes with carbocations were examined [1694]. Intramolecular delivery of a hydride resulted in the reduction of a propargylic alcohol under Nicholas reaction conditions (Eq. (390)) [1695].

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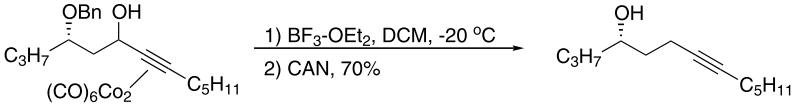
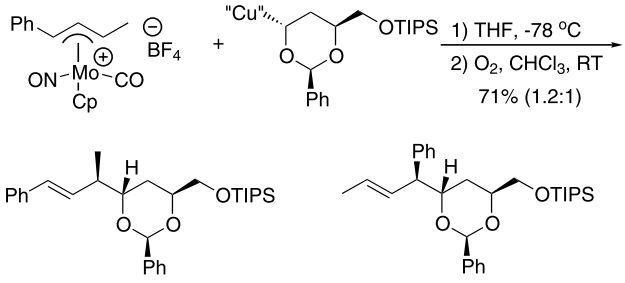



(390)

The diastereoselectivity dependence of the Lewis acid on addition of nucleophiles to formyl substituted iron diene complexes was examined [1696]. Allylation of iron dienal complexes with moderate enantioselectivity was reported [1697]. Wittig reaction of a formyl substituted iron diene complex was used in a stereoselective synthesis of 11*Z*-retinal [1698]. Cyclization of iron diene complexes having a tethered *O*-perfluorobenzoyloximes to give indole derivatives was described (Eq. (391)) [1699]. Intramolecular Friedel–Crafts acylation of iron diene complexes was reported (Eq. (392)) [1700]. Iron diene complexes were used in the synthesis of (−)-spinosyn A [1701], halicholactone [648], and macrolactin

A [1702]. A remote asymmetric induction of an iron diene complex was used in a diastereoselective synthesis of epipatulolide C [1703]. Diastereoselective aldol condensation of tricarbonyl iron complexed dienones was use to prepare 1,2,3-trisubstituted 1,3-diols [1704]. This reaction was employed in the synthesis of carbonolide B and tylonide [1705]. An iron tricarbonyl cycloheptatrienone complex was used to stereospecifically prepared heptitol derivatives [1706]. A stereospecific 1,3-migration of the irontricarbonyl moiety of conjugated trienes was reported [1707].

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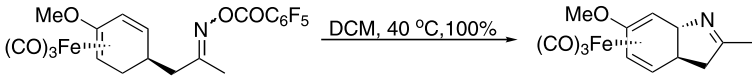


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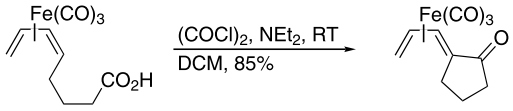


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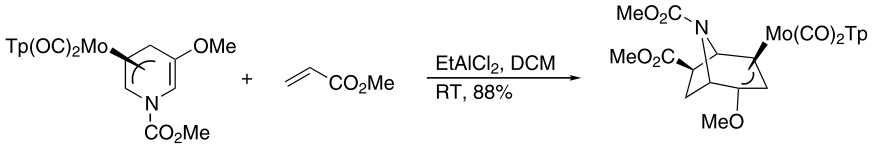
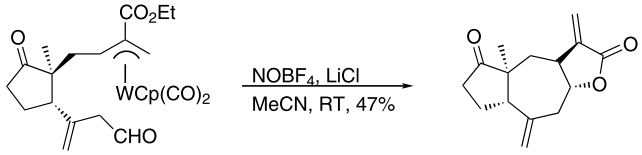


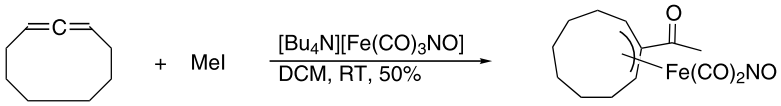

(393)

A procedure for the preparation of $\eta^3$-allyl molybdenum complexes using *cis*-$Mo(CO)_4(Py)_2$ was reported [1708]. $\eta^3$-Pyridinylmolybdenum π-complexes were used in enantiocontrolled synthesis of tropanes (Eq. (393)) [1709] and spirooxindoles [1710] via a [5+2]cycloaddition. Cationic molybdenum dihydropyridine complexes were used as scaffolds for the regioselective synthesis of polyfunctionalized tetrahydropyridines [1711] and related pyranyl complexes to prepare 2,3,6-trisubstituted dihydropyranes [1712]. Addition of a cuprate to a planar chiral $\eta^3$-allyl molybdenum complex was used in the synthesis of cryptophycin 4 (Eq. (394)) [1713]. An approach toward sesquiterpene lactones via intramolecular allylation of $\eta^3$-allyl tungsten complexes was reported (Eq. (395)) [1714]. A stereocontrolled synthesis and reactions of tungsten-$\eta^3$-allyl complexes having two 1-hydroxyalkyl groups was developed. A number of functionalizations were described [1715]. A synthesis of functionalized $\eta^3$-allyl–dicarbonylnitrolsyliron complexes from allenes and an alkyl halide was reported (Eq. (396)) [1716].




(395)

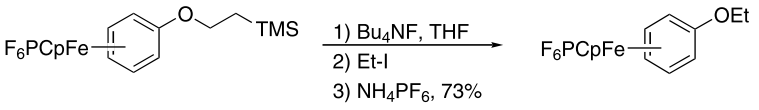
1,7-Induction of chirality using $\eta^3$-allyliron lactone and lactam complexes as chiral templates was observed in Mukayama type aldol condensations [1717]. Stereocomplementary isomers of $\eta^3$-allyliron lactones were prepared from an optically active vinyl epoxide, diiron nonacarbonyl, and ultrasound [1718]. Iron tricarbonyl 1-azabuta-1,3-diene complexes were prepared and used as tricarbonyl iron transfer reagents [1719]. Cationic cyclohexadienyl iron complexes underwent diastereoselective spiroannulation upon reaction with 3-amino-2-propenoic acid derivatives [1720]. Phenoxide complexed to cyclopentadienyl iron was prepared in situ and




(396)

reacted with electrophiles (Eq. (397))) [1721]. Reactions of cyclic $\eta^5$-iron complexes were used in the synthesis of furostifoline [1722], carquinostatin A (Eq. (398)) [1723], and indolo[2,3-*b*]carbazole [1724]. An iron–cyclobutadiene complex was used in the synthesis of (+)-asteriscanolide (Eq. (399)) [1222].

Hydrozirconation of tin, silicon, or selenium substituted alkynes, followed by addition of an acid chloride produced α-tin, -silicon, or -selenium substituted vinylketones [25,1733]. Hydrozirconation of tin substituted alkynes followed by protonolysis gave *cis*-vinylstannanes [1734]. Reaction of the intermediately formed

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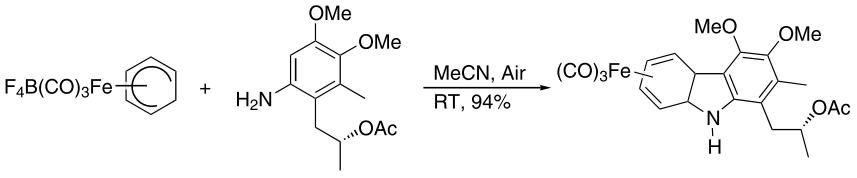


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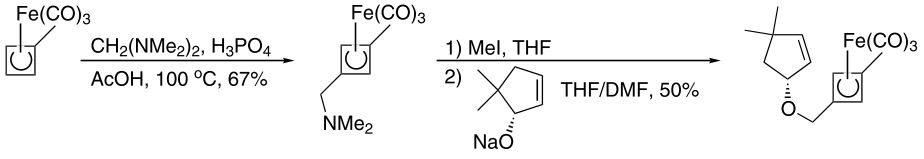
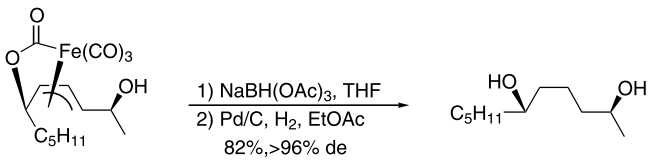


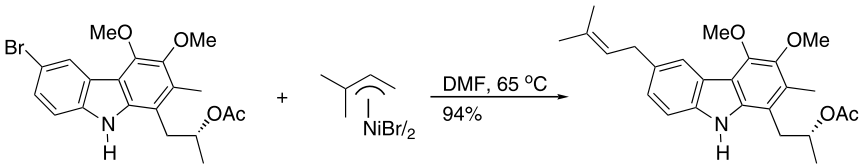

(399)

Synthesis of palladium [1725], platinum [1726], ruthenium [1727], iridium [1728,1729], and titanium [1730] $\eta^3$-allyl complexes was described. $\eta^3$-Allyltricarbonyliron lactone complexes were reductively decomplexed to give acylic 1,5-diols and 1,5,7-triols (Eq. (400)) [1731]. This strategy was used in the synthesis of α- and β-zearalenol [146]. Coupling of a nickel $\eta^3$-allyl complex with an aryl bromide was used in the synthesis of carquinostatin A (Eq. (401)) [1732].




(400)

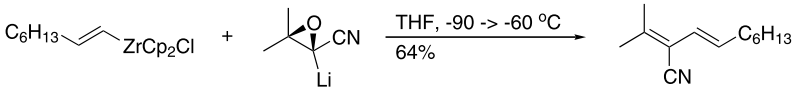
vinylzirconium complex with carbon monoxide followed by HCl gave α-tin substituted α,β-unsaturated aldehydes [24]. Hydrozirconation of alkynyl sulfoxides followed by reaction with electrophiles gave vinyl sulfoxides [408]. Hydrozirconation of terminal alkynes, followed by carbonylation and reaction with diphenyldiselenide, afforded α,β-unsaturated selenoesters [1735], followed by reaction with elemental selenium or tellurium and air oxidation gave symmetrically substituted divinyl diselenides and ditellurides [1736], and followed by diaryldiditellurides produced vinyltellurides [1737]. Reaction of the intermediately formed zirconium species with selenium followed by an alkyl halide gave vinylselenides [1738]. Hydrozirconation of stannylacetylenes followed by reaction with butyltellurenyl bromide gave stannyl–tellurosubstituted alkenes [1739]. Similar hydrozirconation of telluroacetylenes followed by reaction with



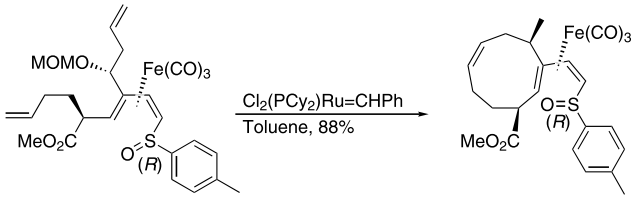

(401)

butylselenyl bromide produced telluro-selenylsubstituted alkynes [1740].

Hydrozirconation of alkynes followed by addition of iodine to produce vinyl iodides [210,1597,1741–1743] was used in the synthesis of the macrocyclic core of apoptoliddin [138]. Addition of lithiated epoxynitriles afforded 2-cyanosubstituted 1,3-dienes (Eq. (402)) [1744], and addition of a variety of lithiated compounds gave allylic products [1745].

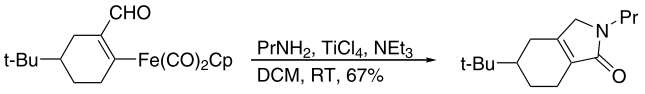



(402)




(403)

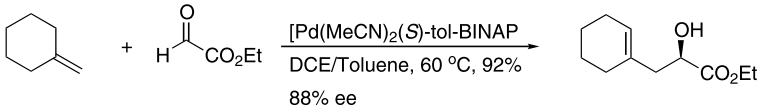
Reaction of terminal alkynes with trimethylaluminum and zirconocene dichloride to give trisubstituted vinyl iodides [225] was used in syntheses of phomactin D [367] and methanophenazine [1746]. Alkene cross-metathesis on alkenes tethered to ferrocene was reported using Grubb's ruthenium catalyst [1747]. Enantiopure $\eta^4$-(1-sulfinyldiene)iron(0) tricarbonyl complexes were used as templates for carbocycle construction via ring-closing metathesis (Eq. (403)) [1748]. α,β-Unsaturated γ-lactones were prepared by an intramolecular carbonylation of iron-substituted enals (Eq. (404)) [1749]. α,β-Unsaturated lactams were prepared in a related fashion [1750].




(404)

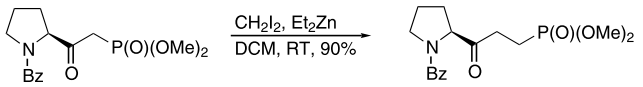
## 5. Miscellaneous

A chiral palladium catalyst was developed for asymmetric glyoxalet-ene reactions (Eq. (405)) [1751]. An interesting zinc-mediated chain extension of β-keto phosphonates has been developed (Eq. (406)) [615]. Intramolecular carbostannation of active methine compounds with unactivated multiple bonds was mediated by tin tetrachloride and triethylamine (Eq. (407)) [1752]. Acetalization of electron-poor alkenes was catalyzed by a palladium–molybdovanadophosphate complex sup-

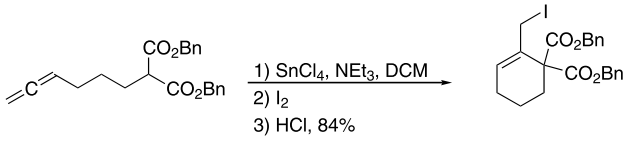



(405)

ported on activated carbon under an oxygen atmosphere [1753]. Palladium catalyzed the reaction of silanes with carboxylic acids to produce silyl esters [1754].




(406)



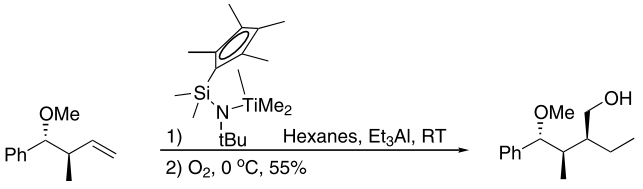

(407)

Titanated alkoxyallenes prepared in situ from 3-alkoxy-2-propyn-1-yl carbonates acted as ester homoaldol equivalents [1755]. A titanium-catalyzed carboalumination of alkenes was reported (Eq. (408)) [1756]. A titanium-mediated coupling of allyl bromide with an alkyne followed by addition of iodine to give a vinyl iodide was used in the synthesis of carbacyclin (Eq. (409)) [368]. A nickel-catalyzed cyclization of 1,3-dienes with a tethered carbonyl group was developed [1757]. A closely related asymmetric cyclization in the presence of silanes was also described (Eq. (410)) [1758].

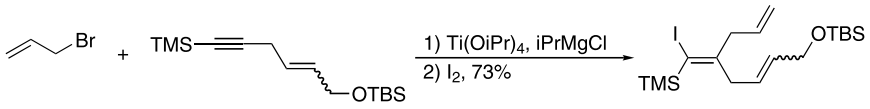
Asymmetric dearomatization of $\eta^2$-arene osmium complexes gave stereodefined cyclohexane derivatives (Eq. (411)) [1759]. $\eta^2$-Furan osmium complexes were transformed into new heterocycles by reaction with either nucleophiles or electrophiles [1760]. Thiolate-bridged diruthenium complexes catalyzed the addition of alcohols, amines, amides, and thiols to propargylic alcohols replacing the OH forming ethers, amines, amides, and thioethers [1761]. Reaction of 2-alkynyl-substituted aromatic ketones with an excess of $W(CO)_5THF$ complex followed by a vinyl ether produced substituted naphthalenes (Eq. (412)) [1762].

## 6. Reviews

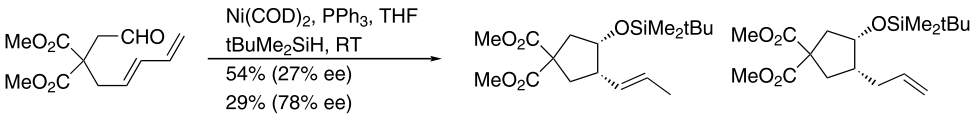
The following reviews appeared in 2000:



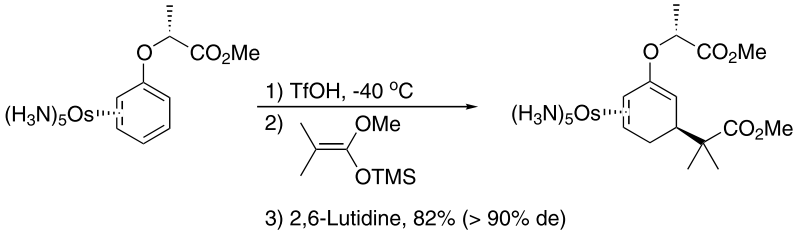

(408)



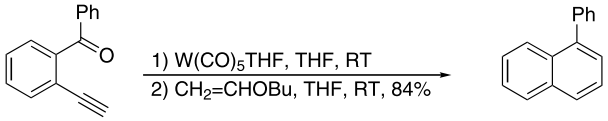

(409)




(410)




(411)




(412)

- Anionic Pd(0) and Pd(II) intermediates in palladium-catalyzed Heck and cross-coupling reactions (17 references) [1763].
- Theoretical studies on reactions of transition-metal complexes (188 references) [1764].
- Applications of stoichiometric organotransition metal complexes in organic synthesis (86 references) [1765].
- Phosphinooxazolidines—a new class of versatile, modular P,N-ligands for asymmetric catalysis (46 references) [1766].
- Chiral monodentate phosphine ligand MOP for transition metal-catalyzed asymmetric reactions (41 references) [1767].
- Nickel-catalyzed cyclizations, couplings, and cycloadditions involving three reactive components (40 references) [1768].
- The application of transition metal catalysis for selective cyclocarbonylation reactions. Synthesis of lactones and lactams (34 references) [1769].
- 1,2-Dihalogenoalkenes: Useful linchpins to unsaturated compounds via palladium or nickel chemistry (127 references) [1770].
- Chemistry of monoorganopalladium complexes relevant to catalysis (48 references) [1771].
- Stereoselective synthesis of axially chiral biaryls utilizing planar chiral (arene)chromium complexes (38 references) [1772].
- Synthetic reactions mediated by a $Ti(O\text{-}I\text{-}Pr)_4/2$ *i*-PrMgX reagent (80 references) [1773].
- Amidocarbonylation—an efficient route to amino acid derivatives (85 references) [1774].
- Acetylenic coupling: A powerful tool in molecular construction (235 references) [1775].
- Olefin metathesis and beyond (161 references) [1776].
- Ionic liquids—new "solutions" for transition metal catalysis (98 references) [1777].
- *N*-Heterocyclic carbenes: State of the art in transition-metal-complex synthesis (123 references) [1778].
- Rhodium(II) mediated cyclizations of diazo alkynyl ketones (61 references) [1779].
- Activation and reactivity of Group 16 inter-element linkage—transition-metal-catalyzed reactions of thiols and selenols (38 references) [1780].
- Rhodium(II) mediated cyclizations of diazo alkynyl ketones (61 references) [1781].
- Development of olefin metathesis catalyst precursors bearing nucleophilic carbene ligands (26 references) [1782].
- Bis(iodozincio)methane—preparation, structure, and reaction (25 references) [1783].
- Recent progress in asymmetric intermolecular C–H activation by rhodium carbenoid intermediates (39 references) [1784].
- Catalysts for metal carbene transformations (39 references) [1785].
- The use of polypyrazolylborate copper(I) complexes as catalysts in the conversion of olefins into cyclopropanes, aziridines and epoxides and alkynes into cyclopropenes (26 references) [1786].
- Nickel-catalyzed intermolecular domino reactions (56 references) [1787].
- Ligand bite angle effects in metal-catalyzed C–C bond formation (215 references) [1788].
- 1,*n*-Dicarbanionic titanium intermediates from monocarbanionic organometallics and their application in organic synthesis (313 references) [1789].
- Synthesis of organotitanium complexes from alkenes and alkynes and their synthetic applications (221 references) [1790].
- Recent advances in the transition-metal-catalyzed regioselective approaches to polysubstituted benzene derivatives (51 references) [1791].
- Transition metal dearomatization reactions (149 references) [1792].
- Efficient synthesis of tricarbonyliron-diene complexes—development of an asymmetric catalytic complexation (168 references) [1793].
- Metal-mediated synthesis of medium-sized rings (258 references) [1794].
- The Heck reaction as a sharpening stone of palladium catalysis (419 references) [1795].
- Palladium-catalyzed reactions of allenes (205 references) [1796].
- Synthesis of heterocyclic and carbocyclic compounds via alkynyl, allyl, and propargyl organometallics of cyclopentadienyl iron, molybdenum, and tungsten complexes (140 references) [1797].
- Synthesis and reactions of allylic, vinylic, and arylmetal reagents from halides and esters via transient organopalladium intermediates (57 references) [1798].
- Metal-catalyzed carbon–sulfur bond formation (135 references) [1799].
- Transition-metal-catalyzed additions of silicon–silicon and silicon–heteroatom bonds to unsaturated organic molecules (152 references) [1800].
- Metal-catalyzed hydrostannations (75 references) [1801].
- Applications of stoichiometric organotransition metal complexes in organic synthesis (86 references) [1802].
- Catalytic asymmetric processes (396 references) [1803].
- Cobalt mediated cyclizations (106 references) [1804].
- Catalytic applications of transition metals in organic synthesis (168 references) [1805].
- Supported catalyst and their applications in organic chemistry (71 references) [1806].
- Recent applications of olefin metathesis and related reactions in carbohydrate chemistry (64 references) [1807].

- Asymmetric functionalization of tricarbonylchromium complexes of arenes by non-racemic bases (42 references) [1808].
- Nickel-catalyzed reactions of nucleophiles with unactivated and partially activated olefins and acetylenes (65 references) [1809].
- Recent advances in the Pauson–Khand Reaction and related [2+2+1] cycloadditions (104 references) [1810].
- Palladium in organic synthesis: Fundamental transformations and domino processes (145 references) [1811].

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