



Review

The chemistry of the carbon–transition metal double and triple bond: Annual survey covering the year 2009[☆]James W. Herndon^{*}*Department of Chemistry & Biochemistry, New Mexico State University, MSC 3C, Las Cruces, NM 88003, USA*

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ABSTRACT

This is a review of papers published in the year 2009 that focus on the synthesis, reactivity, or properties of compounds containing a carbon-transition metal double or triple bond.

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1. Introduction

This survey is intended to be a comprehensive summary of articles that report on the synthesis, reactivity, or properties of compounds featuring a multiple bond between carbon and a transition metal. Reactions that employ metal carbene complexes as transient intermediates generated through well-established routes (e.g. metal catalyzed diazo compound decomposition) are not covered, unless there is significant discussion of the carbene complex intermediate. Several reviews in this area appeared in 2009 [1–5]. Although a determined effort has been made to include patents, in general only patents that focus on the metal–carbene or metal–carbyne complex are included. Patents that appear in 2009 editions of *Chemical Abstracts* in the “Organometallic Compounds” section have been included. Only compounds which feature a multiple bond between a single carbon atom and a single transition metal are discussed in this survey, thus bridging carbene and carbyne complexes are not covered unless there is a multiple bond to at least one transition metal. The complexes of N-heterocyclic (or Arduengo) carbenes (NHC’s) with transition metals have generally not been included. Since the π -donation component of these complexes is usually minimal, there is no formal carbon–metal multiple bond [6], although there is some controversy concerning this issue [7–9]. This area was reviewed several times in 2009 [10–27]. In general, NHC-transition metal complexes are included only if there is discussion of multiple bond character in the carbon–metal bond. This survey has been divided into two sections, metal carbene (or alkylidene) complexes and metal carbyne (or alkylidyne) complexes; the carbene complex section represents the vast majority of this article. The metal carbene section has been organized according to metal, starting from the left side of the Periodic Table. The Ionic Model [28] has been employed for the discussion of oxidation states and ligand electron count throughout this survey. A special section focusing on alkene metathesis has been included prior to the discussion of carbene complexes of individual metals. The metal carbyne section has been organized according to reaction type. Articles from the journals *Angewandte Chemie International Edition*, *Chemistry: A European Journal*, *Tetrahedron*, and *Tetrahedron Letters* are restricted to volumes 48, 15, 65, and 50 respectively, which covers the period of December 2008 to December 2009 according to some search engines.

Abbreviations and monikers (see also the guide to authors for the *Journal of Organic Chemistry* [29])

DFT
NHC
Grubbs catalyst I
Grubbs catalyst II
Grubbs catalyst III
Hoveyda–Grubbs catalyst
Schrock catalyst

Density Functional Theory
N-Heterocyclic Carbene
Structure 1 (Fig. 1)
Structure 2 (Fig. 1)
Structure 3 (Fig. 1)
Structure 4 (Fig. 1)
Structure 5 (Fig. 1)

See also Scheme 1 for abbreviations for distinct modes of metathesis.

2. Metal–carbene or metal–alkylidene complexes

2.1. Review articles and comments

Several reviews/comments covering aspects of metal–carbene complex chemistry appeared in 2009. They have been organized as to whether they focus on olefin metathesis or some other aspect of metal–carbene complex chemistry, and the degree to which carbene complexes are discussed.

Many articles focusing on some aspect of carbene complex-initiated olefin metathesis were published, including the following specific subjects: (1) asymmetric molybdenum-catalyzed olefin metathesis [30,31]; (2) recent advances in high oxidation state molybdenum and tungsten imido alkylidene chemistry [32]; (3) synthesis by metathesis [33]; (4) ruthenium metathesis catalysts where the focus is on the carbene (RCH=) ligand [34]; (5) ruthenium-based olefin metathesis catalysts containing NHC ligands [35]; (6) polar ruthenium carbene metathesis catalysts bearing onium groups [36]; (7) aqueous olefin metathesis [37]; (8) development of aqueous olefin metathesis catalysts [38]; (9) latent olefin metathesis catalysts [39]; (10) well-defined silica-supported carbene complexes [40]; (11) well-defined polymer-supported metathesis catalysts [41]; (12) equilibrium ring closing metathesis [42]; (13) entropy-driven ring opening metathesis polymerization [43]; (14) metathesis and Suzuki couplings for “green” natural products syntheses [44]; (15) synthesis of aromatic compounds via RCM [45,46]; (16) synthesis of five- and six-membered ring heterocycles via metathesis [47]; (17) cross metathesis of acrylonitrile with fatty acid derivatives [48]; (18) metathesis using oleochemicals [49]; (19) end group functionalization of living polymers [50]; (20) use of enyne metathesis for the synthesis of macroheterocycles containing amino acid scaffolds [51]; (21) use of olefin metathesis for site-selective protein modification [52]; (22) use of metathesis for the preparation of natural product-like compound libraries [53]; (23) synergistic combination of enyne metathesis and Diels–Alder reactions [54]; (24) olefin metathesis [55]; and (25) cross metathesis over heterogeneous catalysts [56].

Several review articles report on synthesis of various compounds or compound classes where carbene complex initiated olefin metathesis is a commonly employed synthetic route. Specific compound classes represented include: (1) fully substituted cyclopentenones [57]; (2) 2,5-dihydro furans, pyrroles, and thiophenes [58]; (3) oxepins and benzoxepins [59]; (4) nitrogen heterocycles [60]; (5) spirocyclic compounds [61]; (6) spirocyclic ethers [62]; (7) illicium sesquiterpenes [63]; (8) stemona alka-

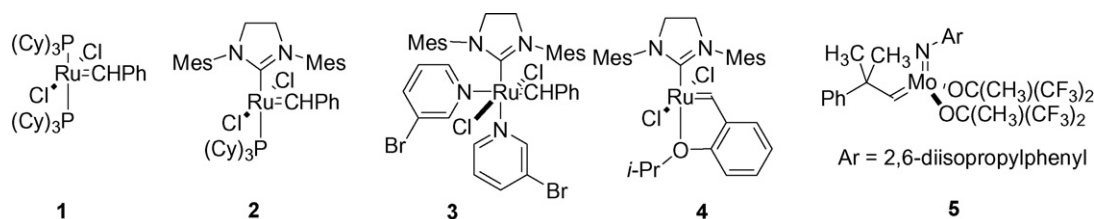


Fig. 1. Structures of alkene metathesis catalysts 1–5.

loids [64]; (9) dihydropyridine-2-carboxylate derivatives [65]; (10) cyclopentitols [66]; (11) cyclopropyl lactone oxylipins [67]; (12) abyssomycins [68]; (13) dolabelides [69]; (14) gem difluorinated heterocycles [70]; (15) unnatural amino acids [71]; (16) functionalized fatty acids [72]; (17) naturally occurring antibiotics [73]; (18) macrocyclic NHC complexes [74]; (19) himandrin [75]; (20) providencin [76]; (21) diverse molecular scaffolds [77,78]; (22) natural product inspired compound collections [79]; (23) cyclic phosphate esters [80]; (24) six-membered ring silacycles [81]; (25) planar chiral metallocenes [82]; (26) antimicrobial polymers [83]; (27) two-dimensional polymers [84]; (28) polymer/carbon nanotube composites [85]; (29) organic monoliths [86]; (30) acetylenic polymers [87]; and (31) polymers of 1,6-diynes [88].

Additional review articles include some segments on carbene complex-initiated metathesis. Articles in this category focus on the following subjects: (1) ruthenium-catalyzed olefin isomerization [89]; (2) microwave-assisted ruthenium-catalyzed reactions [90]; (3) use of chiral NHC complexes in asymmetric catalysis [91]; (4) site isolation of reactive intermediates [92]; (5) immobilized NHC metal complexes in catalysis [93]; (6) auto tandem catalysis [94]; (7) microwave enhanced synthesis [95]; (8) hydrolytic kinetic resolution in organic synthesis [96]; (9) hydrophilic ligands and their applications in aqueous-phase metal-catalyzed reactions [97]; (10) enantioselective catalysis [98]; (11) the synthetic accomplishments of the Nicolaou research group [99]; (12) new pathways in homogeneous catalysis [100]; (13) current challenges and future perspective on topological polymer chemistry [101]; (14) polymerization of species that feature metal–metal bonds [102]; (15) uses of rotaxanes and catenanes for sensing charged species [103]; (16) polymerization reactions in ionic liquids [104]; (17) orthogonal click chemistry in the synthesis of functional soft materials [105]; and (18) morphological studies of ADMET-derived polyolefins [106].

Several reviews on carbene complex chemistry featuring some aspect other than metathesis appeared in 2009. These reviews include the following subjects: (1) Fischer carbene complexes in organic synthesis [107]; (2) use of Group 6 Fischer carbene complexes for the synthesis of optically active molecules [108]; (3) late metal carbene complexes generated through multiple C–H activation processes [109]; (4) carbocyclic carbene complexes [110]; (5) development of catalytic reactions involving allenylidene–metal complexes [111]; (6) allenylidene and higher cumulenyliidene complexes [112]; (7) generation of carbene complexes as catalytic intermediates from alkyne starting materials [113]; (8) non-metathesis uses of Grubbs catalysts [114]; (9) ligating properties of anionic Fischer carbene complexes [115]; (10) synthesis by alkylidenation with metal–carbene complexes [116]; (11) C–H oxidation of methyl ethers via iridium carbene complex intermediates [117]; (12) abnormal NHC–metal complexes [118–120]; (13) niobium-catalyzed activation of ArCF_3 groups [121]; (14) uranium–carbon multiple bonds [122]; and (15) a tribute to the discoveries of Tebbe [123].

Although not specifically focusing on metal–carbene complexes, many review articles place some emphasis on this subject. Subjects reviewed in this category include: (1) platinum and gold-catalyzed

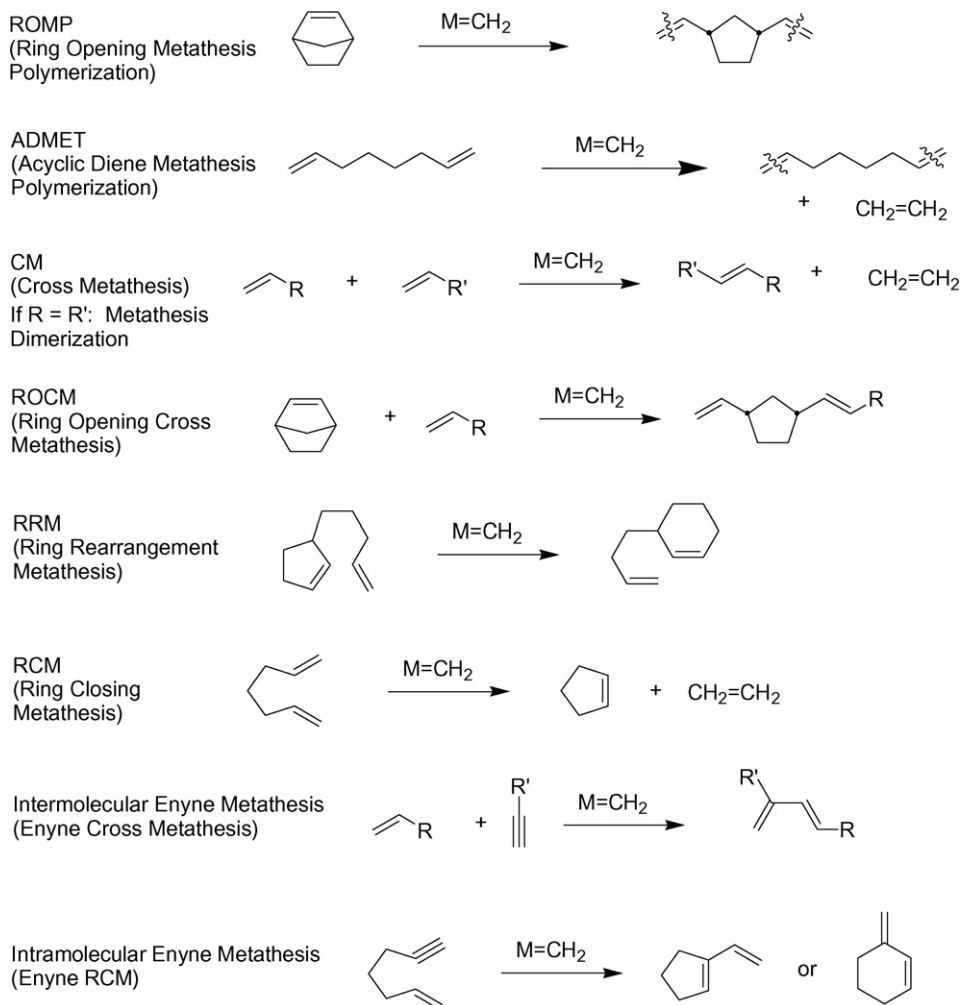
cycloisomerization of polyunsaturated compounds [124]; (2) silver and gold-catalyzed cycloisomerizations [125,126]; (3) transition metal catalyzed propargyl substitution reactions [127–129]; (4) CpRuCl and CpCo catalyzed alkyne trimerization [130]; (5) metal-catalyzed alkyne chemistry [131]; (6) conversion of C–F bonds α - to transition metals into C–H, C–C, and C–heteroatom bonds [132]; (7) aldehyde C–H activation [133]; (8) auto tandem catalysis [94]; (9) syntheses of indole and quinoline derivatives [134]; (10) atom transfer radical polymerization using ruthenium catalysts [135]; (11) the organic chemistry of phosphalkynes [136]; (12) chromium in organic synthesis [137]; (13) the role of electrophilic species in the Fischer–Tropsch reaction [138]; (14) aromaticity of six-membered rings containing one heteroatom [139]; and (15) constrained geometry ruthenium carboranyl complexes [140].

2.2. Alkene metathesis

Alkene metathesis was the most common reaction process reported for metal–carbene complexes in 2009, and this special section is devoted to papers that focus on this process. Many examples of both polymerization [mostly ring opening metathesis polymerization (ROMP)] reactions and small-molecule syntheses appeared. Only metathesis reactions initiated by a discrete transition metal–carbene complex or metathesis reactions that offer significant discussion of the carbene complex intermediates in this reaction have been included here. Distinct modes of alkene metathesis are depicted in Scheme 1.

2.2.1. General studies of alkene metathesis catalysts

Numerous attempts to develop new carbene complex catalysts for alkene metathesis were reported in 2009; some representative examples are depicted in Fig. 2. Also depicted in Fig. 2 are less developed catalysts for olefin metathesis. Several derivatives of the Grubbs and Schrock catalysts (see Fig. 1) were synthesized and tested in their ability to undergo either ROMP or RCM processes. The hexacoordinate bis(trifluoroacetate) analog of Grubbs catalyst I was reported [141]. Reported analogs of both Grubbs catalyst I and II include complexes featuring: (1) a thiophenyl group in place of the carbene phenyl group [142]; and (2) acac ligands in place of chloride ligands (e.g. **6**) (these complexes become active catalysts upon photoirradiation in the presence of $\text{Ph}_3\text{S}^+\text{Cl}^-$) [143]. Reported analogs of Grubbs catalyst II include complexes featuring: (1) indenylidene ligands [144]; (2) indenylidene ligands and sterically hindered NHC ligands [145]; (3) chromanylidene ligands and studies of the carbene–Ru bond rotation [146]; (4) syn- and anti-methyl groups on the NHC ligand and *o*-tolyl groups in place of mesityl groups (e.g. **7**), and evaluation of the relative activities of complexes featuring syn and anti tolyl groups (the syn compound was a particularly effective catalyst for formation of tetrasubstituted alkenes) [147]; (5) chiral NHC ligands [148]; (6) molecular recognition units connected to the carbene phenyl group (e.g. **8**) that recognize chain terminators for the control of ROMP reactions [149]; and (7) analogs of Grubbs catalyst II bound to a silica support through the aryl group of the NHC ligand [150]. Reported analogs of both Grubbs catalyst II and the Hoveyda–Grubbs cata-



Scheme 1.

lyst include complexes featuring: (1) replacement of the mesityl groups of the NHC ligand by substituted naphthyl groups [151] or bulkier aryl groups, which allows for catalyst removal by nanofiltration [152]; and (2) covalent attachments to imidazolium salts [153]. Reported analogs of the Hoveyda–Grubbs catalyst include compounds that feature: (1) a seven-membered ring NHC ligand (e.g. **9**) [154]; (2) covalent attachments to imidazolium salt groups for metathesis in ionic liquids [155,156]; (3) diisopropylphenyl groups in place of the NHC mesityl groups (also features a carbene phenyl ring containing a trifluoroacetamido group) [157]; (4) conformationally restricted naphthyl groups in place of the NHC mesityl groups (e.g. **10**) (anti complexes are better catalysts) [158]; (5) *p*-dimethylaminoaryl groups in place of the NHC mesityl groups (the neutral form is the better ROMP catalyst) [159]; (6) replacement of the chelating oxygen by the sulfur of a sulfoxide group (e.g. **11**) [160] or a simple thioether group (e.g. **12**) (these complexes become good metathesis initiators upon photolysis due to *cis trans* isomerization of the chloride ligands) [161,162]; (7) replacement of the chloride ligands by silyloxy ligands [163]; chelating carboxylate ligands (e.g. **13**) (which become good catalysts in an ionic liquid) [164], or thiocyanates or isocyanates [165]; (8) covalent linkage to amphiphilic components (e.g. **14**) for RCM in brine solutions [166]; (9) a diverse array of NHC groups in place of H_2IMes_2 [167]; (10) a ferrocenyl group attached to the carbene phenyl ring, which dissolves in ionic liquids upon oxidation of the ferrocene group [168]; and (11) a covalent attachment to pyrenyl groups, which

can be immobilized through association with carbon nanotubes [169]. A diverse library of Hoveyda–Grubbs catalyst analogs was reported [170]. Several reports involving formation of heterogeneous analogs of the Hoveyda–Grubbs catalyst appeared in 2009, including complexes featuring covalent linkages to: (1) zirconium phosphate [171]; (2) magnetic nanoparticles [172,173]; (3) silica [174]; (4) polyethylene glycol groups [175]; and (5) polyisobutylene groups [176]. Reported analogs of Grubbs catalyst III include compounds featuring: (1) DMAP ligands in place of pyridine ligands (e.g. **15**), which can be activated by phosphoric acid or deactivated by 1-methylimidazole and is useful for molecular weight control in polymerizations [177]; (2) primary amines in place of the pyridine ligands [178]; and (3) pyridine ligands covalently connected to imidazolium groups [179]. Additional ruthenium carbene complex metathesis catalysts were reported, including: (1) bis(NHC) ruthenium metathesis catalysts (e.g. **16**) [180]; (2) ruthenium carbene complexes featuring unsymmetrical NHC ligands and pyridine ligands (e.g. **17**) [181]; (3) η^6 -arene–ruthenium NHC complexes that feature no additional carbene ligands [182]; (4) indenylidene–ruthenium complexes featuring a chelating salicylimide ligands (e.g. **18**) [183]; and (5) a diruthenium indenylidene complex (e.g. **19**) [184]. New catalysts featuring metals other than ruthenium include: (1) siloxane-bound analogs of the Schrock catalyst [185]; and (2) a diaryloxy imido alkylidene tungsten complex [186]. Additional examples of the design and synthesis of asymmetric molybdenum metathesis catalysts are presented

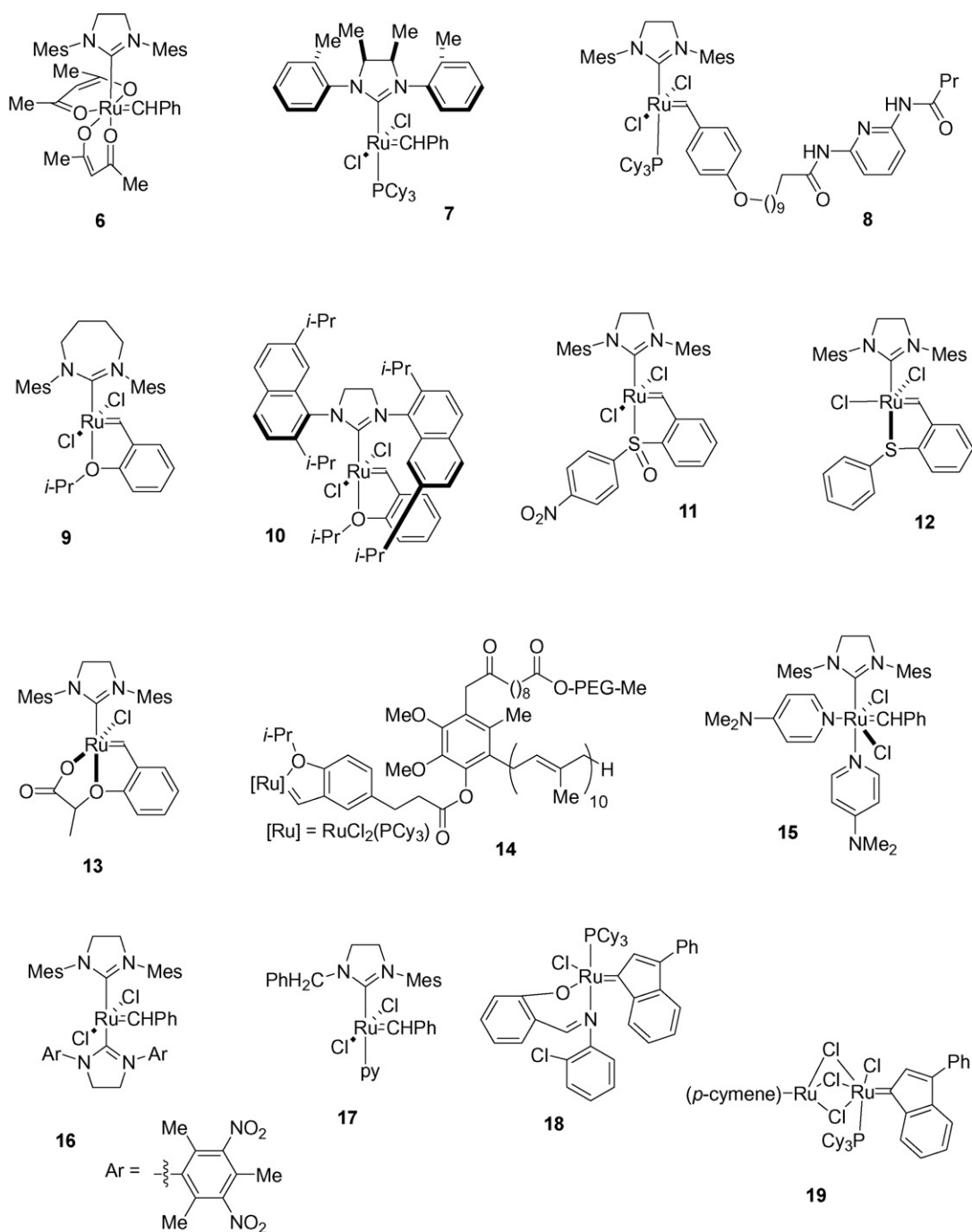


Fig. 2. Representative examples of new catalysts for alkene metathesis.

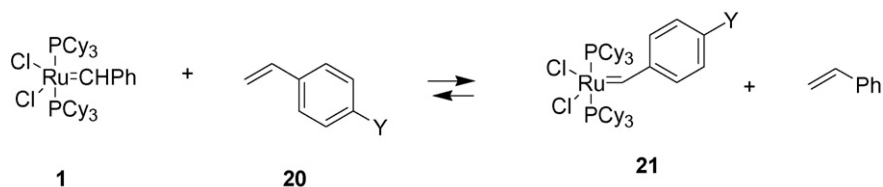
ahead in this section. A bis(trimethylsilylmethyl)imidovanadium complex was an effective ROMP catalyst in the presence of PMe_3 , however attempts to isolate the alkylidene complex were unsuccessful [187]. The structure of aryloxy analogs of Grubbs catalyst II were studied by NMR and computational techniques [188]. Several patents were issued for the synthesis and development of metal–carbene containing olefin metathesis catalysts [189–194].

The equilibrium constants for the reaction of various mono-substituted alkenes and Grubbs catalyst I (**1**) were determined (see Scheme 2) [195]. In the reaction with *p*-substituted styrene derivatives (**20**), the reaction involving styrenes substituted by electron-donating groups was exergonic and that involving elec-

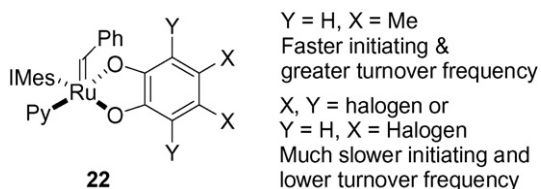
tron deficient substituents was endergonic. The reaction with alkyl-substituted alkenes was controlled by sterics. Only ethylene and propene react exergonically with Grubbs catalyst I.

The correlation between anionic ligand donor ability and metathesis activity was determined for ruthenium metathesis catalysts of general structure **22** (Scheme 3) [196]. The catalysts that feature electron donating aryl substituents undergo initiation at the fastest rate and feature higher turnover frequencies. The higher activity of the electron rich catalyst systems was attributed to decreasing bond strength to the pyridine ligand, which was determined through DFT calculations.

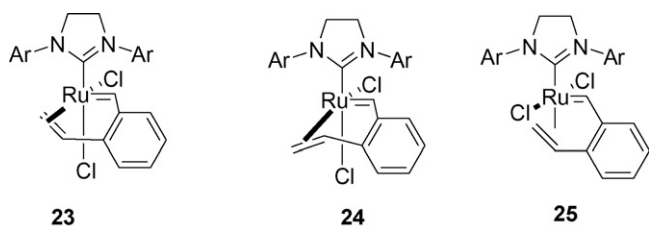
The conformational preferences in ruthenium carbene complexes that feature carbene–alkene chelate ligands (**23–25**,



Scheme 2.



Scheme 3.



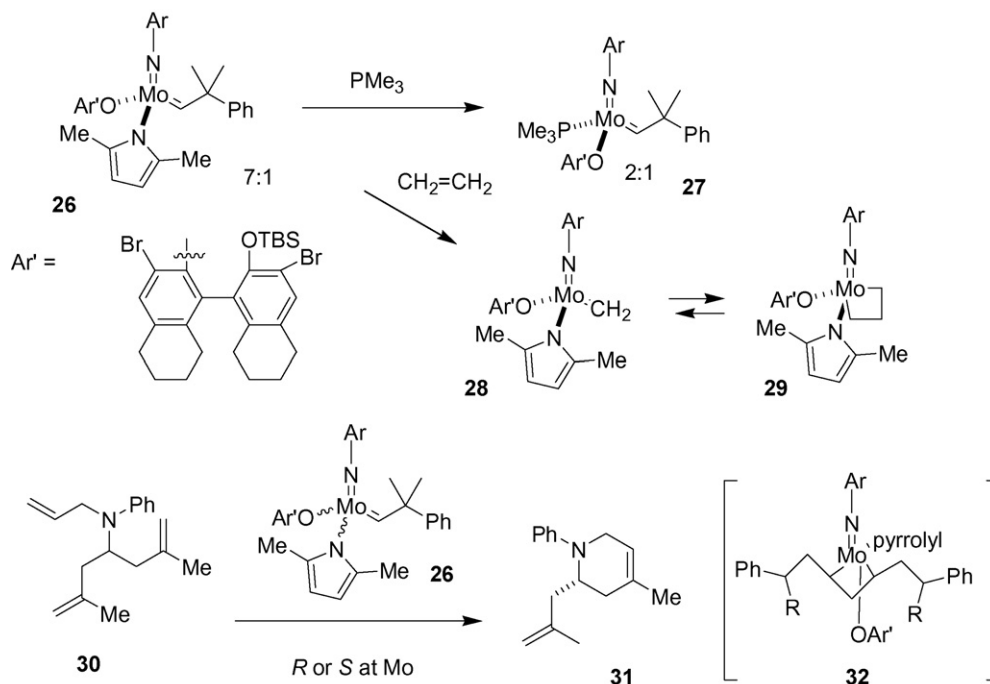
Ar = 2,6-diisopropylphenyl

Scheme 4.

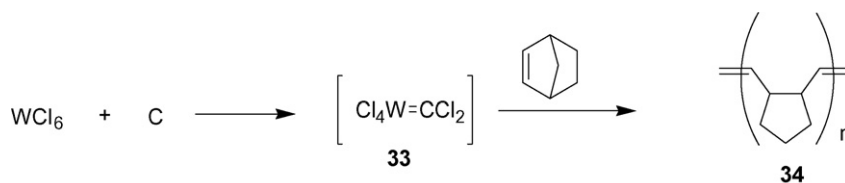
Scheme 4) were evaluated computationally [197]. The focus was on the optimal method to evaluate the relative energies of various conformers by DFT calculations. The M06 method achieved a closer fit to the experimental observations, where conformation **23** is favored over the other two.

The analysis of the bonding of the NHC ligand in Grubbs catalyst II by electron deletion analysis (EDA) revealed that there is significant backbonding by the NHC ligand and that the stronger bonding by the NHC ligand relative to the phosphine ligand is due to a combination of forward and back donation [198]. The NHC ligand was determined to be a poorer sigma donor relative to the phosphine ligand.

Many reports highlight efforts to improve the synthesis, develop new catalysts, and understand the mechanisms for tungsten- and molybdenum-catalyzed olefin metathesis. These studies are depicted in Scheme 5. The configurational stability for chiral-at-molybdenum analogs of the Schrock catalyst (e.g. **26**) was examined [199]. Compound **26**, which also contains a chiral monodentate ligand, catalyzes asymmetric metathesis reactions even when diastereomerically impure. Predominantly inversion of the configuration at molybdenum was observed in the formation of complex **27** through addition of phosphine, which has been attributed to Berry pseudorotation in a five-coordinate intermediate, which in some cases can be isolated. Reaction with ethylene leads to mixtures of the $=\text{CH}_2$ carbene complex (**28**) and the metallacyclobutane (**29**), in a different stereoisomer ratio from the starting complex. Both enantiomers (at molybdenum) of complex **26** show the same direction of enantioselectivity in asymmetric RCM reactions [200]. This phenomenon was attributed to a degenerate metathesis mechanism via a symmetrical (at metallacyclobutane) intermediate (**32**). Tungsten analogs of complex **26** were similarly prepared and their reactivity examined [201].



Scheme 5.



Scheme 6.

Other general studies of alkene metathesis where carbene complexes were discussed include: (1) formation of a metathesis catalyst (**33**, Scheme 6) through reaction of atomic carbon with WCl_6 or $MoCl_6$, followed by ROMP of norbornene [202]; (2) use of polystyrene-bound salicylimines as metal scavengers for molybdenum metathesis reactions (the scavenging process is due to phenoxide ligand replacement) [203]; (3) activation of metathesis reactions via various calixarene additives (calixarenesulfonates are best) [204]; (4) use of Grubbs-catalyst site-isolated polymethyl-disiloxane thimbles for alkene metathesis and syn hydroxylation in a single reaction vessel [205]; (5) use of NHC- CO_2 adducts as sources of NHC ligands for olefin metathesis catalysts [206]; (6) use of a vinyl ether diethylene glycol derivative to deactivate metathesis catalysts [207]; (7) a comparison of mononuclear and binuclear ruthenium metathesis catalysts [208]; (8) monitoring the progress of the ROMP of dicyclopentadiene by differential scanning calorimetry [209]; (9) enhancement of metathesis reactions using second-generation ruthenium complexes and aromatic solvents [210]; (10) determination of microstructural parameters for ROMP reactions in ionic liquids initiated by either Grubbs catalyst I or $[(p\text{-cymene})(PCy_3)Ru=C=C=CPh_2]^+$ [211]; (11) a comparison of FT-Raman and 1H NMR for the determination of ROMP kinetics [212]; (12) use of metathesis reactions to determine double bond position in linear alkenes [213]; (13) ROMP using $[(\mu\text{-}Cl)_2\{Mo(\mu\text{-}Cl)(SnCl_3)(CO)_3\}_2]^{2-}$ and discussion of initial carbene complex generation [214]; (14) ROMP using $Nb(OOCNMe_3)_5$ and MAO, and discussion of initial carbene complex formation [215]; (15) X-ray photoelectron spectra of various ruthenium carbene complex metathesis catalysts [216]; (16) use of mechanical processes to activate ruthenium metathesis catalysts [217,218]; (17) an experimental and computational study of preferences for cis vs. trans disposition of chloride ligands in analogs of the Hoveyda–Grubbs catalyst where other elements replace the oxygen chelate [219]; (18) combined experimental and computational studies of conformation preferences for analogs of the Hoveyda–Grubbs catalyst featuring *o*-tolyl groups in place of mesityl groups, and for olefin chelates [220]; (19) a computational study of metathesis reactions of cis 1,4-diacetoxy-2-butene and revelation that oxygen coordination of a carbene complex intermediate is a key driving force [221]; (20) a computational study of metatheses of fluorinated alkenes [222]; (21) a computational study of the ring closure efficiency for RCM reactions [223]; (22) a computational study of molybdenum-catalyzed cross metathesis of acrylonitrile and propene [224]; (23) surface XPS and vibrational spectra of $\beta\text{-}Mo_2C$ surface (a known metathesis catalyst system) after exposure to various alkenes and detection of alkylidene intermediates [225]; (24) a computational study of olefin metathesis using methylene $Mo(VI)$ complexes and methylene $Mo(V)$ complexes (models for a $Mo/H\beta$ catalyst system) [226]; (25) a computational evaluation of a [2+2] followed by retro-[2+2] cycloaddition route for the formation of methylene-molybdenum complexes from molybdenum oxo complexes and ethylene [227]; (26) a computational study that focuses on C–C bond agostic interaction in titanium metallacyclobutanes that are viable metathesis intermediates [228]; (27) a computational study of 1-octene self metathesis using $[RuCl_2(Phobcat)_2(=CHPh)]$ [229]; (28) a computational study of the metathesis steps for methylene-

molybdenum complexes bound to a zeolite HZSM-5 model system [230]; (29) use of the SCSC-MP2 method to estimate the energetic of reactive intermediates of ruthenium-catalyzed metathesis [231]; and (30) an ESI-MS study of olefin metathesis initiated by Grubbs catalyst I and DFT studies of alkali metal cation adducts with this catalyst [232]. The use of multiple modes of metathesis in the construction of diverse compound libraries was reported [233]. A patent was issued for a method to reactivate metathesis catalysts [234].

2.2.2. Polymerization reactions

Initiation of the ring opening metathesis polymerization (ROMP) (see Scheme 1) reaction using carbene complexes remains a very active area of investigation. The strained alkene norbornene, norbornene derivatives, and copolymerization involving a norbornene derivative and another alkene accounted for a large fraction of all reports of the ROMP reaction in 2009; representative monomers are depicted in Fig. 3. Numerous substituted norbornenes have been subjected to ROMP using metal carbene complexes, including those possessing the following structural features: (1) norbornene in aqueous environments [235]; (2) norbornenes attached to xanthen groups [236]; (3) norbornenes attached silole groups (e.g. **37**) [237]; (4) silylated norbornenes [238] and norbornadienes (e.g. **38**) [239]; (5) norbornenes connected to radical scavenger groups [240]; (6) norbornenes connected to dihydroimidazolium salts [241]; (7) norbornenes connected to ammonium salts (e.g. **39**) [242]; (8) norbornenes connected to guanidine or chloroacetamide groups [243]; (9) norbornenes attached to a triacylglyceride [244–247]; (10) norbornenes linked to gold nanoparticles [248]; (11) norbornenes connected to carbon nanotubes [249] and copolymerizations with ethylenenorbornene [250,251]; (12) norbornenes connected to oligomeric siloxanes [252]; (13) norbornenes connected to polystyrenes or polystyrene–polylactate block polymers [253–255]; (14) a ferrocene-fused norbornene derivative (e.g. **40**) [256]; (15) norbornylmethyl esters [257]; (16) norbornenecarboxylate esters connected to epoxide groups [258], substituted carbazoles (e.g. **41**) [259], azobenzene groups [260], and succinimide groups [261]; (17) bis(norbornenecarboxylate) diesters spaced through a xanthene group [262]; (18) norbornenedicarboxylic acid [263]; (19) norbornene dicarboxylate esters [264–267]; (20) norbornenedicarboxylate esters connected to piperidine groups [268] or unprotected amine groups (e.g. **42**) [269]; (21) norbornene dicarboxylate esters bound to a gold surface and copolymerization with perfluoroalkylnorbornenes [270]; (22) norbornenecarboxamides [271]; (23) norbornene dicarboxamide derivatives derived from α -amino acids [272]; (24) N-substituted norbornenesuccinimides [273,274]; (25) norbornenesuccinimides attached to ammonium ions [275], porphyrins or fullerenes [276], piperazine rings [277]; or N-fluoroaryl groups [278]; (27) norbornenesuccinimides followed by end-capping through cross metathesis with an alkene containing an α -bromopropionate group [279,280]; (28) norbornenesuccinimides bound to functionalized polymers via click reactions (e.g. **43**) [281,282]; (29) norbornenesuccinimides already bound to a ROMP polymer (e.g. **44**) [283]; (30) peptide-bound norbornenesuccinimide deriva-

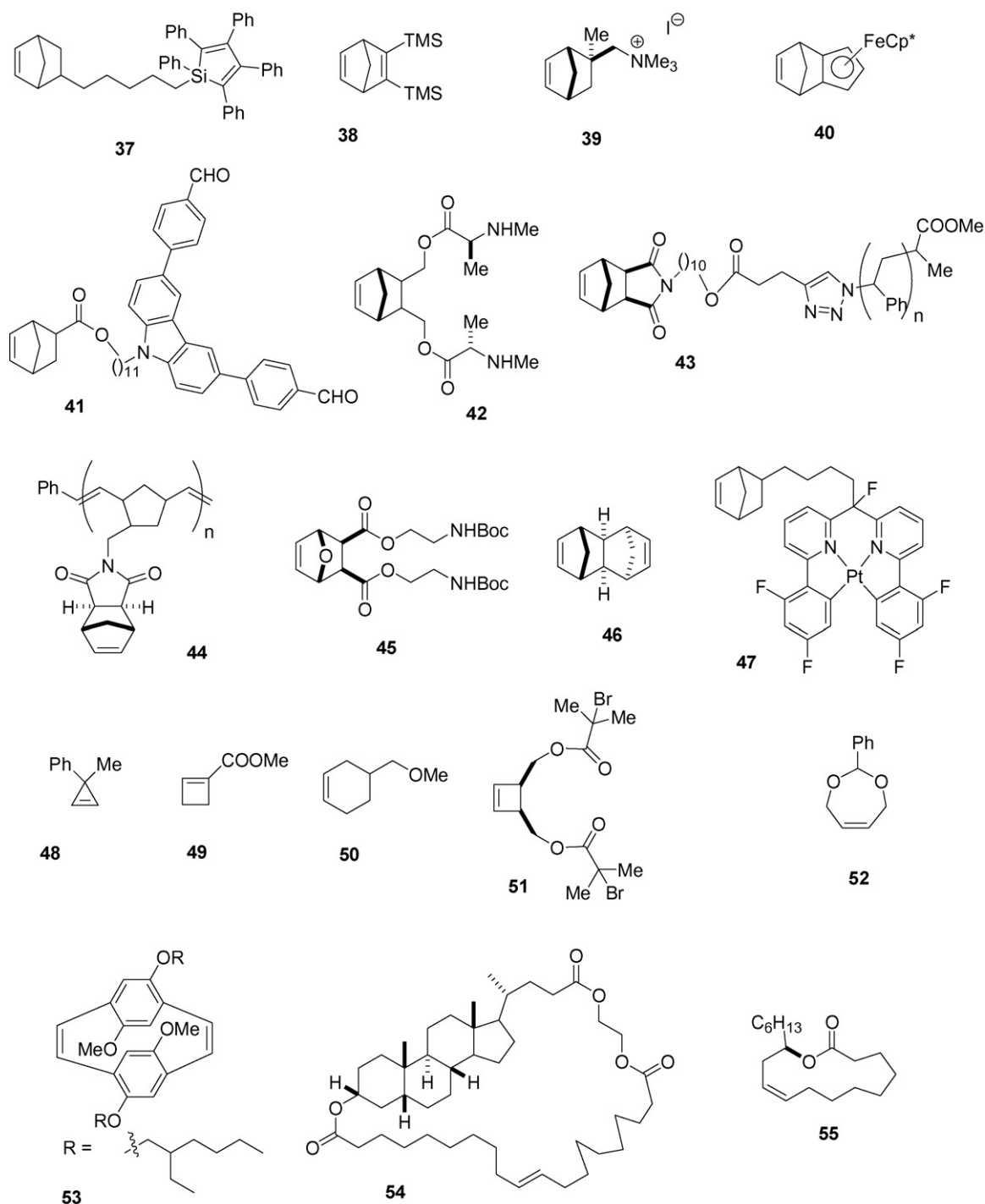
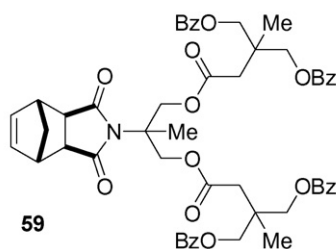
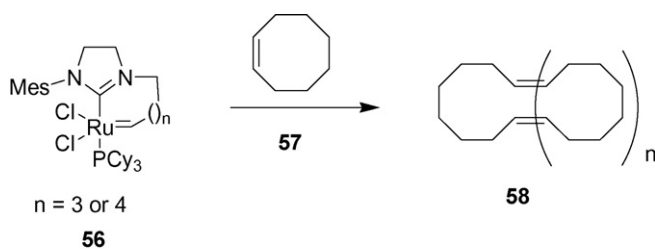


Fig. 3. Representative substrates for the ROMP reaction.

tives [284]; (31) norbornadiene [285]; (32) cis selective ROMP of norbornadiene diesters or cyclooctene using a nonchelating chiral-at-molybdenum complex **26** (see Scheme 5) [286]; (33) oxanorbornenes [287]; (34) aminoethyl esters of oxanorbornenedicarboxylic acid (e.g. **45**) [288]; (35) oxanorbornenedicarboxylate esters connected to TEMPO radical groups [289]; (36) oxanorbornene succinimides connected to α -bromo ester groups [290]; (37) dicyclopentadiene [291,292]; (38) dicyclopentadiene as part of self-healing materials [293]; (39) dicyclopentadiene followed by chain termination via cross metathesis with terpene alkenes [294]; (40) a doubly bridged tetrahydronaphthalene derivative (e.g. **46**) [295–297]; and (41) norbornene connected to a chelating bis(aryl)

platinum complex (e.g. **47**) [298]. Other ring systems that have been subjected to ROMP reactions include: (1) cyclopropenes (e.g. **48**) (and copolymers with norbornenes) and evaluation of the polymerization progress by MALDI [299]; (2) cyclobutene esters (e.g. **49**) and cyclohexenes (e.g. **50**) [300]; (3) cyclobutenes containing remote α -bromo ester groups (e.g. **51**) (either ROMP first or radical polymerization occurring first) [301,302]; (4) cyclooctene [303]; (5) trans cyclooctene [304]; (6) 1,5-cyclooctadiene [305]; (7) cyclooctatetraene [306]; (8) 1,3-dioxepines (e.g. **52**) [307] and sulfur analogs [308]; (9) *p*-cyclophanes (e.g. **53**) [309–311]; and (10) bridged steroids (e.g. **54**) and copolymers with macrocyclic lactones (e.g. **55**) [312]. ROMP polymerization using RuCl₂(Ph₂PBn)L₂



Scheme 7.

complexes alone or with ethyl diazoacetate as an additive (L =piperidine, isonicotinamide, or nicotinamide) was reported [313]. Pulsed addition ROMP was accomplished using Grubbs catalyst III and periodic addition of *cis* 3-hexene to temporarily halt chain growth [314]. Simultaneous or sequential ROMP and atom-transfer radical polymerization was reported [315–318]. The ROMP of cyclooctene was catalyzed using surface-bond ruthenium carbenes generated through cross metathesis [319]. Formation of a diester-linked bis(carbene–ruthenium) complex through cross metathesis followed by use of the *in situ*-generated carbene complex to initiate ROMP of norbornene esters was reported [320]. A ROMP–capture–release strategy for separation of norbornene from non-norbornene containing substances was developed [321]. In the key step of this process, norbornene-containing mixtures are treated with benzene-linked bis(norbornenes) and a ruthenium carbene complex catalyst to form cross-linked copolymers with the norbornene species.

Polymerization of strained alkenes using cyclic carbene complexes (e.g. **56**, Scheme 7) affords cyclic polymers (e.g. **58**). The process has been termed ring expanding metathesis polymerization, or REMP. The relative efficiency of the two catalysts denoted by structure **56** for REMP of cyclooctene was determined [322]. The complex where $n=3$ was determined to be a better initiator and polymerization is through a step growth mechanism. Polymerization using the $n=4$ system was via a chain growth mechanism and resulting in a faster propagation rate and higher molecular weight products. The use of these catalysts to form cyclic dendrimers from norbornene derivative **59** was also reported [323]. Treatment of salen-bound cyclooctenes with Grubbs catalyst III led to cyclic polymers [324].

Several examples using carbene complexes to initiate acyclic diene metathesis polymerization (ADMET, see Scheme 1) were reported. Monomers subjected to ADMET polymerization are depicted in Fig. 4 and include: (1) α,ω -dienes featuring an alkyl group in the tether (e.g. **60**) [325–327], a phosphonate ester group (e.g. **61**) [328], or a benzenesulfonate ester group [329]; (2) 6,6-dimethyl-1,10-undecadiene [330]; (3) alkene-bis(acrylate) esters (e.g. **62**) (and subsequent CM of the unreacted ester group) [331]; (4) hydroquinone-diester spaced α,ω -dienes [332]; (5) 2,5-divinylthiophene derivatives (e.g. **63**) [333,334]; (6) bis(amide)-linked α,ω -dienes [335]; (7) bis(urea)-spaced α,ω -dienes [336]; (8) branched bis(amino acid) spaced α,ω -tetraenes and octa-enes [337]; (9) carbazole-spaced α,ω -dienes (e.g. **64**) [338]; (10) carbohydrate derived dienes (e.g. **65**) [339,340]; (11)

siloxane-spaced α,ω -dienes [341]; (12) a disilacyclobutane-spaced bis(styrene) (e.g. **66**) [342]; (13) a triene derivative containing an azobenzene spacer group (e.g. **67**) [343]; and (14) an α,ω -diene spaced through silyl, ester, and biphenyl groups (e.g. **68**) [344]. Both ADMET polymerization and macrocyclic RCM were demonstrated for bis(alkene–Cp) dimolybdenum complexes (e.g. **69**) [345]. Scrambling of alkene-containing polymers via an ADMET-retro-ADMET process was reported [346].

2.2.3. Nonpolymer-forming ring opening metathesis reactions

Several examples of RO-CM (see Scheme 1) were reported in 2009. Representative examples are depicted in Scheme 8. Co-metathesis of cyclobutene derivative **72** and methallyl alcohol (**73**) proceeded regioselectively to afford diene **74** [347]. The co-metathesis of cyclohexene and 1,4-dichloro-2-butene was reported [348]. The reaction of highly strained cyclobutene **75** and ethylene led to divinyl β -lactam derivative **76** [349]. The RO-CM reaction of bicyclic amide **77** and ethylene to afford **78** was a key step in the total synthesis of alkaloid 223A [350]. Electronic control of regioselectivity was demonstrated for RO-CM reactions of **79** with alkenes featuring electronically diverse substituents (**80**) [351]. The co-metathesis of cyclooctene and halogenated ethylene derivatives was reported using either the Hoveyda–Grubbs or 14-electron carbene complex-phosphonium salts [352]. Successful cross metathesis reactions were also demonstrated using these catalyst systems. In several cases the thermally unstable dimeric carbene complex **84** could be isolated from the reaction. A bidirectional (double) RO-CM (or tandem RO-CM/CM) was observed in the reaction of cycloheptene **85** with a large excess of ethyl acrylate [353]. The RO-CM of cyclopropene **48** and chiral alkenol derivative **87** using was highly stereoselective [354]. Stereocontrol was attributed to H-bonding to a chloride ligand in a metallacyclobutane intermediate. Several examples of enantioselective RO-CM reactions using chiral molybdenum carbene complexes (e.g. **26**, Scheme 9) were also demonstrated [355]. The reaction depicted was also *Z* selective. This same catalyst proved to be a superior for RO-CM and simple cross metathesis reactions involving ethylene [356].

2.2.4. Cross metathesis and metathesis-dimerization reactions

Many examples employing carbene complexes to initiate the cross metathesis (see Scheme 1) of various dissimilar alkenes (usually monosubstituted) were reported in 2009. Representative examples are depicted in Fig. 5. Regrettably it is very difficult to rationally organize these reactions. Several papers report on the cross metathesis of inexpensive low molecular weight electron-deficient alkenes with other more precious alkenes, including cross metatheses of: (1) diallylnickelocenes (**93**) and various monosubstituted alkenes [357]; (2) various electron-deficient ethylene derivatives and 3-butenyl esters of β -ketoacids (e.g. **94**) [358]; (3) an *N*-butenylpyrrolidinone derivative and acrolein for total synthesis of grandisines B, D, and F [359]; (4) 1-octen-3-ol and acrolein [360]; (5) homoallylic alcohol esters and acrolein or ethyl acrylate, which afford the dienoyl systems upon treatment of crude reaction mixtures with base [361]; (6) acrylic acid and an allylic ester for total synthesis of sporostatin [362]; (7) an allylcyclohexenone and ethyl acrylate for total synthesis of lyconadin A [363]; (8) allylamine derivatives and ethyl acrylate [364]; (9) 1,8-nonadien-5-one (**95**) and two moles of methyl acrylate for total synthesis of alkaloid *cis*-223B [365,366]; (10) various C-allylglycosides and methyl acrylate [367]; (11) a homoallylic alcohol and methyl acrylate for total synthesis of pyranone natural products [368]; (12) a homoallylic alcohol derivative and methyl acrylate [369]; (13) a 1-hepten-6-yne derivative and methyl acrylate for preparation of a brasilinolide segment [370]; (14) an allylindole derivative and methyl acrylate for preparation of the strychnos alkaloid skele-

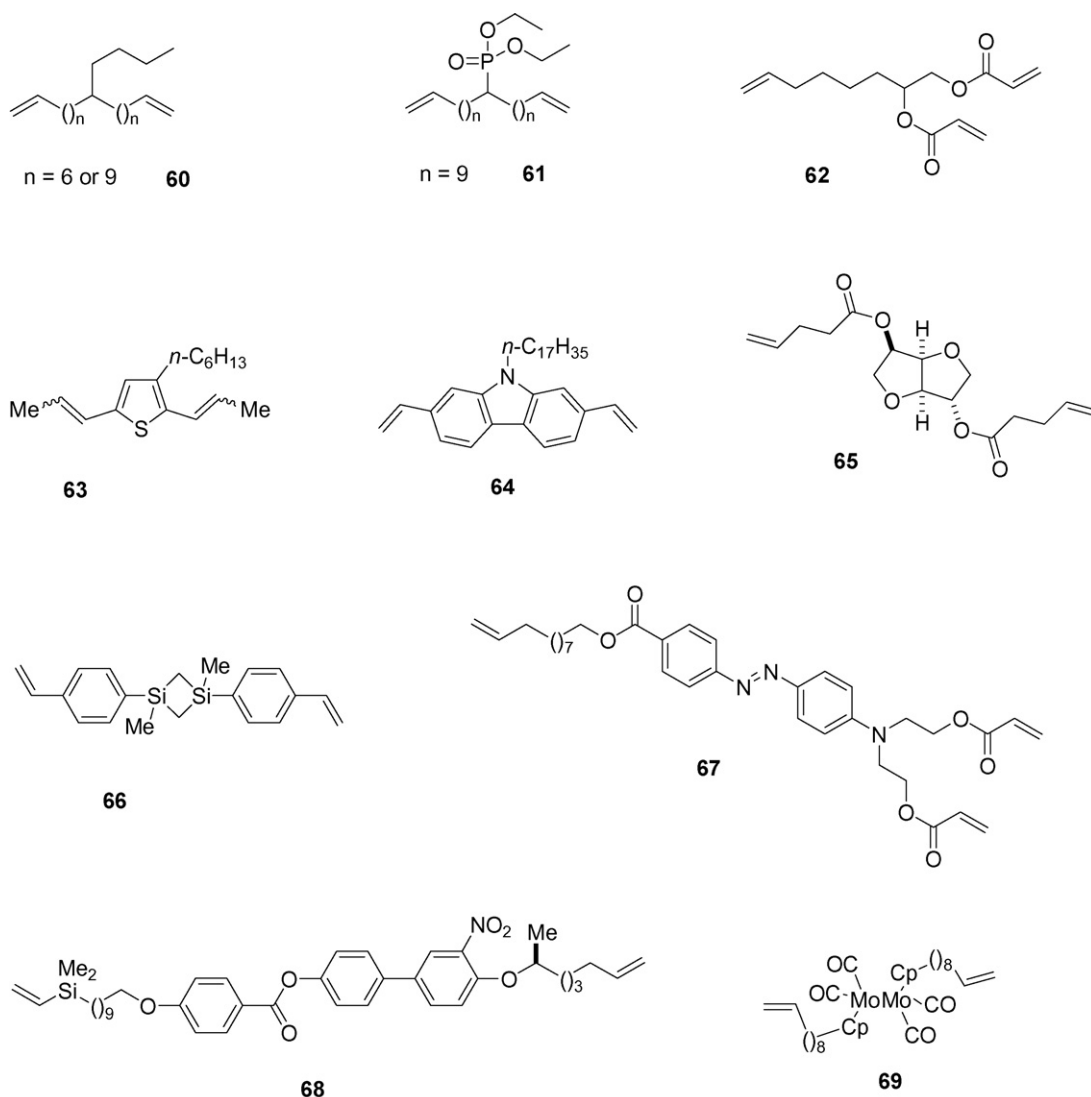
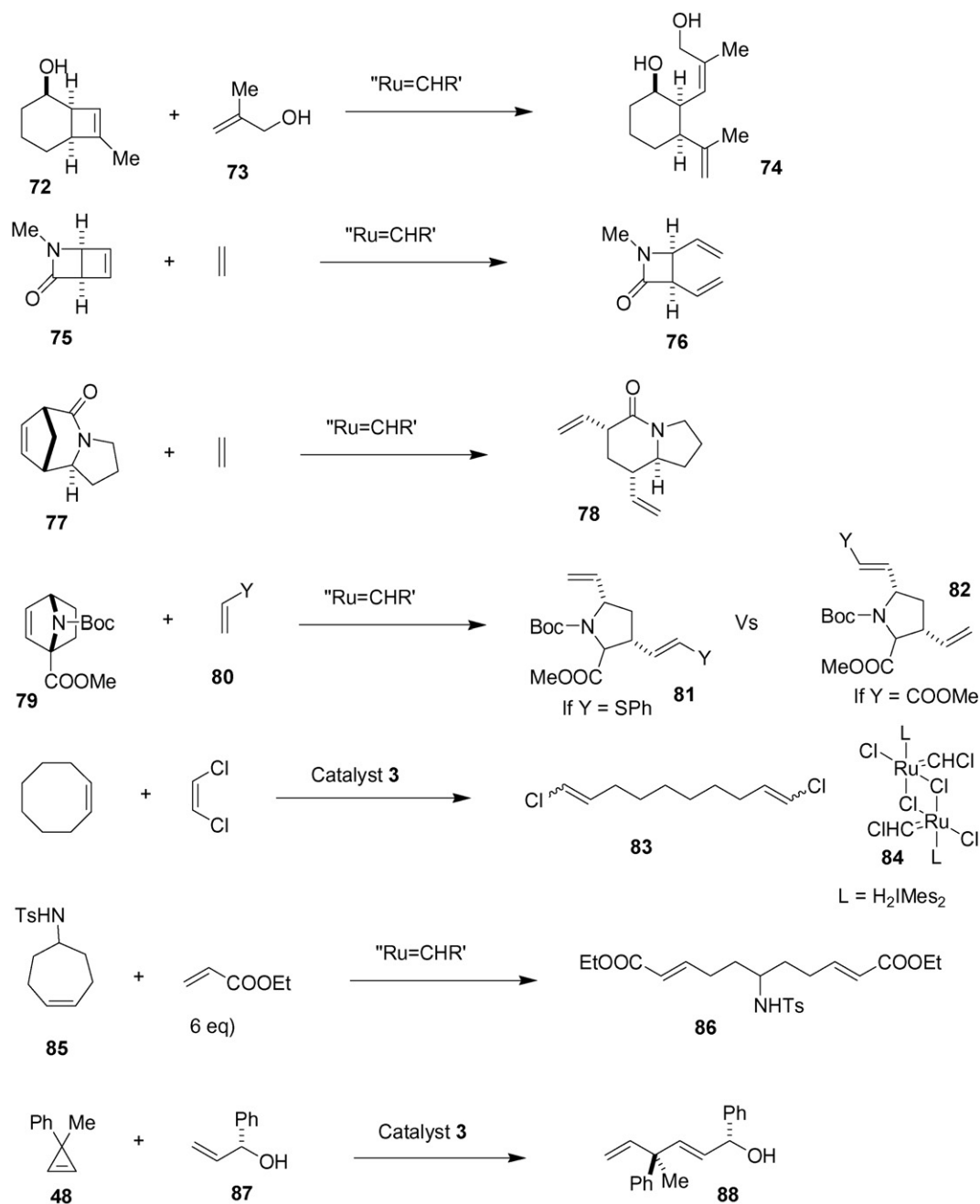


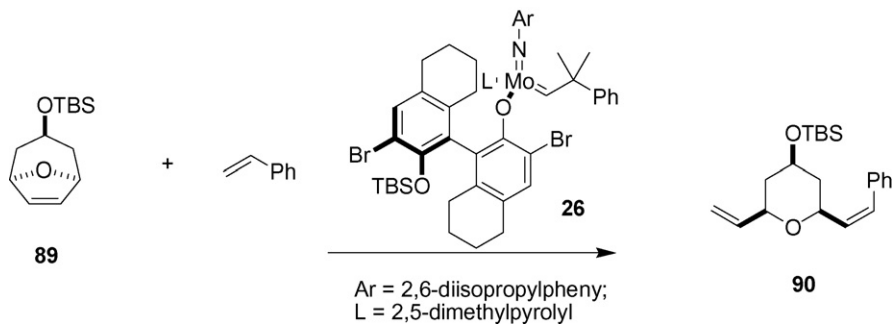
Fig. 4. Representative substrates for ADMET polymerization.

ton [371]; (15) ethyl acrylate and various functionalized allyl derivatives [372]; (16) homoallylic alcohol derivatives and *t*-butyl acrylate for total synthesis of guadinomic acid [373] and negamycin [374]; (17) a homoallylic ether derivative and methyl acrylate for total synthesis of goniothalesdiol A [375]; (18) various acrylate esters and an alkene-connected to a rotaxane structure [376]; (19) alkene-containing phosphinic acid esters and *t*-butyl acrylate [377]; (20) a porphyrin featuring four vinyl groups and methyl acrylate [378]; (21) failed cross metathesis of ethyl acrylate and an alkene-quinoline derivative [379]; (22) various monosubstituted alkenes and acryloyl chloride [380]; (23) acrylonitrile and various alkenes [381]; (24) a homoallylic alcohol derivative and methyl vinyl ketone for total synthesis of 11-methoxycurcularins [382]; (25) a homoallylic tosylate and methyl vinyl ketone for total synthesis of isosolenopsin [383]; (26) a homoallylic amine derivative and methyl vinyl ketone [384]; (27) a homoallylic alcohol derivative and phenyl vinyl ketone for total synthesis of azadiospongins A [385]; (28) a lactone-alkene and a vinyl ketone for total synthesis of rugulactone [386]; (29) Morita–Bayliss–Hillman derived α,β -unsaturated esters and various homoallylic alcohol derivatives [387]; and (30) crotonaldehyde and a diene-ester for total synthesis of etnangien [388]. Several examples employing the cross metathesis of simple inexpensive low molecular weight

functionalized allyl derivatives with more precious alkenes were reported in 2009, including: (1) allylthioprotein derivatives and allyl alcohol [389]; (2) methyl 2,4-hexadienoate and allyl bromide for total synthesis of FR252921 and pseudotrienic acid B [390]; (3) a complex macrocyclic lactone and 1,4-diacetoxy-2-butene for total synthesis of spirastrellolide F methyl ester [391]; (4) vinylpurines and allylmenthol [392]; and (5) a homoallylic alcohol and allyl methyl carbonate for total synthesis of spruce alkaloids [393]. Several examples employing the cross metathesis of simple allylsilane or vinylsilane derivatives with more precious alkenes were reported in 2009, including: (1) an allylglycine derivative and allyltrimethylsilane [394]; (2) various styrene derivatives and monovinylsilsequioxane derivatives [395]; and (3) various *p*-vinylbiphenyl derivatives and vinylsilsequioxane [396]. Several examples employing relatively abundant natural products in cross metathesis reactions were reported in 2009, including (1) methyl ricinoleate and methyl acrylate [397]; (2) methyl oleate and ethylene [398]; (3) fatty acids/esters and acrylonitrile or fumaronitrile [399]; (4) cardanol and vinylated porphyrins [400]; and (5) fatty acid esters and allyl chloride [401]. Other examples of cross metatheses where one of the partners is simple and/or readily available include: (1) 1-pentadecene (or other long chain alkene) and an alkene-aminodiol derivative for of total synthe-



Scheme 8.



Scheme 9.

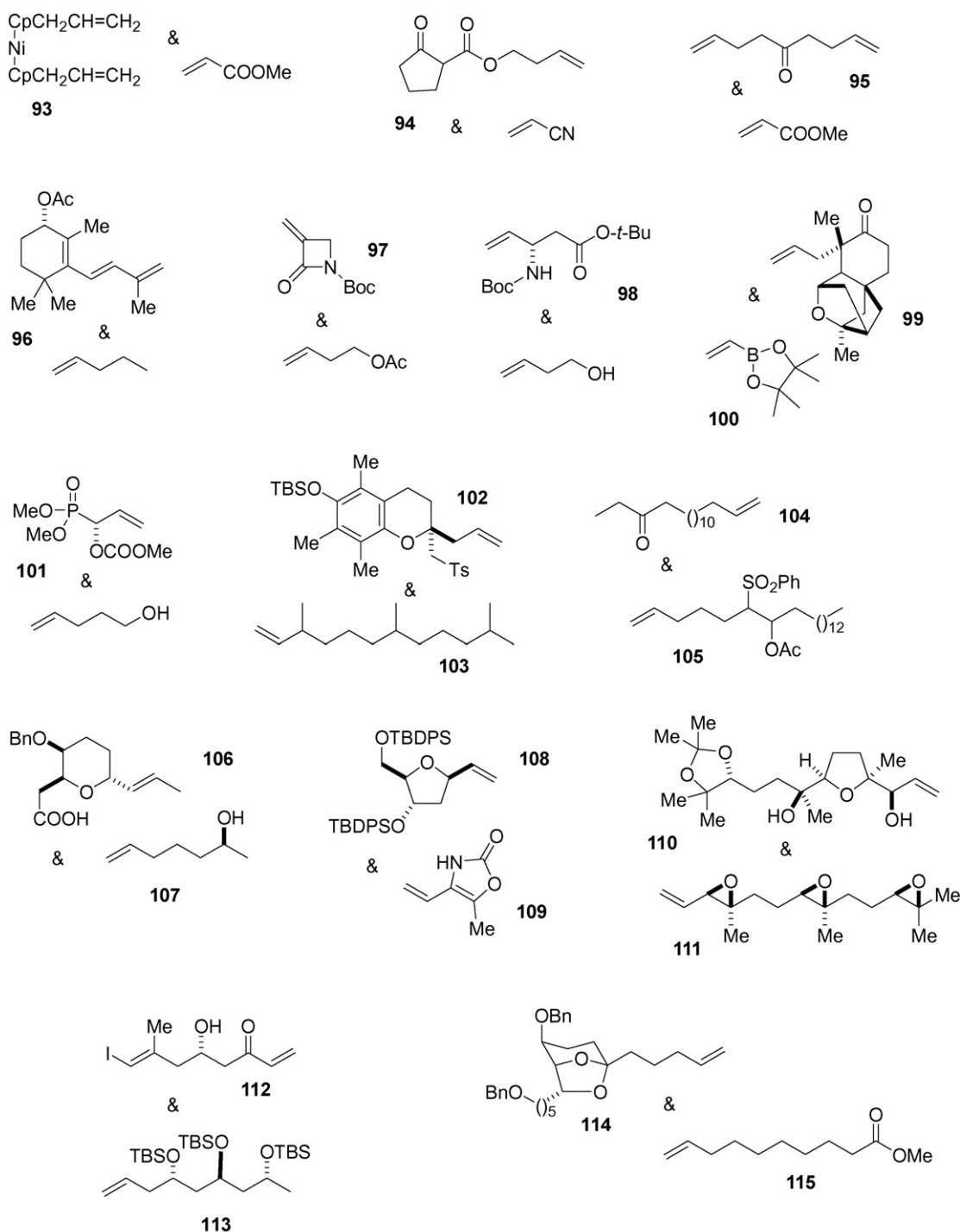
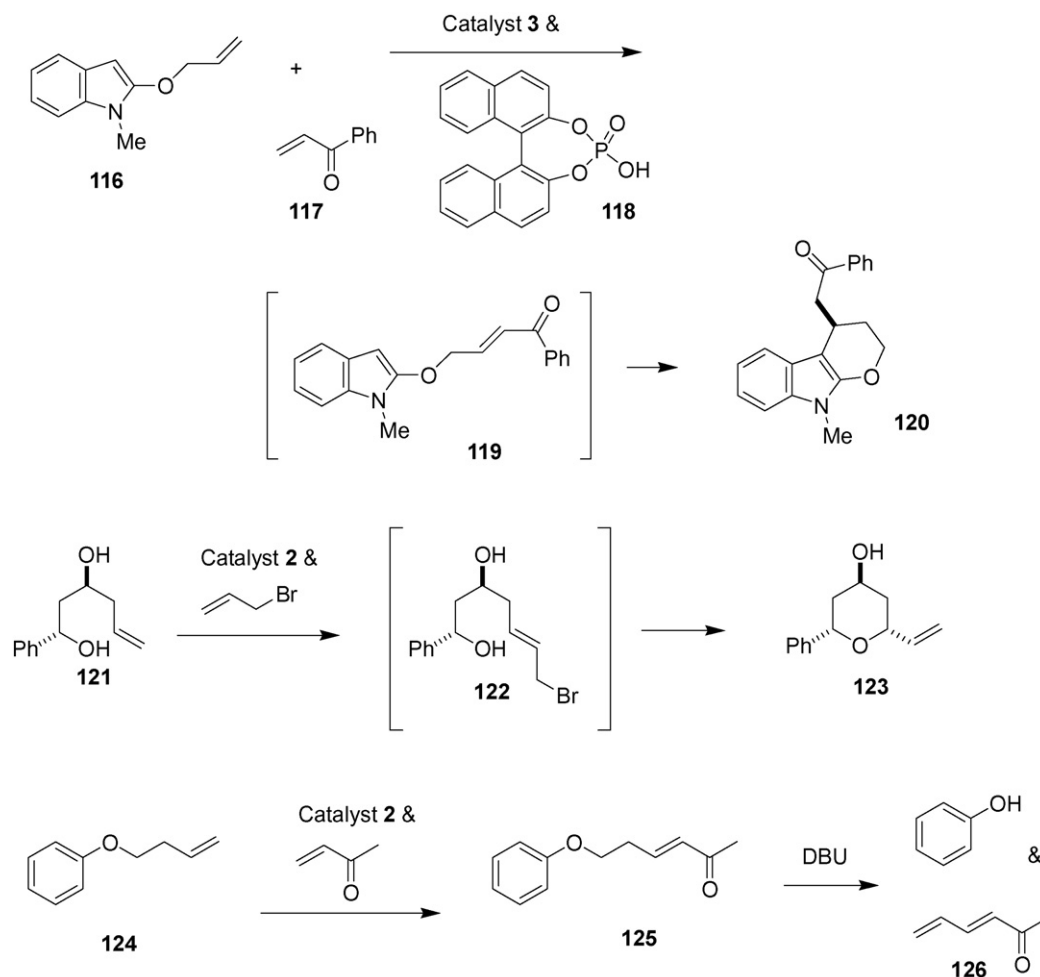


Fig. 5. Represent pairs of alkenes subjected to cross metathesis.

sis sphingosines and sphingosine analogs [402–405], a similar approach to the synthesis of sphingosines using 1-hexadecene [406], and synthesis of sphingosine analogs [407]; (2) a homoallylic ether and 5-methyl-1-hexene for total synthesis of disparlure [408]; (3) a hydronaphthalene-fused vinyltetrahydrofuran and 2-methylpropene for total synthesis of subglutinol [409]; (4) a complex allylphenol derivative and 2-methyl-2-butene for total syntheses of psoralidin [410] and angelmarin [411]; (5) various vinyl-O-heterocycles and various alkenic esters [412]; (6) a bridged vinylidihydropyridine derivative and an optically pure allylic alcohol for total synthesis of awajanomycin [413]; (7) var-

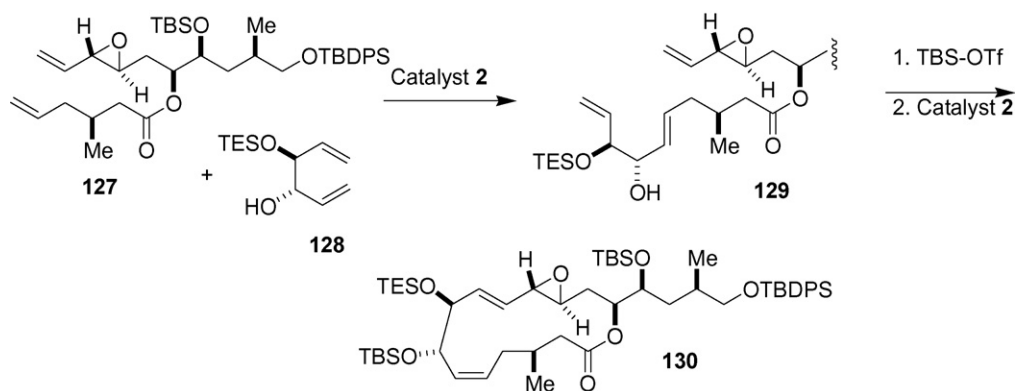
ious monosubstituted alkenes and a carotenoid derivative (96) for preparation of trisporin derivatives [414]; (8) α -methylene- β -lactams (e.g. 97) and various alkenes [415]; (9) optically pure α -vinylbenzyl alcohol and a vinylbutyrolactone derivative for total synthesis of cardiobutanolide [416]; (10) a 4-penten-1-ol derivative and an allylic alcohol for preparation of a sorangicin A segment [417]; (11) a complex alkene (98) and various 4-butenyl derivatives for the total synthesis of largazole and analogs [418,419]; (12) lignin-derived allylbenzenes and styrene derivatives [420]; (13) a 3-propenoate ester and an allylamine derivative [421]; (14) a diene derivative and benzyl-protected cis 2-butene-1,4-diol for total syn-



Scheme 10.

thesis of amphidinol 3 [422]; (15) a complex polycyclic alkene (**99**) and vinylborane **100** for total synthesis of platencin [423] and plansimycin [424]; (16) a vinylborane and a vinylnucleoside derivative [425]; (17) a vinylborane and an alkene-diester [426]; (18) an amine-chelated vinylboronate and various monosubstituted alkenes [427]; (19) an alkene-containing spinosin analog and various monosubstituted alkenes [428]; (20) a vinylphosphonate ester and an N-allyluridine derivative [429]; (21) allylphosphonate esters (e.g. **101**) and 4-pentyn-1-ol [430]; (22) porphyrin-bound alkenes and 1-hexene [431]; (23) vinylpyrenes and ruthenium-nanoparticles containing α,β -unsaturated ester groups [432]; (24)

steroidal alkenes and alkenol derivatives [433,434]; (25) steroidal alkenes and alkenes containing fluorinated side chains [435]; (26) an allyl derivative of FK506 and various monosubstituted alkenes [436]; and (27) β -lactams bound to a solid support through an alkene linkage and various allylbenzene derivatives [437]. Many examples of successful cross metatheses involving two different and structurally complex/precious alkenes were reported in 2009, including: (1) various alkene carbohydrate derivatives [438]; (2) two relatively unfunctionalized alkenes for preparation of all of the stereoisomers of a moth pheromone [439]; (3) threefold CM of tris(4-vinylphenyl)amine and a complex styrene deriva-



Scheme 11.

tive [440]; (4) a complex diene and an allylic amine derivative for total synthesis of an ADDA analog [441]; (5) hydrocarbon alkenes and a α,ω alkene-acetate derivative for total synthesis of naturally occurring pentadecanoate esters [442] and trogodermal [443]; (6) β -carotene and 3-methyl-2,4-hexadienoate esters [444]; (7) a hydrocarbon alkene (**103**) and an allyl-dihydropyran (**102**) for total synthesis of vitamin E and side chain stereoisomers [445]; (8) vitamin E derivatives featuring an alkene group and butenyl glycosides [446]; (9) a methyleneindene derivative and an allylpyridine derivative [447]; (10) alkene-ketones (e.g. **104**) and alkenes that contain a β -acyloxysulfone group (e.g. **105**) for preparation of alkenone natural products [448]; (11) two different allylic alcohol derivatives for total synthesis of pinellidic acid natural products [449,450]; (12) an allylic alcohol and an alkene-lactone for total synthesis of aspergillide B [451]; (13) an allylic alcohol and a vinylidihydropyrene derivative for total synthesis of strictifoline [452]; (14) an allyl alcohol and a complex conjugated diene for total synthesis of apoptolisin A [453]; (15) an alkene-alcohol (**107**) and a tetrahydropyran derivative (**106**) for total synthesis of aspergillide B [454]; (16) a complex allylic alcohol and another complex monosubstituted alkene for synthesis of an iriomoteolide-1a fragment [455]; (17) a homoallylic alcohol derivative and an allyltetrahydropyran derivative for total synthesis of ethyl deoxymonate B [456]; (18) a homoallylic alcohol and a heptenol derivative for total synthesis of a naturally occurring trienoate ester [457]; (19) a homoallylic alcohol derivative and a complex polyol derivative for preparation of an RK-397 segment [458]; (20) a homoallylic alcohol derivative and alkene-tetra-ol derivative for total synthesis of cladospolide A [459]; (21) a vinyl-tetrahydrofuran (**108**) and a vinyloxazolone (**109**) for preparation of nucleoside analogs [460]; (22) complex alkene-tetrahydrofuran derivative and a complex alkene-furanone for total synthesis of sylvaticin [461]; (23) two complex oxygenated partners (**110** and **111**) for total synthesis of omaezakianol [462]; (24) an alkyne-tetrahydrofuran derivative and a complex enone for total synthesis of montanacin E and stereoisomeric compounds [463]; (25) a complex homoallylic ether derivative (**113**) and a complex enone (**112**)

for total synthesis of marinomycin A [464]; (26) an alkene-bicyclic ketal derivative (**114**) and methyl 9-decenoate (**115**) for total synthesis of didemniserinolipid 13 [465]; (27) a complex acetylated triol and a vinylidihydropyranone derivative for total synthesis of synargentolide A [466]; (28) benzodiazepine-alkenes and various electron-deficient alkenes [467]; (29) a butenylpyrrolidine derivative and an alkene-ketone for total synthesis of broussonetines C, O, and P and analogs [468,469]; (30) a hexenylsilane and a hexenyl-oxy-functionalized ruthenium biphenyl-based chelating phosphine oxide complex [470]; and (31) an O-pentenyl glycoside and an allylglycine derivative [471]. Tandem cross metathesis and enantioselective Michael addition were observed in the reaction of indole **116** (Scheme 10) with an enone (e.g. **117**), a ruthenium carbene complex, and a chiral phosphoric acid derivative (e.g. **118**) [472]. Tandem cross metathesis and S_N2' reaction was observed in the reaction of alkene **121** and allyl bromide in the presence of a metathesis catalyst; this step was key for the total synthesis of diospongin A [473]. Removal of butenyl groups from aryl ethers (e.g. **124**) was accomplished through methyl vinyl ketone cross metathesis followed by elimination [474]. A combination of chemoselective cross metathesis of dienes **127** and **128** (Scheme 11) and RCM was employed for the total synthesis of iriomoteolide 3a and analogs [475]; the same diene (the smaller one) was also employed in a double metathesis-based approach to cladospolide D [476]. The use of chiral-at-tungsten carbene complexes for Z selective cross metathesis and metathesis dimerization was reported [477]. Degradation of a complex polyene into simpler fragments through cross metathesis with ethylene was key in the structure determination of symbiodinolide [478–481].

Several examples of dimerization via metathesis (see Scheme 1) were reported in 2009. Compounds subjected to carbene complex-catalyzed metathesis dimerization are depicted in Fig. 6, and include: (1) pentenyl ethers (e.g. **131**) [482]; (2) allylic amine derivatives (e.g. **132**) [483]; (3) steroidal alkenes (e.g. **133**) [484]; (4) isoquinolines (e.g. **134**) (and macrocyclizations using tethered dimeric analogs) [485]; (5) vinylthiophenes (e.g. **135**) [486]; (6) carotenoids (e.g. **136**) [487]; (7) C-vinylglycosides (e.g. **137**)

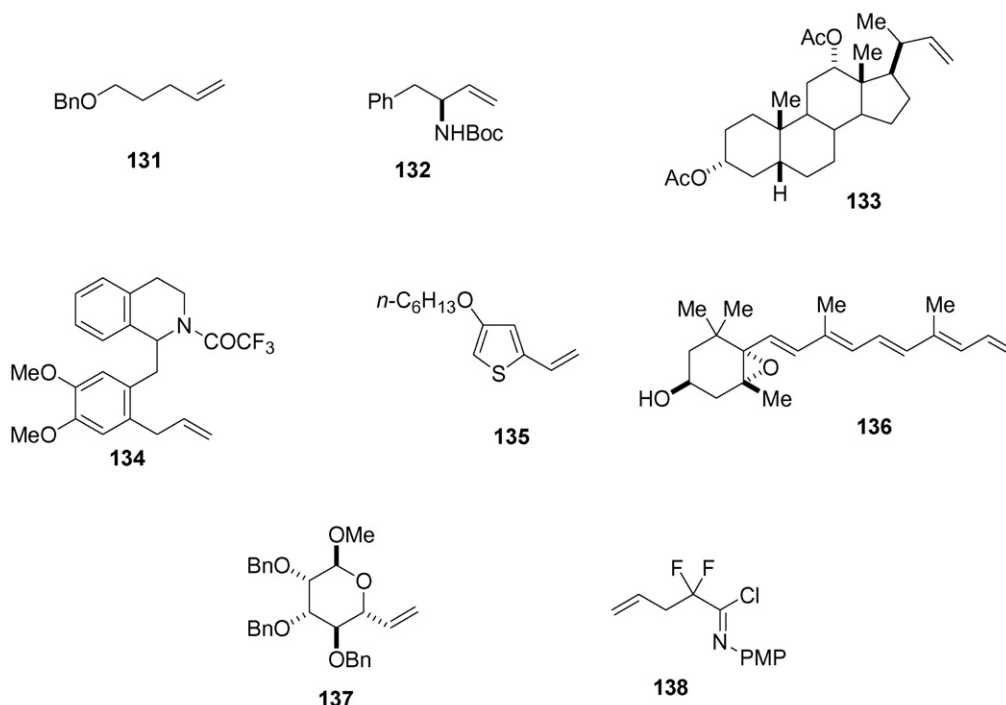
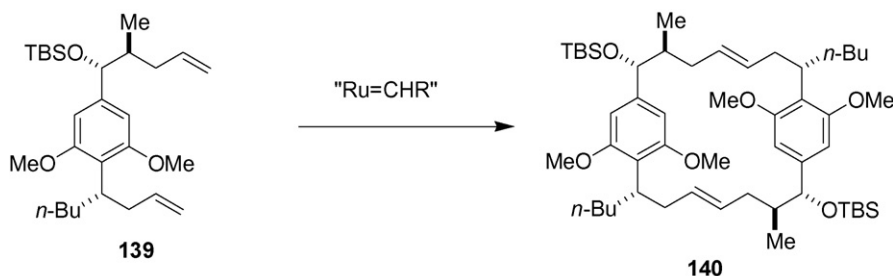


Fig. 6. Representative alkenes subjected to metathesis dimerization.



Scheme 12.

[488]; (8) *p*-acetoxy styrene [489]; (9) 3,5-dimethoxystyrene [490]; (11) a vinyl group-containing polyphenyl system [491]; and (12) 11-undecenal (and CM with various vinyl-EWG species) [492]. Metathesis dimerization, cross metathesis, and cross metatheses of the dimer were reported for alkenes that contain a fluorinated iminoyl chloride group (e.g. **138**) [493]. Metathesis dimerization and methyl acrylate cross metathesis were examined for oleate esters [494].

Additional examples feature cross metathesis in tandem with some other metathesis mode. Tandem dimerization-RCM was observed for phenyl tethered α,ω -diene derivative **139** (Scheme 12) was employed as a key step in the synthesis of cylindrocyclophane A [495].

2.2.5. Ring closing metathesis

The ring-closing metathesis reaction (RCM) (see Scheme 1) has emerged as a very important method for organic synthesis. Numerous carbene complexes (see Figs. 1 and 2) initiate RCM reactions. Many examples forming diverse ring sizes have been reported in 2009, including macrocycles and medium-size rings, as well as the traditional five- and six-membered ring-forming reactions. Reactions have been classified according to the type of ring system formed as a result of RCM.

The RCM reaction has been employed for the synthesis of a variety of carbocyclic ring systems (Fig. 7, the indicated bond was formed via the RCM reaction). Examples include: (1) cyclopentenones [496–500], including those employed for total syntheses of carbocyclic nucleoside analogs (see **144**) [501–510], conformationally constrained S1P₁ agonists [511], jatrophone segments [512], neuraminidase inhibitors [513], and valienamines [514]; (2) cyclopentenones spiro-fused to pyridones [515]; (3) five- to seven-membered rings spiro-fused to cyclohexene-diones [516]; (4) bicyclo[3.2.1]cyclooctenones [517], including an intermediate for total synthesis of platensin analogs (see **145**) [518]; (4) cyclohexenes fused to pyridine rings and use of benzoquinone as an additive to prevent alkene isomerization [519]; (5) cyclohexenones [520,521]; (6) cyclohexenes [522], including those employed in total syntheses of gabosines A and N (see **146**) [523], pancratistatin (see **147**) [524], shikimic acids [525], carissone [526], valienamine [527], platensimycin (**148**) [528], aspidofractinine [529], and the vinigrol skeleton (see **149**) [530]; (7) six-membered ring benzene precursors [531]; (8) a phenanthrene for total synthesis of isohasubanan alkaloids [532]; (9) helicenones (e.g. **150**) [533]; (10) six- to eight-membered rings fused to the bicyclo[2.2.2]octane ring system [534]; (11) cycloheptenes fused to both rings of a naphthalene (e.g. **151**) [535]; (12) cycloheptenes for a synthetic approach to frondosins [536]; (13) cycloheptenes for total syntheses of epsilicine (see **152**) [537], cyathins A₃ and B₂ [538], englerin A [539], calystegine A₃ (see **153**) [540]; and daphnacyclidine alkaloid segments (see **154**) [541]; (14) tricyclic cycloheptenes for total synthesis of echinopine A (**155**) [542]; (15) seven- and eight-membered ring allylsilanes for total synthesis of teucladiol and poitediol (e.g. **156**) [543]; (16) cyclooctenes for synthesis of

the core ring system YW3699 [544,545]; (17) a cyclooctenone for total synthesis of euphococcinone and adaline (e.g. **157**) [546]; (18) fluorinated cyclooctenes (e.g. **158**) via a relay approach [547]; and (19) a cyclodecene derivative (**159**) for total syntheses of diversifolin [548]. An unusual allylic oxidation product was a significant byproduct in the preparation of cyclopentapiperidines via an RCM reaction [549]. The tandem aza-Claisen rearrangement RCM reaction using diene-imino esters (e.g. **160**, Scheme 13) was reported [550,551].

Numerous examples of the formation of nitrogen heterocycles using the RCM reaction (Fig. 8) were reported in 2009, including: (1) dihydropyrroles [552–554]; (2) direct formation of indoles (e.g. **164**) in a tandem alkene isomerization/RCM sequence starting from *N*-allylic aniline derivatives (e.g. **163**) [555]; (3) direct formation of pyrroles in a one-pot RCM-oxidation sequence [556]; (4) optically pure five- and six-membered rings [557]; (5) five- and six-membered ring α,β -unsaturated lactams fused to a proline derivative for total synthesis of monomorine (a cross metathesis approach was also reported) [558]; (6) five- and six-membered rings fused to isoindolones [559]; (7) various five- to eight-membered ring cyclic amines [560,561]; (8) tetrahydropyridines [562–566], including total syntheses of securinine (see **165**) [567], castanospermine [568], meloscine (see **166**) [569], epiquinamide [570], conhydrine [571,572], both rings of conhydrine and epiquinamide (see **167**) [573], epi CP-99994 [574], 8-oxypseudopalmitine and 8-oxypseudoberberine [575], 3-hydroxy-2-phenylpiperidine [576], deoxyallonojirimycin (see **168**) [577], deoxyaltronojirimycin [578], lasubine II [579], lentiginosine [580], and the cannabistatine core [581]; (9) α,β -unsaturated six-membered ring lactams [582–585], including total syntheses of various lycopodium alkaloids (see **169**) [586]; (10) a one-pot synthesis of β,γ -unsaturated six-membered ring lactams followed by treatment with Lawesson's reagent to afford the corresponding thiolactams [587]; (11) an ansa-bridged tetrahydropyridine (**170**) for quebrachamine total synthesis formed through asymmetric RCM using a chiral molybdenum catalyst [588]; (12) six-membered ring NO, NS, OO, and OS heterocycles (e.g. **172**) fused to a benzene ring using a tandem alkene isomerization-RCM strategy of the corresponding allyl derivatives (e.g. **171**) [589]; (13) six-membered ring cyclic acylhydrazines (e.g. **173**) and lactams (and subsequent conversion to pyridazines and pyridines) [590,591]; (14) six-membered ring cyclic hydrazines [592]; (15) six-membered ring *N*-sulfonyl lactams [593]; (16) six-membered ring cyclic pyridinium salts (e.g. **174**) [594]; (16) 8-azabicyclo[3.2.1]oct-2-enes for preparation of calystegine analogs (e.g. **175**) [595]; (17) six- and seven-membered ring cyclic amines [596]; (18) six- and seven-membered ring lactams (e.g. **176**) [597]; (19) six- and seven-membered ring cyclic ureas [598]; (20) six- to eight-membered ring cyclic amines [599,600] and lactams [601]; (21) seven-membered ring amines [602], including those employed in total syntheses of SB 462795 [603], norsecurinine and securinine [604], the reported structure of norcardimicin B [605], and IBR₂ analogs [606]; (22) seven-membered ring α,β -unsaturated lactams [607],

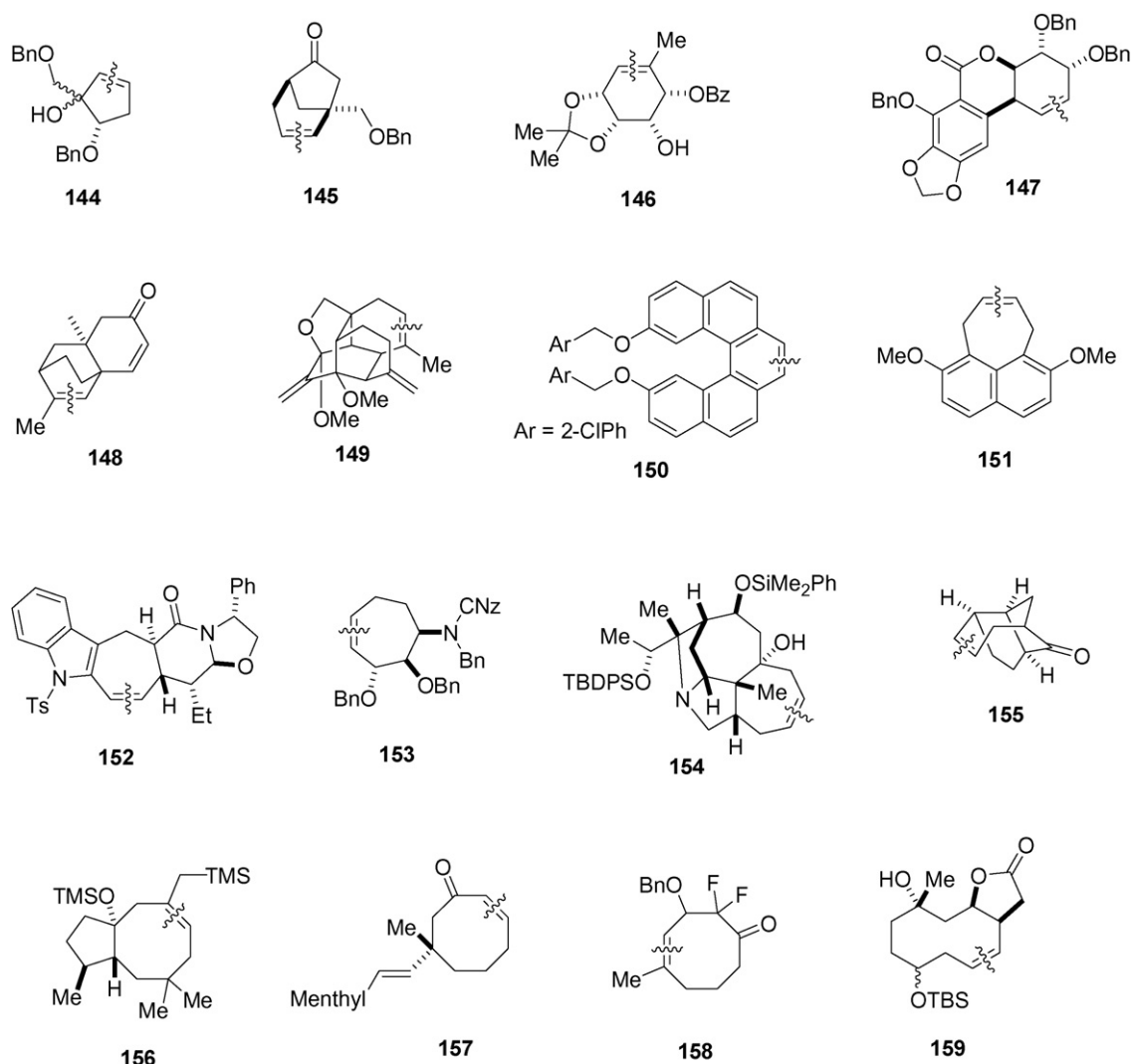
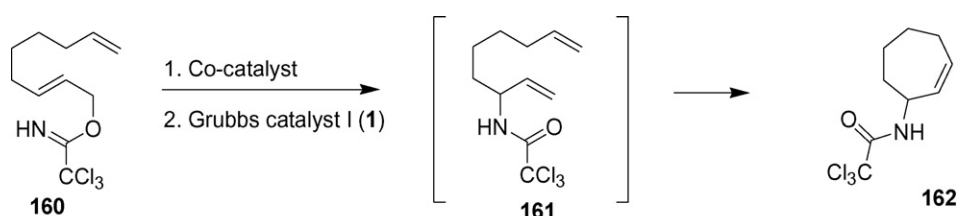


Fig. 7. Representative carbocycles produced through an RCM reaction (bond constructed through RCM indicated).

including syntheses of glycosidase inhibitors (see **177**) [608]; (23) seven-membered ring lactam-hydrazides (e.g. **178**) [609]; (24) 1-azabicyclo[4.4.1]undec-3-enes [610]; (25) highly oxygenated 10-azabicyclo[4.3.1]dec-3-enes (e.g. **179**) [611]; (26) seven-membered ring cyclic guanidines [612]; (27) seven-membered ring O,N-heterocycles (also via enyne metathesis) [613]; (28) seven- to eight-membered rings fused to the indole ring system (e.g. **180**) [614]; (29) seven- to nine-membered rings fused to a naphthalene ring system [615]; (30) eight- to ten-membered ring amines [616]; (31) an eight-membered ring cyclic amine for total synthesis of apparacine (**181**) [617,618]; (32) eight-membered ring N,O-heterocycles (e.g. **182**) [619]; (33) a nine-membered ring amine for total synthesis of castanospermine (**183**) [620]; and (34) a ten-membered dinitrogen-bridged indolocarbazole for prepara-

tion of staurosporine analogs [621]. A one-pot RCM-hydroboration sequence employing diene **184** (Scheme 14) was employed for the total synthesis of deoxynojirimycin [622]. Tandem rhodium vinylcarbene N–H insertion and RCM using vinyl diazo compounds (e.g. **186**) and amino-alkenes (e.g. **187**) was employed for the synthesis of five- to eight-membered ring nitrogen heterocycles [623]. The carbene insertion process generates diene intermediate **188**, which undergoes the RCM event prior to product isolation.

Many diverse oxygen heterocycles were synthesized via the RCM reaction in 2009 (Fig. 9), including: (1) five- and six-membered ring ethers fused to vitamin D₃ [624]; (2) five- to seven-membered ring ethers fused to benzene rings [625]; (3) six-membered ring ethers [626,627], including those employed in total syntheses of laulimalide (see **190**) [628], neopeltolide



Scheme 13.

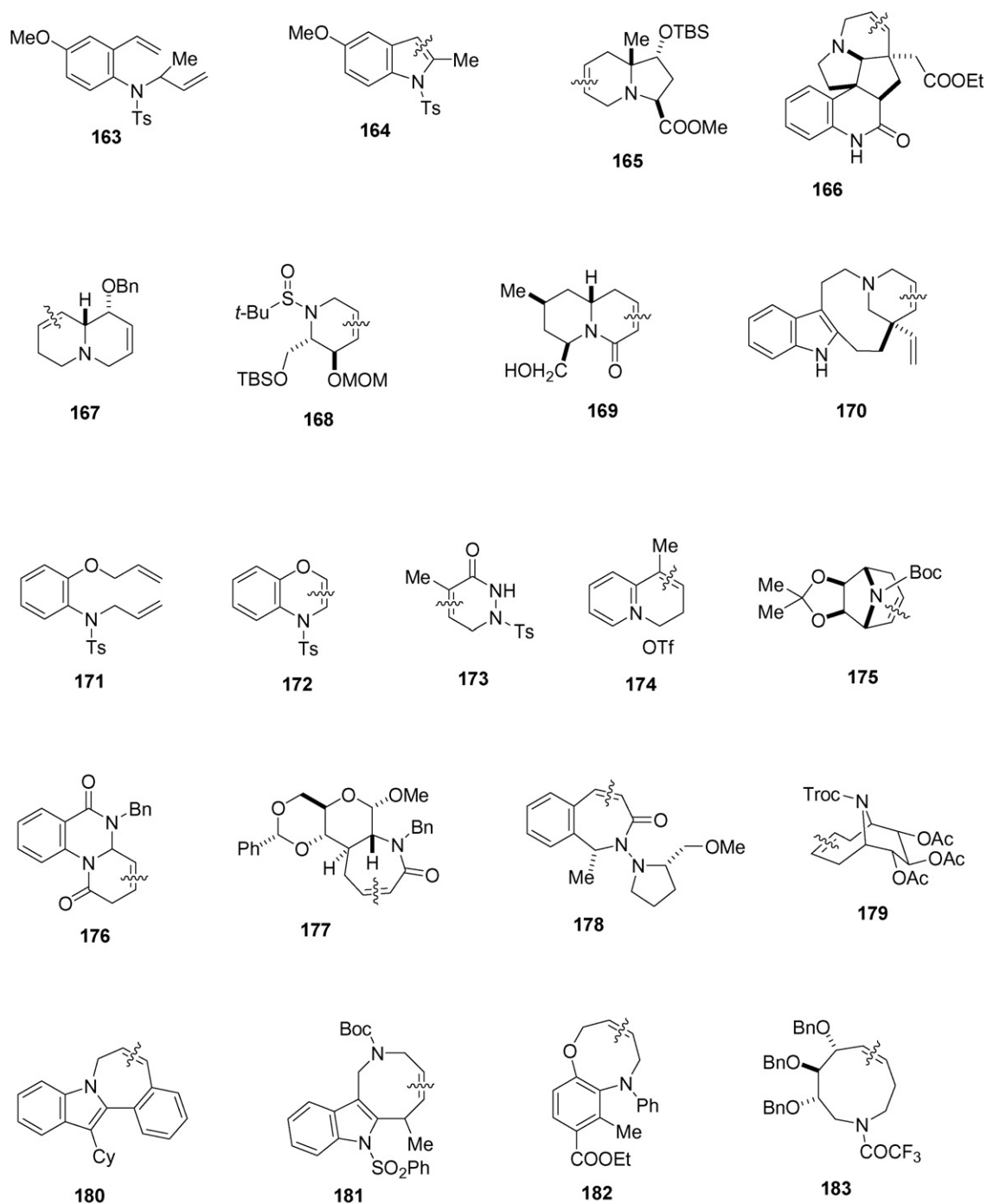
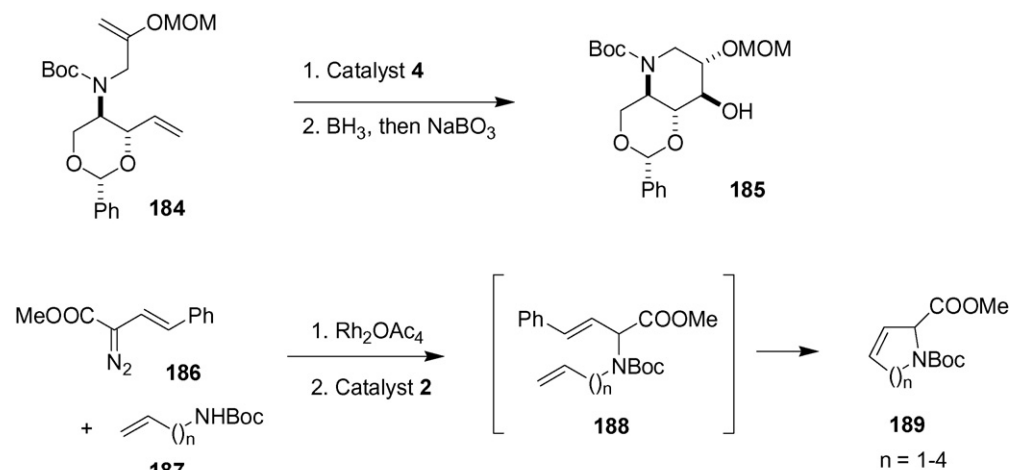


Fig. 8. Representative N-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).

(and a structural correction) [629], fostriecin [630], spirocyclic steroids [631]; hagin E and related natural products [632], and all stereoisomers of centrolobine (see **192**) employing tandem alkene isomerization of allyl ether **191** [633]; (4) six-membered ring α,β -unsaturated lactones [634,635], including those employed for total syntheses of 6-acetoxy-5-hexadecanolide (see **193**) [636], goniothalamine [637], fostriecin and phoslactomycin B [638], EBC 23–25 and EBC 72–76 [639], dodoneine [640,641], kurzilactone [642], lasubine I and subcosine I [643], rugulactone [644], phoslactomycin B (via a relay approach) [645], cryptocarya diacetate [646], cryptocarya triacetate (see **194**) [647], cryptocaryalactone [648], denosomin [649], neopeltolide [650], polyrhacitide A [651], spicigerolide [652], leiocarpin A and galantinic acid [653], an

antifungal pyrone [654], clavulactone [655], dumetorine and epihydridropinidine [656], and other pyranone natural products [657]; (5) an 8-oxabicyclo[3.2.1]oct-2-ene derivative (**195**) for total synthesis of a calystegine B₂ derivative [658]; (6) six- to nine-membered rings present in brevioxins and related marine-derived toxins (e.g. **196**) [659–663], including a total synthesis of brevenal [664]; (7) a seven-membered ring benzo-fused ether for total synthesis of radulanin E [665]; (8) seven-membered ring oxygen heterocycles (e.g. **197**) fused to carbohydrate systems [666]; (9) an eight membered ring ether for total syntheses of heliannuol [667]; (10) eight- and nine-membered ring oxacyclic alkyne-cobalt complexes (e.g. **198**) for total synthesis of obtusan [668]; (11) eight- to eleven-membered rings fused to a furan ring system [669]; (12) a nine-membered ring



Scheme 14.

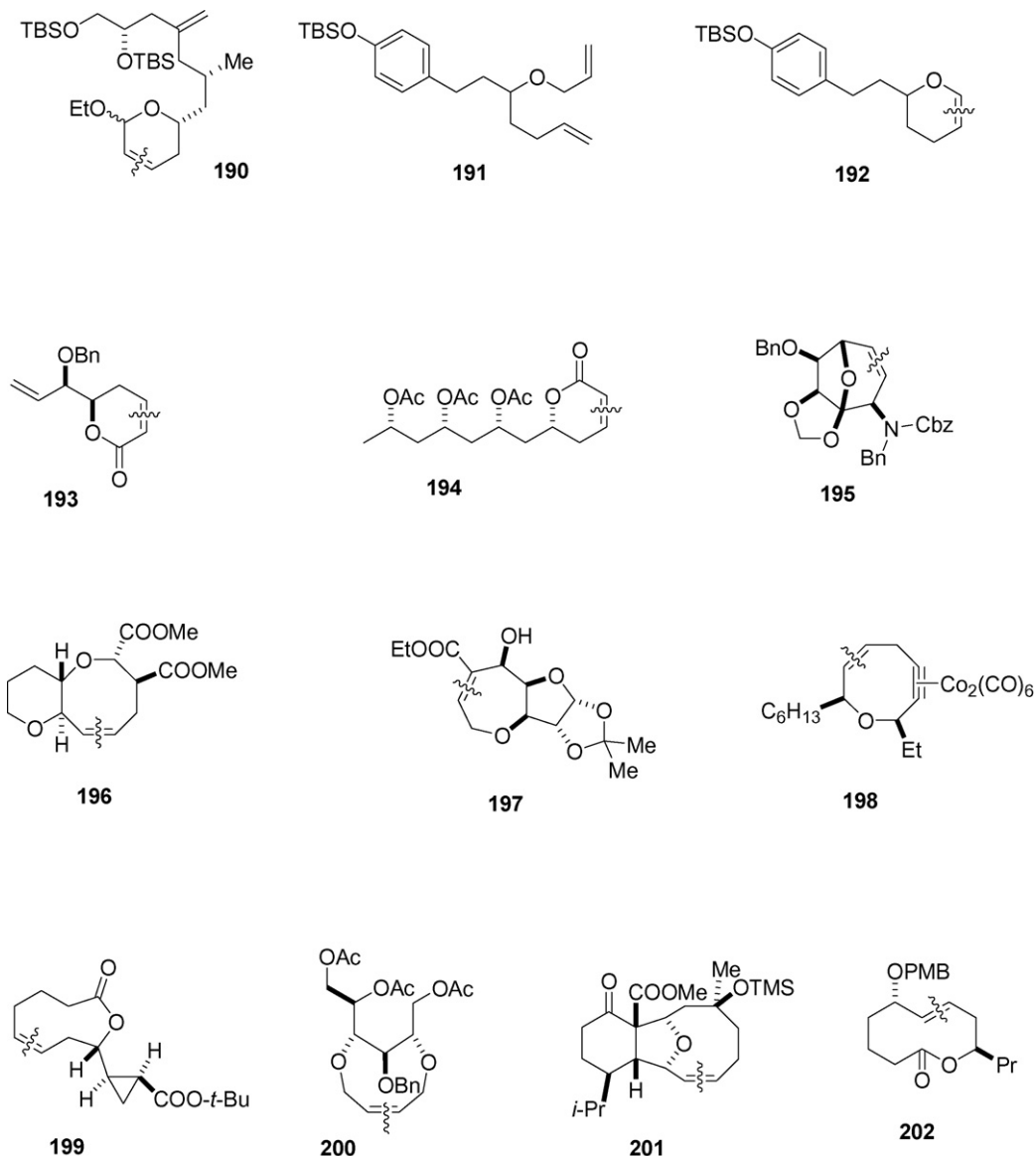
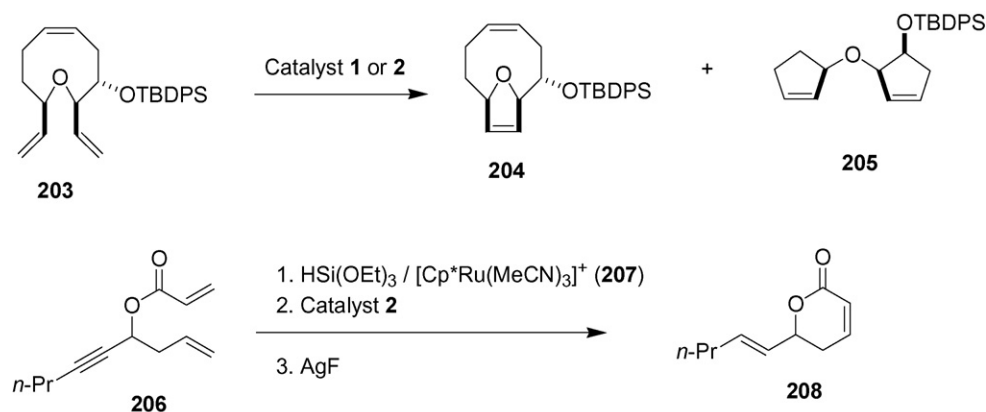


Fig. 9. Representative oxygen-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).



Scheme 15.

lactone (e.g. **199**) for total synthesis of halicholactone [670]; (13) a nine-membered ring diether bridged glucopyranose (**200**) [671]; (14) a furan-bridged nine-membered ring ether (**201**) for total synthesis of polyanthellin A [672]; (15) failure to form a furan-bridged nine-membered oxygen heterocycle required for briarellins E and F total synthesis [673]; and (16) ten-membered ring lactones for total syntheses of putaminoxin (see **202**) [674], decarestrictines C₁, C₂ [675] and J [676], aspinolide [677], stagonolide C [678,679], stagonolide F [680], nonenolide [681], and herbarumin I [682,683]. A total synthesis of pyranicin employed three different metathesis events, formation of an α,β -unsaturated lactone (via relay metathesis), formation of a six-membered ring ether via RCM, and cross metathesis of two complex alkene molecules [684]. Attempts to prepare eleutherobin analogs from triene **203** (Scheme 15) led to the desired RCM product (**204**) as well as a secondary metathesis product (**205**) [685]. A one-pot hydrosilylation-RCM-protodesilylation sequence using diene-yne **206** was employed in the total synthesis of pironetin [686].

Heterocyclic compounds involving elements other than N and O were also constructed via the RCM reaction (Fig. 10). Examples include: (1) five- and six-membered ring cyclic silanes (e.g. **209**) (metathesis dimerizations were also reported) [687]; (2) asymmetric formation of a cyclic siloxane (**210**) for total synthesis of epi-citreoviral via asymmetric RCM using a chiral ruthenium catalyst [688]; (3) asymmetric formation of cyclic phosphonates (e.g. **211**) using a chiral molybdenum carbene complex catalyst [689]; (4) cyclic sulfides (e.g. **212**) [690]; (5) cyclic sultams (e.g. **213**) [691]; (6) cyclic monothiocarbonates (e.g. **214**) [692]; (7) nitrogen/selenium heterocycles (e.g. **215**) [693]; and (8) bridged zirconocenes (e.g. **216**) [694].

Numerous macrocyclic compounds (rings with ≥ 11 atoms) were synthesized using the RCM reaction in 2009 (Fig. 11), including: (1) a macrocycle-bridged benzofuran (**217**) for total synthesis of kendamycin [695]; (2) a macrocyclic N-bridged bis(benzo) azanorbornene [696]; (3) a tetrahydrofuranophane-ether [697]; (4) macrocyclic lactams for synthesis of Asp90 inhibitors [698], and fluviricins B₂–B₅ (the synthesis also employs cross metathesis) [699]; (5) macrocyclic bis(lactams) [700]; (6) macrocyclic tris(lactam) for total synthesis of jaspakinolide (the RCM event employs relay metathesis) [701]; (7) a macrocyclic lactam *m*-cyclophane for total synthesis of N-methylmaysenine [702]; (8) macrocycle-bridged peptides (e.g. **218**) [703–718], including total synthesis of BILN 2061 [719,720] and failed attempts to form this class of compounds [721]; (9) macrocycle-bridged glycopeptides [722]; (10) macrocycle-bridged β -lactams [723,724]; (11) macrocycle-bridged bis(β -lactam)s [725]; (12) a macrocyclic bis(lactam) bis(lactone) for total synthesis of cryptophycin-52 and analogs [726]; (13) macrocyclic ketones for total synthesis of

several different amphidinolides [727] and metacycloprodigiosin (see **219**) [728]; (14) macrocyclic bis(ketone)s for total syntheses of characiol [729] and 17-norcharaciol [730]; (15) a macrocyclic tris(ketone) and a six-membered ring ether (via RCM) for total synthesis of methyl sarcophytoate [731]; (16) a macrocyclic ketolactone for total synthesis of methmycin (see **220**) [732]; (17) a carbohydrate-bridged macrocyclic lactone for total synthesis of ipomoeassin F (see **221**) [733]; (18) macrocyclic lactones [734], including those employed for syntheses of aspecyclides B and C (see **222**) [735], compounds stereoisomeric to tulearin A [736], various resorcyclic lactones [737,738], the lyngbouilloside macro-lactone core [739], the plecomarolide macrolactone core [740], brefeldin A (see **223**) [741], the ring system of latrunculin B [742], amphidinolactone [743], aspiciin [744], the auriside core [745], aigialomycin D [746,747], Sonic Hedgehog inhibitors [748], the macrocyclic ring of iriomoteolide 3a (an earlier step employs cross metathesis) [749], zearalenone analogs [750], and fluorinated salicylhalamide analogs [751]; (19) macrocyclic bis(lactone)s for total syntheses of amphidinolide X [752], bridged steroids (see **224**) [753], colletallol [754], stereoisomers of tanikolide dimer (early steps employ cross metathesis) [755], acremodiol stereoisomers [756], and as part of a strategy to control cross metathesis reaction using two different but structurally complex partners in polyether antibiotic synthesis [757]; (20) macrocyclic tris(lactone)s for total synthesis of macrocyclic anticancer agents [758] and macrospherulides A and B [759]; (21) a failed attempt to prepare triazene *m*-cyclophanes [760]; (22) a macrocycle-bridged tricyclic ring system for total synthesis of nakadomarin A [761]; (23) optically active *p*-cyclophanes (e.g. **225**) [762]; (24) doubly-bridged phenanthrolines (e.g. **226**) [763]; (25) a macrocycle-bridged disaccharide (**227**) for synthesis of ipomoeassin analogs [764]; (26) a macrocycle-bridged bipyridyl ligand (**228**) [765]; (27) macrocycle-bridged carbohydrates [766,767]; (28) macrocycle-bridged trisaccharides [768]; (29) bridged ferrocene- β -lactams (e.g. **229**) [769]; (30) a macrocycle-bridged nucleoside-centered dendrimeric molecule [770]; (31) a macrocyclic ansa-naphthalene system [771]; (32) macrocyclization of hyperbranched glycerols [772]; (33) rotaxanes and/or catenanes [773–776]; (34) catenane-like macrocycle-bridged calixarenes [777]; (35) interlocked calixarene dimers [778]; (36) daisy chains [779]; (37) macrocyclized dimeric compounds [780]; and (38) cyclic polycaprolactone derivatives (ring closure was also achieved via enyne metathesis) [781].

Several examples of ring rearrangement metathesis (RRM) were reported in 2009 (see Scheme 16). These examples include: (1) rearrangement of the cyclobutene derivative **230**, which was a key step in the total synthesis of erystotramidine [782]; (2) conversion of norbornene derivative **232** into the corresponding tricyclic fused ring system (**233**) [783]; (3) conversion of norbornenes into

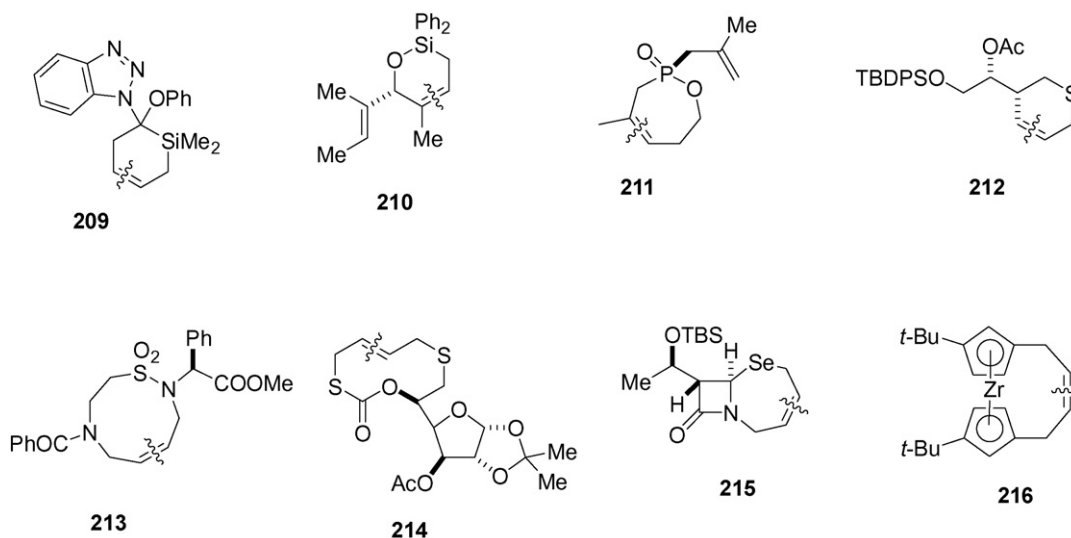


Fig. 10. Representative examples of other heterocycles prepared via the RCM reaction (bond constructed through RCM indicated).

the corresponding fused ring systems as part of a total synthesis of bakkenolide A [784]; (4) conversion of carbohydrate-templated norbornenes into the corresponding fused ring systems [785]; (5) synthesis of nine-membered ring eleutherobin analogs using RRM of a norbornene derivative [786]; (6) conversion of oxanorbornenes (e.g. **234**) to the corresponding fused ring ethers (e.g. **235**) [787,788]; (7) conversion of oxabicyclo[3.3.1]nonene derivative **236** into the fused ring system (**237**) as part of a total synthesis of norhalichondrin B (a later step of the synthesis employs cross metathesis) [789]; and (8) conversion of dihydropyran derivatives (e.g. **238**) into two different heterocyclic ring systems (e.g. **239**) [790]. A tandem RRM-CM sequence involving the conversion of sultam-fused norbornenes (e.g. **240**) and *t*-butyl acrylate to bridged sultams (e.g. **241**) was also demonstrated [791,792].

2.2.6. Alkene metathesis involving alkyne components

Several examples of the synthesis of conjugated dienes through the intermolecular (enyne CM) or intramolecular (enyne RCM) metathesis of enynes (see Scheme 1) using carbene complexes were reported in 2009. Examples of intermolecular enyne metathesis are depicted in Scheme 17 and include: (1) reaction of monosubstituted alkenes (e.g. 1-decene) with propargylic esters (e.g. **245**) [793]; (2) coupling of alkyne **247** and 5-bromo-1-pentene (**248**) as a key step in the total synthesis of actinophyllic acid [794]; (3) reaction of alkyne-benzene derivatives with ethylene [795]; (4) coupling of alkynylborane **250** and alkene **251**, which was a key step in the total synthesis of amphidinolide K [796]; (5) synthesis of α,β -unsaturated aldehydes (e.g. **254**) via one-pot enyne metathesis using an enol ether followed by hydrolysis of the resulting alkoxydiene (e.g. **253**) [797]; (6) one pot enyne-metathesis and cation cyclization to afford tetrahydropyridines (e.g. **257**) from alkynes and amine-alkynes (e.g. **255**) [798]; and (7) one-pot tandem enyne metathesis using alkynes and enol ethers followed by hetero-Diels–Alder reaction for synthesis of carbohydrates (e.g. **259**) [799,800]. Examples of intramolecular enyne metathesis are depicted in Scheme 18 and include: (1) preparation of vinylcyclopentenes (e.g. **261**) [801], including total syntheses of valerenic acid [802], and an approach to hexacyclenic acid [803]; (2) formation of alkenyl-substituted five- and six-membered rings [804]; (3) formation of spiro-fused vinylcyclopentenes (e.g. **263**) [805]; (4) formation of densely functionalized cyclohexenes (e.g. **265**) [806]; (5) selective formation of six-membered rings or five-membered rings from dienyne in a hydroxyl group directed process [807];

(6) formation of cyclic sulfoximines (e.g. **267**) [808]; (7) formation of macrocyclic lactone-dienes (e.g. **269**) [809]; (8) formation of carbocycle-bridged nucleosides (also via RCM) (e.g. **271**) [810]; (9) enantioselective and endo selective enyne metathesis employing chiral molybdenum carbene complexes (e.g. **273**) and dienes featuring enantiotopic alkene groups (e.g. **272**) [811]; and (10) direct formation of styrene derivatives (e.g. **277**) through enyne metathesis followed by dehydration or enolization [812]. A net ring rearranging enyne metathesis was observed in: (1) the reaction of oxanorbornene-dialkyne **278** (Scheme 19) with Grubbs catalyst I [813], and (2) the reaction of hydrophenanthroline **280** with ethylene in the presence of a ruthenium carbene complex [814].

Tandem intramolecular enyne metathesis and cross metathesis [e.g. conversion of **282** (Scheme 20) and 1-tetradecene to **284** in the presence of Grubbs catalyst II] was employed for synthesis of sphingosine analogs [815]. Representative examples of tandem enyne metathesis–RCM are depicted in Scheme 21. The tandem enyne metathesis/RCM reactions for dienyne **285** and related compounds was observed to be very catalyst and substrate dependent [816]. The direction of tandem enyne metathesis/RCM of compounds of general structure **290** was dependent on the alkene substitution pattern, where the less substituted alkene reacts first with the catalyst [817]. A silicon-tethered tandem enyne metathesis/RCM sequence was employed for synthesis of the illudin core from dienyne **293** [818] and a related process was employed for total synthesis of acylfulvene and irofulven [819]. A similar silicon-tethered tandem enyne metathesis/RCM sequence, followed by protidesilylation was employed as a key step in the total synthesis of cochleamycin A [820]. Tandem enyne metathesis/RCM was employed for the synthesis of annulated carbolines from indole-dienynes (e.g. **295**) [821]. The same paper also includes many examples of simpler enyne metatheses and RCM's designed to produce annulated carbolines.

Enyne metathesis reactions involving conjugated diynes and triynes often proceed with 1,3-propargyl shifts of the carbene intermediates; examples are depicted in Scheme 22. A relay metathesis-rearrangement sequence was employed for total syntheses of asperpentyn, harveynone, and tricholomenyn A from dialkyne-diene starting materials (e.g. **297**) [822]. More elaborate examples involving additional alkyne groups are depicted in Scheme 23. The reaction of ene-triynes **301** with ruthenium carbene complexes resulted in simple enyne metathesis products (e.g. **303**) using internal alkynes, however the use of a terminal alkyne

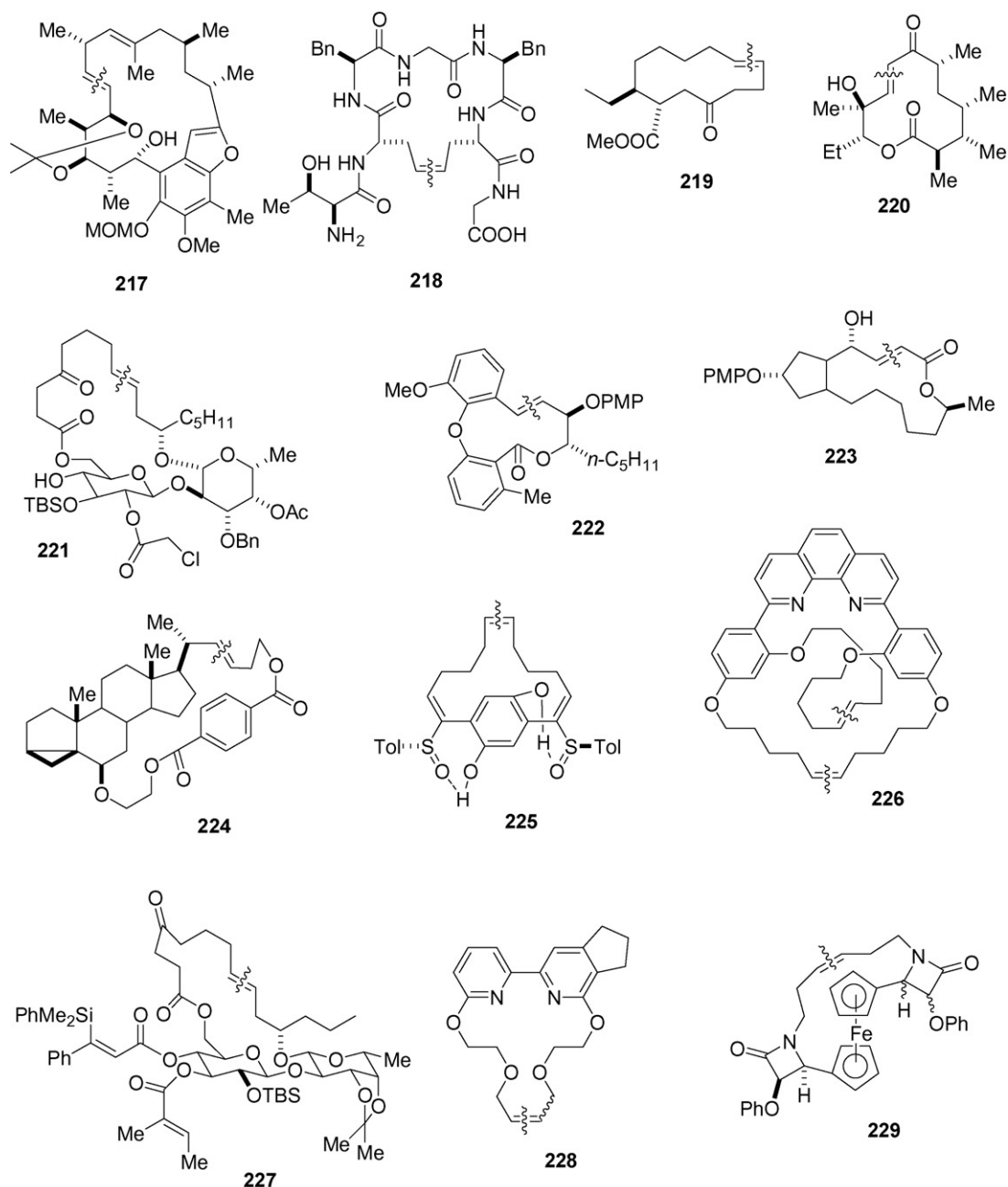


Fig. 11. Representative macrocycles (ring size > 10) prepared using the RCM reaction (bond constructed through RCM indicated).

resulted in a stable carbene complex (**304**), obtained through a double propargyl shift after the initial enyne metathesis event [823]. Formation of stable carbene complex **308** was observed in the reaction of conjugated diyne–enyne derivative **305** with Grubbs catalyst II [824]. The final carbene complex was an effective RCM catalyst in the case where $n = 0$.

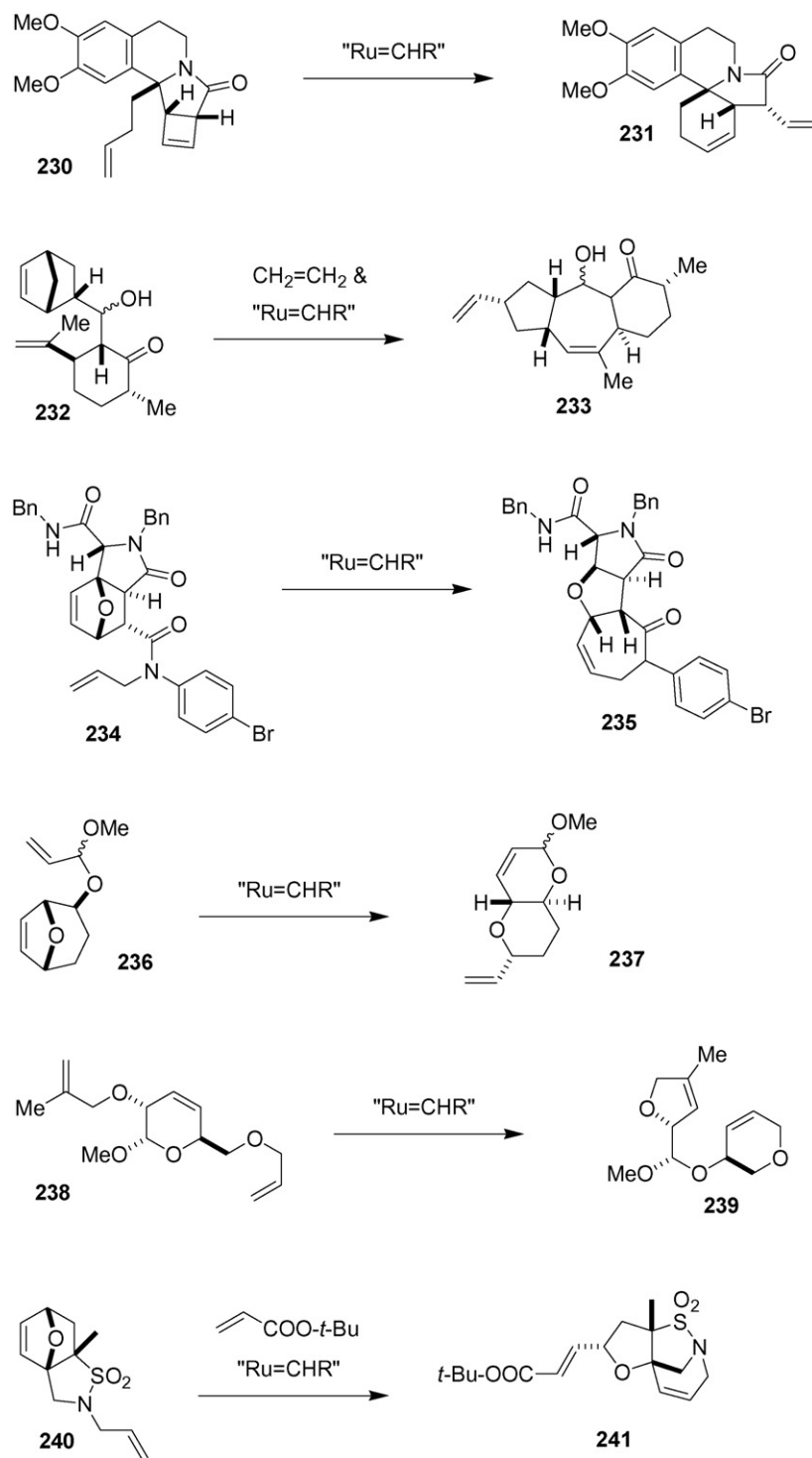
Several examples of dialkyne polymerization were reported in 2009 (see Scheme 24). The polymerization reaction for dialkynes of general structure **309** using analogs of the Hoveyda–Grubbs catalyst where fluorinated carboxylate ligands replace chlorides (**310**) led to mixtures of the five-membered ring polymer (**311**) and alkyne trimerization product (**312**) [825]. The degree of trimerization was highly dependent on the composition of the tether, and was more severe with amine tethers than ether tethers. Stepwise assembly of oligomeric analogs (e.g. **315**) was accomplished through reaction of triene derivative **313** with the Schrock

catalyst (**5**) followed by subsequent reaction of the resulting bis(carbene–molybdenum) complex (**314**) with additional triene **313** [826]. This reaction sequence repeated to make compounds containing up to 23 conjugated alkene groups. The general use of Grubbs catalyst I for alkyne trimerization was also reported [827].

2.2.7. Non-metathesis reaction processes involving the Grubbs and related catalysts

Several publications in 2009 report on processes unrelated to metathesis that are initiated by ruthenium carbene complex catalysts **1–4** and structurally related carbene complexes.

A frequent side reaction during metathesis is alkene isomerization. This side reaction has been attributed to the formation of metal hydride complexes under the conditions necessary for metathesis. The reaction of alkene **318** (Scheme 25) with Grubbs catalyst II and vinyl trimethylsilyl ether led to the alkene isomerization product



Scheme 16.

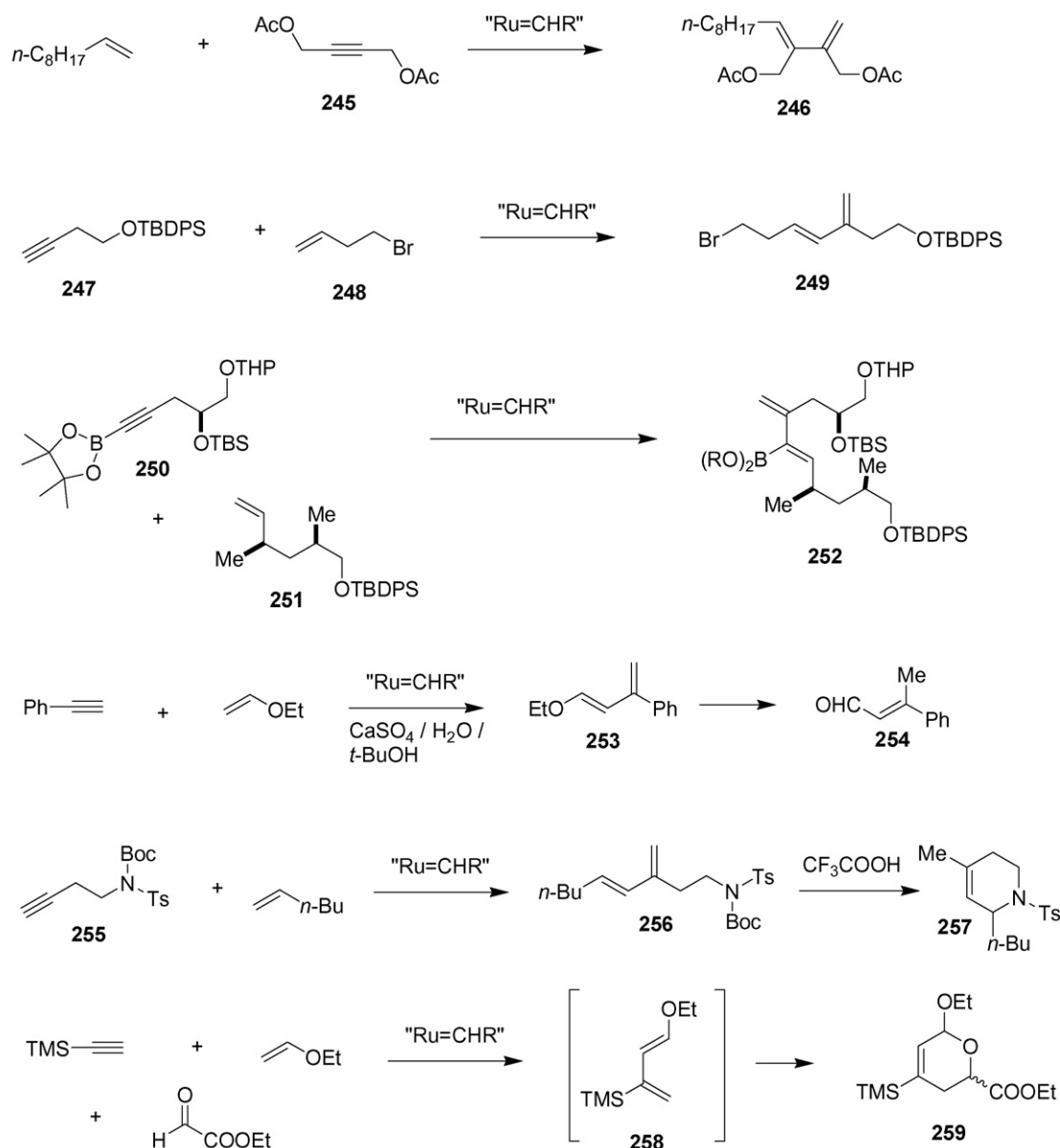
320 [828]. Subsequent conversion to the diene **321** and RCM were essential steps in the preparation of the epicoccin core ring system. Several of the RCM processes previously reported involve alkene isomerizations [555,589,633].

The use of ruthenium metathesis catalysts **2** and **4** to initiate polymerization of hydroxyl-containing terminal alkynes (e.g. **323**, Scheme 26) was reported [829]. The living polymer carbene complex groups were identified when following the reaction by proton NMR. The propagating species depicted (**324**) was proposed. Computational studies suggested that oxygen-chelated complexes were involved in the polymerization process. Several metathesis cata-

lysts were investigated for the polymerization of phenylacetylene derivatives [830] and acetylene [831].

Alkane metathesis was effected using a dual catalyst system consisting of an iridium hydride (**325**, Scheme 27) and the Schrock carbene complex (**5**) or various derivatives (e.g. **326**) [832,833]. The role of the iridium catalyst is to effect a C–H activation and formation of an alkene. Once formed, the alkene immediately undergoes olefin metathesis dimerization. Subsequent hydrogenation affords the alkane products.

Grubbs catalyst was employed for the cycloisomerization of some alkynols (e.g. **329**, Scheme 28) [834]. In many of the cases



Scheme 17.

examined, enyne metathesis competes with the cycloisomerization process.

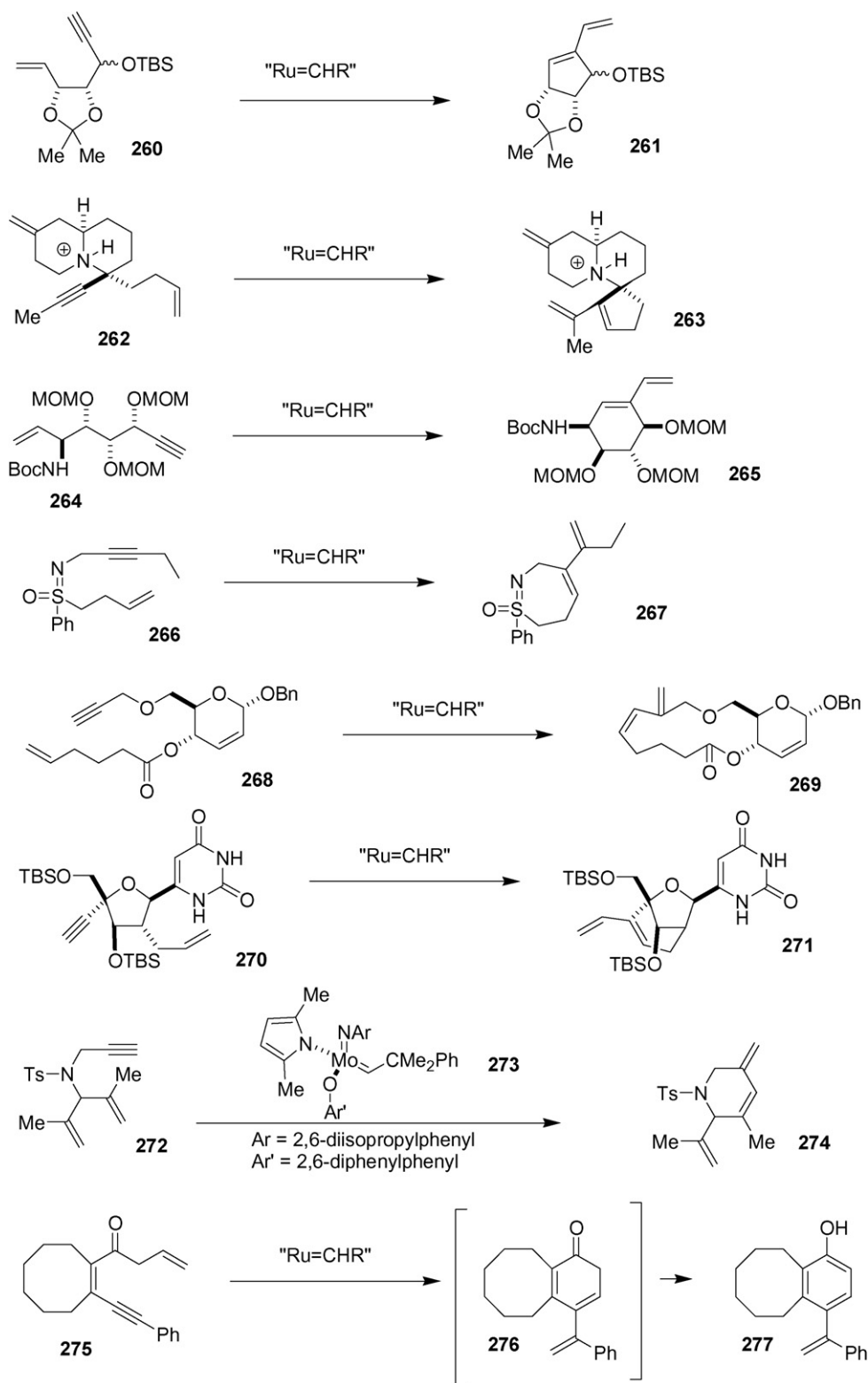
Several noncatalytic reactions were reported for ruthenium metathesis catalysts (see Scheme 29). The reaction of Grubbs catalyst I with hydridoborate ligand **331** resulted in the novel bridging hydride structure **332** [835]. The reaction of chelating silver–NHC carbene complex **333** and the Hoveyda–Grubbs catalyst (**4**) led to the expected ligand substitution product **334** accompanied by the carbene substitution product **335** [836]. The reaction of the ruthenium carbene complex metathesis catalysts (e.g. **4**) with triphenylphosphine followed by isocyanides or CO led to carbene C–C insertion products (e.g. **337**) [837]. Reactions employing electron-rich carbene complexes (e.g. **338**) led to the simple ligation products (e.g. **339**). These processes were also studied computationally [838]. The lowest energy reaction pathway involves ligation to afford the octahedral complex (e.g. **340**), followed electrophilic attack of the carbene at the mesityl ring (affording **341**) and formation of the cyclopropane (**342**), followed by electrocyclic ring opening of the norcaradiene system.

2.3. Individual carbene or alkylidene complexes classified according to metal

Some studies of carbene complexes occur over a variety of metal classes. A ligand knowledge base graph for a variety of carbene ligands was developed [839]. Ligands examined include Schrock, Fischer, and Arduengo carbene ligands. Molecular covalent bond radii were computed for a variety $\text{M}=\text{CH}_2$ derivatives ($\text{M}=\text{Li}$ to element 112) [840]. Pincer carbene complexes of zirconium (**346**, Scheme 30) and palladium (**348**) were prepared and the resulting complexes studied by X-ray crystallography and DFT studies [841]. These complexes feature strong σ C–M bonds and considerably weaker π -bonds.

2.3.1. Group 4 metal–carbene complexes

Both isolable titanium–carbene complexes and reactions that involve titanium alkylidene complexes as intermediates are covered in this section.

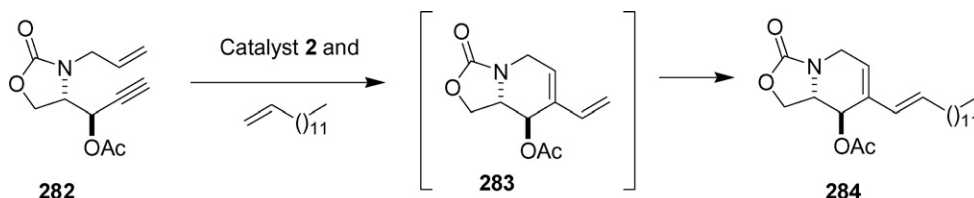
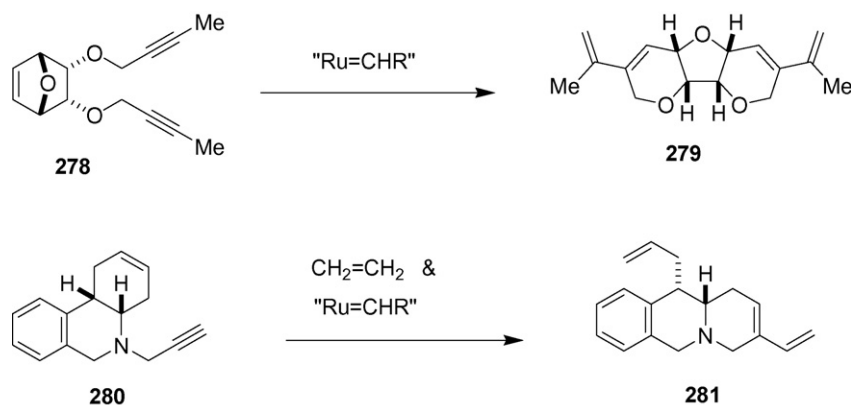


Scheme 18.

The coupling of titanium carbene complexes (e.g. **350**, Scheme 31) with aromatic fluorides was reported [842]. The reaction initially produces the C–H activation products (e.g. **353**) in a mechanism involving generation of a carbyne complex (**351**) followed by C–H oxidative addition and hydrogen migration. Thermolysis of these complexes leads to the carbene-fluoride complex

355 accompanied by formation of benzyne intermediate (**357**), which was successfully trapped in a Diels–Alder reaction.

Several examples employing *in situ*-generated titanium-carbene complexes in synthetic organic chemistry were demonstrated in 2009; representative examples are depicted in Scheme 32. Several examples of titanium-mediated alkene-



carbonyl group comethathesis (e.g. the conversion of **358** to **359**, **360** to **361**, or **362** to **363**), which likely involves titanium alkylidene intermediates, were reported [843–845]. The Petassis reagent (Cp_2TiMe_2) or the Tebbe reagent [$\text{Cp}_2\text{Ti}(\mu\text{-Cl})(\mu\text{-CH}_2)\text{AlMe}_2$] was employed in several carbonyl olefinations (e.g. conversion of **364** to **365**) [685,699,846–851]. Tandem Petassis olefination and Ferrier rearrangement (e.g. conversion of **366** to **368**) was a key step in the total synthesis of okilactomycin; the same synthesis also employs a macrocyclic RCM event [852]. The tandem Petassis olefination and Ferrier rearrangement process was also employed in the total synthesis of spongistatin 1 [853].

2.3.2. Group 5 metal–carbene complexes

The generation and reactivity of vanadium carbene complexes (e.g. **371**, Scheme 33) was reported [854]. Dialkylvanadium(III) complex **370** serves as a source of alkylidene complex **371**, which reacts with a variety of reagents. Reaction of complex **370** with calcogenide sources (e.g. S₈) led to the corresponding vanadium(V) alkylidene complexes (e.g. **372**). The selenide and telluride complexes were considerably less stable. The corresponding imido complex (**373**) was obtained through reaction of complex **370** with trimethylsilyl azide. The reaction with nitriles under nitrogen resulted in nitrile–alkylidene coupling product **376**. The mechanism for formation of this product involves [2+2]-cycloaddition (affording **374**) followed by ring opening to produce the imido complex (**375**), followed by reaction with nitrogen. Reaction with azobenzene resulted in the imido(iminoacyl)vanadium complex **378** in a process involving [2+2]-cycloaddition followed by ring opening through N–N bond cleavage.

Niobium pincer carbene complex **380** (Scheme 34) was prepared through deprotonation of pincer alkyl complex **379** [855]. The X-ray structure of **380** revealed that the Nb=C double bond was significantly shorter than the Nb–C single bond. The double bond was attributed to σ -donation and π -donation from the carbene carbon. The carbene complex was inert to further deprotonation—a bis(carbene) complex could not be generated.

The reaction of deuterium labeled compound **381** (Scheme 35) with the organolithium reagent **382** afforded a pentaalkyltantalum species, which evolved to the carbene complex at 25 °C [856].

The pentaalkyltantalum complex was observable by NMR. The deuterium isotope effect for the α -hydride elimination process was 14. Isolation of the ^1H analog of complex **383** was considerably more difficult.

The synthesis and reactivity of benzylidene-tantalum complexes (e.g. **386**, Scheme 36) was reported [857]. Heating the dibenzyl complex **385** results in its conversion to the carbene complex **386**, which exists as equilibrating syn and anti rotamers. The carbene complex was reacted with a variety of organic compounds. Reaction with protic substances led to addition products (e.g. **387**). Reaction with CO led to the η^2 -ketene complex (**388**). Reaction with ketones, and esters led to carbonyl olefination products (e.g. **390**) and an oligomeric tantalum oxo species (**389**). Reaction with carbodiimides led to stable [2+2]-cycloadducts. Reaction with nitriles led to vinylimido complexes (e.g. **392**) in a process involving [2+2]-cycloaddition followed by ring opening.

Additional examples pertaining to Group 5 metal carbene complexes are depicted in [Scheme 37](#). Niobium carbene complexes (e.g. **394**) were suggested as likely intermediates in niobium-catalyzed cyclization of isoindolines containing *o*-trifluoromethylphenyl groups (e.g. **393**) [858]. Carbene complex formation was not observed in a study of the reaction of tantalum(V) complexes with diphenyl diazomethane [859].

2.3.3. Group 6 metal–carbene complexes (further classified according to structure and reaction type)

2.3.3.1. *Schrock-type carbene complexes.* A significant portion of this subject material has already been presented in the alkene metathesis section; the Schrock catalyst (**5**) belongs to this class of compounds. Additional studies unrelated to olefin metathesis are depicted here. The propagating species in phenylacetylene poly-

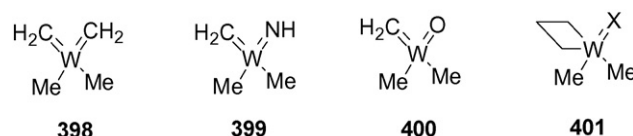
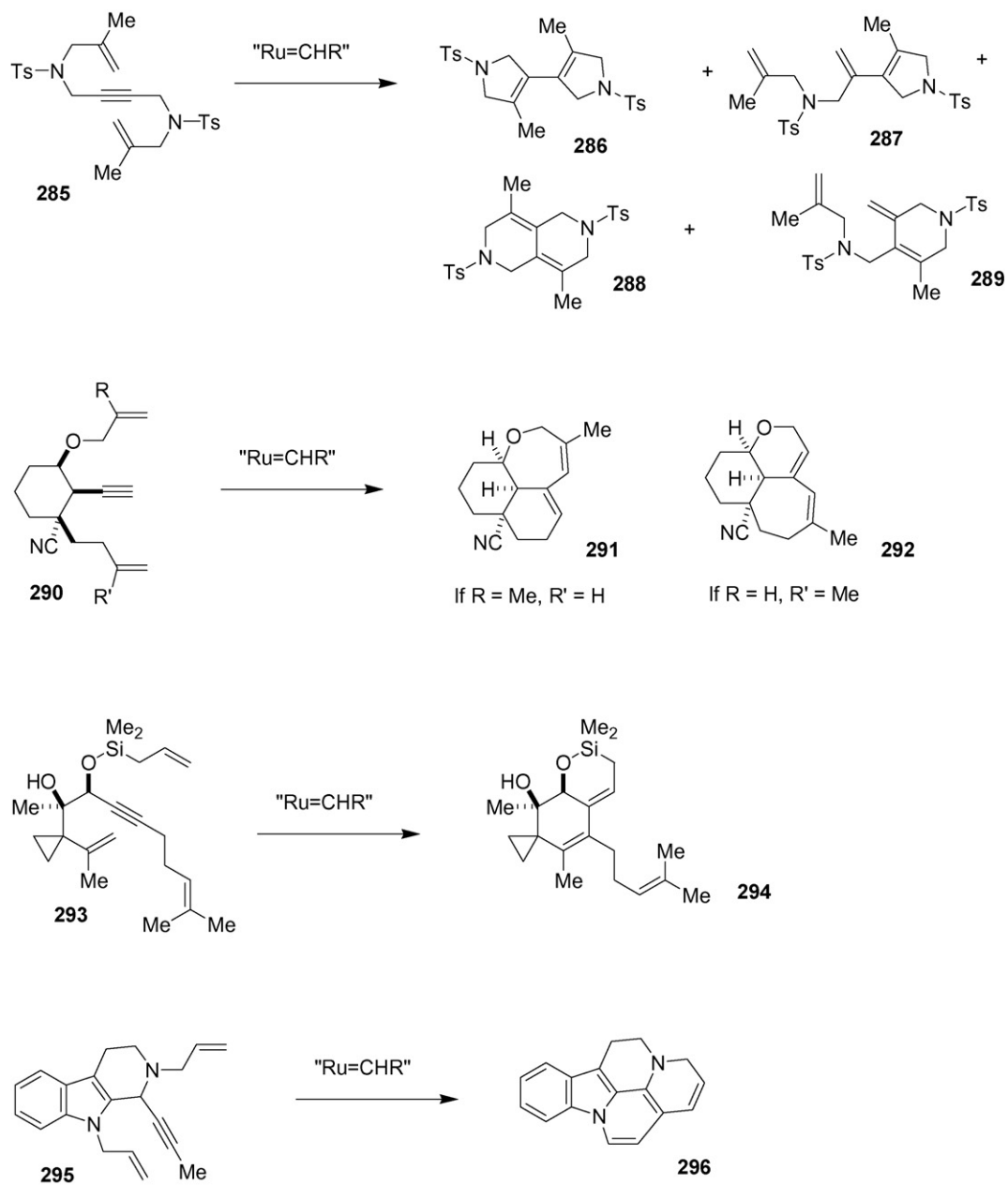
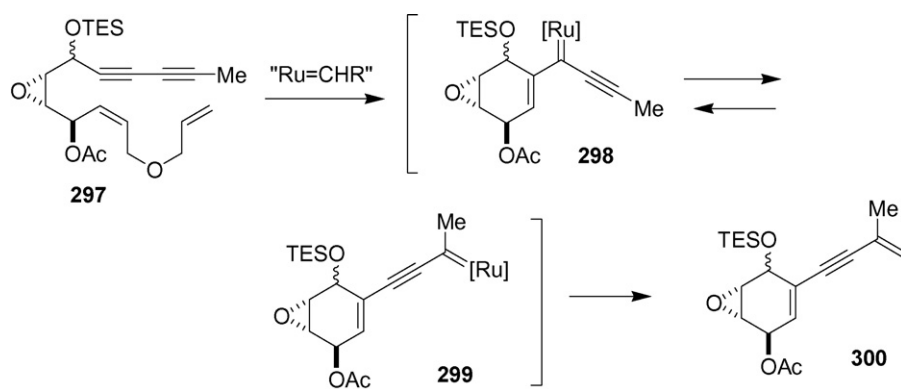


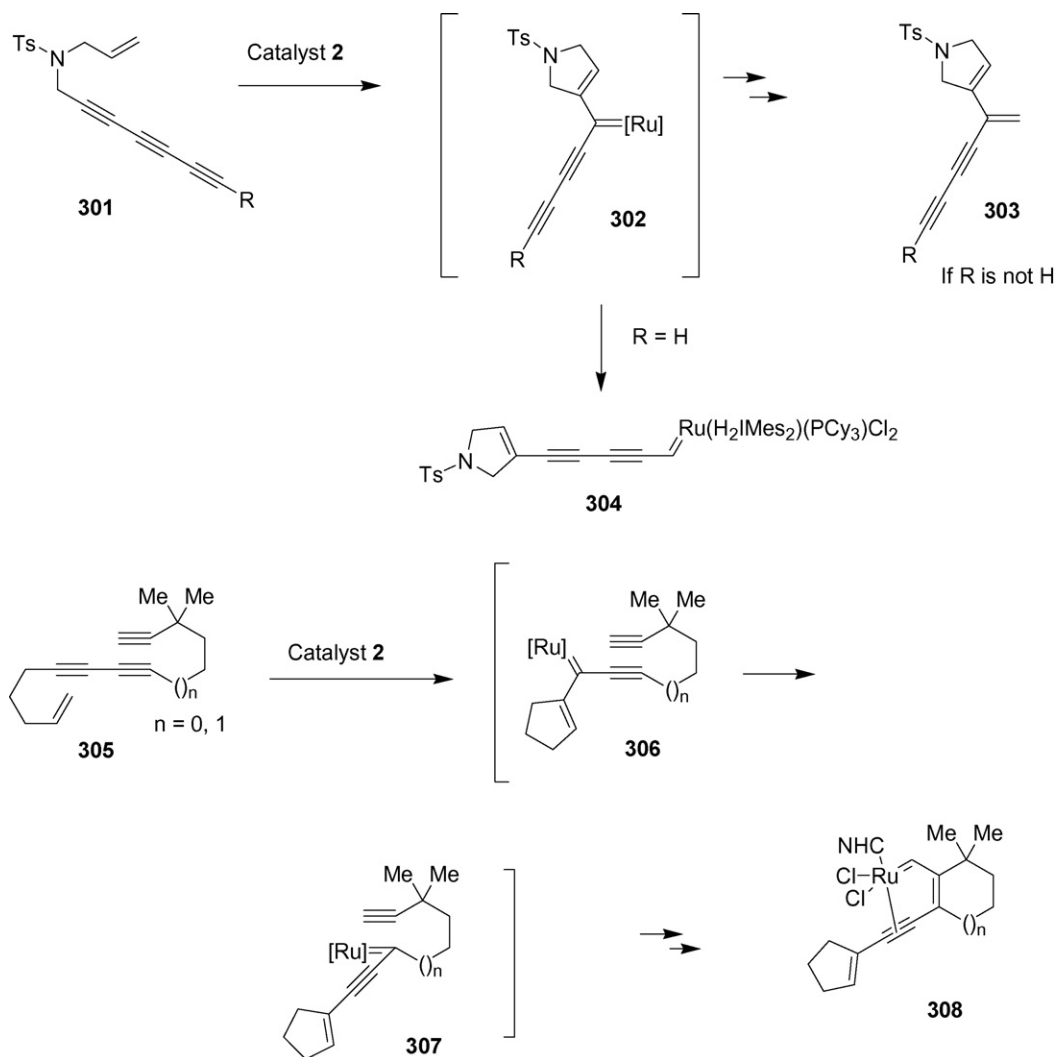
Fig. 12. Tungsten–carbene complexes and ethylene addition products.



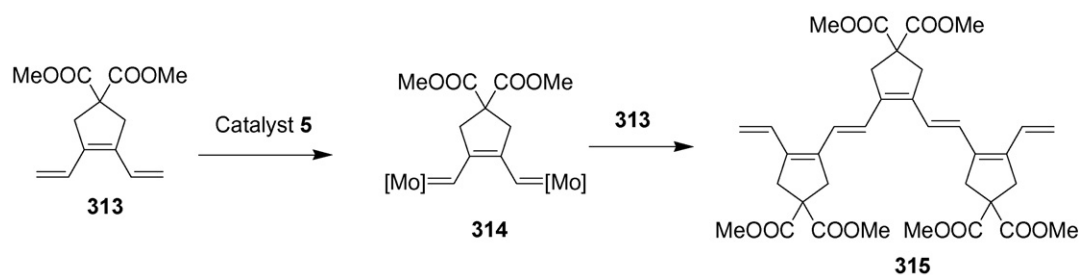
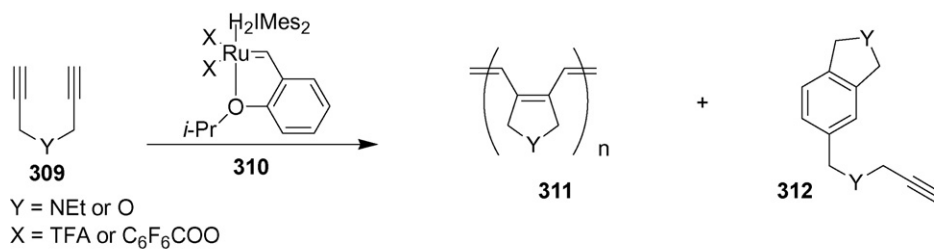
Scheme 21.



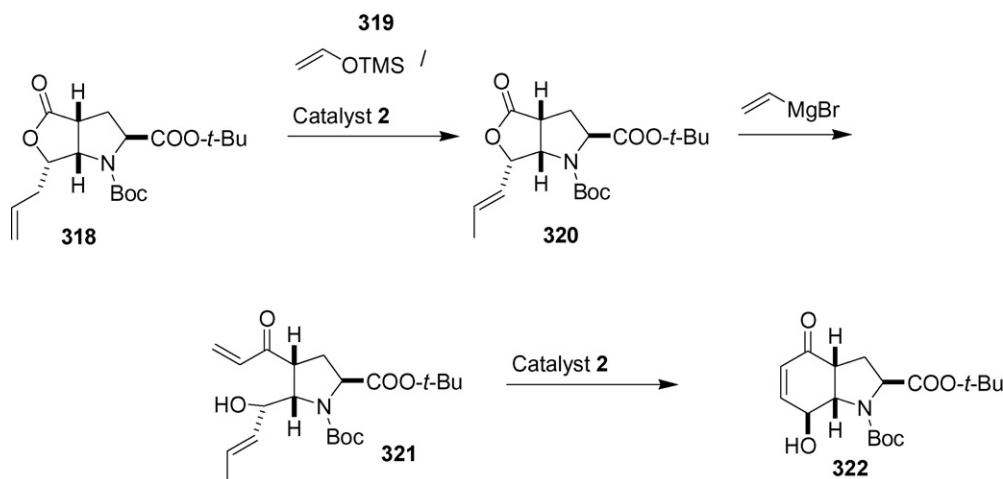
Scheme 22.



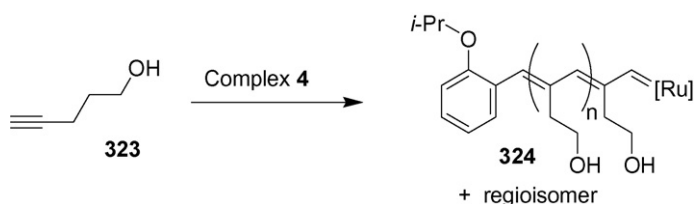
Scheme 23.



Scheme 24.



Scheme 25.



Scheme 26.

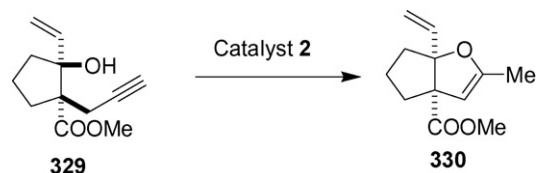
merization by $\text{Na}_2[\text{W}_2(\mu\text{-Cl})_3\text{Cl}_4(\text{THF})_2]x(\text{THF})_3$ was identified by ^1H NMR signals in the range of δ 11–13 [860]. The reaction of tungsten methylene complexes **398–400** (Fig. 12) with ethylene was evaluated computationally [861]. In this case the preferred pathway was [2+2]-cycloaddition. Addition to the carbene ligand was kinetically and thermodynamically more favorable than addition to the tungsten-heteroatom double bond.

2.3.3.2. Publications focusing on synthesis, formation, or physical properties of Fischer carbene complexes of Group 6 metals. Bridging carbyne complexes of Group 6 metals (e.g. **403**, **404**, **406**, see Scheme 38) were produced in the coupling of dimetal carbonyl complexes (e.g. **402**) with alkyl triflates of acyl halides [862]. Reactions with alkyl halides were more complex. Reaction with acryloyl chloride (**405**) led to the bridging acyloxy carbyne complex **406** and a vinylmolybdenum com-

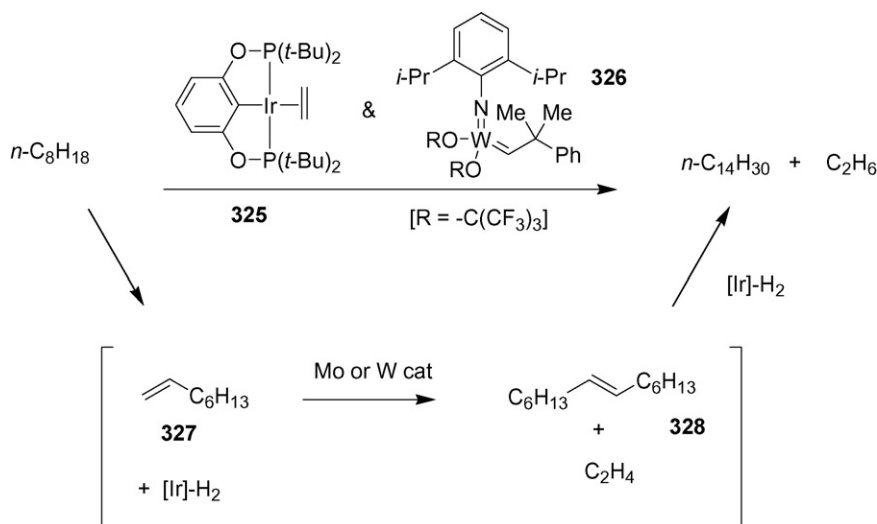
plex (**407**) obtained through direct acylation at molybdenum followed by decarbonylation [863]. Reaction of similar bridging carbyne–dimolybdenum complexes with cationic gold complexes leads to complexation at the phosphine–molybdenum linkage [864].

2.3.3.3. Reaction of Group 6 metal–carbene complexes with alkenes and dienes. This section focuses on reactions of Group 6 metal–carbene complexes involving coupling with alkenes at the carbene carbon. Other examples of the coupling of carbene complexes with alkenes where the reactive site is elsewhere can be found ahead under the heading: cycloaddition reactions occurring at the C–C π -bond of α,β -unsaturated metal–carbene complexes (Section 2.3.3.6).

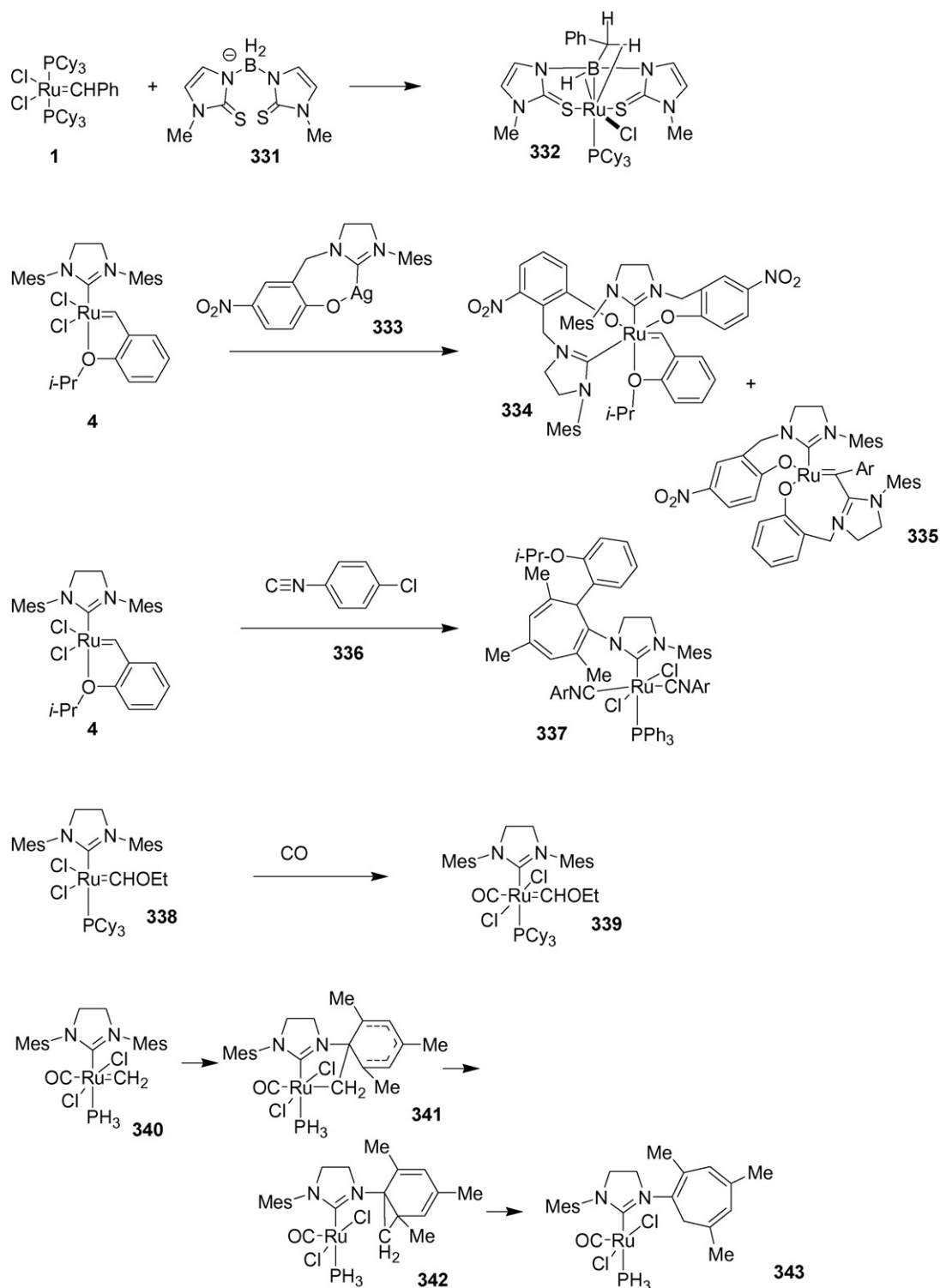
The tandem reaction of alkynylcarbenes (e.g. **408**, Scheme 39) with a strained alkene (e.g. **409**) and another alkene (e.g.



Scheme 28.



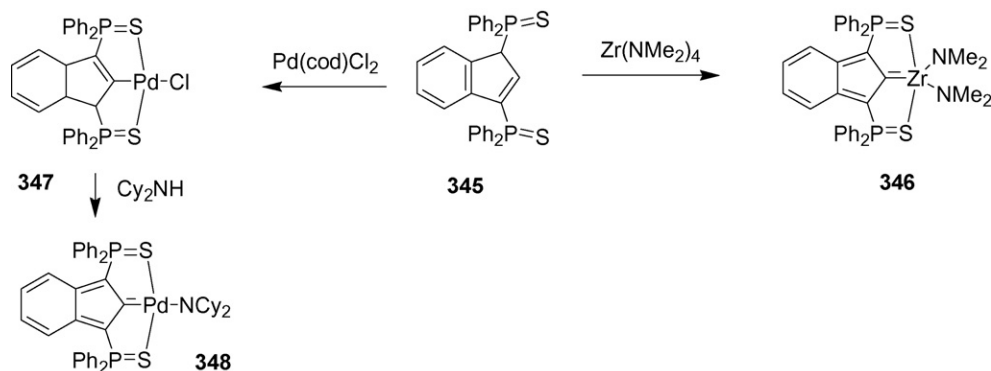
Scheme 27.



Scheme 29.

cyclopentene) was reported [865]. The reaction is a net tandem [2+2+1]/[2+1]-cycloaddition process that results in cyclopropylcyclopentene derivatives (e.g. **410**). A mechanism involving formation of a metallacyclobutene (**411**), followed CO insertion to afford the metallacyclopentanone (**412**), followed by a propargyl shift resulting in an unstabilized carbene complex (**413**) was proposed. Capture of the carbene complex intermediate through a cyclopropanation reaction affords the observed product. A variety of different intramolecular tethering schemes were tested.

2.3.3.4. Reaction of Group 6 metal–carbene complexes with alkynes—benzannulation. Many examples of benzannulation using α,β -unsaturated chromium–carbene complexes (see Scheme 40) and alkynes (commonly known as the Dötz reaction) were reported in 2009. Examples are include: (1) synthesis of naphthylcarbene–chromium complexes (e.g. **415**) (and higher nuclearity analogs) and subsequent benzannulation reactions [866]; (2) synthesis of the individual naphthyl rings of VAPOL ligands from arylcarbene complexes (e.g. **417**) [867]; (3) synthesis of arylcarbene complexes



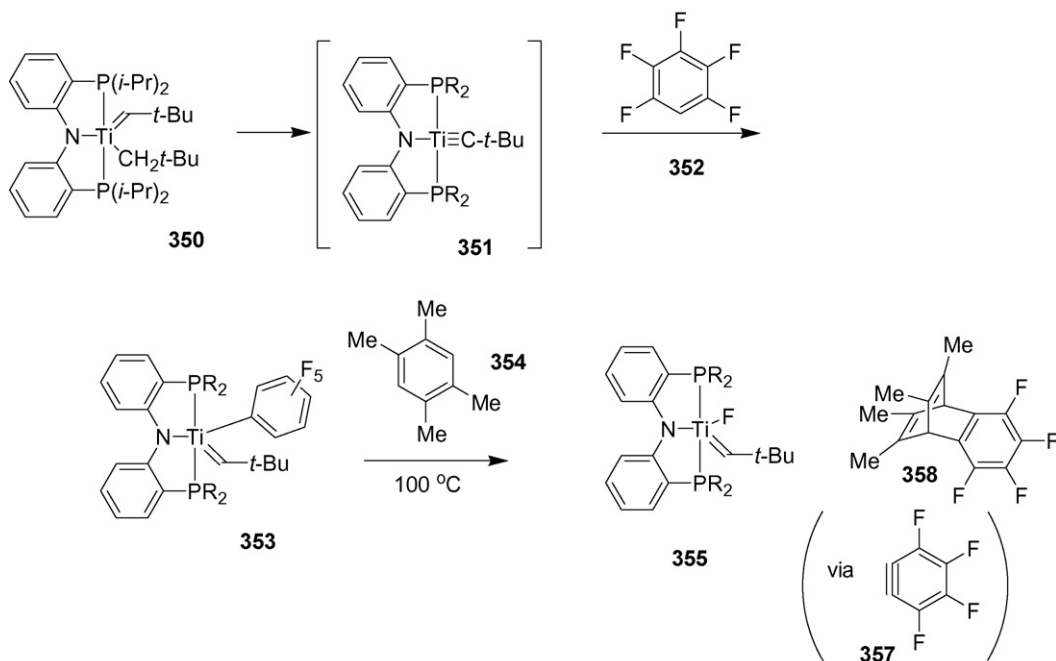
Scheme 30.

containing a cymantrene group (e.g. **421**) and subsequent benzannulation reactions with alkynes, followed by investigation of haptotropic rearrangements [868]; (4) synthesis of phenanthrene- $\text{Cr}(\text{CO})_3$ complexes (e.g. **424**) and subsequent studies of ligand exchanges and haptotropic rearrangements [869]; (5) selective monobenzannulation and dibenzannulation reactions for various bis(carbene) complexes fused to five-membered heteroaromatic ring systems (e.g. **426**) (dimerization and redox reactions were also reported) [870]; (6) an intramolecular benzannulation process involving 2-alkynylaryloxycarbene complexes (e.g. **428**) [871]; and (7) synthesis of calixarenes (e.g. **432**) in a double-benzannulation – macrocyclization process [872]. A computational study of the Dötz reaction was reported (see Scheme 41) [873]. These studies emphasize the effect of substituents on the energetic and predicted benzannulation or cyclopentannulation reaction pathways. Several conclusions emerged from these studies. The studies suggest that benzannulations involving alkenylcarbene complexes (e.g. **433**) proceed through chromahexatrienes (**435**), while those involving phenylcarbene complexes (e.g. **438**) proceed through vinylketenes (**440**). The optimal pathways in each case are depicted in Scheme 41.

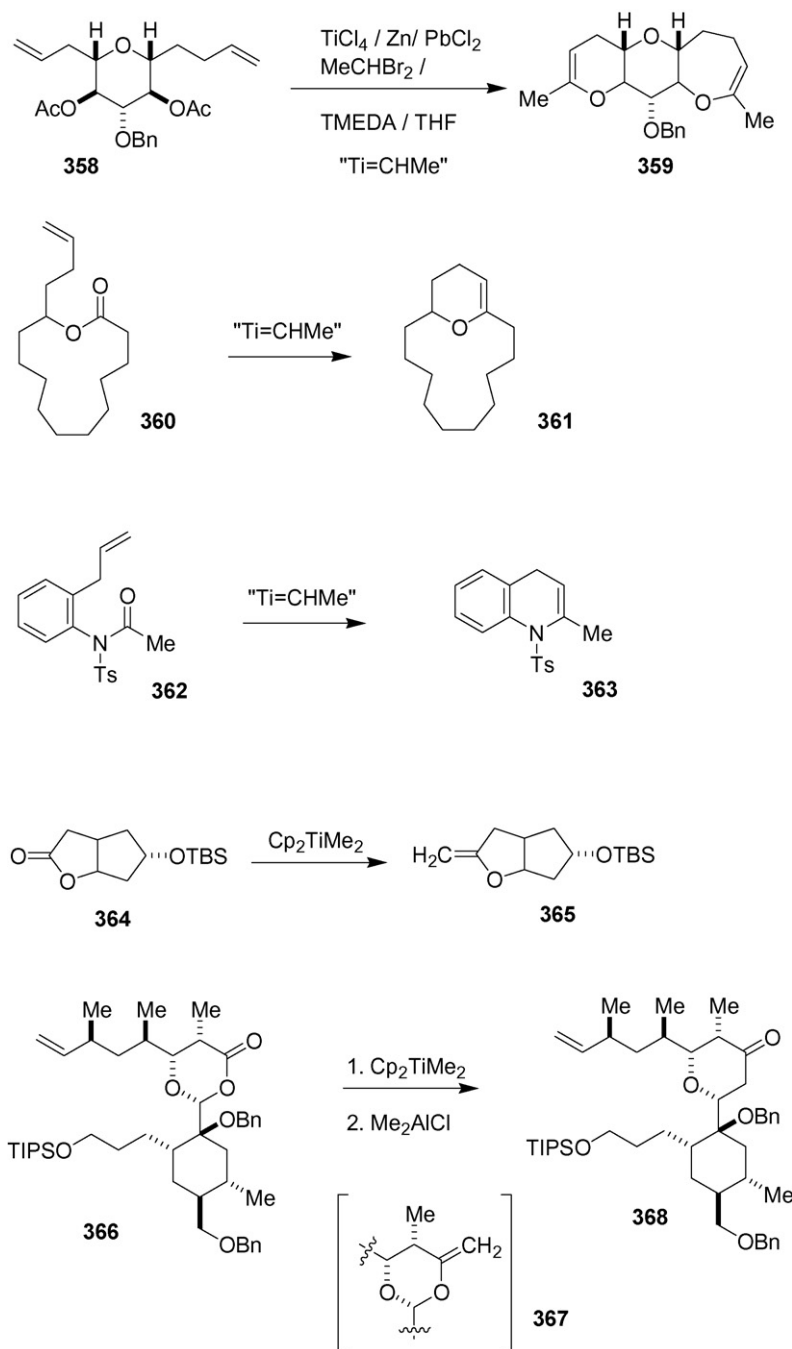
2.3.3.5. *Nonbenzannulation reactions of Group 6 metal–carbene complexes with alkynes.* Several processes other than benzannulation that involve the capture of vinylcarbene complexes generated from the coupling of carbene complexes and functionalized alkynes were reported in 2009.

The coupling of arylpropargyl alcohols (e.g. **443**, Scheme 42) and Fischer carbene complexes under solvent-free conditions led to alkylidene- γ -lactones (e.g. **446**) [874]. Subsequent treatment with acid in the air afforded the corresponding furanones (e.g. **447**). This reaction process likely involves the formation of a vinylketene intermediate (**445**), which reacts with the alcohol to afford the lactone.

The coupling of carbene complexes and 3-alkynylpyridine-4-carbaldehydes (e.g. **448**, Scheme 43) and ketones was reported [875]. The reaction leads to the formation of azaisobenzofuran intermediates (**451**), which undergo subsequent inter- and intramolecular Diels–Alder reactions to afford isoquinoline derivatives (e.g. **452**). The reaction employing γ,δ -unsaturated carbene complexes (e.g. **453**) proceeded through a similar series of events to afford aza-hydrophenanthrenes (e.g. **455**). Quinoline analogs of the starting alkynes were also reported [876].



Scheme 31.



Scheme 32.

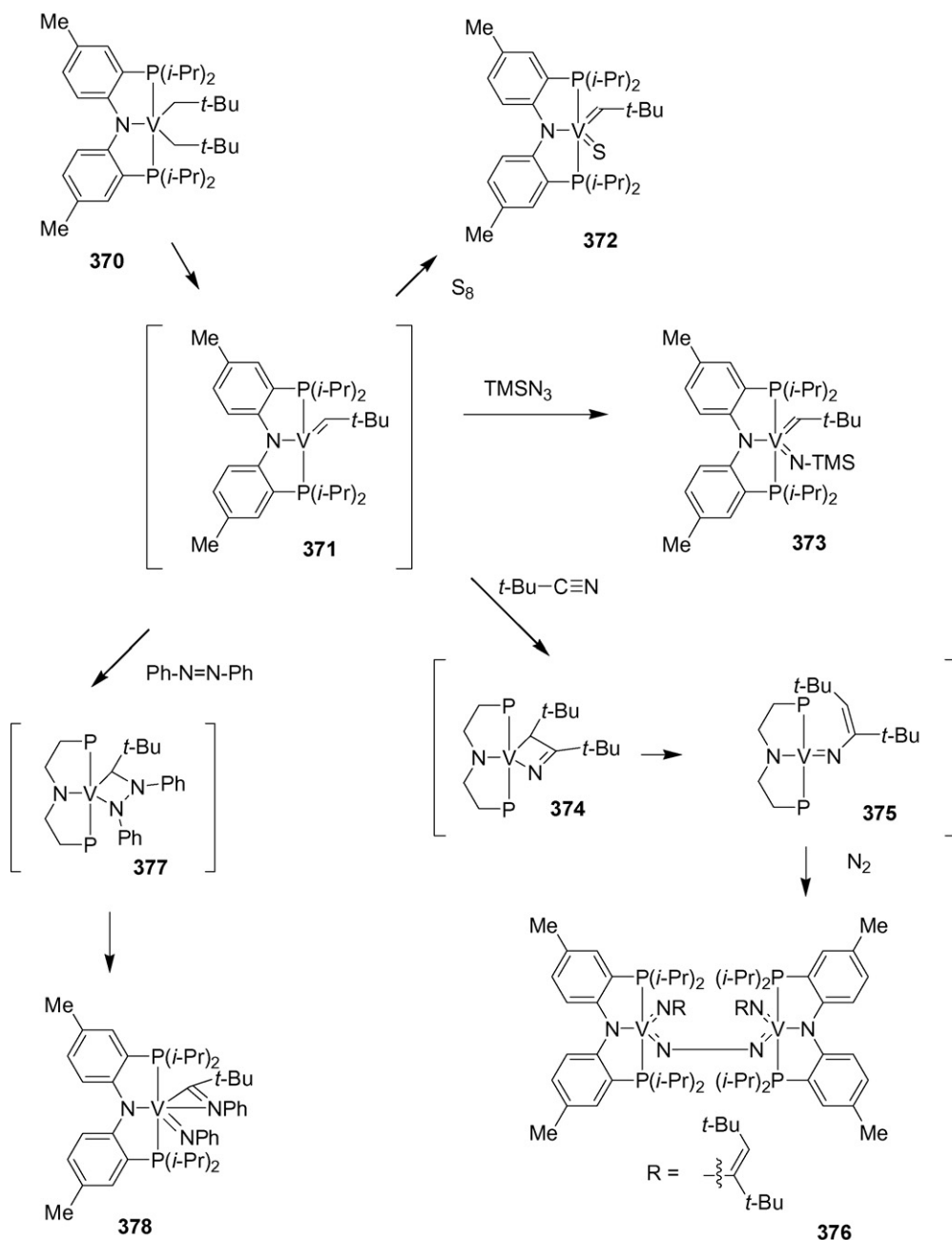
2.3.3.6. *Reactions occurring at the conjugated C–C π -bond of α,β -unsaturated Group 6 metal-carbene complexes.* The coupling of alkynylcarbene complexes (**457**, Scheme 44) with tropolone imines (e.g. **456**) was reported [877]. The reaction proceeds through a net [8+2]-cycloaddition. When the R group is unsaturated, secondary cyclization processes to afford phenols (e.g. **459**) was observed. Aminocarbazoles were observed if isocyanides were added to these reactions.

Use of alkynylcarbene complexes (e.g. **460**, Scheme 45) to accomplish three-carbon annulation onto an enamine or an indole ring (e.g. **461**) was reported [878]. The mechanism involves Michael addition followed by ring closure to a cyclic allene complex (**463**). Protonation/hydrolysis on silica gel affords the final product. Enantiomerically pure products were obtained if the carbene complex

contains a chiral alkoxide group. The addition of stabilized carbanions (e.g. **466**) to enynylcarbene–chromium complexes (e.g. **465**) led to cyclized compounds (e.g. **469**) [879]. A mechanism involving Michael addition followed by cyclization with chromium migration, followed by protonolysis of the C–Cr bond was proposed.

The base-catalyzed reaction of alkynylcarbene complex **470** (Scheme 46) (and the tungsten analog) and dithranol (**471**) resulted in a variety of product types (**472–475**), all derived from Michael's addition of a phenolic species to the alkynylcarbene complex [880]. The choice of reaction conditions has a dramatic effect on the observed reaction pathways.

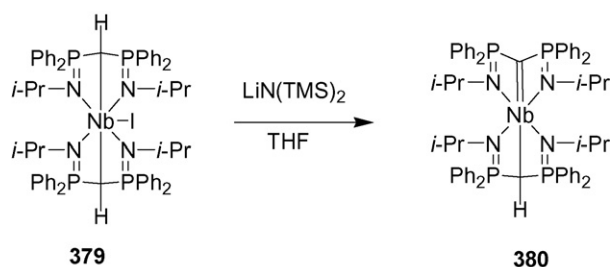
The hydride reduction of pyranlydene metal complexes (e.g. **476**, Scheme 47) led to cyclopentenones (e.g. **481–483**) [881]. The proposed mechanism involves hydride addition to the γ -position



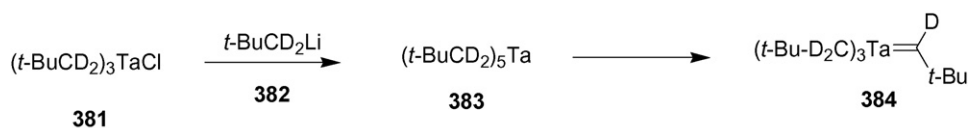
Scheme 33.

followed by elimination to form a dienoyl–acylate complex (478), followed by cyclization and reductive elimination to form a five-membered ring extended enolate species (e.g. 480). This reaction mechanism was supported through deuterium labeling studies. Addition of acid affords a mixture of conjugated (e.g. 482) and unconjugated (e.g. 481) cyclopentenones. The product could also be quenched with acid chlorides to afford the α -acylcyclopentenones (e.g. 483). Similar products could also be obtained from dienoyl acylate complexes generated from dienyllithium reagents and Group 6 metal hexacarbonyls.

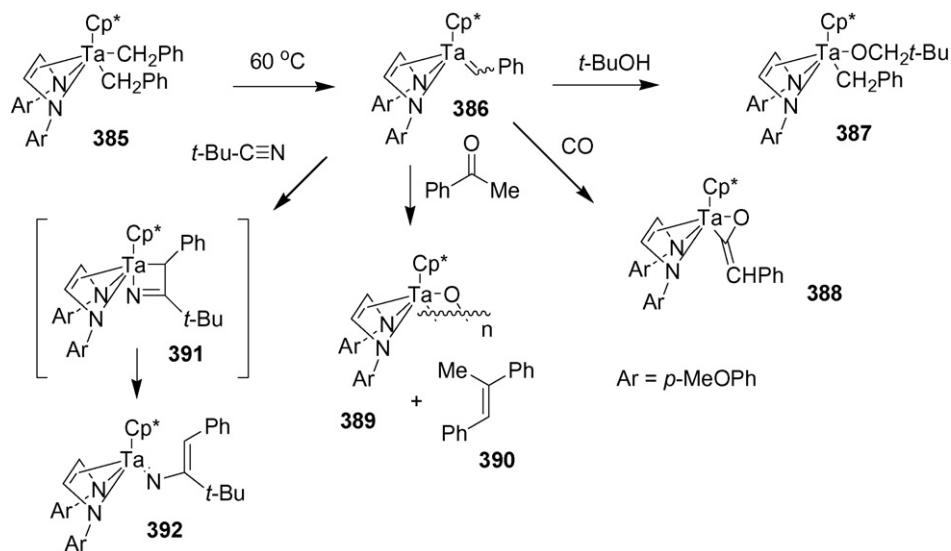
2.3.3.7. Synthesis and reactivity of Group 6 metal–vinylidene complexes, and reactions that involve vinylidene–metal complexes as intermediates; also includes other process that involve the formation of a carbene complex from an alkyne and a noncarbene metal complex. The synthesis and reactivity of ferrocenyl-substituted



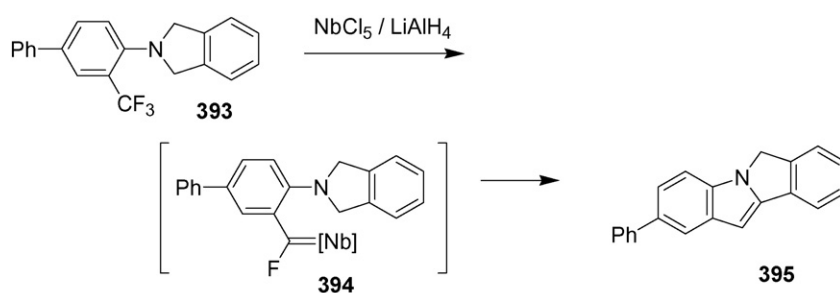
Scheme 34.



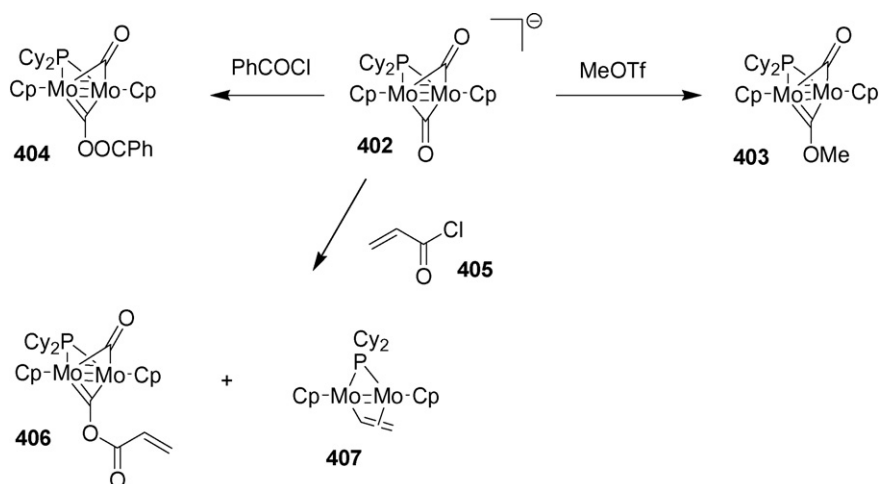
Scheme 35.



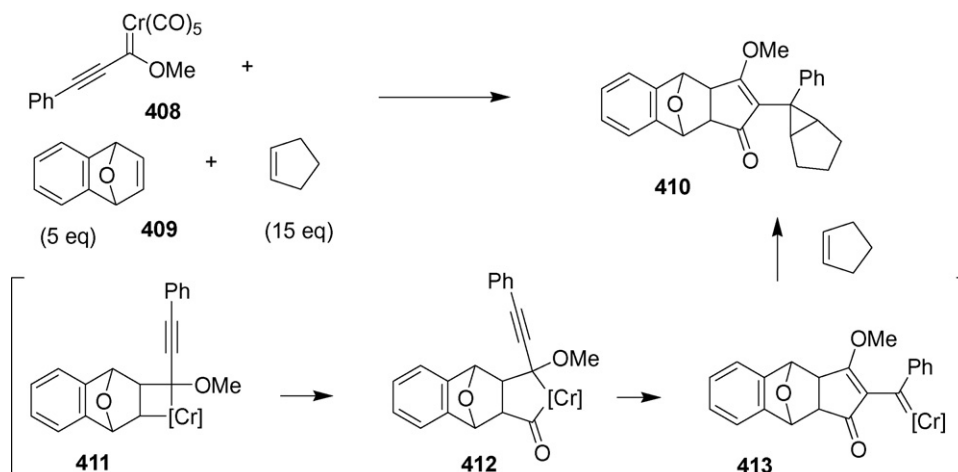
Scheme 36.



Scheme 37.



Scheme 38.



Scheme 39.

allenylidene–chromium, molybdenum, and tungsten complexes (see Scheme 48) was reported [882]. The bis(ferrocenyl) complexes (**485**) were prepared via sequential treatment of propargylic alcohol **484** with butyllithium and M(CO)_5 sources. The three routes depicted were employed for the synthesis of monoferrocenylallenylidenes. Additional analogs were prepared through net substitution reactions involving amino (e.g. **490**) or alkoxyallenylidenes (e.g. **491**).

Tungsten–butatrienylidene complexes (e.g. **496** and **497**, Scheme 49) were prepared from the reaction of tungsten–dinitrogen complex **494** with stannylbutadiynyl derivatives (e.g. **495**) [883]. Reaction of the initially formed stannylated complexes (**496**) with water leads to the protiodestannylation product (**497**). After extended time at room temperature, the butatrienylidene complex converts to a dimeric complex (**500**) featuring two tungsten(I) groups. A mechanism involving formation of the bis(alkylidene)cyclobutene derivative (**498**) through [2+2]-cycloaddition followed by ring opening was proposed. The bis(alkenylidene) complex features resonance contribution from a bis(alkynyltungsten) complex (**499**) where each tungsten is in the odd-electron +1 oxidation state. The dimeric complex **500** undergoes an irreversible two-electron reduction.

Tungsten vinylidenes complexes (e.g. **502**, Scheme 50) and cyclic carbene complexes (e.g. **503**) are key intermediates in the cycloisomerization of alkynols to cyclic enol ethers (e.g. **504**). This process was a key step in the preparation of saccharomycins [884].

A computational study of the tungsten-catalyzed reaction of 2-alkynylbenzaldehyde derivatives (e.g. **505**, Scheme 51) and vinyl ethers (e.g. **506**) to afford tricyclic compounds (e.g. **509** and **510**) was reported [885]. These studies support the key step in this process where heterocyclization affords an intermediate 1,3-dipole derivative (**507**), which undergoes cycloaddition with an enol ether to form the bicyclic carbene complex (**508**). This process is stereoselective due to secondary molecular orbital interactions. Subsequent intramolecular C–H insertion or intermolecular Si–H insertion with silane additives then affords the observed products.

Tungsten vinylidene complexes were briefly mentioned as potential intermediates for the tungstoenzyme acetylene hydratase hydration of acetylene, but the major focus was on an η^2 -alkyne complex based mechanism [886].

2.3.3.8. Reactions involving carbanions derived from deprotonation of Group 6 metal–carbene complexes. The attempted alkylation of various Group 6 methyl methoxycarbene complexes (**511**, Scheme 52) under phase transfer conditions was reported [887]. The course of the reaction was highly dependent on the metal, the amount

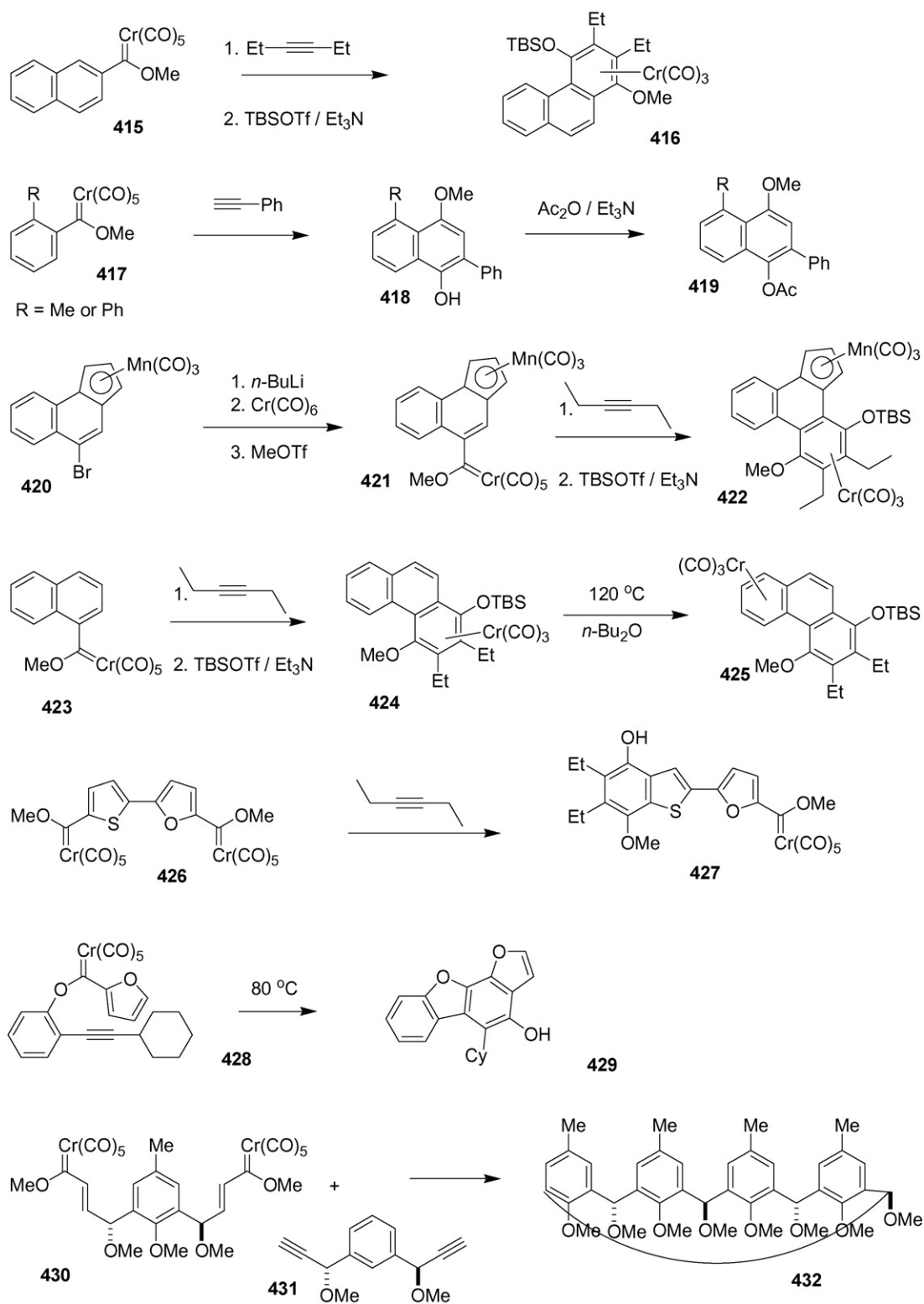
of phase transfer catalyst employed, and the base concentration. Molybdenum and tungsten complexes underwent either alkylation (e.g. formation of **512**) or formation of polyoxometallates (**513**). Optimal formation of alkylation products employs 50% NaOH solution and 0.1 mol% of the phase transfer catalyst *n*-Bu₄NBr. Optimal formation of the polyoxometallates was realized using 1 equiv. of the phase transfer catalyst and 33% NaOH solution. The polyoxometallates were also successfully produced using the metal hexacarbonyls. Polyoxometallates were not produced from chromium complexes. Vinylstannanes (e.g. **516**) were prepared through deprotonation of alkoxy carbene complexes followed by quenching with tributyltin triflate [888]. Presumably this process involves stannylation of the intermediate anion derivative (**515**).

2.3.3.9. Reactions involving the addition of nucleophiles to the carbene carbon. Ferrocenyl thioamides (e.g. **519**, Scheme 53) were obtained through aminolysis of ferrocenyl alkoxy carbene complexes (e.g. **517**) followed by reaction of the resulting aminocarbene complex (**518**) with sulfur [889]. Aminolysis using silylated amines (e.g. **520**) was employed for the grafting of ferrocenyl carbene complexes onto a silicon surface [890].

The three-component coupling of Fischer carbene complexes (e.g. **523**, Scheme 54), enolates (e.g. **524**), and allyl–Grignard reagents was evaluated computationally [891]. The calculations emphasize the effect of the R group on formation of either a five-membered ring (when $\text{R} \neq \text{H}$) through direct cyclization of the key intermediate **525** or a six-membered ring through CO insertion and cyclization in cases where $\text{R}=\text{H}$.

Coupling of carbene complexes (e.g. **523**, Scheme 55) with alkynyllithiums followed by reaction with furfural imines (e.g. **528**) led to substituted benzofurans (e.g. **534**) [892]. The initial reaction affords a non-heteroatom stabilized carbene complex (e.g. **529**) that has a reasonable lifetime at low temperatures. Subsequent treatment with furfural imine proceeds via Michael's addition the furan ring to the alkynyl carbene complex (affording **531**) followed proton transfer to afford a carbene complex–imine (**532**), followed by [2+2]-cycloaddition and β -hydride elimination.

2.3.3.10. Reactions that involve transfer of a Fischer carbene ligand to another metal. Transfer of a Fischer carbene ligand from chromium to rhodium or gold was reported (see Scheme 56) [893]. The reaction of β -aminoalkenyl carbene complex **535** with terminal alkynes led to cyclic carbene complexes that are not directly stabilized by a heteroatom (e.g. **536**). Subsequent reaction with rhodium or gold salts led to the carbene transmetallation products (e.g. **538**, **540**). The reaction of aminoalkenyl carbene complexes

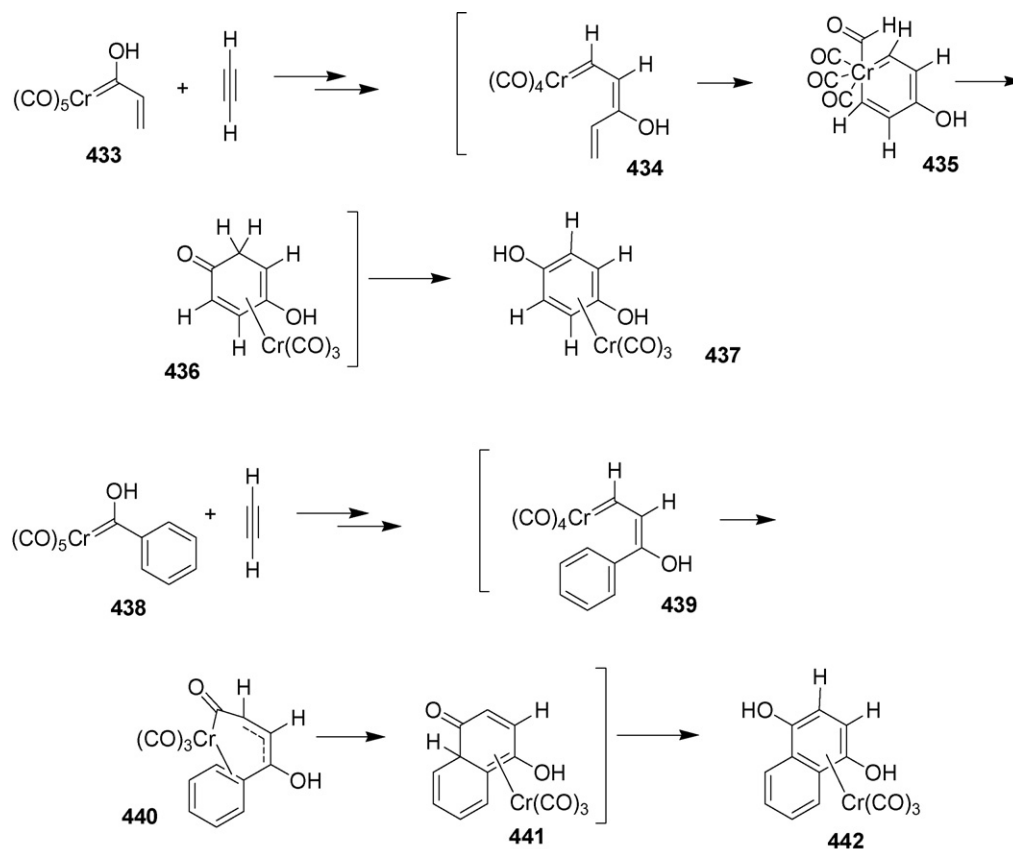


Scheme 40.

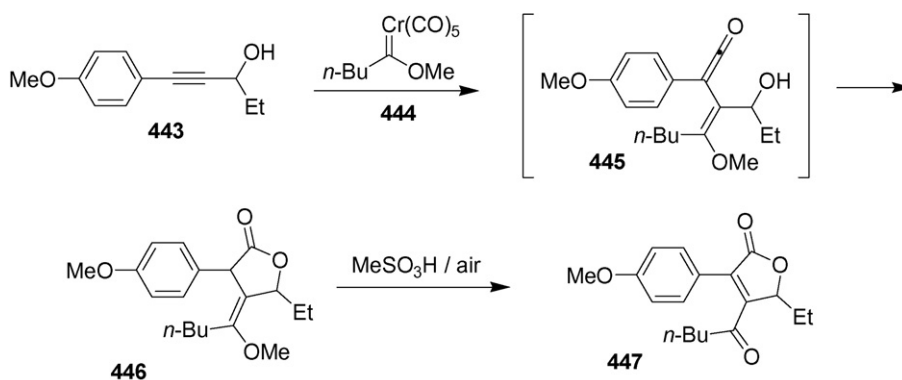
(e.g. **541**) with palladium salt **543** or platinum(II) chloride led to the bis(carbene)-Group 10 metal complexes (e.g. **544**) [894]. The electrochemical and electronic properties of the resulting complexes were studied experimentally and computationally. The reaction of α,β -unsaturated carbene complexes (e.g. **545**) with gold was reported [895]. The gold carbene complex **546** could be isolated as characterized, including an X-ray structure. Gold

complexes catalyze cyclopropanation and carbene dimerization reactions using α,β -unsaturated carbene complex **547**. Treatment of the tungsten carbene complex **547** with a gold complex led to dimerization/cyclization products (e.g. **548** and **549**).

2.3.3.11. Other reactions of Group 6 metal-carbene complexes. Click reactions were demonstrated for alkynylcarbene complexes that



Scheme 41.



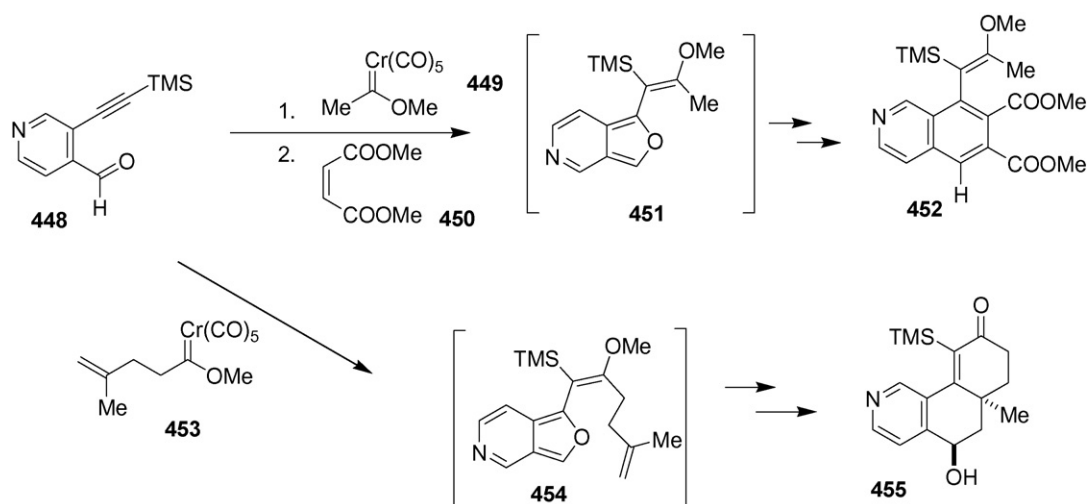
Scheme 42.

feature distant alkynyl groups (e.g. **550**, Scheme 57) [896]. Coupling of complex **550** with bis(azides) (e.g. **551**) led to bimetallic carbene complexes (e.g. **552**). The resulting complexes were studied by cyclic voltammetry.

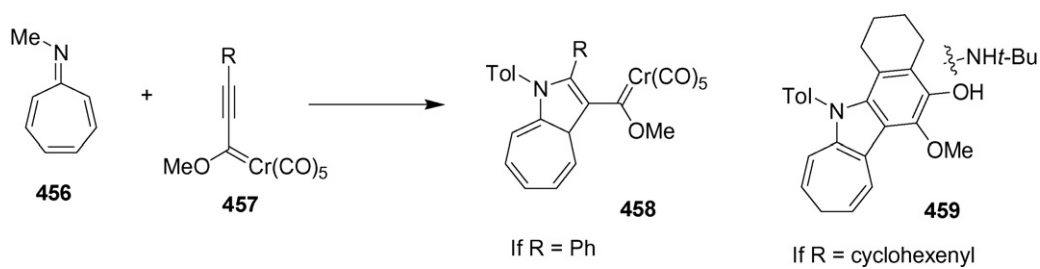
Computational and experimental studies of the photochemical reactions of metallocene-substituted chromium carbene complexes (e.g. **553**, Scheme 58) were reported [897]. Calculations predicted that the ruthenocene-substituted complexes would engage in ketene formation, while the ferrocene analogs were already known to fail. The ruthenocene complex was prepared and found to undergo ketene-like reactions. Irradiation of complex **553** ($\text{M} = \text{Ru}$) in the presence of imine derivative **554** led to the β -lactam derivative **556**. Related ketene-forming processes were also

demonstrated for carbene complexes tethered to β -lactam groups (e.g. **557**, **561**) [898].

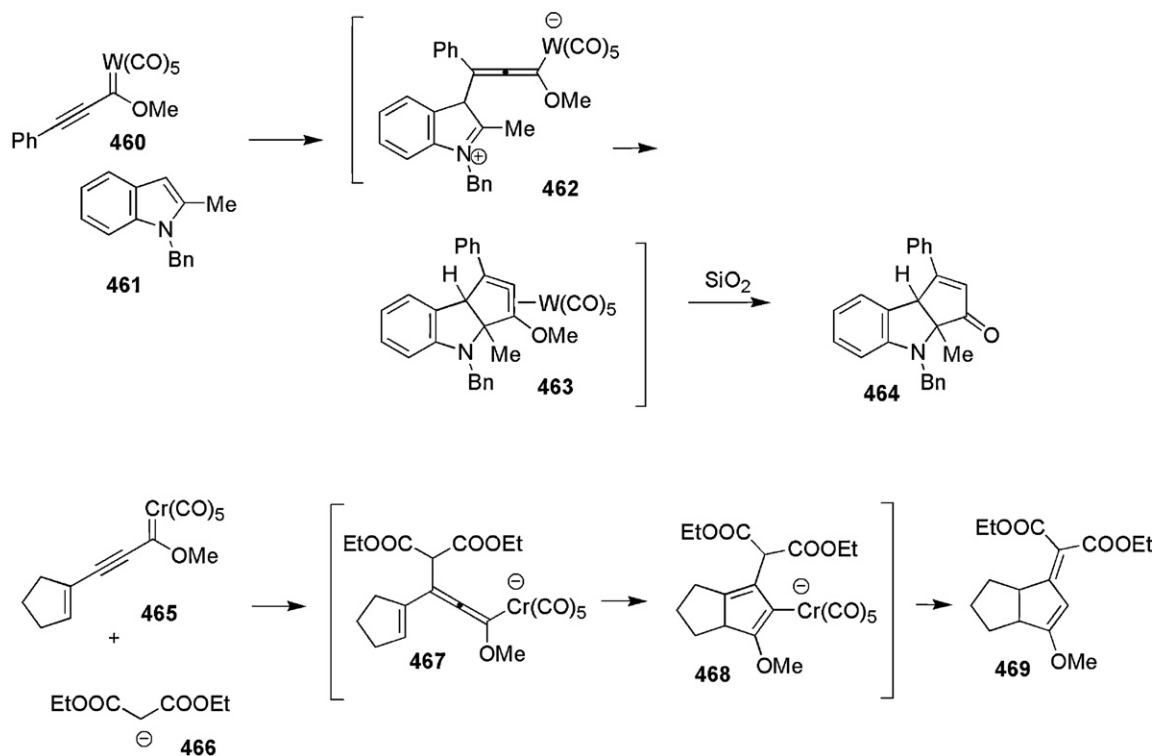
The coupling of tungsten dialkyl complex **563** (Scheme 59) with the alkene groups of various heterocyclic rings was reported [899]. All of the observed adducts were proposed to arise via the carbene complex intermediate **564**, obtained through α -hydride elimination and reductive elimination from **563**. The reaction with dihydrofuran (**570**) led to the metallacyclobutane (**571**) via a mechanism involving [2+2]-cycloaddition with carbene complex intermediate **564**. Reaction with dihydropyran (**568**) led to the heterocyclic ring opened adduct **569**. A similar type of product (**575**) was obtained through the coupling of η^2 -alkenylcarbene complex **572** with dihydropyran. The divergent behavior of dihydrofuran



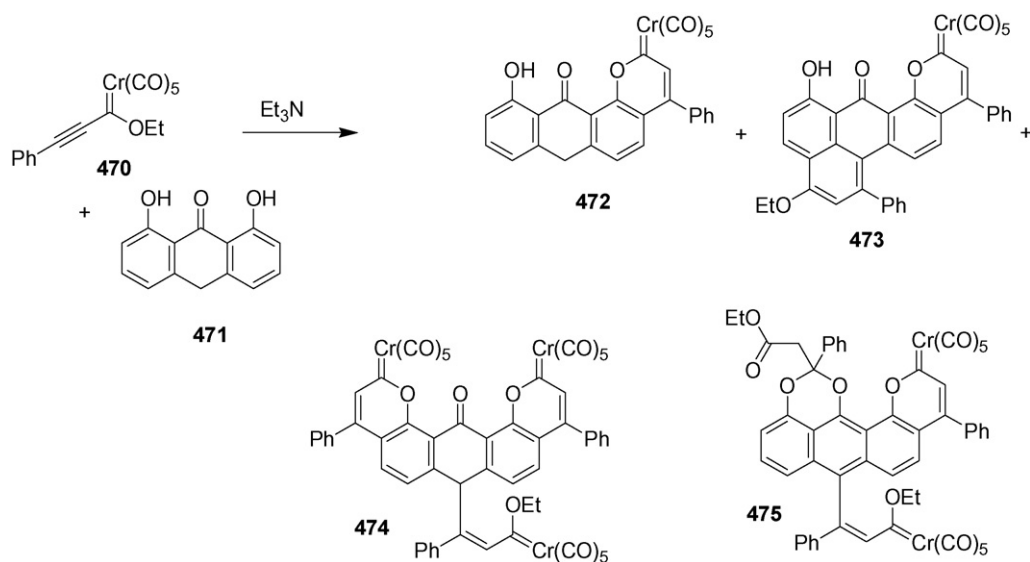
Scheme 43.



Scheme 44.



Scheme 45.

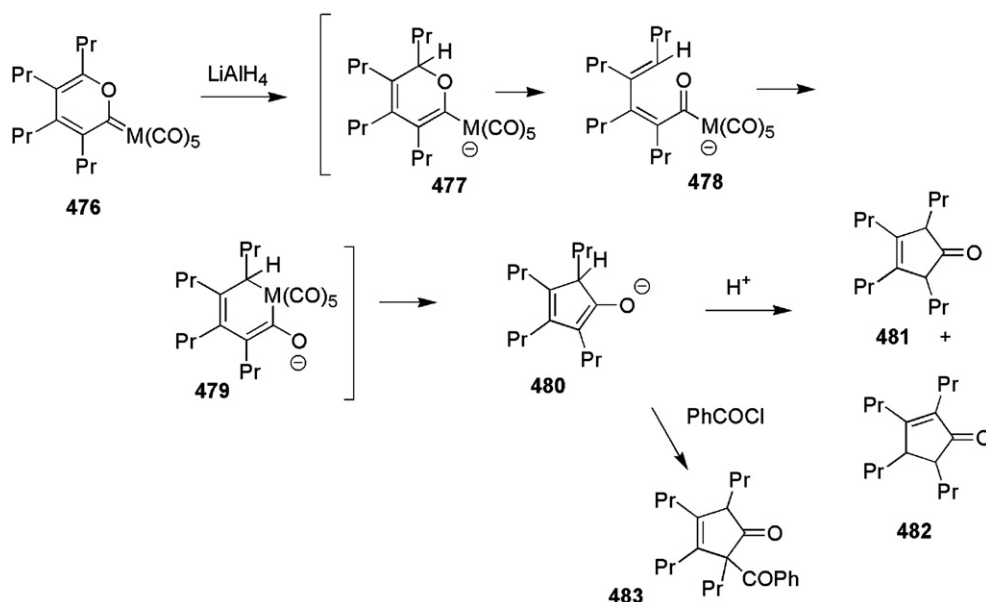


Scheme 46.

and dihydropyran was attributed to conformational flexibility in the heterocyclic ring which allows coordination of oxygen to the metal in the larger ring system. The reaction with tetrahydropyridine (**565**) led to the N–H activation products **566** and **567** as a mixture of stereoisomers.

Additional studies of Group 6 metal–carbene complexes are depicted in Scheme 60 and include: (1) reaction of aminocarbene complexes (e.g. **576**) with chlorophosphane **576** to afford the azaphosphirene complex **577** [900]; (2) possible involvement of chromium carbene complexes in the conversion of trichloromethyl allylic acetates (e.g. **578**) to dienes using chromium(II) chloride [901], addition of carbon tetrabromide/CrCl₂-derived bromomethylene to α,β -unsaturated amides [902], and a net [3+2]-cycloaddition involving RCH₂CCl₃, α,β -unsaturated ketones, and CrCl₂ [903]; (3) formation of polymetallic alkoxy carbene complex (**582**) through the reaction of tungsten carbonyl derivative **581** (or the molybdenum analog) with an yttrium hydride cluster (reaction with rhenium, rhodium, and iridium carbonyls

was also reported, however these reactions did not afford carbene complexes) [904]; (4) generation of palladium nanoparticles through the reaction of tungsten carbene complex acylates with palladium salts in ethylene glycol [905]; (5) tautomerization of an η^2 -iminoacyl allyl tungsten complex (**583**) and an aminocarbene complex (**584**) [906]; (6) the crystal structure for zirconoxycarbene–chromium complex (**585**) [907]; and (7) computational studies of thiophene/selenophene oxidative addition products, including chromathiabenzene derivatives (**588**) [in both cases, complexation of the chromium tricarbonyl fragment to arene (**586**) is more favorable than the C–E oxidative addition (affording **587/588**)] [908]. The negative ions ESI-MS of several aminocarbene complexes was studied experimentally and computationally [909]. Ionization to [M–H][–] involves acceptance of an electron from the ESI source followed by transfer of a hydrogen atom to an additive (e.g. tetrathiafulvene). Fragmentation includes loss of CO ligands and loss of alkynyl groups from alkynylcarbene complexes.



Scheme 47.

2.3.4. Group 7 metal–carbene complexes

The reaction of manganese formyl complexes (e.g. **591**, Scheme 61) with electrophiles leads to carbene complexes in a series of reactions designed to mimic C–C bond formation from syngas [910]. The boryloxycarbene complexes (e.g. **592**) are quite stable but undergo retro CO insertion upon photolysis. These complexes were not reduced by LiEt_3BH or NaEt_3BH . The traditional Fischer carbene complexes (e.g. **593**) were prepared by methylation of the formyl complexes. These complexes were reactive to hydrides to form methoxymethyl complexes (e.g. **594**), which could also be prepared through the direct reaction of metal formyl complexes with acidic methanol. The latter reaction was most effective for the synthesis of monophosphine complexes due to the instability of the metal formyl complex precursors. Subsequent carbonylation generates a methoxyacetyl complex (**595**), which upon further alkylation affords a dialkoxyethylene derivative (**596**) via formation of the carbene complex followed by a hydride shift. The silyloxycarbene complexes proved to be very unstable.

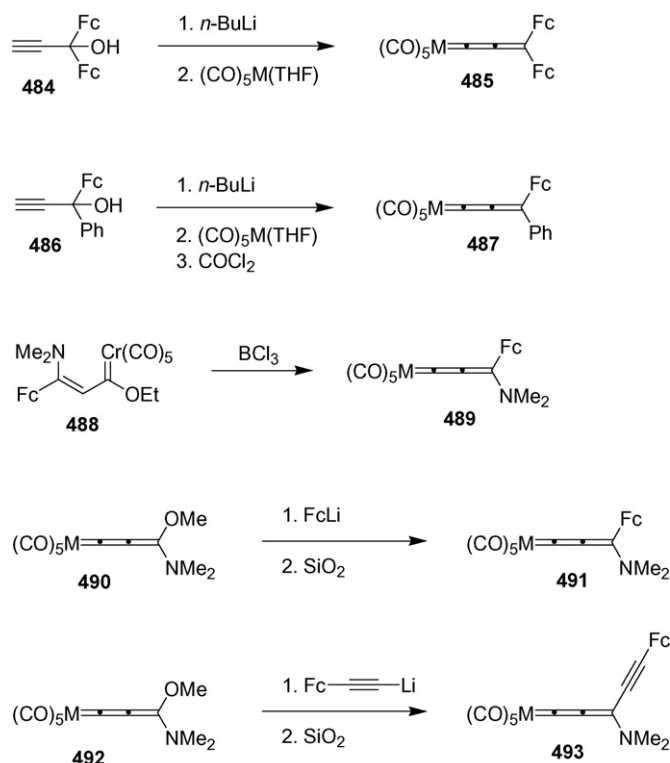
Cationic rhenium carbene complexes (e.g. **599**, Scheme 62) were generated through protonation of alkenylrhenium complexes (e.g. **598**) [911]. The protonation process was accompanied by fluoride ligation. A variety of alkenylrhenium complexes were tested in this reaction.

The formation and reactivity of cationic rhenium vinylidene complexes (e.g. **606**, Scheme 63) was reported [912]. The reaction of complex **600** with acetylene led to a mixture of acetylide complexes **601** and **602**, however when this reaction was conducted in the presence of 2,6-dimethylpyridine only the simple acetylide complex **601** was produced. The mechanism for the formation of complex **602** involves formation of the hydride alkynyl complex **603**, followed by NO insertion and addition of oxygen to the complexed acetylide ligand. This mechanism was supported by DFT studies. Protonation of acetylide complex **601** afforded the vinylidene complex (**606**), which afforded the Fischer carbene complexes (e.g. **607**) upon treatment with thiophenol or benzene-selenol. The reaction of rhenium vinylidene complex **608** with either $\text{Pd}(\text{PPh}_3)_4$ or $\text{Pt}(\text{PPh}_3)_4$ led to the bridging bimetallic complexes (**609**) [913,914].

The reaction of $\text{H}_2\text{C}=\text{ReO}_3$ with ethylene was studied computationally [915]. The thermodynamically and kinetically favored cycloaddition pathway is [3+2]-cycloaddition of C–Re–O to the alkene.

2.3.5. Group 8 metal–carbene complexes

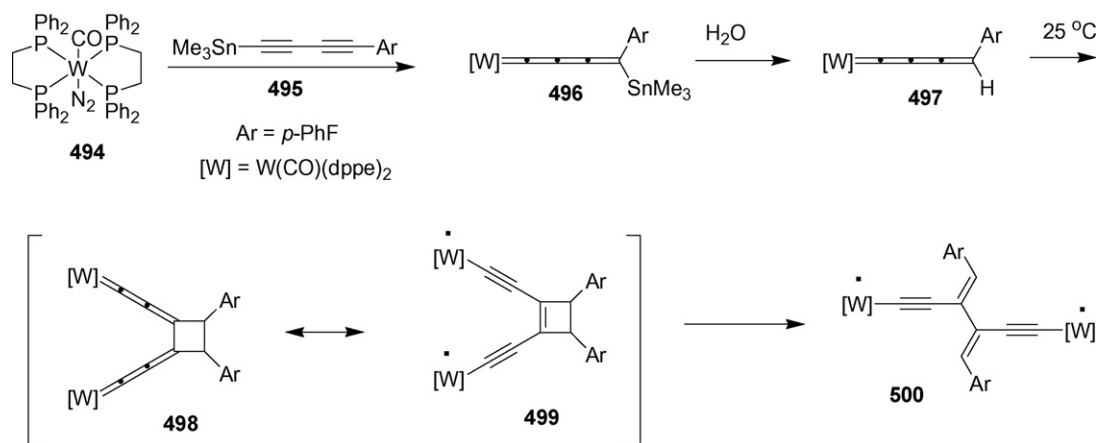
2.3.5.1. Non heteroatom-stabilized carbene complexes. Numerous additional examples of the synthesis and reactivity of this class of



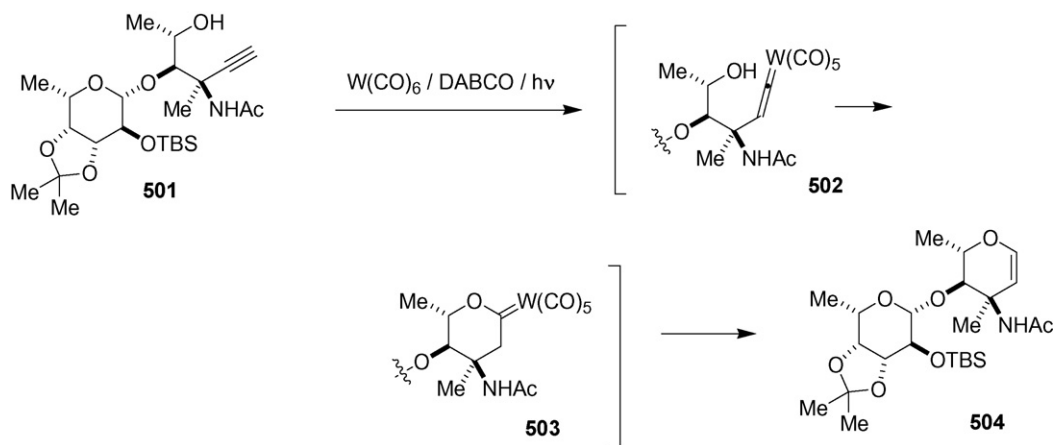
Scheme 48.

compounds have been presented in the alkene metathesis section. The Grubbs catalysts fall into this classification.

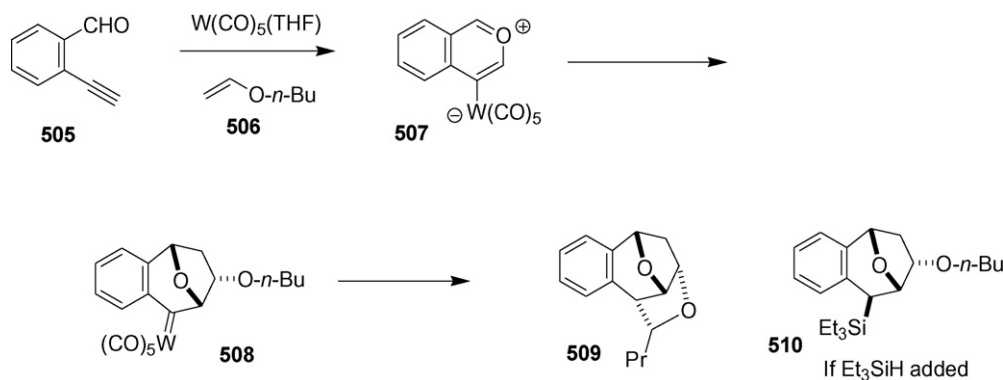
Several examples of the formation of Group 8 carbene complexes from diazo compounds were reported in 2009 (see Scheme 64). Ruthenium carbene complex (**614**) was prepared through direct reaction of complex **613** with phenyldiazomethane [916]. The role of ruthenium carbene complexes (e.g. **617**) in ruthenium–PNNP-complex (**615**) catalyzed aziridination reactions of imines by diazo compounds was clarified [917]. Although the ruthenium PNNP complex **615** catalyzed the formation of aziridines from indenones and ethyl diazoacetate, the isolated carbene complex **617** does not react with imines (e.g. **618**) to form aziridines except in the presence of an excess of diazoacetate. The reaction of carbene complex **617** with ethyl diazo acetate leads to diethyl maleate (**620**). Based on these observation aziridine formation was attributed to the NMR-observable ruthenium diazo complex (**616**) and not the carbene complex **617**. Ruthenium and



Scheme 49.



Scheme 50.



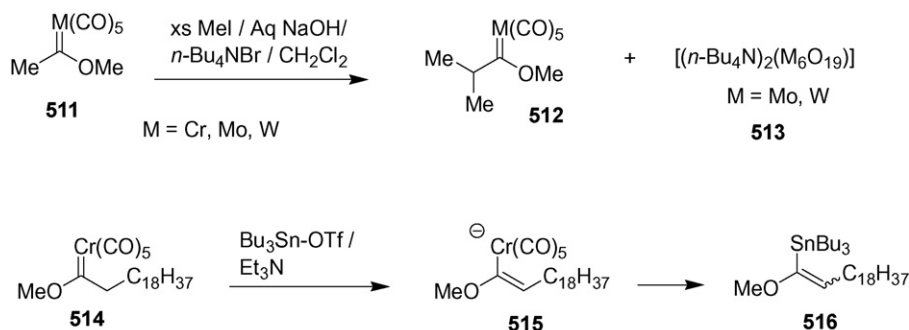
Scheme 51.

osmium carbene complexes (**622**) were prepared from the reaction of metal–porphyrin complex **621** with diazo compounds [918]. The carbene complex **622** could be isolated and characterized. Reaction of the carbene complex with styrene led to the cyclopropanation product. The initial complex also serves as a catalyst for cyclopropanation.

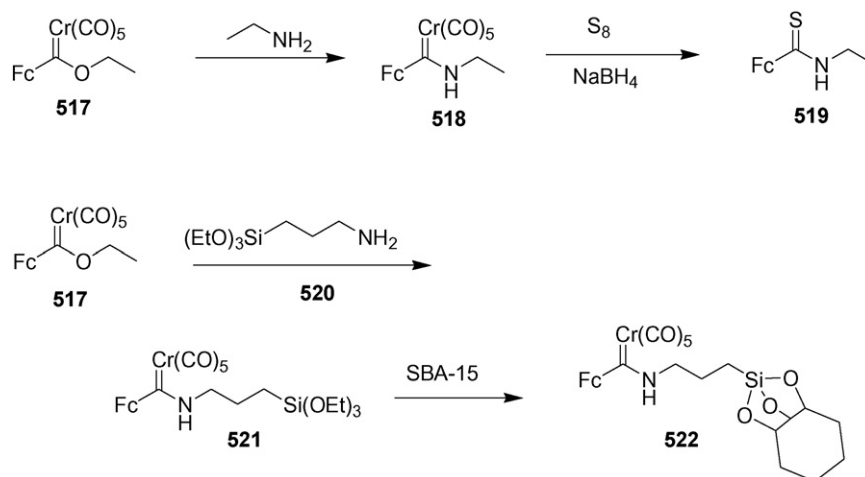
Ruthenium carbene complexes (e.g. **628** and **630**, Scheme 65) were suggested as intermediates in the reaction of propargylic esters (e.g. **624**) with diazo compounds in the presence of a ruthenium catalyst (**625**) to afford diene derivatives (e.g. **626**) [919]. Formation of the diene product was attributed to either of two mechanistic possibilities: (1) formation of a carbene complex (**628**) through rearrangement of the alkyne complex (**627**) followed by coupling with the diazo compound, or (2) formation of a carbene complex from the diazo compound (**629**) followed by alkyne insertion, acetate migration, and demetallation.

Other examples of diazo compound generated Group 8 metal carbene complexes are depicted in Scheme 66, and include: (1) competing diazo coupling, cyclopropanation and metathesis in the ruthenium-catalyzed reaction of diazo compounds (e.g. **631**, **636**) with various alkenes (the reaction pathway depends on the alkene) [920]; (2) catalysis of asymmetric cyclopropanation reactions using iron porphyrins [921]; (3) generation of ruthenium carbenes at an electrode surface through reaction of surface-bound ruthenium species with α -diazo esters [922]; (4) experimental and computational studies of asymmetric cyclopropanation catalyzed by ruthenium salen complexes [923]; and (5) possible involvement of iron carbene complexes in iron porphyrin catalyzed cyclopropanation [924,925] and ketene generation [926].

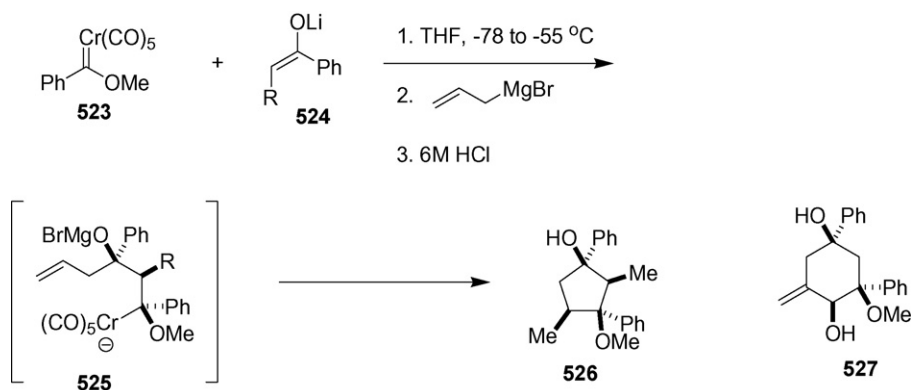
Additional studies of nonheteroatom-stabilized Group 8 carbene complexes are depicted in Scheme 67 and include: (1) contribution of a carbene resonance form (**641**) to the structure



Scheme 52.



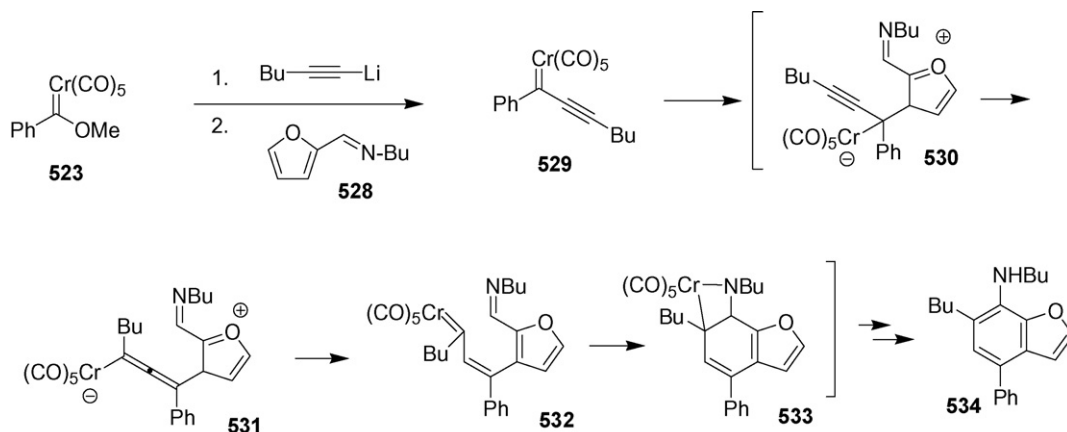
Scheme 53.



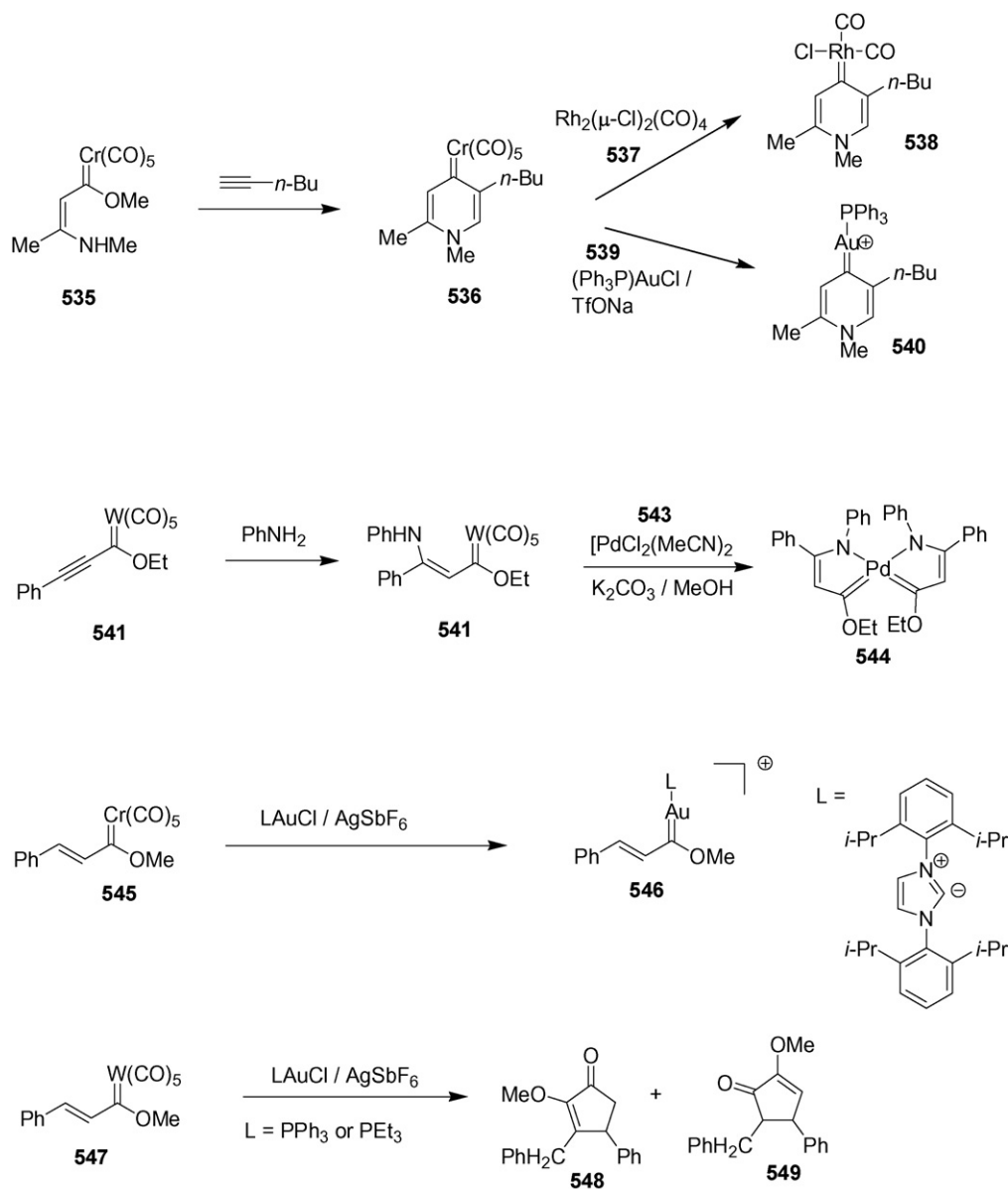
Scheme 54.

of the one-electron oxidation product of ruthenavinylferrocene complex **639** [927]; (2) ruthenium-catalyzed reaction of alkenes with α,α -dichloroesters and magnesium to afford cyclopropane derivatives [928]; (3) a computational study of the molecular and electronic structure of iron carbene complexes of the general formula $[\text{L}_2\text{CpFe}=\text{CR}_2]^+$ ($\text{L}=\text{CO}$ or phosphines) [929]; and (4) DFT calculation of the structure and NMR properties of $(\text{CO})_4\text{Fe}=\text{CF}_2$ [930].

2.3.5.2. *Bis(carbene)ruthenium complexes from coupling of two alkynes and a ruthenium complex.* Competitive allenylidene formation (e.g. **644**, Scheme 68) and cyclic bis(carbene) complex (e.g. **646**) generation were observed in the coupling of diynes of general structure **642** with ruthenium complex **643** [931]. The reaction employing the diyne where $\text{R}=\text{H}$ led to the allenylidene complex **644**. The reaction employing the methyl analog led exclusively to the π -allyl carbene complex **647** via the bis(carbene) complex



Scheme 55.

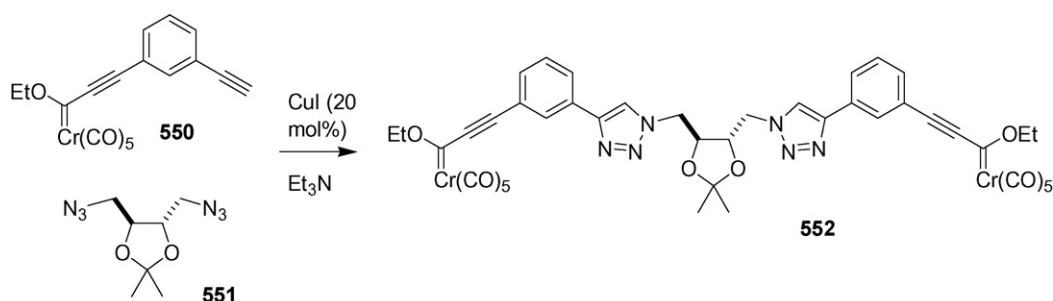


Scheme 56.

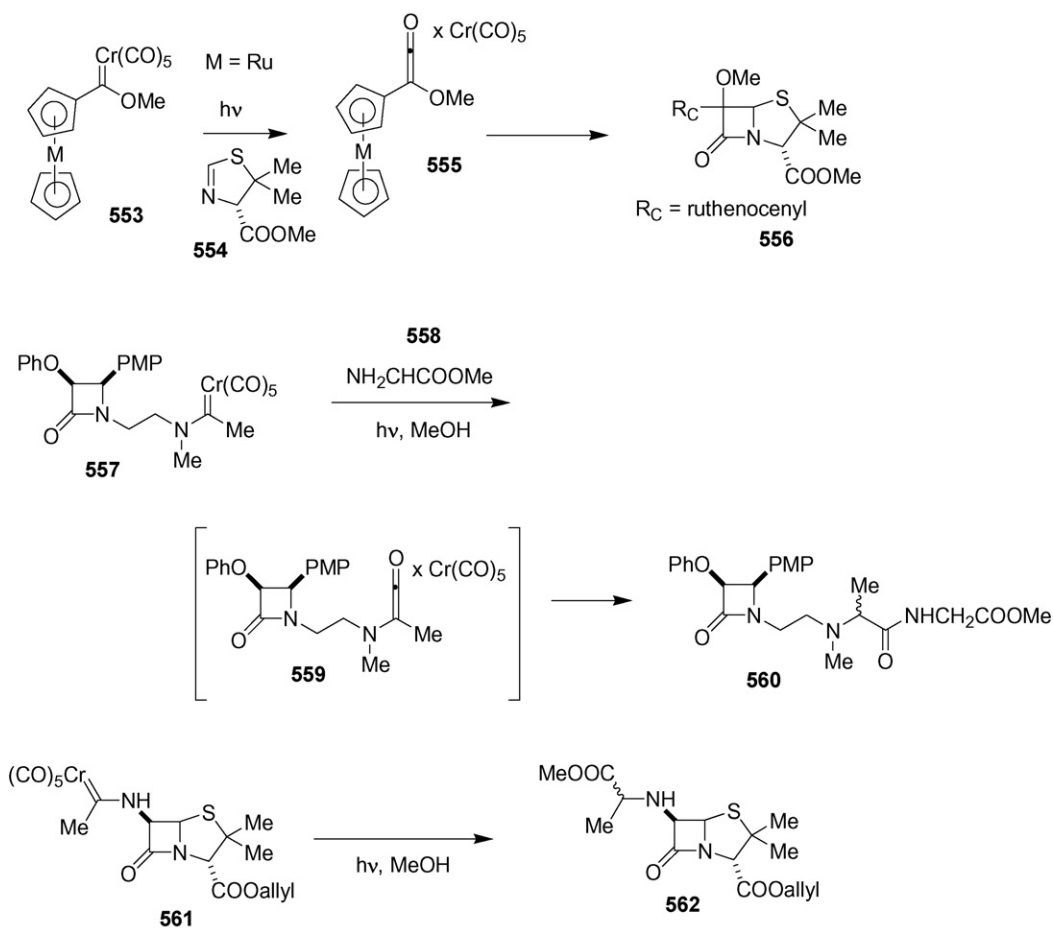
intermediate **646**. Other derivatives led to mixtures of products from both reaction pathways.

Ruthenium bis(carbene) complexes are likely intermediates in ruthenium-catalyzed alkyne trimerization reactions. Several examples of this process were reported in 2009, and representative examples are depicted in Scheme 69. Ruthenium-catalyzed

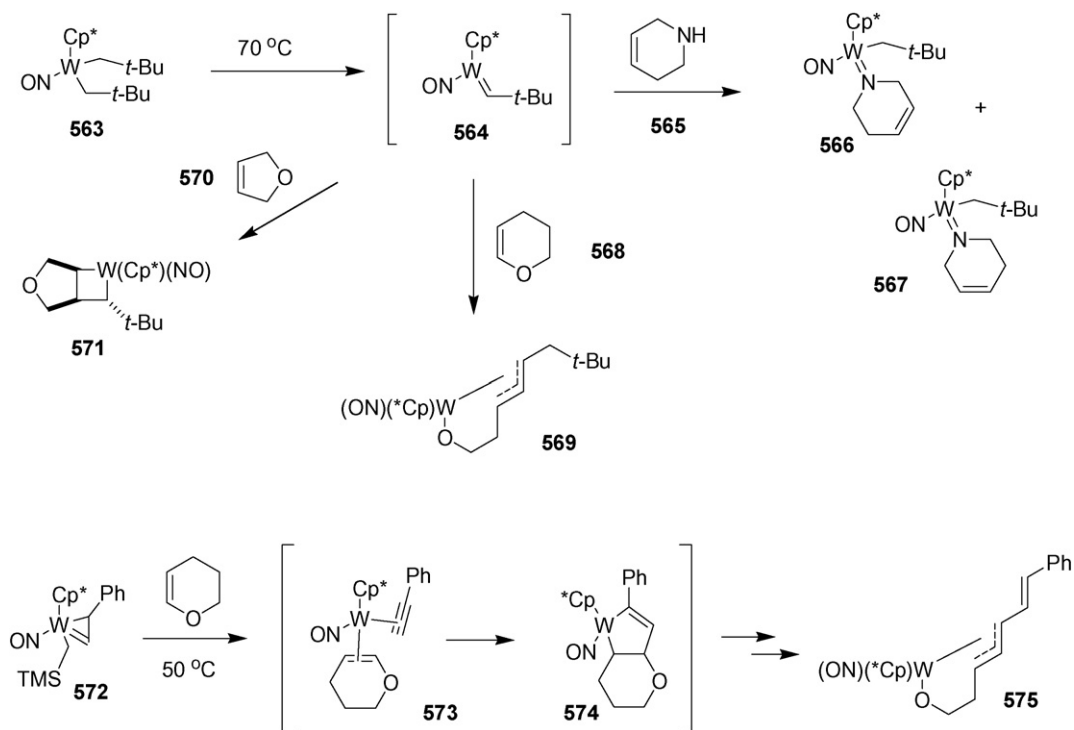
coupling of dialkyne **648** and alkyne **649** was a key step in the total synthesis of sporolide B [932]. Ruthenium-catalyzed alkyne trimerization of bis(pyridinium) salt **651** led to dicationic alkyne trimerization product **652** [933]. Alkyne trimerization of bis(iodoalkyne) derivative **653** and acetylene followed by a subsequent RCM event was also reported [934].



Scheme 57.

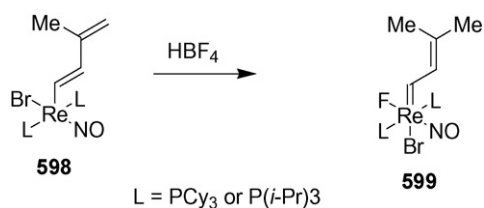


Scheme 58.



Scheme 59.





Scheme 62.

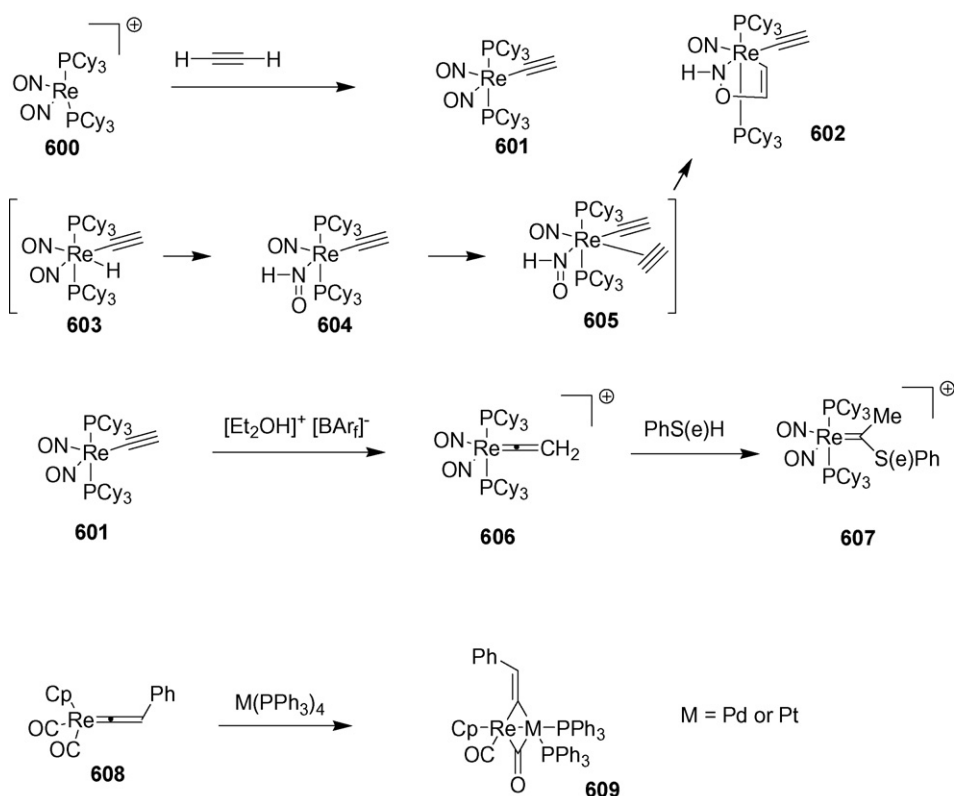
Several reaction processes invoke cyclic ruthenium bis(carbene) complexes as reactive intermediates (see Scheme 70). A novel alkyne coupling process leading to dienylketones (e.g. **662**) likely proceeds through bis(carbene) complex intermediates (**660**) [935]. Ruthenium carbene complexes might be involved in alkyne trimerization and diazo additions catalyzed by Cp*RuCl(cod) [936]. Ruthenium bis(carbene) complexes (e.g. **663**) were proposed as intermediates in the three-component coupling of two alkynes and an alcohol to afford an alkoxydiene (e.g. **664**) [937,938].

2.3.5.3. Metalla-aromatics. Osmabenzenes (e.g. **667** and **668**, Scheme 71) were prepared via the two-step reaction of osmium complex **665** with 1,4-pentadiyn-3-ol (**664**) [939]. The coupling reaction initially affords chelated alkenylosmium complex **666**, which is converted to the osmabenzene complex upon treatment with nucleophiles. In several cases the nucleophile also replaced the chlorine ligands. The tertiary alcohol analog **669** was also converted to an osmabenzene derivative (**672**) [940]. In this case the initial coupling leads to the triene chelate **670**. Reaction with chloroform in the presence of benzonitrile at room temperature leads to the ligand substitution product **671**, which at higher temperature converts to the osmabenzene (**672**). The mechanism of this process

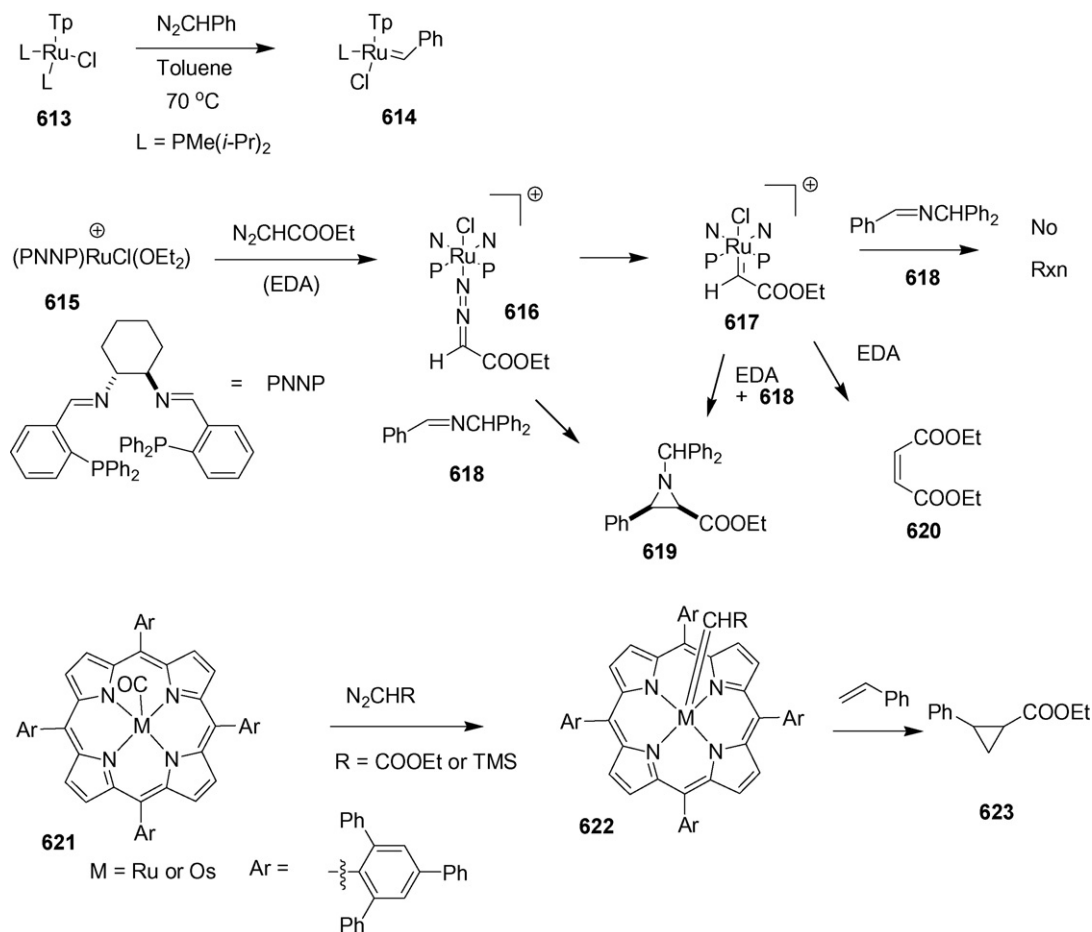
was studied by DFT calculations, and the most reasonable pathway involves conversion to the chelated allene complex followed by C–H oxidative addition. Base-induced cyclization reactions were reported for osmabenzenes containing thiocyanate ligands (e.g. **673**) [941]. The reaction proceeds via an aromatic substitution pathway to afford the initial product **674**. Reaction with chloroform led to the hydrolysis product **675**. Silver oxidation led sulfur oxidation product **676**.

The synthesis and reactivity of ruthenabenzofurans (e.g. **677**, **679**, and **681**, Scheme 72) was reported [942]. Protonation of the ruthenabenzofuran **677** led to the tethered ruthenabenzene derivative **678**. This process was reversible. Reaction of complex **677** with HCl led to predominantly the neutral ruthenabenzofuran derivative **679** accompanied by a minor amount of the Cp-complex **680**. An actual ruthenabenzofuran complex (**681**) was generated upon treatment of complex **679** with an isocyanide. Osmaphenols (e.g. **683**) were generated in the reaction of osmium–enone complex **682** with dry chloroform [943]. An osmafuran (**686**) was formed from the reaction in wet chloroform. The proposed mechanism for formation of the osmafuran is a complex multistep process where the vinyl group undergoes hydration/oxidation to form a cyclic metal acyl complex (**684**), which then undergoes retro CO-insertion and protonation to afford the observed product. The synthesis and reactivity of related osmafuran **687** was also reported [944]. Subsequent reaction with phenylacetylene led to the nine-membered ring carbene complexes **688** and **689**. Thermolysis led to osmafuran derivative **690**. Reaction with trimethylphosphine afforded a ligand substitution product. A mechanism involving sequential insertion (metallacyclobutene formation followed by ring opening) of two alkyne units was proposed.

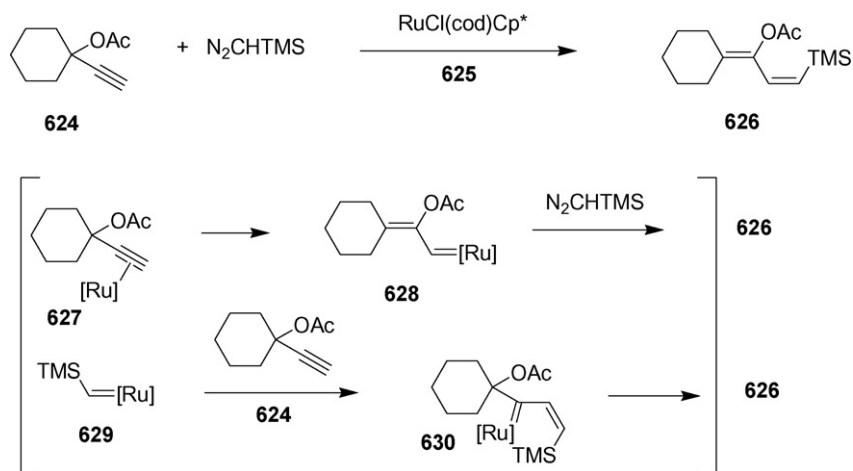
Ruthenaindolizines (e.g. **692**, Scheme 73) were prepared through dehydrogenation of dihydorruthenaindolizine complex **691** [945]. Thermolysis of the dihydro complex **691** in toluene leads to the metal-aromatic species **692**. The ruthenium–carbon dou-



Scheme 63.



Scheme 64.



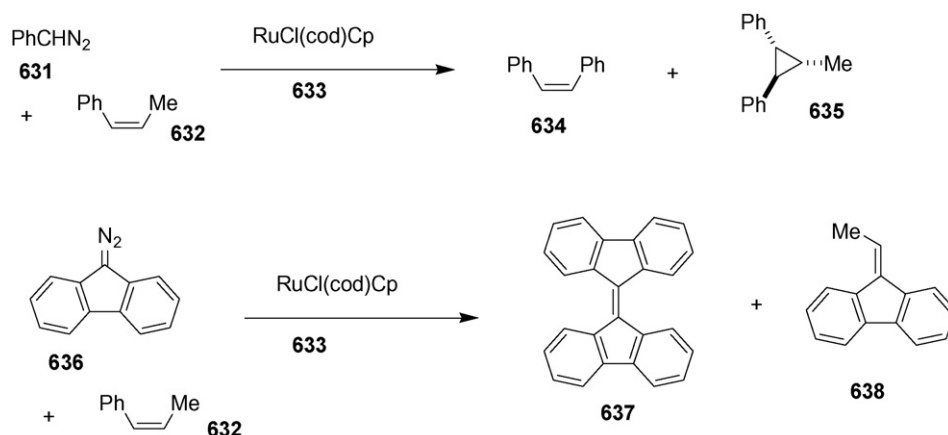
Scheme 65.

ble bond was intermediate between a single and a double bond according to the X-ray structure. The ^{13}C NMR spectrum and MO calculations also supports this formulation.

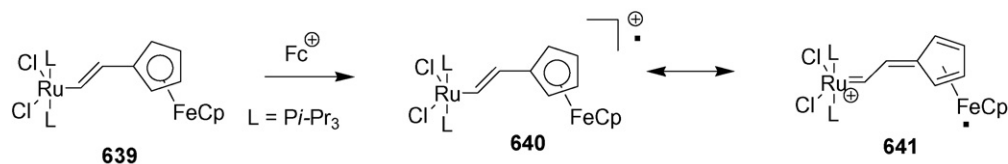
2.3.5.4. Heteroatom-substituted Group 8 metal–carbene complexes. The coupling of arylcarbene-iron complexes (e.g. **693**, Scheme 74) with perfluoroalkynes was reported [946]. The reaction leads to predominately furan derivatives (e.g. **694**). Attempted Fischer synthesis of a carbene complex from α -styryllithium reagent **695** led instead to the stable vinylketene complex (**696**), which coupled

with hexafluoro-2-butyne to afford the benzannulation product **697**.

Chelating aminocarbene-osmium complexes (e.g. **700**, Scheme 75) were prepared through a C–H oxidative addition reaction [947]. Reaction of osmium complex **698** with aminophosphine derivative **699** led to the carbene complex **700**. Reaction of carbene complex **700** with CO or isocyanides led to complexation of the ligand and opening of the chelate, affording complex **702**. Reaction with terminal alkynes also proceeded with breakage of the chelate to afford the vinylidene complex (**701**).



Scheme 66.

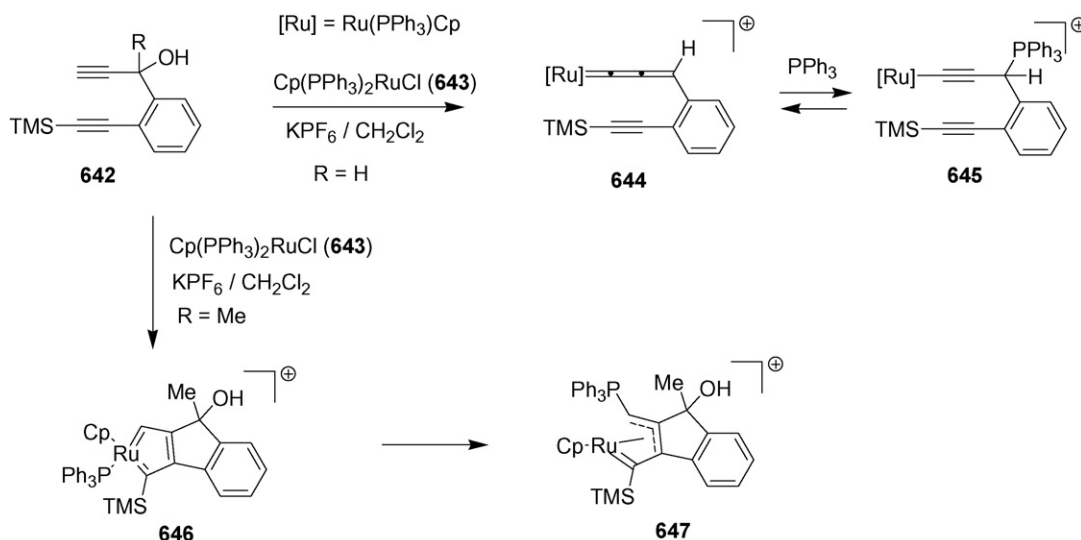


Scheme 67.

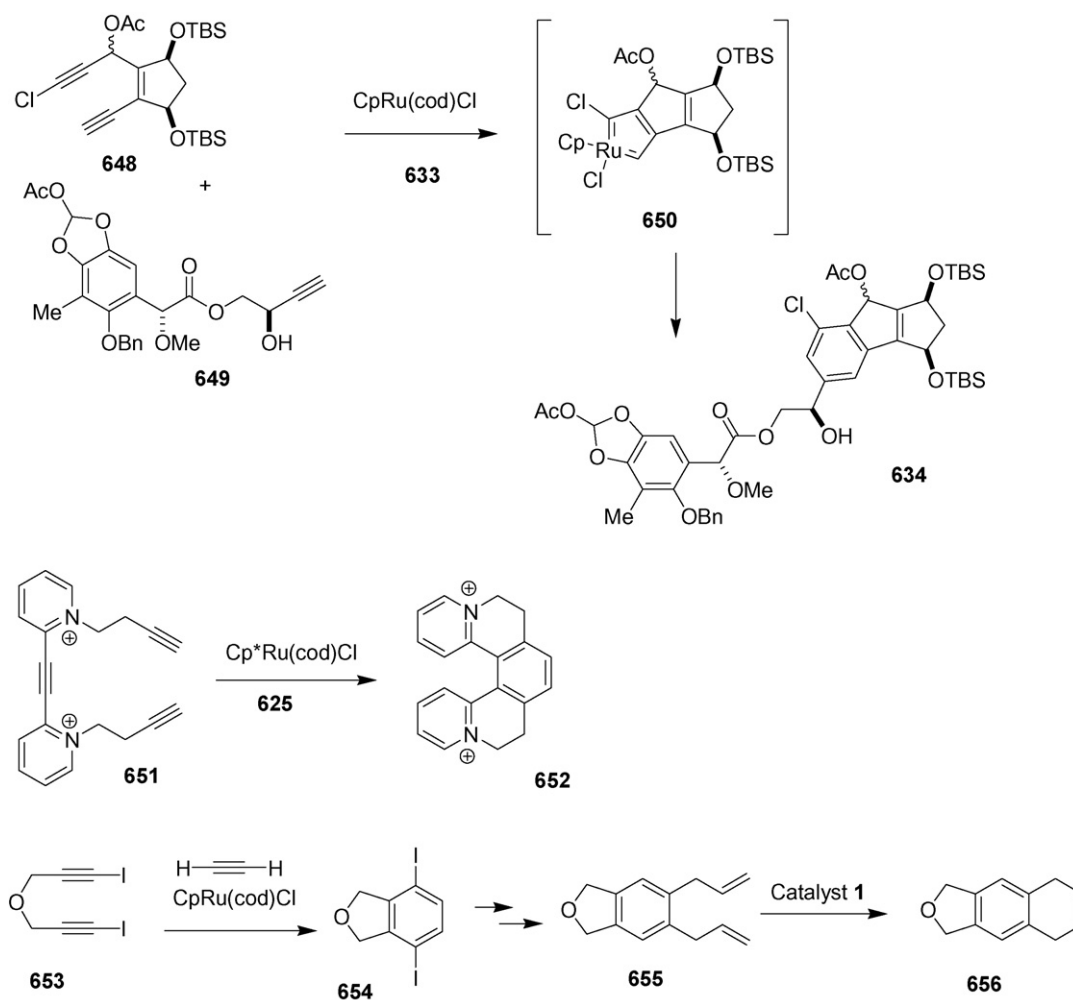
Several papers report on the synthesis of carbene complexes through C–H activation processes involving pyridine derivatives (Scheme 76). The synthesis of ruthenium- or osmium-carbene complexes (e.g. **706**) through net C–H activation of vinylpyridine derivatives was reported [948]. The reaction of either the osmium or ruthenium complex **703** with vinylpyridine (**704**) initially leads to the chelating complex **705**, which slowly converts to an equilibrium mixture of complex **705** and the chelating carbene complex **706**, favoring the carbene complex. The position of equilibrium was highly dependent on the ligands. This process is catalyzed by base in a mechanism involving addition to the alkene followed by elimination. The individual intermediates in this transformation (**707**–**709**) can be prepared and isolated using an excess of base to initiate the first step of the reaction. Alkylidenecyclopropane analogs (e.g.

710) afford ring-expanded carbene complexes (e.g. **714**) upon treatment with complexes **703** [949]. The proposed mechanism involves formation of the chelating alkene complex (**711**), followed by rearrangement to the cyclopropylmetal complex (**712**), followed by alkyl migration to afford the carbene complex. An iron carbene complex (**717**) was produced in the reaction of tris(pyridylthio) derivative **715** with iron salt **716** [950]. The complex features a very short iron–carbon bond (1.77 Å). DFT calculations suggest extensive back-bonding in this complex, even though it is stabilized by two heteroatoms.

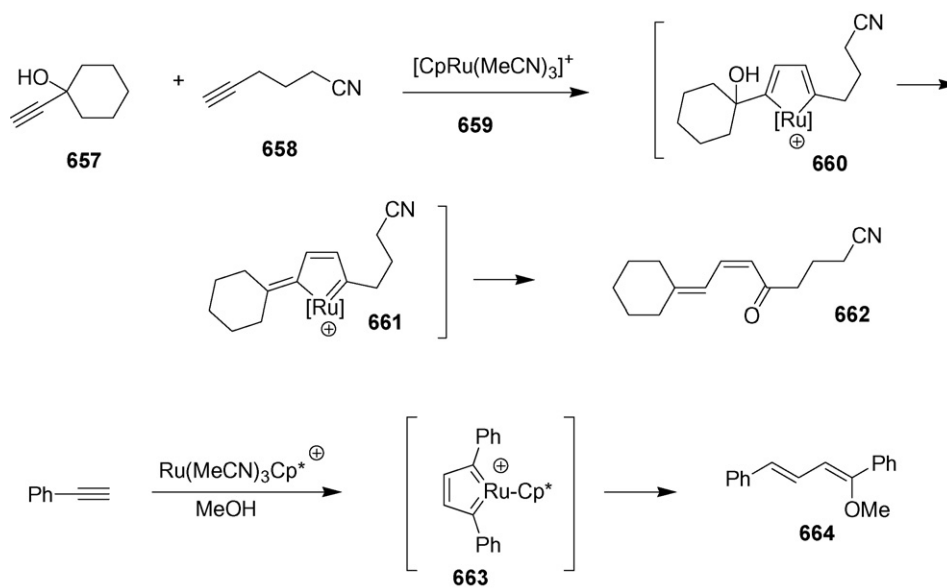
The reactivity of chelating aminocarbene-ruthenium complexes (e.g. **718**, Scheme 77) was reported [951]. The complex exists as a fluxional compound in equilibrium with the aminoalkyl form (**719**). The equilibrium mixture was converted to the nonfluxional



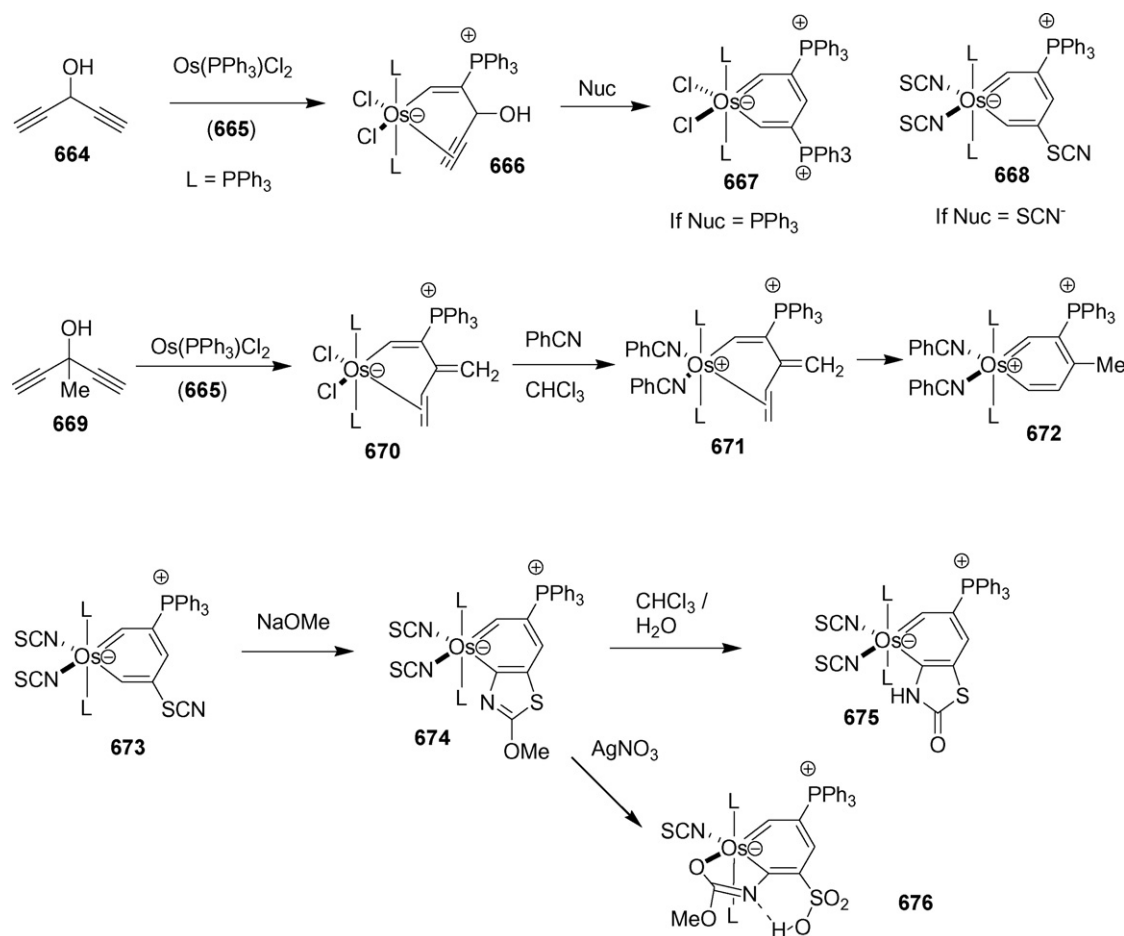
Scheme 68.



Scheme 69.



Scheme 70.



Scheme 71.

aminoalkyl complexes **720** upon treatment with ligand additives. The reaction with silanes led to the silylruthenium analogs (**721**). The mechanism for this latter process involves the direct addition of H and Si across the Ru–C double bond as revealed through deuterium labeling studies.

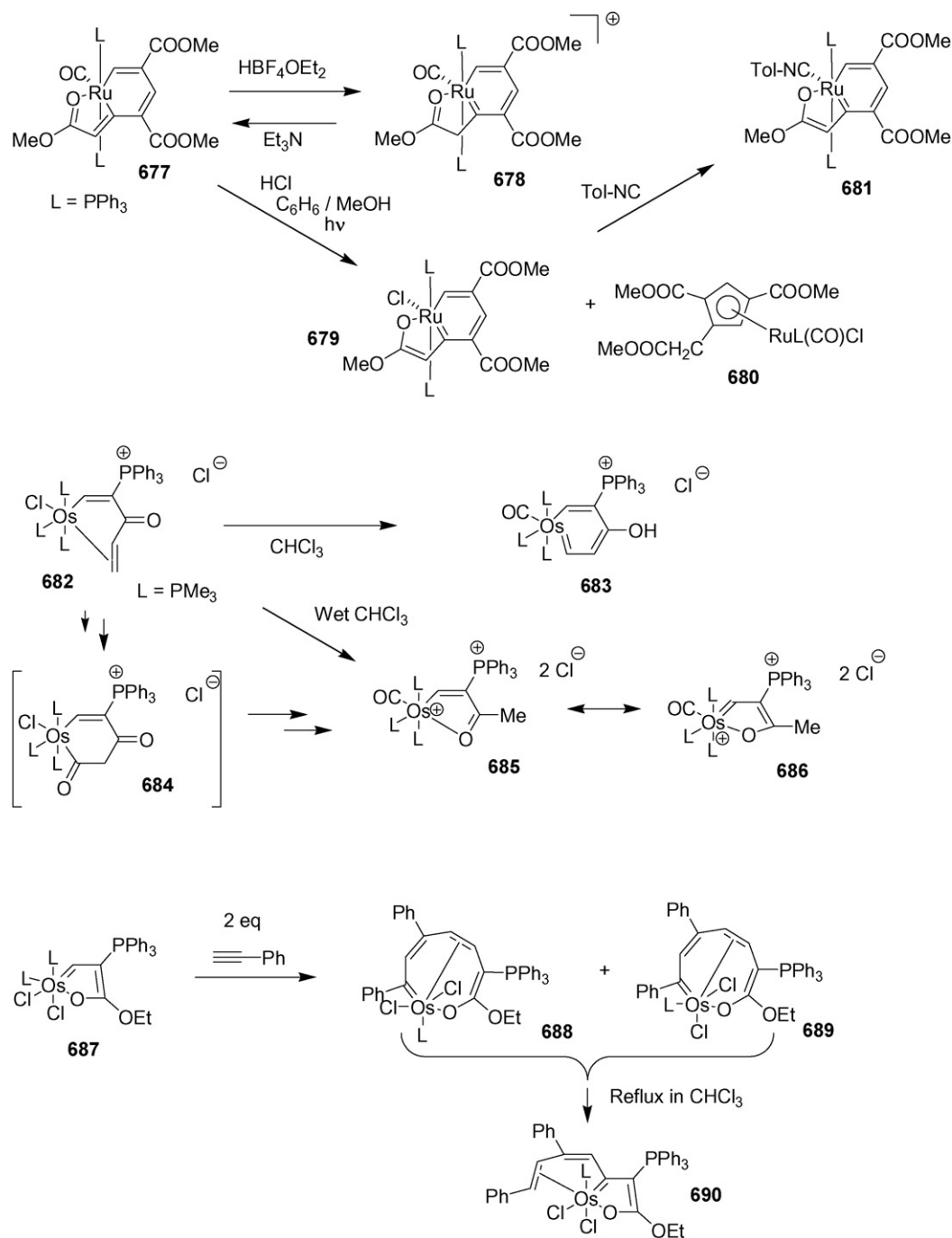
The reactivity of bridging aminocarbene-iron complexes substituted by sulfur (e.g. **722** and **724**, Scheme 78) was examined [952]. Reaction of the thiophenyl complex **722** with sodium borohydride led to the bridging allylcarbene complex (**723**). Reaction of the zwitterionic complex **724** with the titanocene derivative **725** led to the diiron–titanium–complex species **726**.

Additional studies of heteroatom-stabilized Group 8 metal carbene complexes are depicted in Scheme 79 and include: (1) a computational study of the conversion of alkynals (**727**) to cyclic compounds (**729**) and CO, which suggests the intermediacy of carbene complexes (**728**) [953]; (2) formation of chelating aminocarbene–iron complexes (e.g. **733**) from chelating isocyanide complexes [954]; and (3) generation of phosphorus-substituted carbene complex **736** through reaction of Ru–P doubly bonded species **734** with phosphalkyne **735** below 0 °C (upon warming to room temperature, the phosphorus heterocycle **737** was obtained) [955].

2.3.5.5. Group 8 metallacumulene complexes. Many examples of the formation of metal vinylidene complexes (**740**, Scheme 80) via coupling of coordinatively unsaturated Group 8 metal complexes with terminal or silylated alkynes were reported in 2009. Representative examples are depicted in Fig. 13. Common reaction pathways for these complexes include reaction with nucleophiles to form

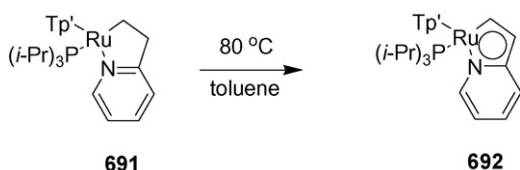
vinylmetal species (**743**), reaction with alcohols (or amines) to form Fischer's carbene complexes (**744**) or water to form metal acyls (**742**), and deprotonation at the β-position to form alkynylmetal complexes (**741**). Other common synthetic routes to metal vinylidenes included addition of electrophiles to metal acetylide complexes (e.g. the reverse of the reaction synthesizing **741**), and treatment of acylmetal complexes with dehydrating agents (*i.e.* the reverse of the reaction synthesizing **742**). Metal-higher cumulene complexes (**746**, **751**) are produced from the coupling of coordinatively unsaturated Group 8 metal complexes with propargyl alcohols, or by addition of electrophiles to the δ-carbon of alkenylethynyl–metal complexes (**750**). Common reaction pathways for these complexes include reaction with nucleophiles at the γ-position, resulting in alkynylmetal complexes (**748**), or attack at the γ-position, resulting in allenylmetal complexes (**749**). Reaction with alcohols or amines can lead to α,β-unsaturated Fischer's carbene complexes (**747**). Representative examples of this class of compounds are depicted in Fig. 13.

Specific reports which highlight the reaction pathways of Scheme 80 are depicted in Fig. 13 and include the following examples of vinylidene complexes: (1) formation of iron vinylidene complexes (e.g. **753**) via photochemical reaction of complex **752** with chelating phosphines and terminal alkynes [956]; (2) formation of dicationic bis(iron-vinylidene) complexes from *p*-diethynylbenzene and subsequent transformation to the neutral bis(alkynyliron) complexes (includes electrical conductivity studies of the complexes) [957]; (3) formation of ruthenium vinylidene complexes and subsequent transformation to alkynylruthenium complexes [958]; (4) formation of a cationic ruthenium vinylidene



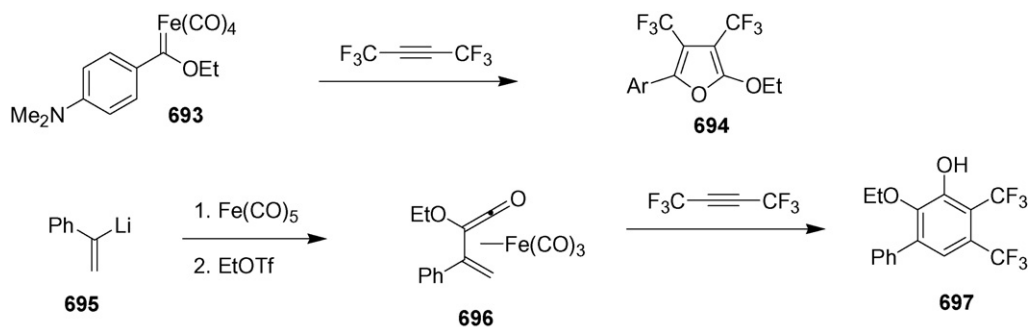
Scheme 72.

complex featuring a cobaltocene bis(phosphine) ligand through protonation of the alkynylruthenium complex [959]; (5) formation of tricationic trimetallic bis(vinylidene) complexes (e.g. **754**) and subsequent transformation to the monocationic bis(alkynyl)

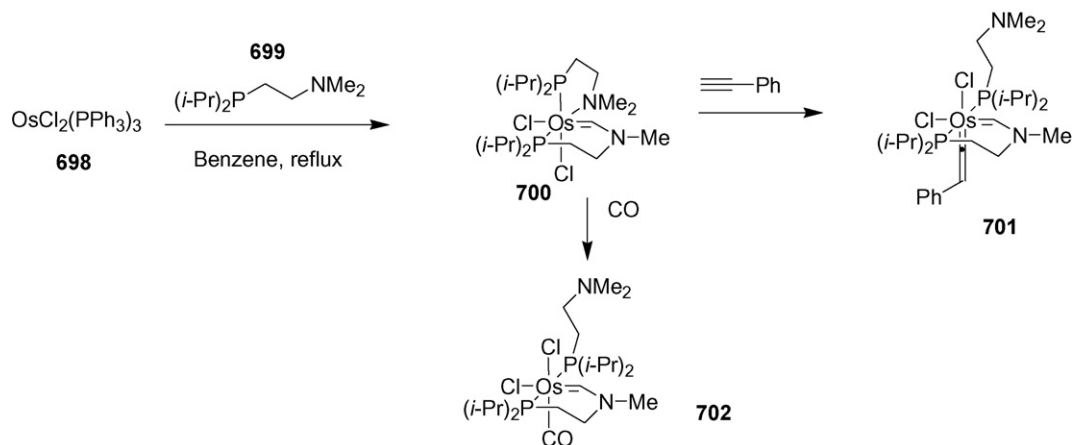


Scheme 73.

complexes [and electrochemical oxidation thereof to afford a bis(vinylidene)] [960]; (6) formation of bis(ruthenium–vinylidene) complexes connected through thiophene units (e.g. **755**) and subsequent NLO studies [961]; (7) formation of dicationic ruthenium–vinylidene complexes from 4-ethynylpyridinium salts and subsequent transformation to the alkynyl complexes [962]; (8) double protonation of bimetallic pyridylethynylruthenium complex **756** to afford the dicationic vinylidene complex (**757**) [963]; (9) formation and reactivity of vinylidene complexes connected to uracil groups (e.g. **758**) and self-assembly of ruthenium vinylidene as well as derived Fischer carbene (e.g. **759**) and alkynyl complexes [964]; (10) formation of cationic ruthenium vinylidene complexes connected to bis(dithiolene) groups [965]; and (11) formation of bromovinylidene complexes (e.g. **760**) through electrophilic



Scheme 74.



Scheme 75.

bromination of alkynylruthenium complexes [966]. Several processes likely involve metal vinylidene intermediates synthesized by these pathways, including: (1) formation of polymetallic alkynyl-ferrocene derivatives from the bis(ethynyl)ferrocene derivatives [967]; (2) preparation of iron acetylides via reaction of alkynyl gold complexes with $\text{FeCl}(\text{dppe})\text{Cp}$ in the presence of ammonium hexafluorophosphate to generate a cationic vinylidene complex (e.g. **761**) followed by deprotonation to afford the neutral acetylide complex [968]; (3) preparation of alkynylruthenium complexes [969]; (4) ruthenium-catalyzed alkyne dimerization [970]; and (5) ruthenium-catalyzed cycloisomerization of alkynols [971]. Examples that highlight higher cumulenes are also depicted in Fig. 13 and include: (1) enantioselective formation of allenylidene complexes (e.g. **763**) from ruthenium complex **762** and propargyl alcohols [972]; and (2) formation and nucleophilic addition to various vinylidene and allenylidene ruthenium complexes (e.g. **764**) [973]. Several processes reported in 2009 invoke metal-higher cumulene complexes as intermediates, including: (1) conversion of bis(alkynyl)ruthenium complexes to enynylruthenium complexes through protonation [974]; and (2) ruthenium-catalyzed addition of carboxylates to terminal alkynes [975]. Metal-vinylidene resonance contribution to alkynylmetal complexes was noted in several cases involving electron deficient metals, including: (1) cationic alkynylruthenium(III) complexes generated through electrochemical oxidation of the corresponding neutral alkynylruthenium(II) complexes [976]; and (2) phosphonioacetylide–ruthenium complexes [977]. In many cases, the electrochemical oxidation of alkynyl–metal complexes generates metallacumulene complex structures [978]. A computational study assessed that the allenylidene ligand is a poorer π -acceptor than a nitrile group [979]. Comparisons between the bonding in vinylidene complexes and isoelectronic borylene complexes were noted [980].

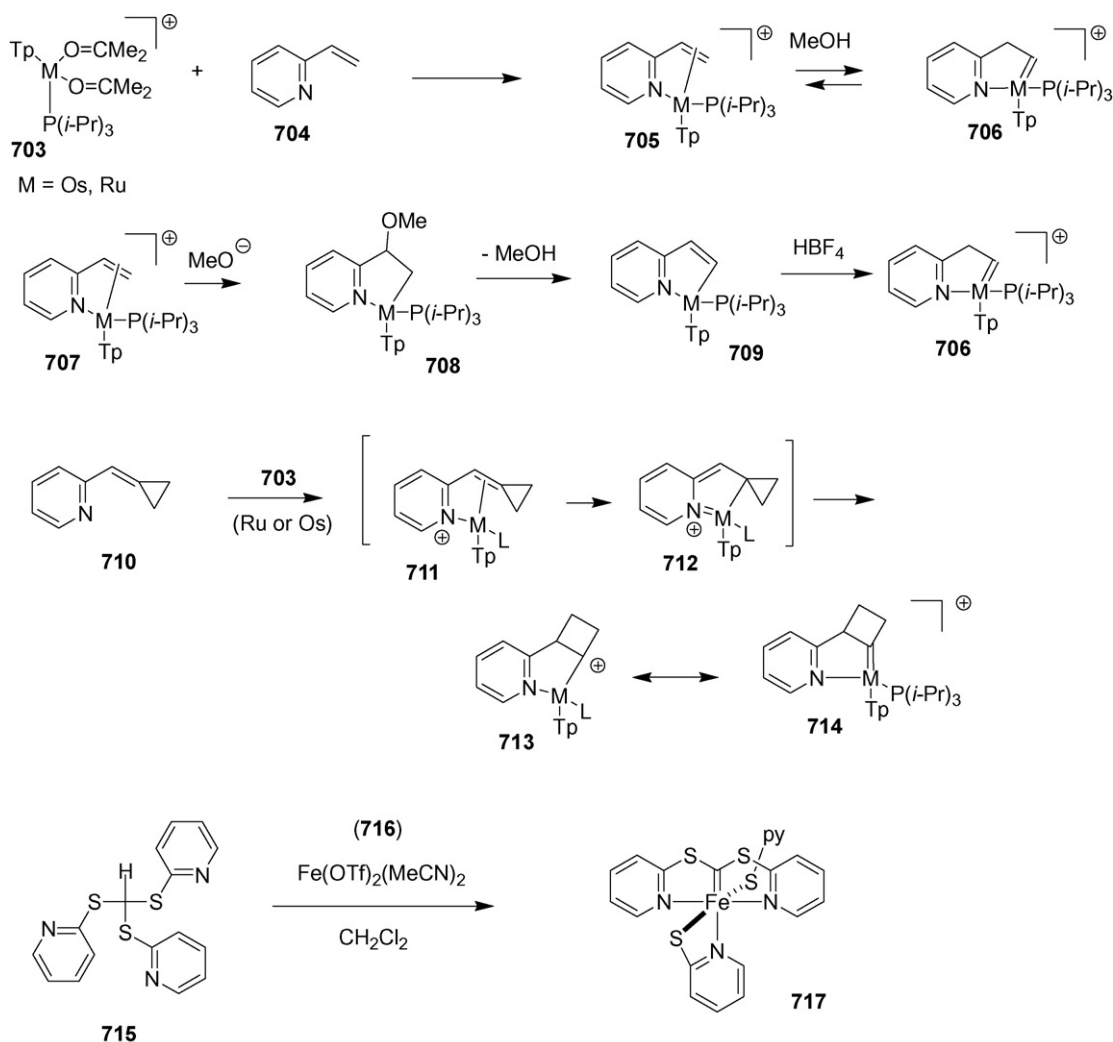
The direct formation of vinylidene–ruthenium and iron complexes (e.g. **768**) from diarylacetylenes (e.g. **766**) was reported [981]. Carbon-13 labeling studies reveal that the more electron-deficient aromatic ring preferentially migrates (Scheme 81).

The reaction of ruthenium linear nitrosyl complex **769** (Scheme 82) with acetylenes led to the corresponding vinylidene complex (**771**) featuring a bent nitrosyl ligand [982]. Subsequent reaction with methyl propiolate in the presence of acid led the [2+2]-cycloaddition product **772**, which features a linear nitrosyl ligand. The reaction of alkynylruthenium complex **773** with highly polarized alkyne **774** also resulted in a [2+2]-cycloadduct (**776**) via an ionic mechanism [983].

The reaction of complex **643** (Scheme 83) with bis(trichloromethyl) propargyl alcohol derivative **777** afforded a stable vinylidene complex (**778**) that did not transform to an allenylidene complex [984]. Reaction with alumina led to the expected formation of an alkynylruthenium complex (**780**). Reaction with hydroxide ion led to a ketoalkynylruthenium complex (**779**) via a deprotonation and haloform elimination process.

The reaction of neutral ruthenium vinylidene complexes (e.g. **781**) and tetracyanoethylene (**782**) led to the substituted vinylidene complex **785** [985]. A mechanism involving nucleophilic addition to tetracyanoethylene to afford a carbyne complex intermediate (**783**) followed by elimination of HCN was proposed. A very short Ru–C bond was attributed to contribution from a carbyne complex resonance form (**786**) (Scheme 84).

The low-temperature reaction of complex **787** (Scheme 85) and terminal alkynes led to the cationic vinylidene complex (**788**) [986]. The reaction at room temperature was more complex, and led to vinylidene complex **788** in addition to the alkynyl vinylidene complex **789** and the enone complexes **790** and **791**. The enone complex **790** was the major product. The enone complex was also obtained



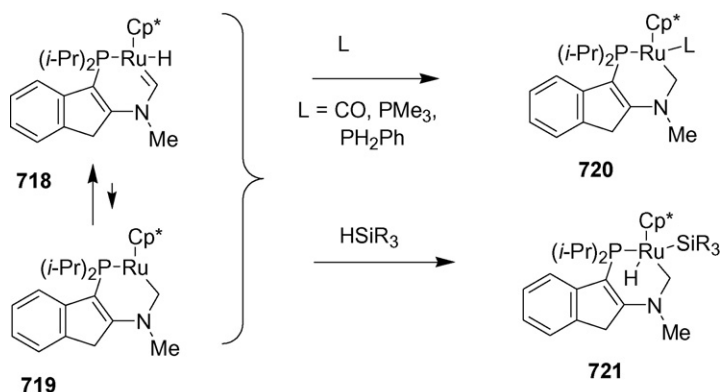
Scheme 76.

from the reaction of complex **789** with aqueous acetone. Formation of enynyl complex **790** involves alkyne migration followed by hydration of the enynyl system **792** followed by enolization and complexation. Enynyl complex **791** was formed via hydration of the vinylidene complex to an acylmetal complex followed by alkyne insertion.

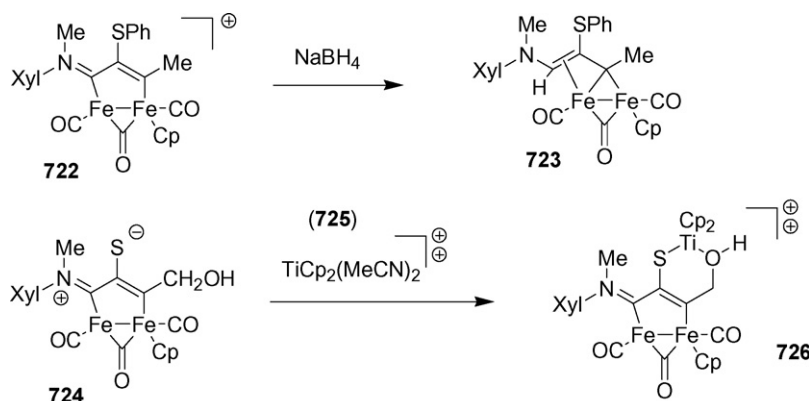
The formation of neutral ruthenium vinylidene complexes containing two acetate ligands (e.g. **796**, Scheme 86) was reported [987]. Reaction with propargyl alcohols leads to sta-

ble hydroxyvinylidene complexes (e.g. **797**), which could not be transformed to allenylidene complexes under a variety of conditions. Extended storage led to the carbonyl derivative (**799**) and 1,1-diphenylethylene (**800**). A mechanism involving formation of the four-membered ring ether (**802**) followed by retro-[2+2]-cycloaddition was suggested for the latter process.

Several cyclization reactions involving terminal alkynes invoke vinylidene intermediates; examples are depicted in Scheme 87. The reaction of enynes (e.g. **801**) with bimetallic complex **802** led to



Scheme 77.

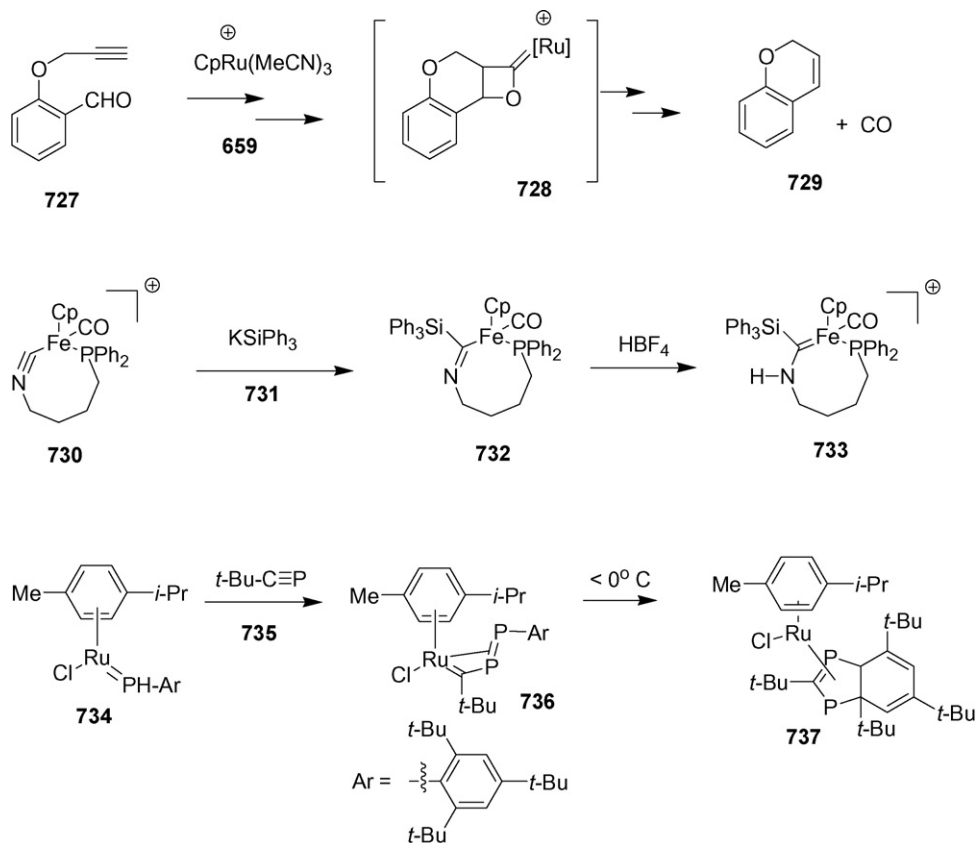


Scheme 78.

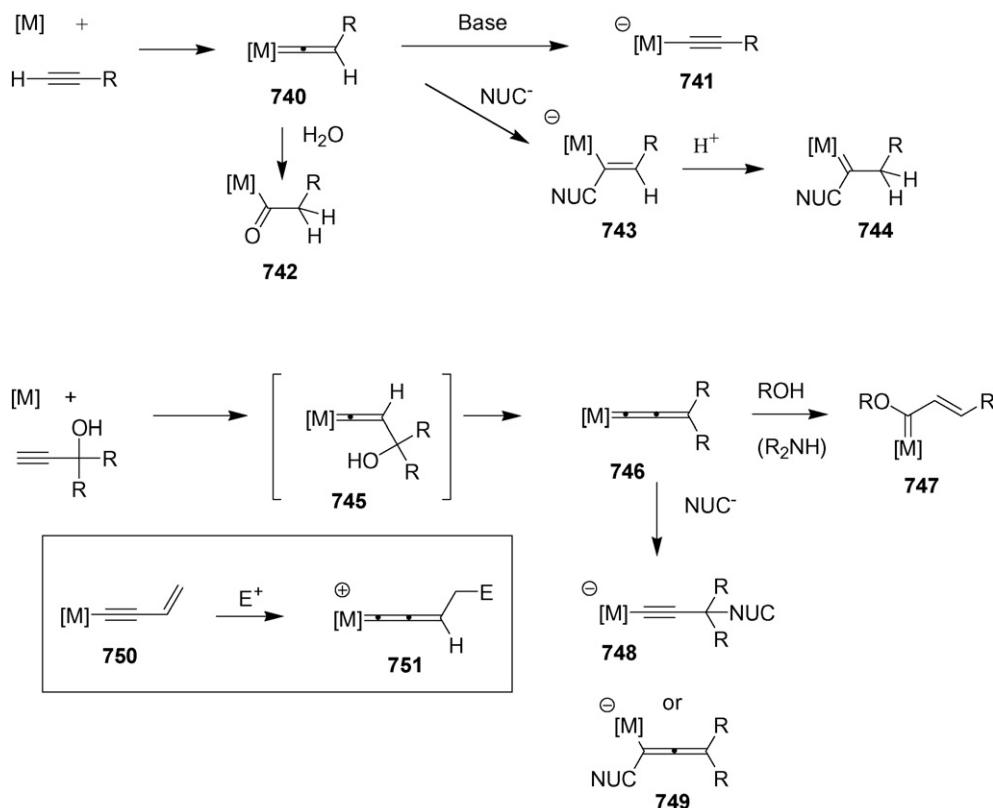
cyclohexadienes (e.g. **805**) [988]. A mechanism involving formation of a vinylidene complex (**803**) followed by electrophilic addition to the alkene group was proposed. Deuterium labeling studies support the intermediacy of vinylidene complexes. The reaction of enyne-nitrones (e.g. **806**) with ruthenium complex **807** led to pyridones (e.g. **810**) [989].

Additional studies of Group 8 metal vinylidene complexes are depicted in Scheme 88. The reaction of cationic ruthenium vinylidene complex **811** with HX led to double HX addition products (e.g. **812**) [990]. Carbene complexes **814** and **815** were prepared via the reaction of ruthenium ketophosphine complex **813** with terminal alkynes [991]. Carbene complex **814** was formed in methanol, and cyclic carbene complex **815** was produced in dichloromethane. Thermolysis of the cyclic carbene complex led to the alkene complex **816**.

The formation and protonation of (dienyl)allenylidene–osmium complexes (e.g. **820**, Scheme 89) was reported [992]. The dienyl complex was prepared through hydrometallation of enyne **819**. Reaction of the dienyl complex **820** with HBF_4 led to the dicationic carbene(allenylidene) complex **821**, which converted to the carbyne complex **822** upon treatment with sodium acetate. The same carbyne complex could also be obtained through a protonation/ligand substitution reaction involving the reaction of acetic acid with complex **820**. Similar formation of a carbyne complex and ligand substitution pathways were observed in the reaction of complex **820** with oximes or 1,3-diketones. Reaction of alkenyl–allenylidene complexes (e.g. **823**) with CO led to the alkenyl migration product **824** [993]. Subsequent reaction with HBF_4 led to the cyclopentenylidene complex **826**. A Nazarov-type mechanism involving divinylcarbene complex inter-



Scheme 79.



Scheme 80.

mediate **825** was proposed to account for formation of the carbene complex.

The synthesis of ruthenium allenylidene complexes that feature pendant alkene and anthracenyl groups (e.g. **830**, Scheme 90) was reported [994]. The allenylidene complex **830** was transformed to the tricyclic-vinylidene complex **834** by treatment with a Grignard reagent (**831**) to afford the substituted alkynylruthenium complex (**832**), which was then converted to the vinylidene complex by reaction with allyl iodide. The resulting allylvinylidene complex (**833**) then undergoes intramolecular Diels–Alder reactions with the anthracene unit to afford the bridged structure **834**. Treatment of ruthenium complex **643** with a variety of 4,4-disubstituted-1,6-enynes featuring a propargylic alcohol (e.g. **835** and **839**) or ether group was reported [995]. A wide variety of product types were produced in this reaction, depending on the substituents and the atoms in the tether. In the simplest case (**835**), the cationic allenylidene complex (**837**) was formed, which further rearranged to the branched vinylidenes complex **838** upon further heating. The deuterium labeling revealed that the shift of the allyl group is predominantly via the 1,3-shift process and not via a Cope rearrangement. Many additional enyn-ol derivatives were tested in their reactions with ruthenium complex **643**. More highly substituted alkene analogs (e.g. **839**) afforded cyclization products (e.g. **841**) in addition to simple allenylidene complexes due to the greater nucleophilicity of the alkenes. Products were frequently the result of rearrangement processes of allylic systems.

The synthesis and reactivity of cationic ruthenium allenylidene (e.g. **843**, Scheme 91) and dicationic ruthenium carbyne complexes (e.g. **844**) was reported [996]. Ruthenium allenylidene complex **843** was generated from ruthenium chloride **842** and propargyl alcohols. Reaction of allenylidene complexes with 1,3-diketones (e.g. **845**) led to the cycloaddition products (e.g. **846**). Protonation of allenylidene complex **843** led to dicationic carbyne complex **844**. Reaction of this complex with resorcinol (**848**) led to cycloaddition

products, cyclic carbene complexes (e.g. **849**). Reaction of dicationic carbyne complexes with electron rich aromatics (e.g. methylfuran, **850**) led to the electrophilic aromatic substitution products, vinylidene complexes (e.g. **851**).

Bis(ruthenium) allenylidene complexes (e.g. **854**, Scheme 92) were prepared and their electrochemistry and electronic spectra were studied [997]. The bis(allenylidene)diruthenium complexes were further reacted with the butadiynylruthenium complex **855** to afford the C7-linked tetrakis(ruthenium) complexes (e.g. **856**). A key step in this transformation is proton transfer to afford the enynylruthenium complex **857** and the butatrienylidene complex (**858**) pair, which couple to afford the observed product.

Ruthenium allenylidene complexes (e.g. **861** and **866**, Scheme 93) were proposed as intermediates in various propargyl substitution reactions. The alkoxypropargylation of electron-rich alkenes using propargyl alcohols (e.g. **843**) and diruthenium catalyst **860** in ethanol was reported [998]. Catalysts where the chloride ligands have been replaced by bromide or iodide ligands were less effective in promoting propargyl substitution reactions [999]. Enantioselective intramolecular aromatic substitution reactions (e.g. conversion of **864–867**) were reported using chiral complexes that contain bridging sulfide complexes (e.g. **865**) [1000]. Ruthenium-catalyzed substitution of propargyl alcohols was studied computationally [1001]. Ruthenium allenylidene complexes were identified as important intermediates in these processes.

The conversion of cationic ruthenium-butadiyne complexes (e.g. **868**, Scheme 94) to butatrienylidene complexes (e.g. **871**) was studied computationally [1002]. The overall process was found to be highly exothermic ($\Delta H = -13.1$ kcal/mol). The most favorable reaction pathway involves conversion to the ethynylvinylidene complex (**869**) via a 1,2-hydrogen shift followed by conversion to the butatrienylidene complex through a deprotonation/protonation sequence.

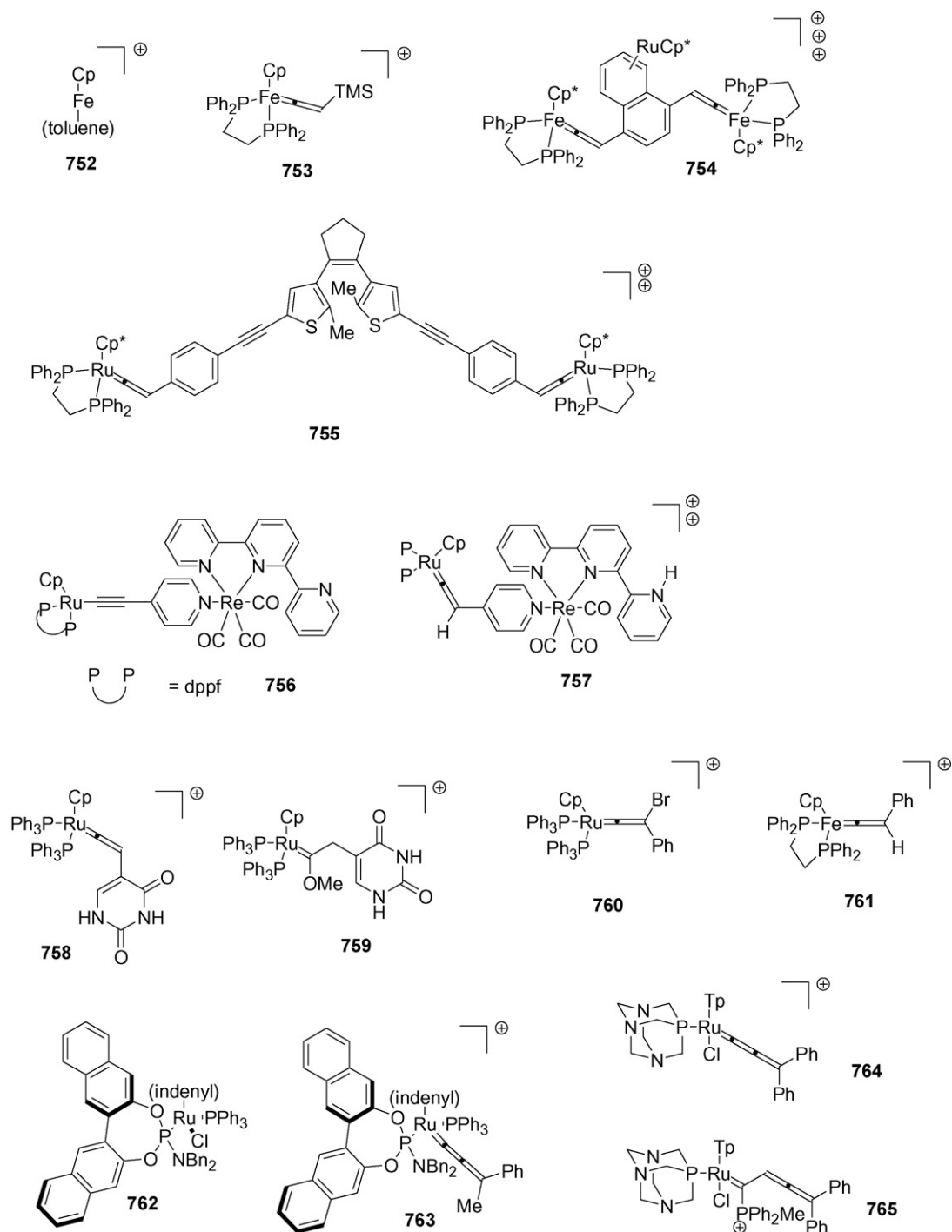
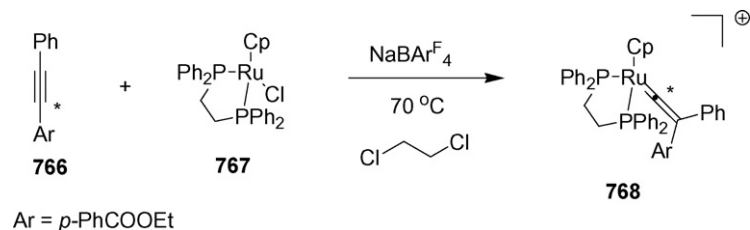
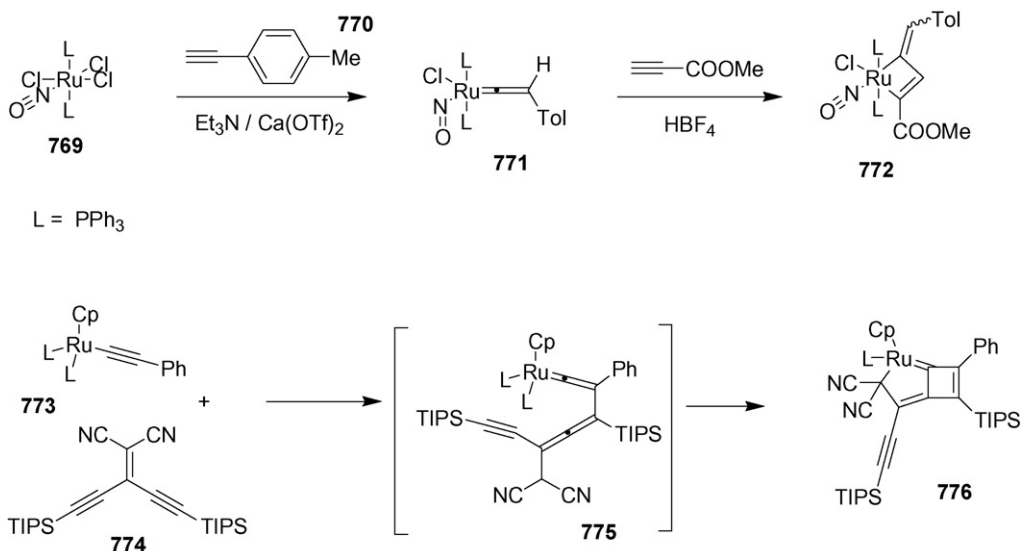
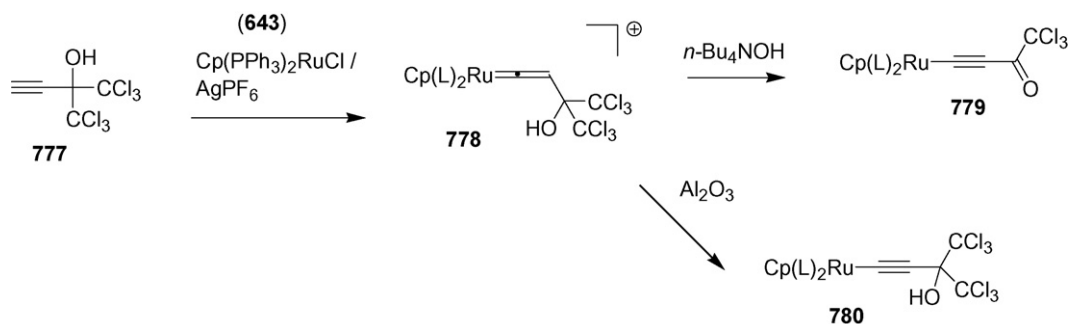


Fig. 13. Representative Group 8 metallacumulene complexes, precursors, and reaction products reported in 2009.





Scheme 82.



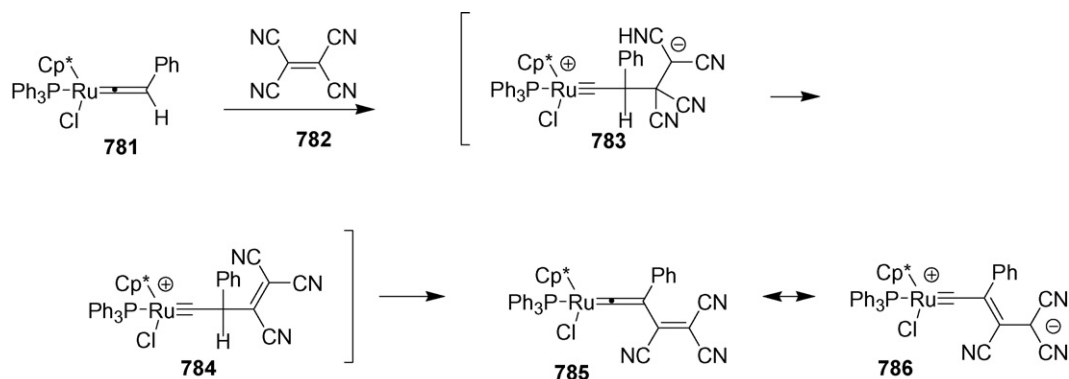
Scheme 83.

2.3.6. Group 9 metal–carbene complexes

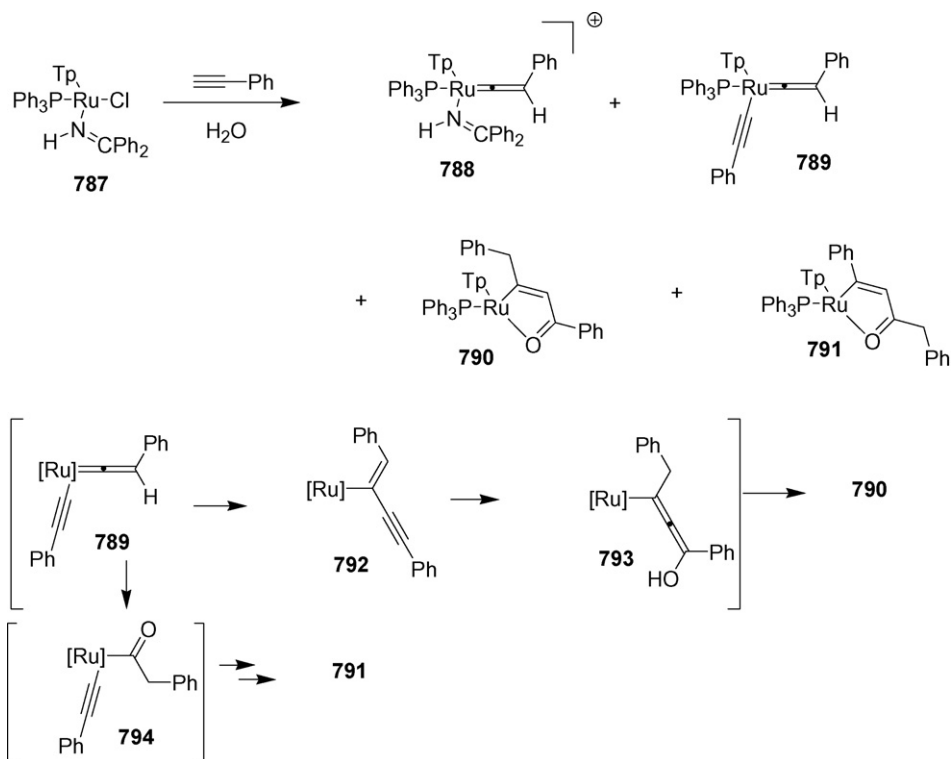
2.3.6.1. Simple carbene complexes. Examples of carbene complexes derived from predominantly donor carbene ligands are depicted in Scheme 95. Cyclopropenylidene–rhodium complexes (e.g. **877**) were prepared through an oxidative addition process from dichlorocyclopropene **875** [1003]. These complexes were tested as hydroformylation catalysts, however were not effective unless zinc metal was added. Iridium–NHC complexes where the nitrogens are acylated (e.g. **880** and **881**) were prepared through a ligand substitution process employing iridium complex **879** [1004]. Additional ligand substitution processes were demonstrated for the initially-formed carbene complexes. Spectral properties

suggest that in this complex the NHC ligand is not a pure donor ligand.

A variety of carbene complexes (e.g. **884** and **886**, Scheme 96) were formed from the reaction of dihydrido-iridium complex **882** with various ethers [1005]. Reaction of complex **882** with THF and various methyl alkyl ethers proceeds via $\alpha, \alpha-C-H$ activation to afford the carbene complex. A mechanism involving C–H activation, followed by α -hydride elimination to afford the trans dihydrido-carbene complex **884**, followed by release of H_2 was proposed to explain the formation of carbene complexes. The α -hydride elimination process kinetically affords the trans dihydride (**884**), which could be isolated, and presumably converts to the cis dihydride



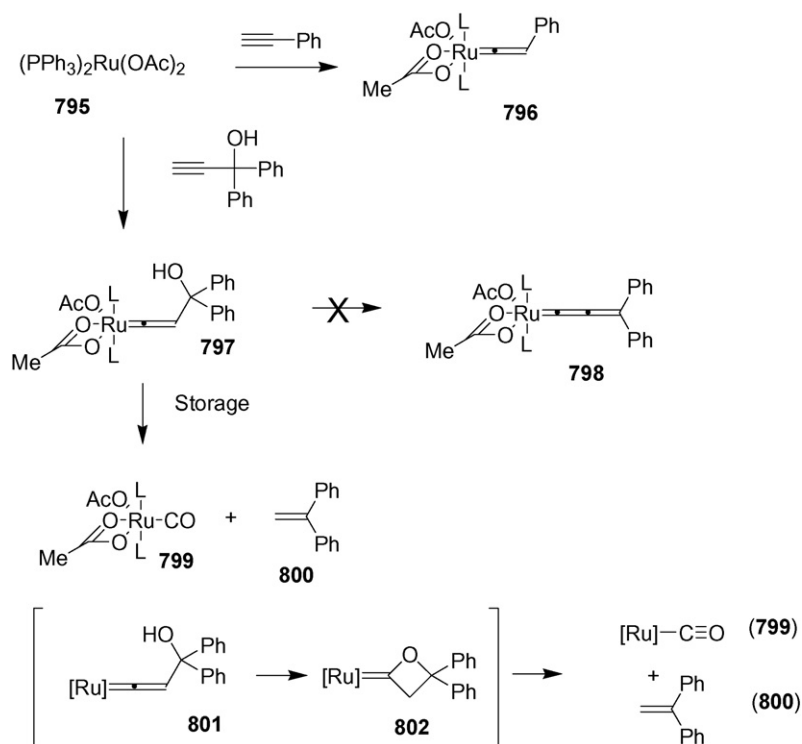
Scheme 84.



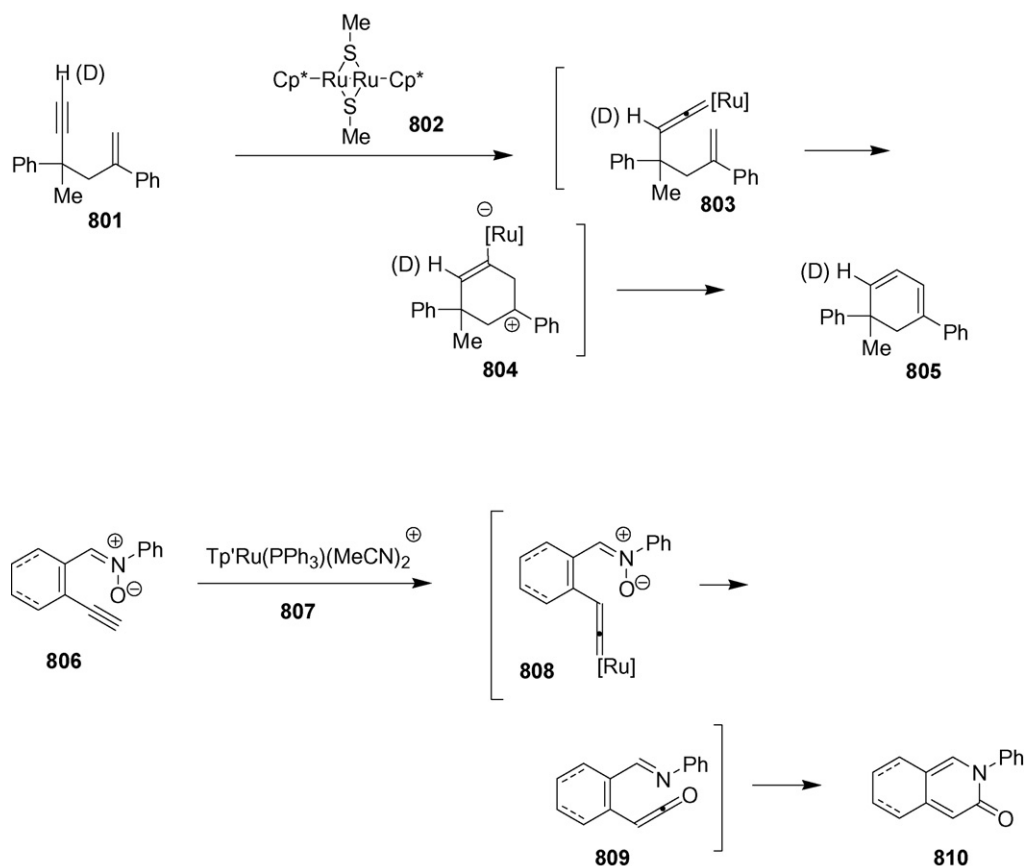
Scheme 85.

(**855**) prior to release of H_2 . Reaction with diethyl ether and dioxane proceeds via α,β -C–H activation to afford iridium complexes of the unsaturated ethers (e.g. **887**). Reaction with benzyl methyl ether led to the decarbonylation product **891**. A mechanism involving benzylic C–H activation, release of benzaldehyde, C–H activation of benzaldehyde, and decarbonylation as key steps was proposed.

The reaction of an iridium carbene complex **892** (Scheme 97) with heterocumulenes was reported [1006]. The reaction of carbene complex **892** with carbon disulfide at room temperature led to the double addition product **893**. Thermolysis of this complex led to the carbon monosulfide complex **894** and the thioester. Slow addition of carbon disulfide at room temperature led directly to the



Scheme 86.

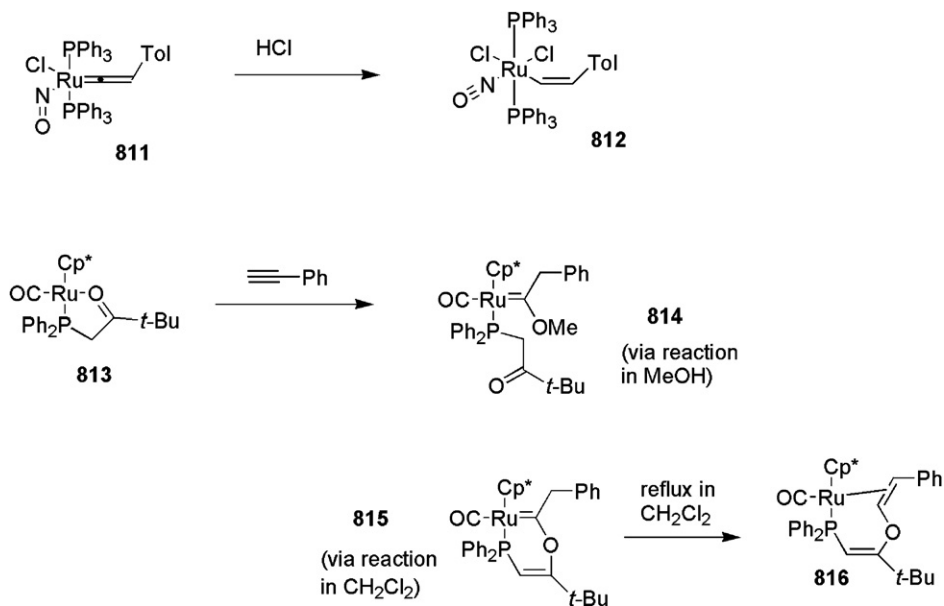


Scheme 87.

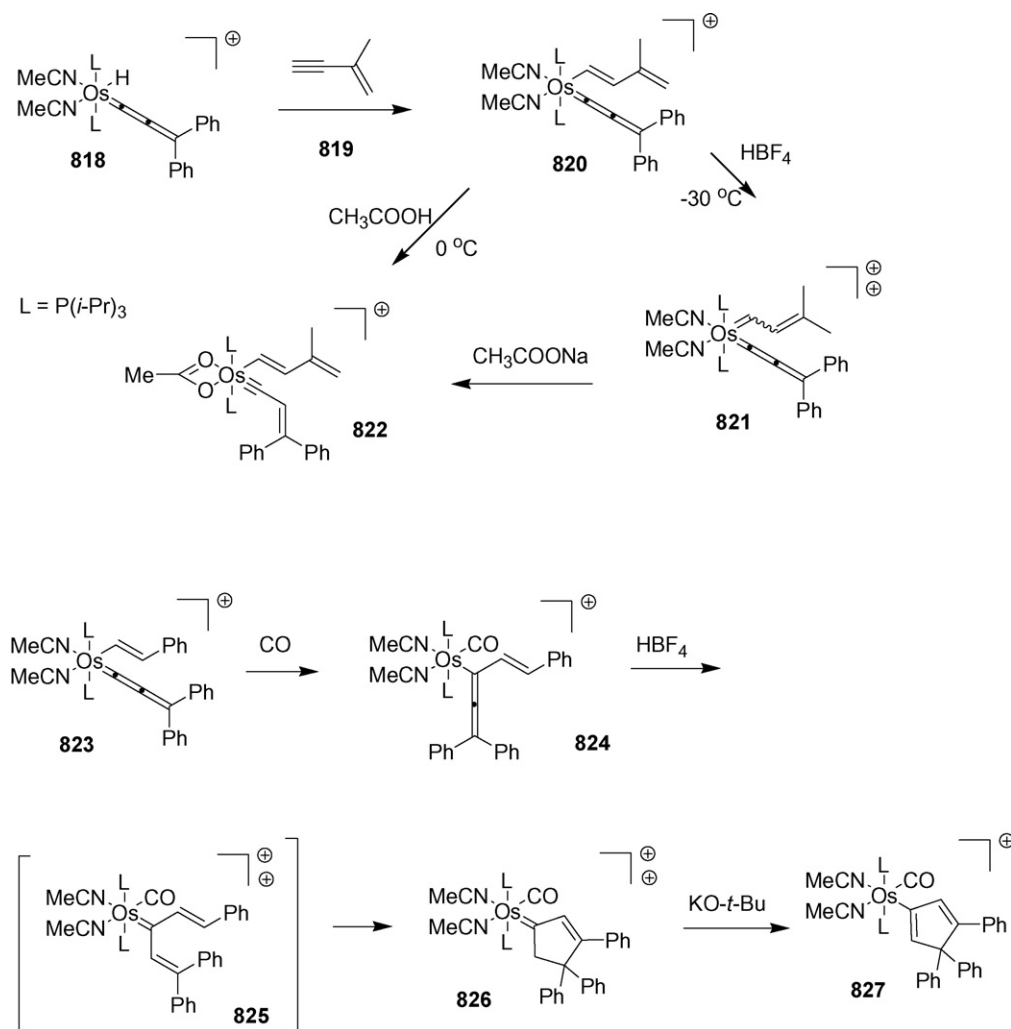
carbon monosulfide complex. These observations were attributed to initial formation of carbon disulfide bis adduct **895**, which reacts with additional carbon disulfide to afford the double- CS_2 adduct (**893**), or converts to the CS complex (**894**) when the concentration is CS_2 is minimal. A similar set of reactions were reported using phenyl isothiocyanate. The reaction of iridium carbene complexes related to **892** with heterocumulenes was studied computationally

[1007]. Transformations are consistent with [2+2]-cycloaddition followed by retro-[2+2]-cycloaddition.

The reaction of cyclooctenyliridium complex **898** (Scheme 98) with THF led to the carbene complex (**899**) in a double C–H activation process [1008]. Kinetics analysis of the process revealed dissociated of cyclooctene to be the rate determining step. The complex could also be prepared direct from iridium–cyclooctene



Scheme 88.



Scheme 89.

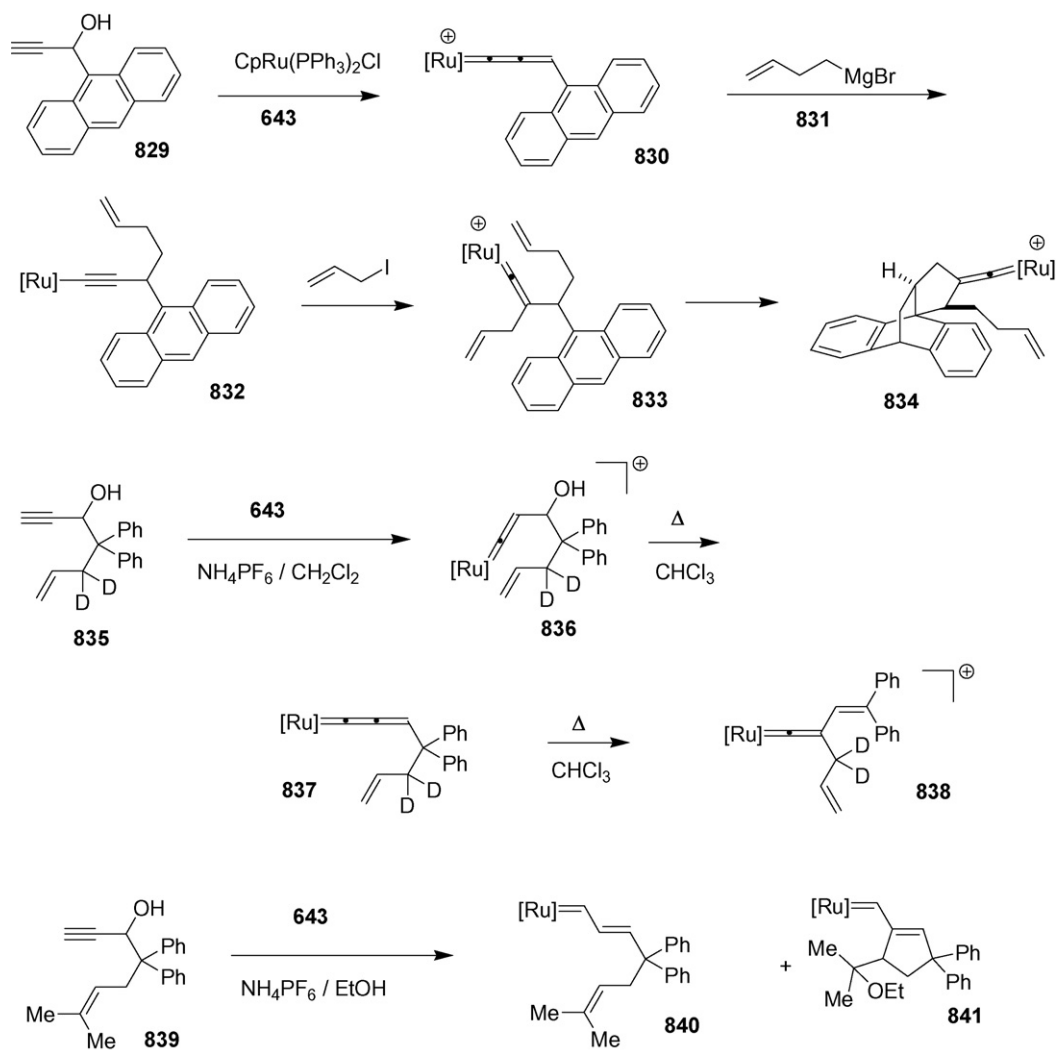
complex **901**. Iridium carbene complexes (e.g. **904**) were also prepared through C–H activation of pyridine derivatives (e.g. **903**) [1009].

An experimental and computational study of the formation of iridium carbene complexes through C–H activation of alkyl phenyl ethers or *o*-alkylphenols was reported (see Scheme 99) [1010]. Anisole (**905**) yielded two types of carbene complexes, the expected C–H activation product **907** and the rearranged carbene complex **908**. Ortho-hydroxytoluene (**909**) affords the “rearranged” carbene complex **908** upon reaction with complex **906**. Carbene complexes derived from ethyl ether derivatives (e.g. **912**) undergo an additional reaction at higher temperature, conversion to the alkene–iridium complexes (e.g. **913**). Likely mechanistic pathways were evaluated computationally. Additional experimental and computational studies of the interconversion of the above carbene complexes and alkenes were also reported [1011].

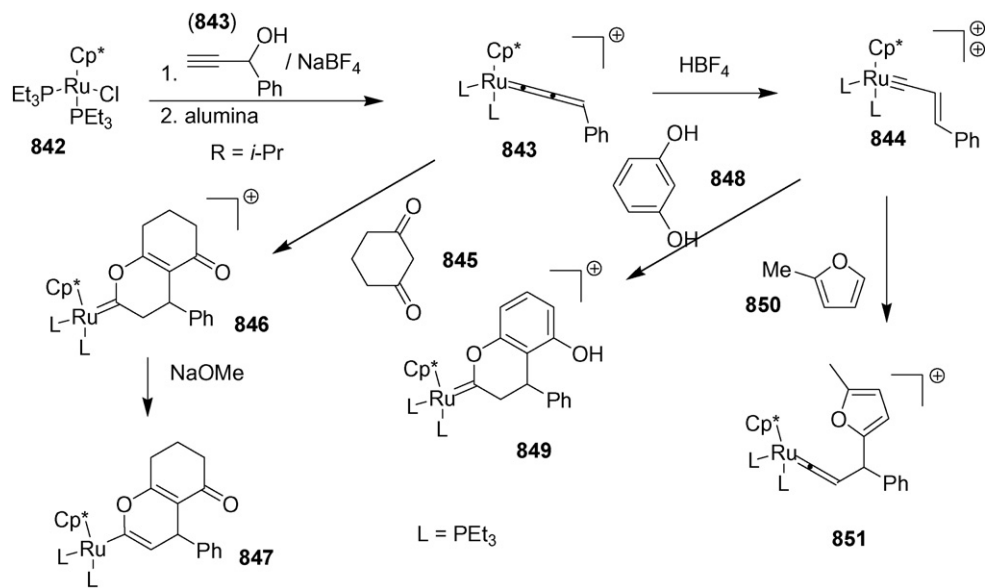
The reaction of ethyl diazoacetate with imines catalyzed by dicobalt octacarbonyl (see Scheme 100) led to amide-esters (e.g. **916**) [1012]. A key step in this transformation is carbonylation of a cobalt carbene complex intermediate (e.g. **917**). The resulting ketene (**918**) undergoes [2+2]-cycloaddition followed by ring opening of the β -lactam (**919**) to afford the final product.

The reaction of iridium complex **921** (Scheme 101) with quino-linecarboxaldehyde derivative **920** led to the hydroxycarbene complex **922** [1013]. Various ligand exchange and oxygen complexation to other metals were reported.

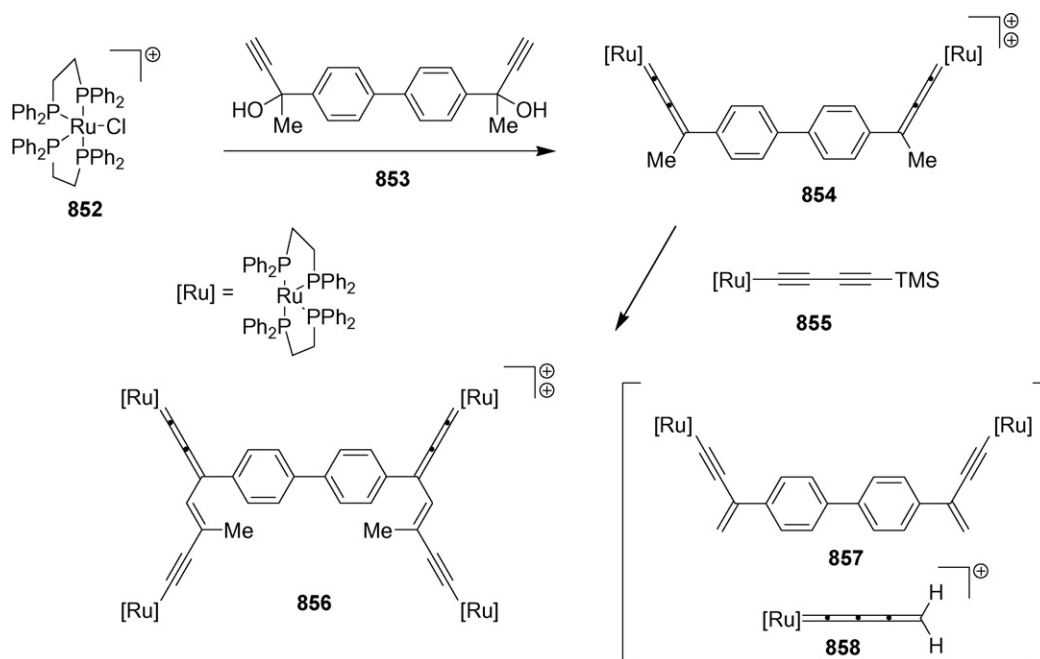
Other studies of Group 9 metal–carbene complexes (excluding metallacumulenes) are depicted in Scheme 102 include: (1) involvement of metal carbene complexes (e.g. **926**) in rhodium- and copper-catalyzed co-arcate cyclization reactions [1014]; (2) possible involvement of cobalt carbene complexes (e.g. **931**) in the reaction of stable cobaltacyclobutenes (e.g. **928**) with nitroso compounds to afford cobalt azametallacycles (e.g. **929** and **930**) [1015]; (3) possible involvement of cobalt carbene complexes in Co–S insertion of diazo-derived carbene units into cobalt dithiolene complexes [1016]; (4) possible involvement of rhodium carbene complexes (e.g. **934**) as intermediates in a nitrene initiated cascade reaction of aromatic ring containing alkynyl sulfonamides (e.g. **933**) [1017]; (5) possible involvement of cyclopropylcarbene–rhodium complexes (e.g. **937** in cycloisomerization of diene-ynes (e.g. **936**) to bis(cyclopropane)s (e.g. **938**) [1018]; (6) carbene resonance contribution to a heterobimetallic system where the carbonyl oxygen of an acyliridium carbene complex is complexed to ruthenium [1019]; (7) use of sulfoxonium ylides as a carbene source in iridium-catalyzed carbene insertion reactions [1020]; and (8) a computational study of the Fischer–Tropsch reaction on a cobalt surface [1021]. Several papers report significant mechanistic discussions of rhodium carbene complexes generated through rhodium-catalyzed diazo decomposition. Specific examples include: (1) determination of kinetic isotope effects for insertion of rhodium carbenoids into aromatic C–H bonds [1022]; (2) a computational study of donor–acceptor substituted car-



Scheme 90.



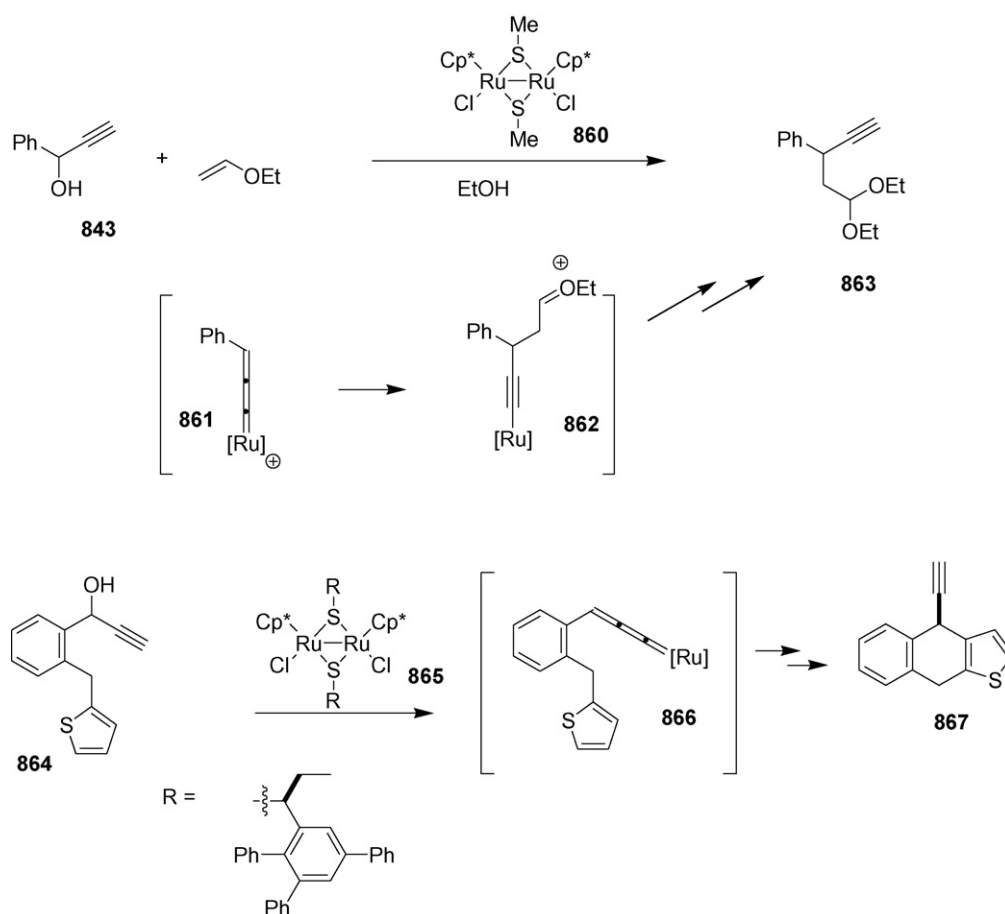
Scheme 91.



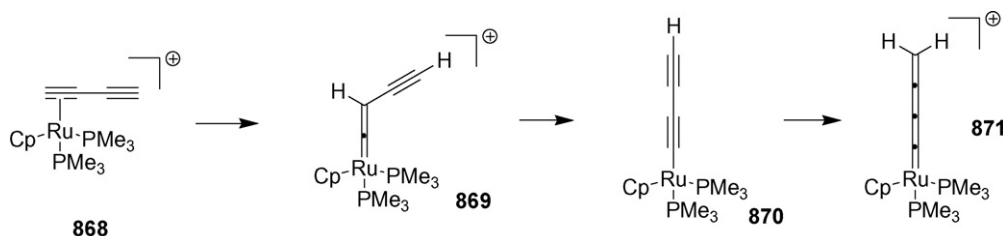
Scheme 92.

benoids with emphasis on stereoselectivity and chemoselectivity issues in comparison to more reactive carbenoids derived from simple diazoacetates [1023]; (3) an experimental and computational study of carbenoid formation and reactivity using Rh–Bi bimetallic

complexes [1024]; (4) formulation of stereoselectivity models for chiral rhodium complex-catalyzed enantioselective cyclopropanation reactions [1025,1026]; and (5) DFT studies of carbene complex intermediates from rhodium–NHC complex catalyzed



Scheme 93.



Scheme 94.

decomposition of diazoacetamides [1027]. The backbonding contributions from $\text{H}_2\text{C}=\text{Ir}(\text{Cp})(\text{CH}_2=\text{CH}_2)$ and $\text{H}_2\text{C}=\text{Ir}(\text{Cp})(\text{CO})$ were evaluated computationally [1028]. The methylene and ethylene ligands compete for backbonding, thus decreasing the bonding to the ethylene ligand. The donor properties of several NHC ligands to various iridium(I) complex fragments were also evaluated computationally.

2.3.6.2. Metalla-aromatics. The thermal equilibration of metallocycloheptatrienes (e.g. **941**, Scheme 103) and metallabicyclo[3.2.0]heptadienes (e.g. **942**) was reported [1029]. The interconversion of **941** and **942** is governed by the amount of water present in the reaction. Reaction of the metallabicyclo[3.2.0]heptadiene complex (**942**) with ligand additives led to a stable metallacycloheptatriene system (**943**). Reaction of complex **943** with *t*-butyl hydroperoxide led to the chelating metallacycloheptadiene complex **944**. The reaction of metallacyclopentadiene **940** with bis(trimethylsilyl)acetylene followed a somewhat different course leading to internally-coordinated π -allyl complex **945**. In this reaction vinylidene complex intermediates (e.g. **946** and **948**) were suggested. Two successive vinylidene formations followed by alkenyl group migrations, followed by protodesilylations were proposed. Other double insertion products were obtained using various terminal alkynes.

Sulfur-substituted iridabenzene (e.g. **951–953**, Scheme 104) were prepared from the iridacyclopentadiene-thiocarbonyl complexes (e.g. **950**) [1030]. Thermolysis of complex **950** affords the iridithi-irene derivative **951**. The three-membered ring was opened to afford thiomethyl derivative **952** by treatment with methyl triflate in dichloromethane/acetonitrile solutions. Protonation of **951** in the presence of a nitrile (acetonitrile or *p*-tolunitrile) led to the iridabenzothiazolium complex **953**. The expected thiol analog of **952** could be observed if the reaction process was followed by NMR. Addition of base (e.g. triethylamine) transforms this derivative back into the complex **951**.

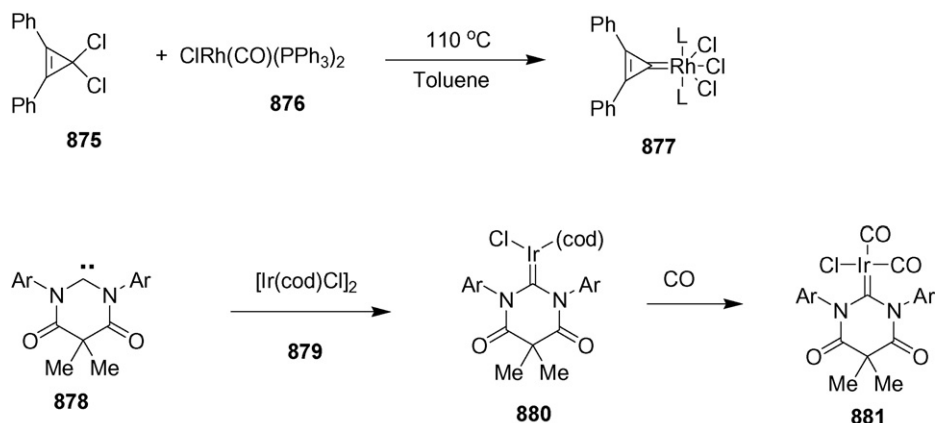
2.3.6.3. Metallacumulene complexes. Rhodium–vinylidene complexes were proposed as intermediates in the net [4+1]-cycloaddition of terminal alkynes and α,β -unsaturated imines (e.g. **954**, Scheme 105) to afford pyrrole derivatives (e.g. **957**) [1031]. The reaction could also be initiated using actual rhodium vinylidene complex **956**.

2.3.7. Group 10 metal–carbene complexes

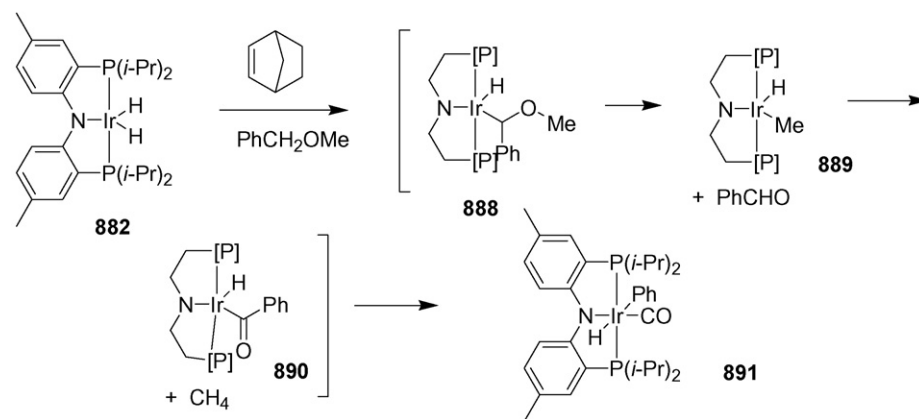
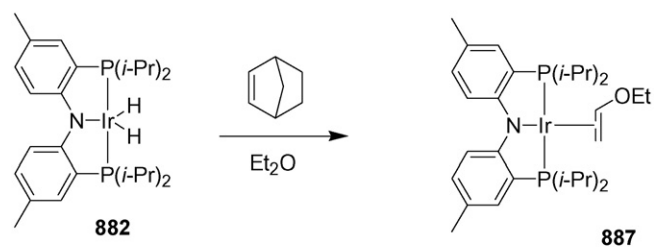
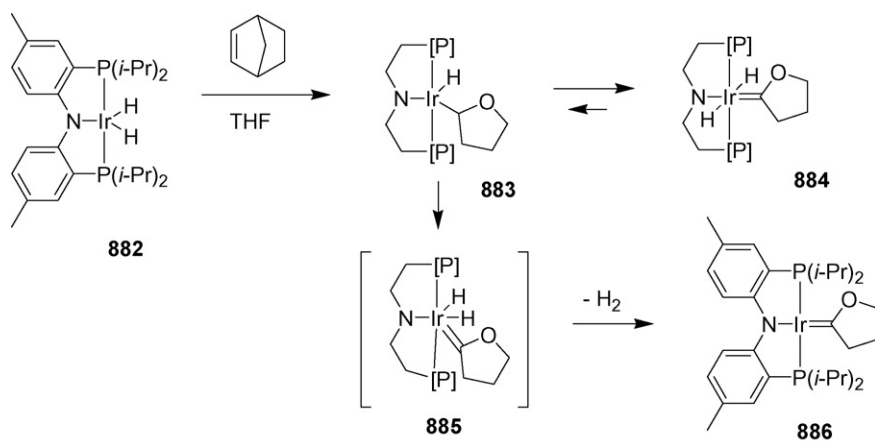
The reactions of nickel carbene complexes (e.g. **960**, Scheme 106) were investigated [1032]. Reaction with azides led to the corresponding imines (e.g. **961**) and the azide–nickel complex (e.g. **962**). Reaction with nitrous oxide led to the nickel η^2 -ketone complexes (e.g. **964**). A reaction mechanism involving formation of the diaza-metallacycle (**963**) followed by loss of nitrogen was proposed. The processes were also evaluated computationally.

Bimetallic aminocarbene–platinum complexes (see Scheme 107) were prepared from the reaction of platinum carbene complexes (e.g. **967** and **970**) with anionic metal carbonyl complexes [1033]. The reaction of alkoxycarbene complex **968** with the molybdenum anion **971** led to the alkenylplatinum complex **972**. These types of complexes could also be prepared through deprotonation of the isolated carbene complexes using amine bases. In many cases the metal could be displaced from the coordination sphere at platinum using phosphine ligands (e.g. reaction of complex **973** with triphenylphosphine to afford ionic complex **974**).

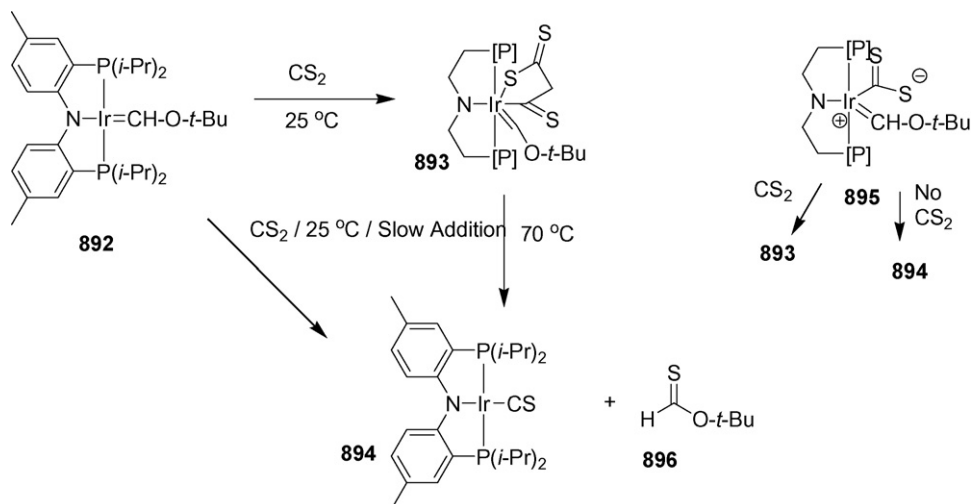
Additional examples of stable and isolable Group 10 metal carbene complexes are depicted in Scheme 108. A palladium allenylidene complex (**976**) was prepared through alkylation of a palladium propiolamide complex **975** [1034]. A cycloheptatrienylpalladium complex (**979**) was prepared from the corresponding diazo compound **977** [1035]. These complexes feature an agostic interaction with the ortho aromatic hydrogens. The reaction of protonated diacetylplatinum(II) complexes (e.g. **980**) with various reagents was reported [1036]. Treatment with



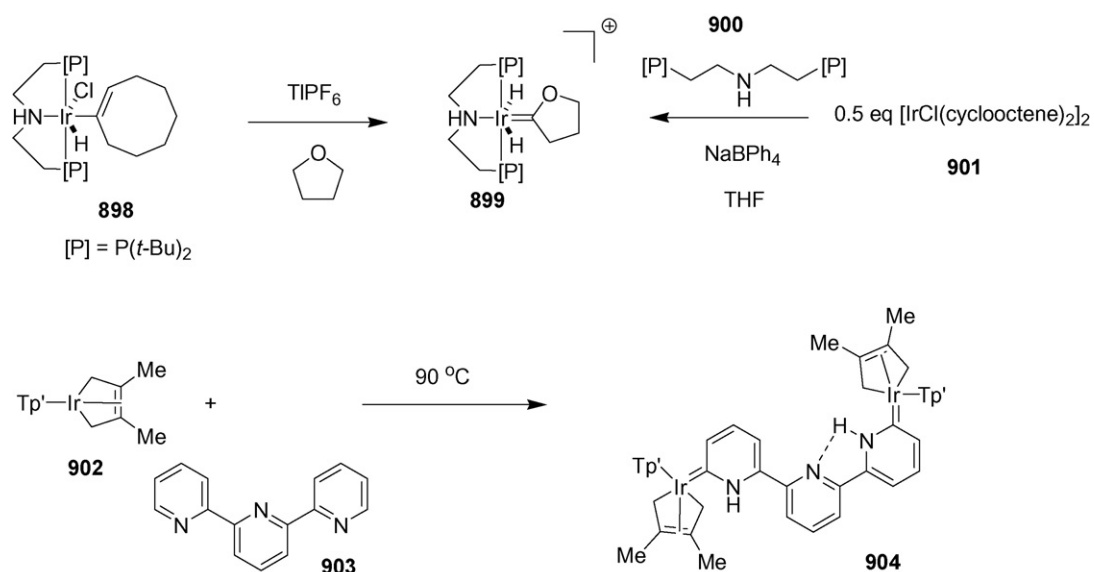
Scheme 95.



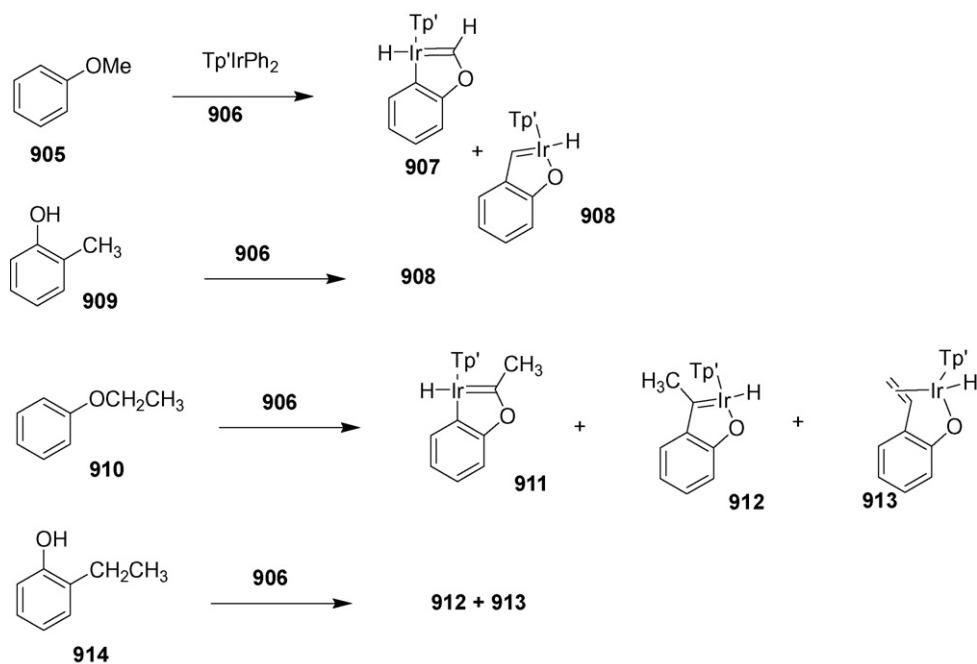
Scheme 96.



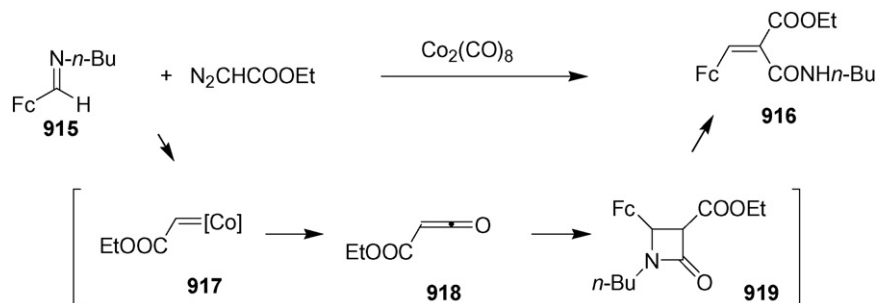
Scheme 97.



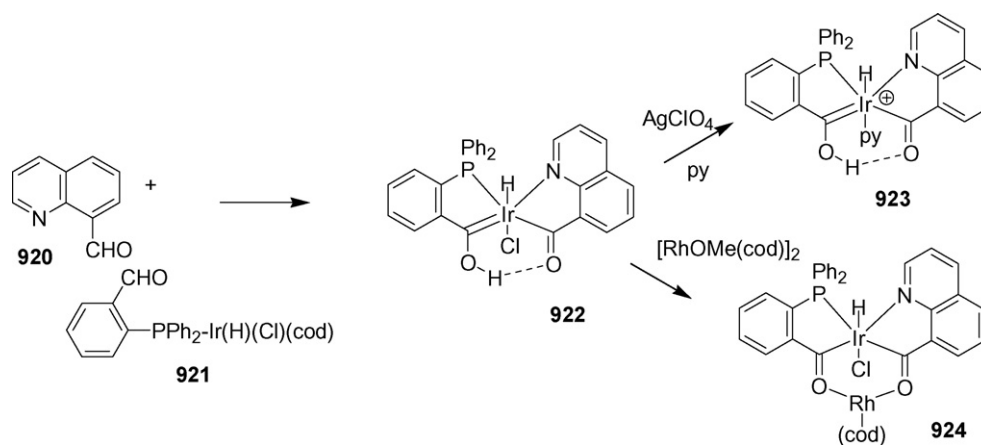
Scheme 98.



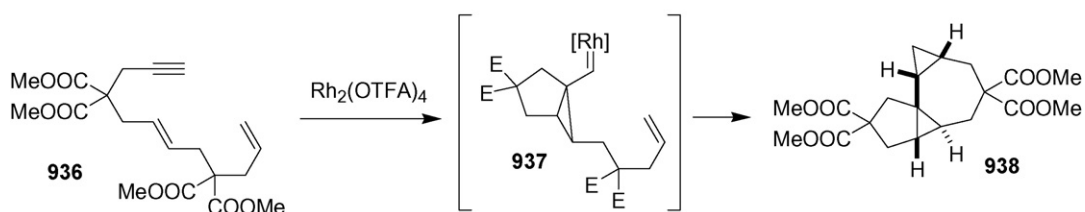
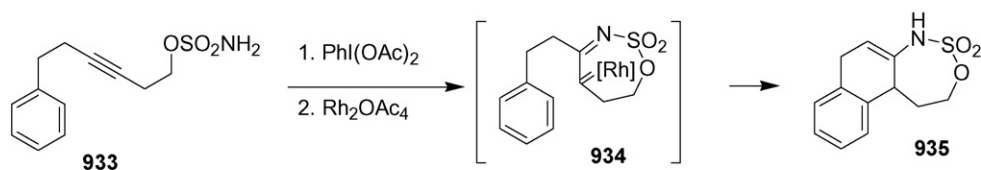
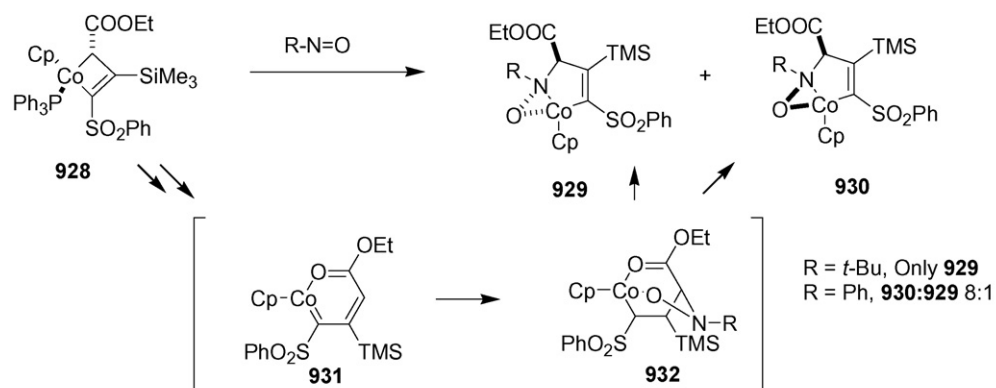
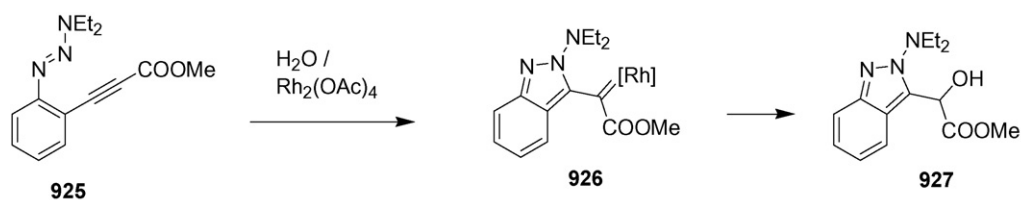
Scheme 99.



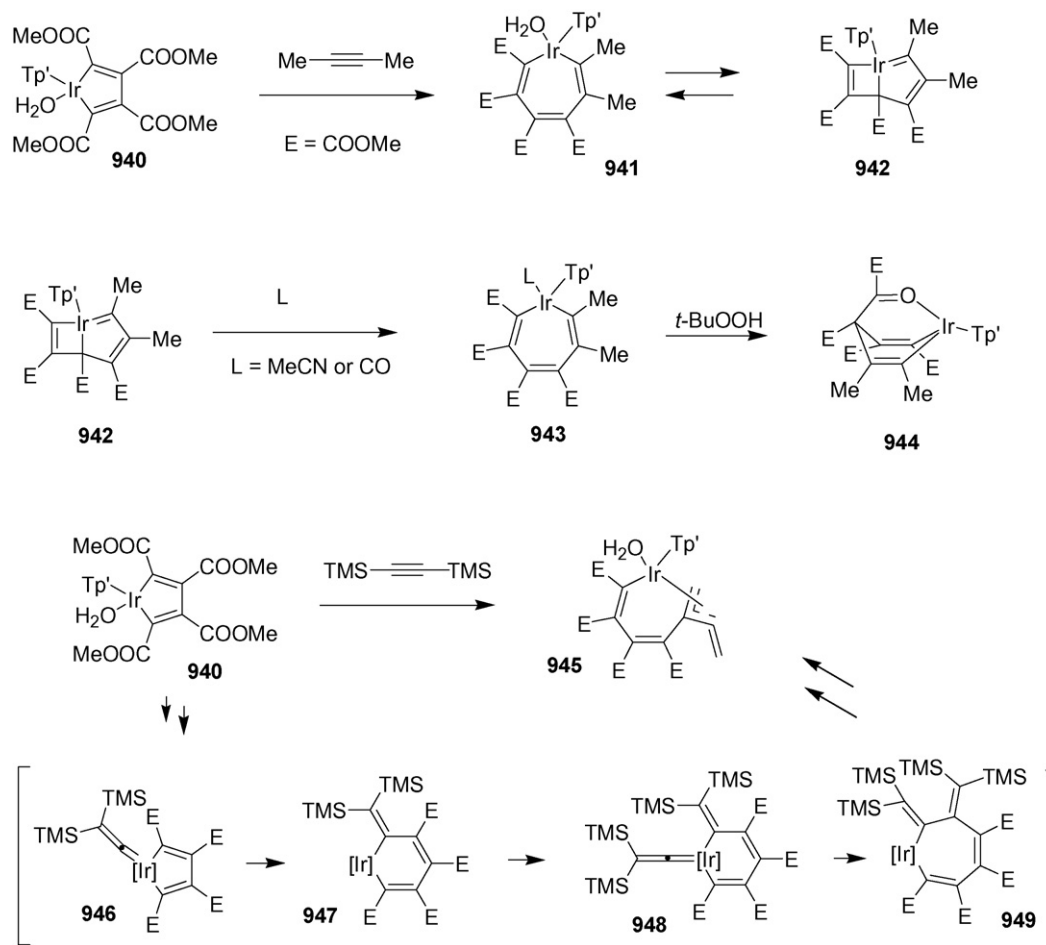
Scheme 100.



Scheme 101.



Scheme 102.



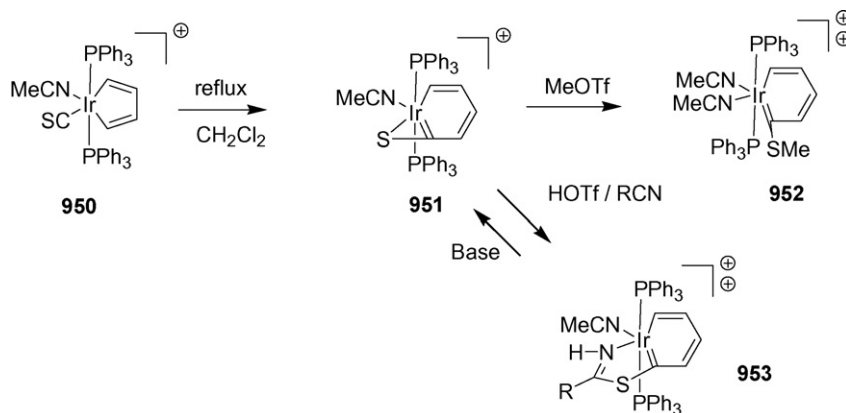
Scheme 103.

bis(trimethylsilyl)amine led to a polymeric diacetylplatinum(II) complex (**981**). Treatment of the polymeric complex with proton sources regenerates cationic carbene-like species (e.g. **982** and **983**).

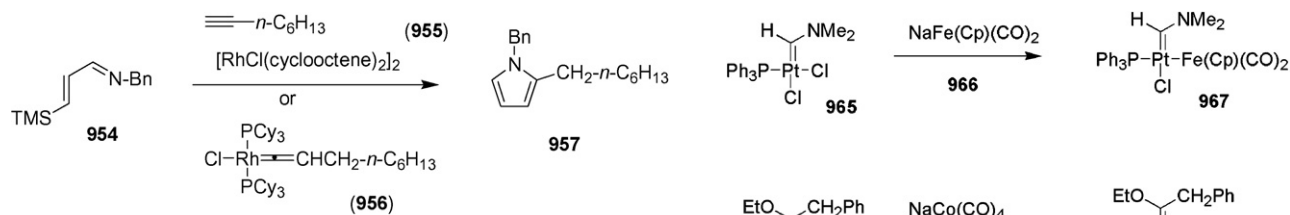
The synthesis and reactivity of platinabenzene (e.g. **985**, **986** and **989**, Scheme 109) was reported [1037]. Complex **985** was produced from the reaction of the organolithium reagent derived from iodide **984** with platinum halides. The reaction with the platinum-cod complex resulted in the Cp-platinabenzene complex **985**. A Cp*-platinabenzene complex (**989**) was obtained directly using the Cp* platinum complex **987**. In some cases the simple transmetal-

lation product (**988**) could be obtained, which readily rearranges to the platinabenzene. The reactivity of platinabenzene complexes was briefly probed. The complexes are thermally very stable and do not decompose even at 100°C . Treatment with bromine or oxygen leads to decomposition. Complex **989** undergoes Diels-Alder reaction with maleic anhydride (**990**) to afford platinabicyclic **991** but not with DMAD. The metalla-arene ring also undergoes molybdenum complexation to afford a metalla-arene complex (**993**).

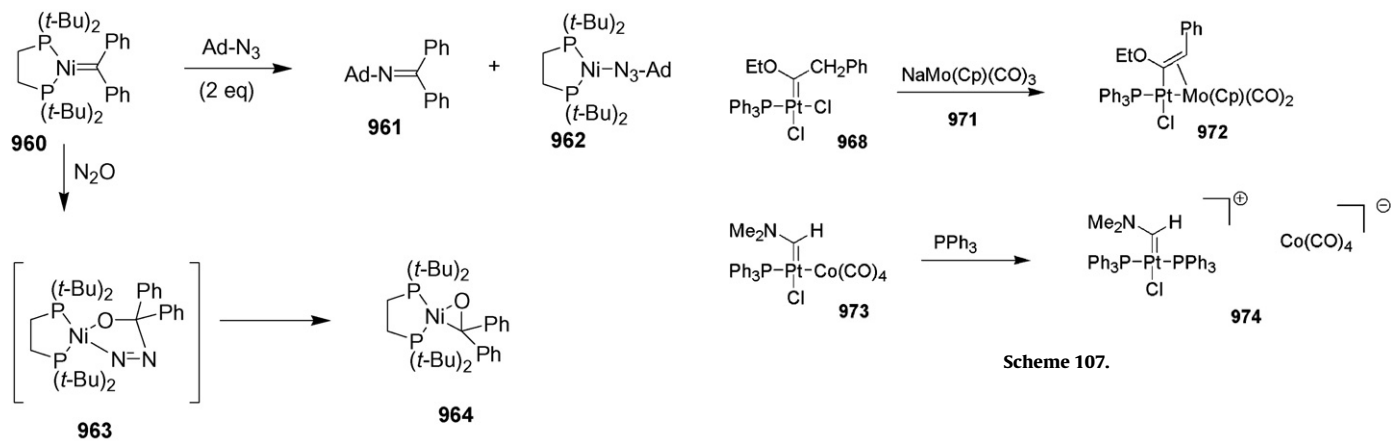
Several processes reported in 2009 invoke Group 10 metal carbene complexes (see Scheme 110). Nickel carbene complexes (e.g. **997**) were proposed as intermediates in the formation of



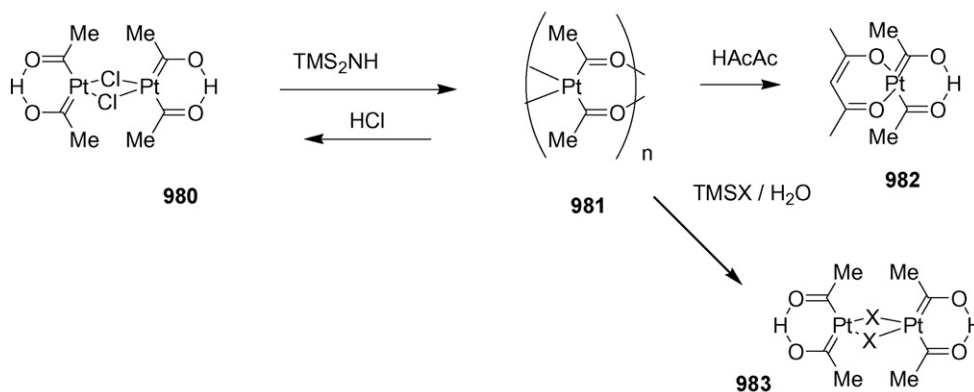
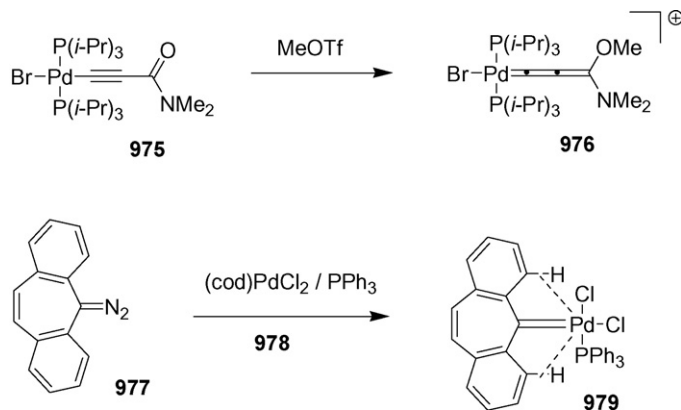
Scheme 104.



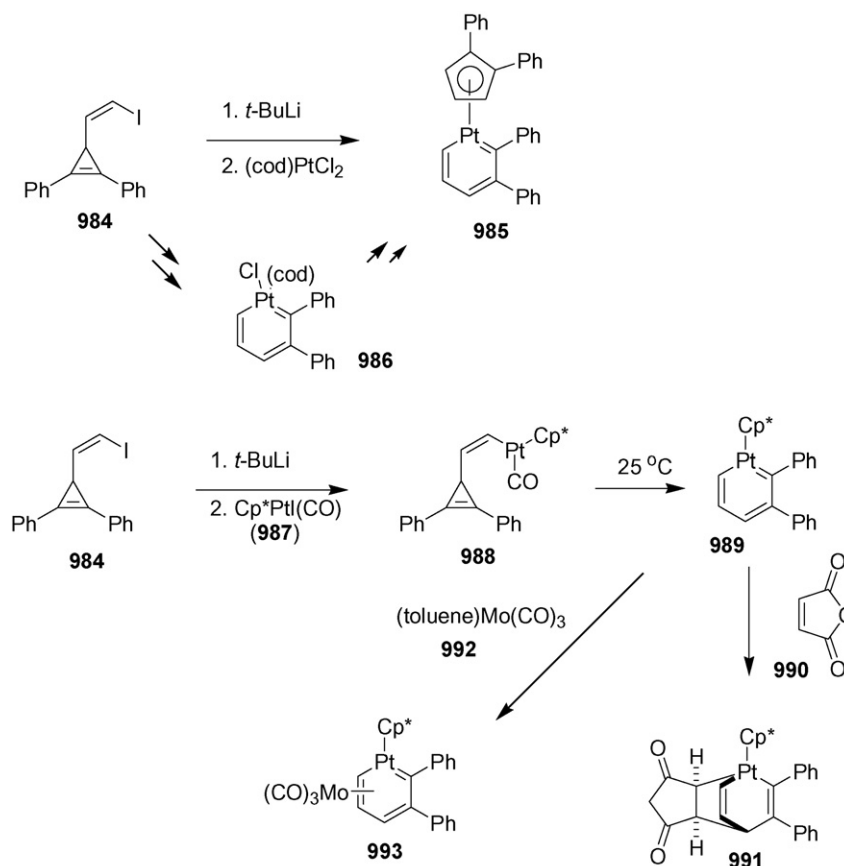
Scheme 105.



Scheme 106.



Scheme 108.



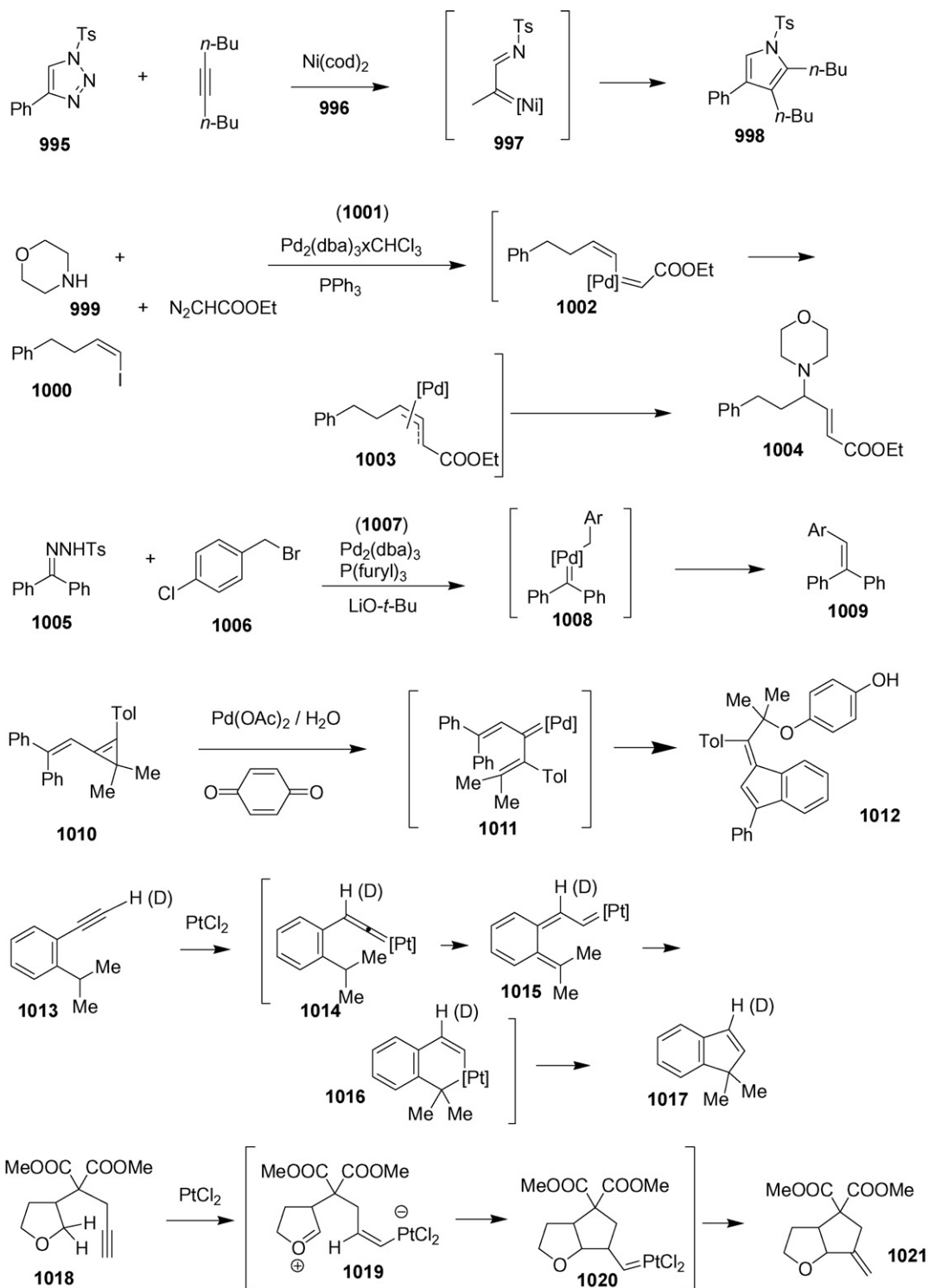
Scheme 109.

pyrroles (e.g. **998**) from the nickel-catalyzed coupling of triazoles (e.g. **995**) with alkynes [1038]. Palladium carbene complexes (e.g. **1002**) were proposed as intermediates in the reaction of ethyl diazoacetate with alkenyl iodides (e.g. **1000**) and amines in the presence of palladium complex **1001** [1039]. The proposed mechanism involves formation of alkenylpalladium–carbene complex **1002**, migration to form an allylpalladium complex (**1003**), followed by reaction with the amine. Palladium carbene complexes (e.g. **1008**) were suggested as intermediates in the palladium-catalyzed formation of alkenes (e.g. **1009**) from tosylhydrazones (e.g. **1005**) and halides [1040]. Palladium carbene complexes (e.g. **1011**) were suggested as intermediates in the palladium-catalyzed ring expansion/cyclization of vinylcyclopropanes (e.g. **1010**) in the presence of benzoquinone [1041]. Palladium carbene complexes were suggested as intermediates in palladium-catalyzed ring opening reactions of alkylidenecyclopropane-ketones [1042]. Platinum vinylidene complexes (e.g. **1014**) were suggested as intermediates in a cyclization/C–H activation process [1043]. Deuterium labeling studies support the proposed mechanism. Platinum vinylidene and carbene complexes (e.g. **1020**) were suggested as likely intermediates in cyclization/C–H activation reactions of alkyne-tetrahydrofurans (e.g. **1018**) [1044]. Deuterium labeling studies support this mechanistic pathway. Platinum carbene complexes were suggested as intermediates in platinum-catalyzed ring expansion of alkylidenecyclopropanes [1045]. Platinum carbene complexes were suggested as intermediates in E/Z isomerization of alkenylplatinum complexes [1046]. A substantial backbonding contribution was suggested for palladium oxadiazoline complexes, obtained through dipolar cycloaddition of nitrones and palladium isocyanide complexes [1047,1048]. Several papers report on mechanistic aspects of Group 10 metal-catalyzed cyclopropana-

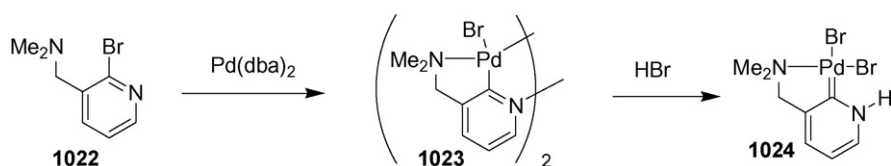
tion reactions [1049,1050]. A computational study of nickel(0) and nickel(II)-catalyzed cyclopropanation was also reported [1051].

For all of Group 10 metals, atoms generated through laser ablation were reacted with methane derivatives and the adducts isolated in an argon matrix. Products were identified through their infrared spectra and comparison with calculated spectra for geometry-optimized structures. Nickel carbene complexes were generated through reaction of perhalomethane derivatives with nickel atoms [1052]. The carbene complexes were accompanied by C–X simple insertion products. The carbene complexes were of similar energy when configured in the singlet and triplet configurations. Only the insertion products were obtained from trihalomethane and dihalomethane derivatives. Palladium carbene complexes were generated from the reaction of palladium atoms with various halomethane (X = Cl, F) derivatives [1053]. The reaction of perhalomethanes led to carbene complexes [X₂C=PdX₂] and/or insertion products [X₃C–PdX₃], while reaction with trihalomethanes and dihalomethanes led to only insertion products. Platinum carbene complexes were generated through reaction of platinum atoms with methane, ethane, or halomethane derivatives [1054]. The carbene complex H₂C=PtH₂ was detected in the reaction with methane, however H₃C–PtH was the overwhelmingly major product. The carbene complex H₂C=PtHF was identified in the reaction of platinum atoms with CH₃F, and this carbene complex is likely an intermediate in the conversion of initially-formed H₃C–PtF to the thermodynamically more stable H₂FC–PtH. Carbene complexes were also identified in the reaction of platinum atoms with CH₂F₂, CH₂ClF, and CHX₃ (X = Cl, F).

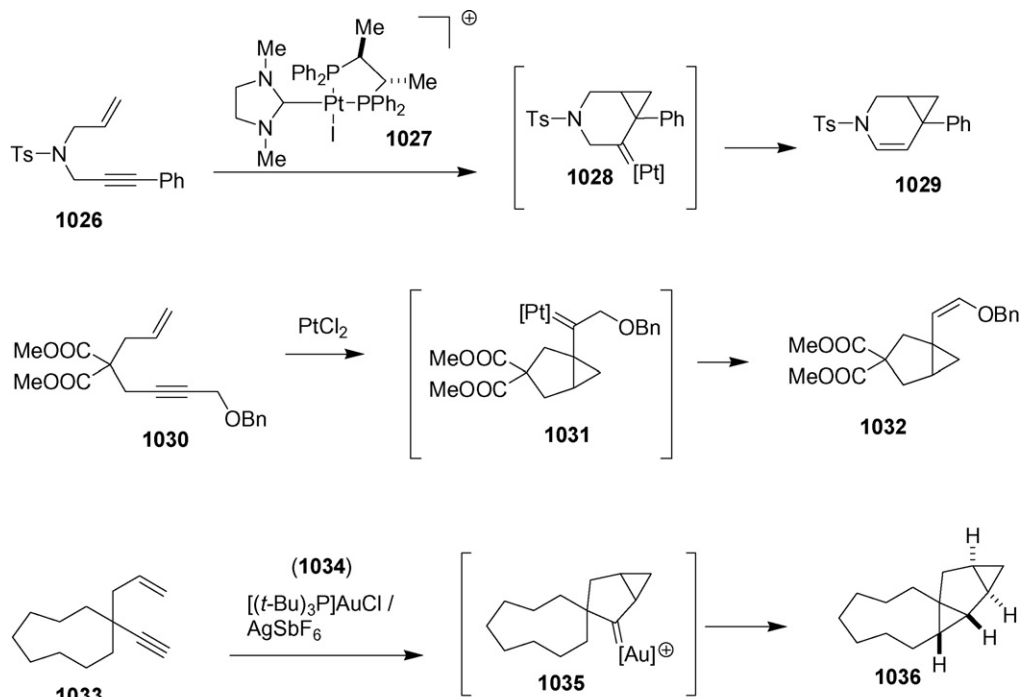
Additional studies of Group 10 metal carbene complexes are depicted in Scheme 111. A palladium carbene complex (**1024**) was prepared through protonation of 2-pyridylpalladium complex



Scheme 110.



Scheme 111.



Scheme 112.

1023 [1055]. There was however minimal evidence of a Pd–C double bond. Computational studies of the bonding in pyridinylidene (and benzo analogs) Group 10 metal complexes were reported [1056]. The failure to observe palladium(II) vinylidene complexes by collision-activated ion-molecule reactions from the palladium-catalyzed coupling of phenylacetylene and norbornadiene was noted [1057]. The interconversion of palladium alkynyl complexes and vinylidene complexes were evaluated computationally. The binding of halocarbene fragments to a palladium surface was evaluated computationally [1058]. The demetallation of alkenylplatinum complexes through protonolysis, which may involve platinum carbene complexes, was examined through deuterium labeling studies [1059].

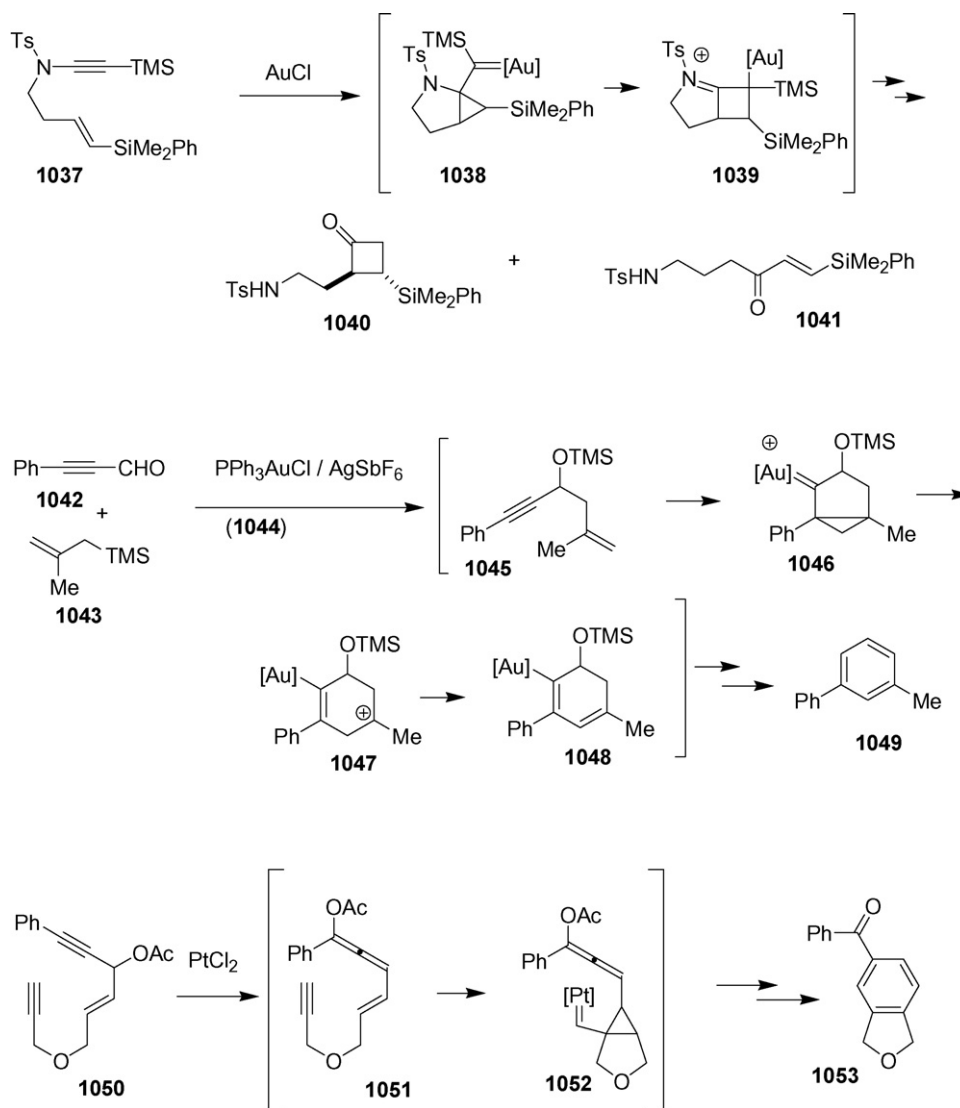
2.3.8. Generation of platinum and gold carbene complexes from alkynes or allenes

Several papers report on the development of new reaction processes using carbenes generated from alkynes or allenes and either platinum or gold complexes. Some controversy exists as to whether the intermediates of these reactions are actual carbene complexes or metal-stabilized carbocations [1060].

Reaction of simple enynes with gold or platinum compounds frequently results in the generation of cyclopropylcarbene–metal complexes. Examples that retain the initially-produced ring system are depicted in Scheme 112. Asymmetric cycloisomerization of enynes (e.g. **1026**) using a chiral platinum complex (**1027**) was reported [1061]. Related enantioselective cycloisomerizations using gold complexes that contain chiral phosphine ligands [1062] or chiral platinum chelating –NHC complexes were also reported [1063]. Platinum chloride catalyzed enyne cycloisomerization of propargyl ethers (e.g. **1030**) led to the selective formation of cyclopropyl enol ethers (e.g. **1032**) [1064]. Enyne cycloisomerization accompanied by trapping of the carbene complex intermediate through intramolecular C–H insertion reactions [e.g. conversion of enyne **1033** to bis(cyclopropane) **1036**] was also demonstrated [1065]. Computational studies of platinum-catalyzed enyne cycloisomerization were also reported [1066].

In many of these reactions, secondary processes that involve opening of the cyclopropane ring are observed; representative examples are depicted in Scheme 113. The use of ynamines (e.g. **1037**) in this reaction led to acyclic products (e.g. **1041**) and cyclobutanones (e.g. **1040**) [1067]. In this case, the intermediate cyclopropylcarbene–gold complex (**1038**) undergoes rearrangement to an iminium salt (**1039**), which then hydrolyzes to afford the cyclobutanone and the corresponding ring opened product. The gold-catalyzed coupling of propargyl aldehydes (e.g. **1042**) and allylsilanes (e.g. **1043**) leads to aromatic rings (e.g. **1049**) in a formal [3+3]-cycloaddition process [1068]. In this reaction, a Lewis acid-catalyzed allylation affords an enyne (**1045**), which cyclizes to afford a cyclopropylcarbene complex (**1046**), which affords the observed product after ring opening and elimination. A novel five-membered ring annulation pinacol rearrangement sequence likely involves cyclopropylcarbene–gold complex intermediates [1069]. Cycloaromatization (e.g. formation of **1053** from **1050**) was observed in the reaction of diene–propargyl acetates (e.g. **1050**) with PtCl₂ [1070]. The six-membered ring was obtained through rearrangement of allenylcyclopropylcarbene complex intermediate **1052**.

In several cases, the cyclopropylcarbene complex intermediates were intercepted with nucleophiles. Representative examples are depicted in Scheme 114. Gold-catalyzed cyclization of enynes (e.g. **1055**) in the presence of electron-rich aromatic rings (e.g. **1056**) (including heterocycles) led to monocyclic products (e.g. **1059**) from nucleophilic ring opening of the cyclopropylcarbene complex intermediate (**1057**) [1071]. Other reports involving capture by an electron-rich aromatic ring include a moderately enantioselective process where the cyclopropylcarbene complex was intercepted through reaction with indoles [1072]. A net double cycloaddition process was observed in the reaction of enyne **1060** with a gold complex to afford tetracyclic compound **1064** [1073]. In this process, a methoxy group nucleophilically attacks the cyclopropylcarbene complex intermediate (**1061**) to afford oxonium ion (**1062**). Subsequent elimination followed by Nazarov-type cyclization leads to the observed product (**1064**). Asymmetric formation



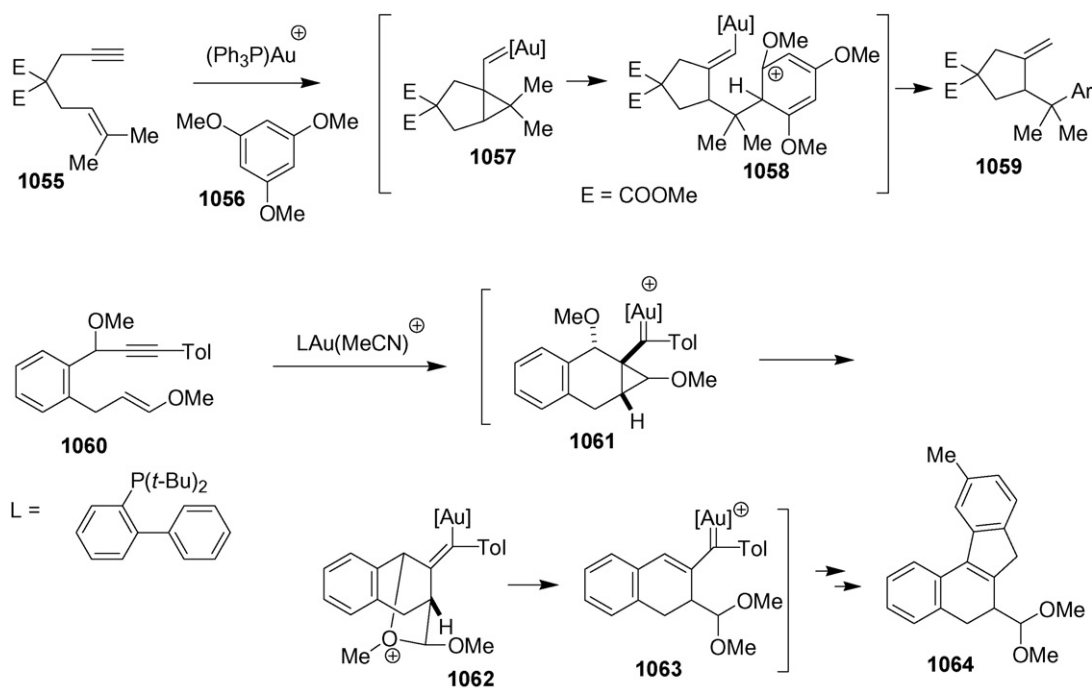
Scheme 113.

of cyclopropylcarbene–gold complexes followed by interception with alcohol nucleophiles was also reported [1074]. Capture of the cyclopropylcarbene complex with allylic alcohols affords compounds where additional rings can be formed through RCM [1075]. Several examples involving the capture of cyclopropylcarbene complex intermediates through reaction with carbonyl groups were reported. Representative examples are depicted in Scheme 115. A net double cyclization process was observed when enyne–ketone **1065** was treated with a gold complex [1076]. The proposed mechanism involves nucleophilic attack of the carbonyl oxygen to open the cyclopropylcarbene complex (**1066**) followed by coupling of vinylgold and oxonium ion functionalities in the resulting intermediate (**1067**). A net [2+2+2]-cycloaddition process (e.g. conversion of enyne **1055** and benzaldehyde to **1071**) was reported [1077]. In this case, a net cycloaddition of the carbonyl compound to the cyclopropylcarbene complex intermediate (**1059**) affords an intermediate (**1069**) containing oxonium ion and vinyl-gold functionalities, which either cyclizes to afford the cyclic ether **1071**, which can undergo retrocycloaddition eliminating acetone to form diene **1072**. A net intramolecular [3+3]-cycloaddition reaction was observed in the reaction of ynamine-carbamates (e.g. **1073** with gold catalysts [1078]. Nucleophilic attack by the carbonyl oxygen in the cyclopropylcarbene complex intermediate (**1074**) was

a key step. Reaction of cyclopropylcarbene–metal intermediates with an ester carbonyl group was also reported [1079]. In some cases the carbonyl incorporation products result from reversible opening of the cyclopropylcarbene complex to an allylcarbene complex [1080].

Platinum- and gold-catalyzed enyne metathesis (see Scheme 1) and alkyne + alkene [2+2]-cycloaddition likely involves metal cyclopropylcarbene complex intermediates. Examples are depicted in Scheme 116. The platinum catalyzed enyne metathesis of bis(phosphonate) **1077** (and several other organotransition metal-based transformations for this compound) was followed by ³¹P NMR [1081]. A chiral intermediate, possibly the cyclopropylcarbene complex **1078**, was detected. Use of gold catalysts for [2+2]-cycloaddition of enyne systems was reported [1082]. Gold-catalyzed [2+2]-cycloaddition to afford net alkene isomerization products (e.g. conversion of enyne **1080** to cyclobutene **1082**) was proposed to involve cyclopropylcarbene–gold complexes (**1081**) [1083].

Several examples employing platinum or gold carbene complexes generated through intramolecular cyclization of furan–alkyne derivatives were reported in 2009. Representative examples are depicted in Scheme 117. A moderately enantioselective cyclization reaction was observed in the



Scheme 114.

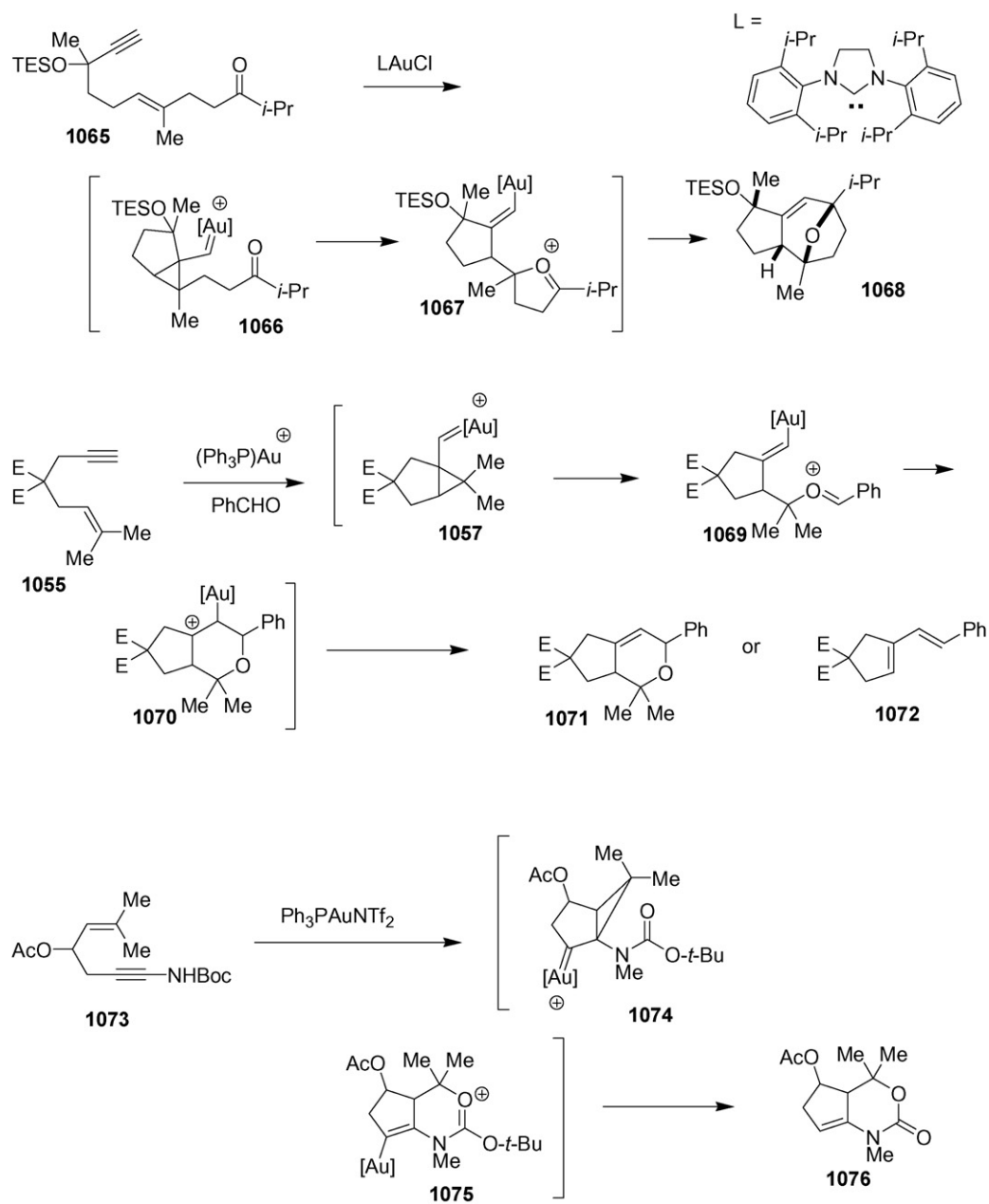
reaction of furan–dialkyne derivatives featuring enantiotopic alkyne groups (e.g. **1083**) with chiral gold carbene complexes [1084,1085]. A diastereoselective reaction employing a substrate with diastereotopic alkyne groups was also reported [1086]. Annulation reactions resulting in benzofurans (e.g. **1088**) were observed for propargyl alcohol–furans featuring a shorter tether (e.g. **1086**) [1087]. Reaction of furan–alkoxyalkyne derivative **1089** with a gold complex led to the cyclized product **1093** in a process involving cyclopropylcarbene formation (**1090**) followed by intramolecular electrophilic addition to the benzene ring [1088]. In this case the regiochemistry of ring formation was reversed relative to previous studies due to the alkoxy group on the acetylene. The course of the reaction using arylacetylene–ynamides (e.g. **1094**) was dependent on the length of tether [1089]. The gold-catalyzed reaction of enyne–alcohols (e.g. **1098**) and methylfuran led to the arylenone derivatives (e.g. **1101**) [1090]. The proposed mechanism involves electrophilic aromatic substitution to afford a furan–alkyne (**1099**) and gold-catalyzed cyclization. Opening of the cyclopropylcarbene complex intermediate (**1100**) affords the desired product.

Gold- and platinum carbene complexes were also generated from various heterocyclization processes. Several examples involving the generation of carbonyl ylides from enyne–carbonyl compounds were reported in 2009. Representative examples are depicted in Scheme 118. Alkynylbenzaldehyde derivatives (e.g. **1102**) afforded cyclized products (e.g. **1105** and **1106**) upon treatment with PtCl_2 [1091]. Key steps involve formation of the carbonyl ylide intermediate (**1103**), followed by dipolar cycloaddition to afford the carbene complex (**1104**), followed by C–H insertion. Spirocyclic compound **1106** arises from an acid-catalyzed rearrangement of compound **1105**. Related compounds (e.g. **1107**) undergo intermolecular coupling with alkene dienophiles to afford 8-oxabicyclo[3.2.1]octenes (e.g. **1112**) [1092]. In this case the carbonyl ylide intermediate (**1108**) reacts as a diene in a Diels–Alder reaction. A phenyl shift of the resulting oxonium ion (**1110**) affords a platinum carbene complex (**1111**), which converts to the alkene. Several examples involving a net transfer of an oxygen atom were reported in 2009. Representative examples are depicted in Scheme 119. The reaction of sulfoxide enynes (e.g. **1113**) with a gold

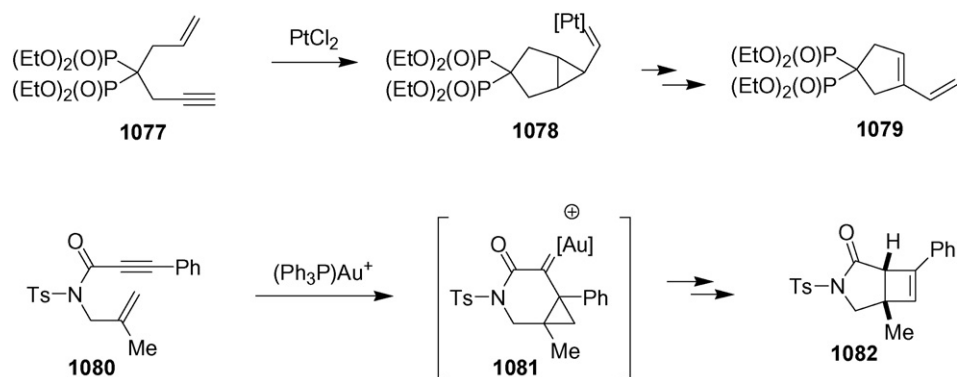
catalyst led to cyclic ketones (e.g. **1117**) in a process involving formation of the α -ketocarbene complex (**1115**) through oxygen atom transfer, followed by ylide (**1116**) formation, followed by [2,3]-sigmatropic rearrangement [1093]. Gold carbene intermediates generated through heterocyclization (e.g. **1121**) were proposed as key intermediates in a novel oxy-transfer/alkyl shift/aromatization process employing α -acetylenic ketones (e.g. **1118**) [1094]. The formation of the cyclic ketone (e.g. **1127**) from amine–alkynes (e.g. **1123**) and mCPBA was proposed to involve formation of the amine oxide, followed by heterocyclization and fragmentation to form the α -ketocarbene complex (**1126**) [1095]. Subsequent C–H insertion then leads to the observed product. The same reaction sequence was employed as a key step in the total synthesis of cernizine C [1096].

Carbene complexes were also generated from 1,2-shift of propargyl ester derivatives in the presence of platinum and gold complexes. In many cases, the reactions result in cyclopropanation products (see Scheme 120). The reaction of propargyl acetate–diene derivative **1130** with gold complexes was studied experimentally and computationally [1097]. The cyclopropane derivatives **1133** and **1134** arise through 1,2-acetate shift–carbene (**1131**) generation followed by intermolecular cyclopropanation. The alternate cyclopropanation product **1136** arises through rearrangement to the allene (**1132**), followed by rearrangement to a carbene complex (**1135**) and intramolecular cyclopropanation. Similar reactions were accomplished using ionic liquids as solvents [1098]. The acetate shift process was employed as a key step in the total synthesis of cubebol from enyne derivative **1137** [1099]. Asymmetric tandem acetate shift–cyclopropanation (e.g. formation of **1140**) was also reported [1100]. A tandem acetate shift and carbene addition process was observed in the platinum- and palladium-catalyzed reaction of propargyl acetates (e.g. **1141**) and furans to afford trienones (e.g. **1144**) [1101]. In this case the initially formed cyclopropane (**1143**) undergoes ring opening under the reaction conditions.

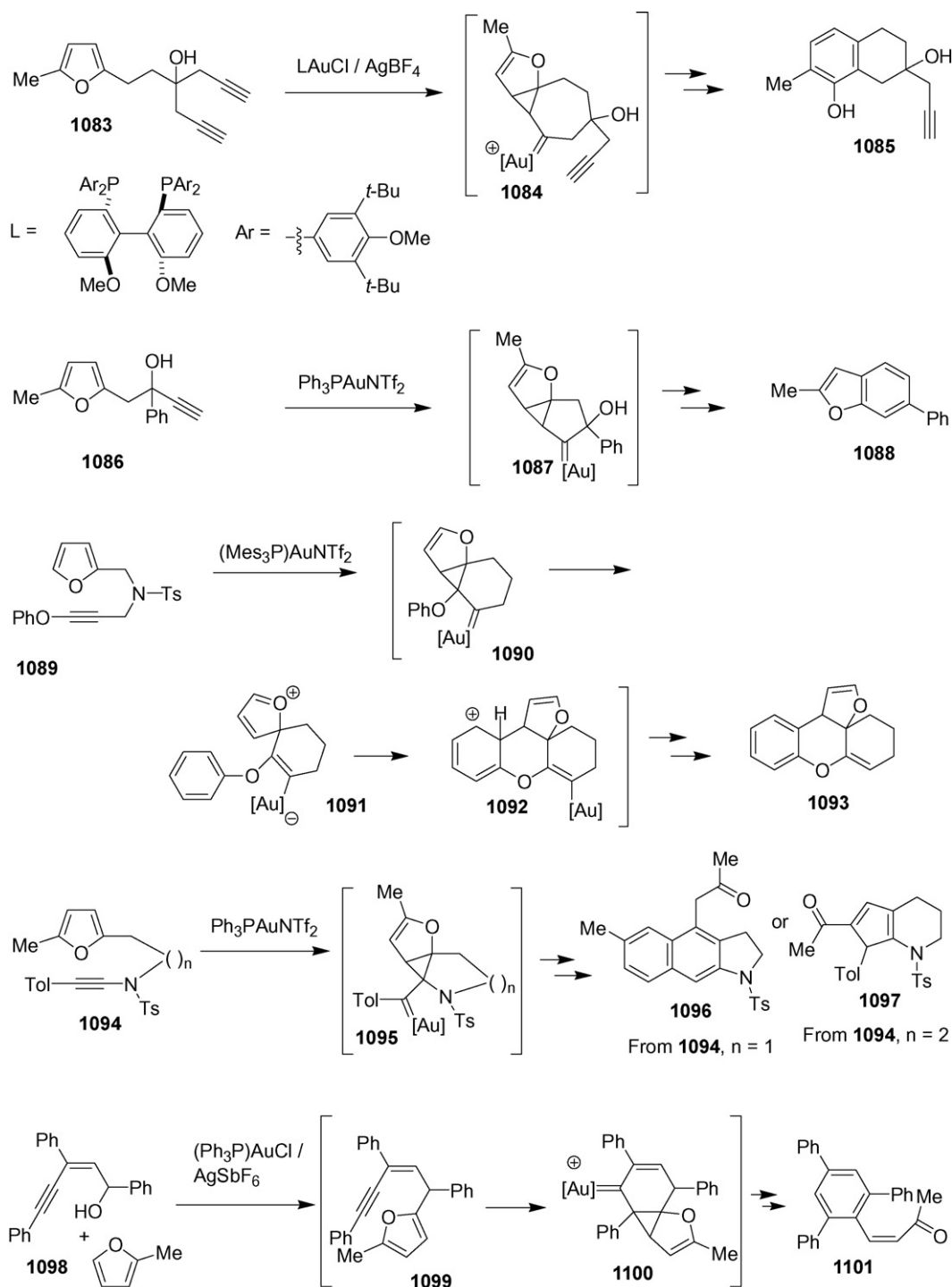
In many cases, the carbene complex intermediate is trapped in other processes. Representative examples are depicted in Scheme 121. The 2-butyne-1,4-diol derivative **1145** afforded diene



Scheme 115.



Scheme 116.

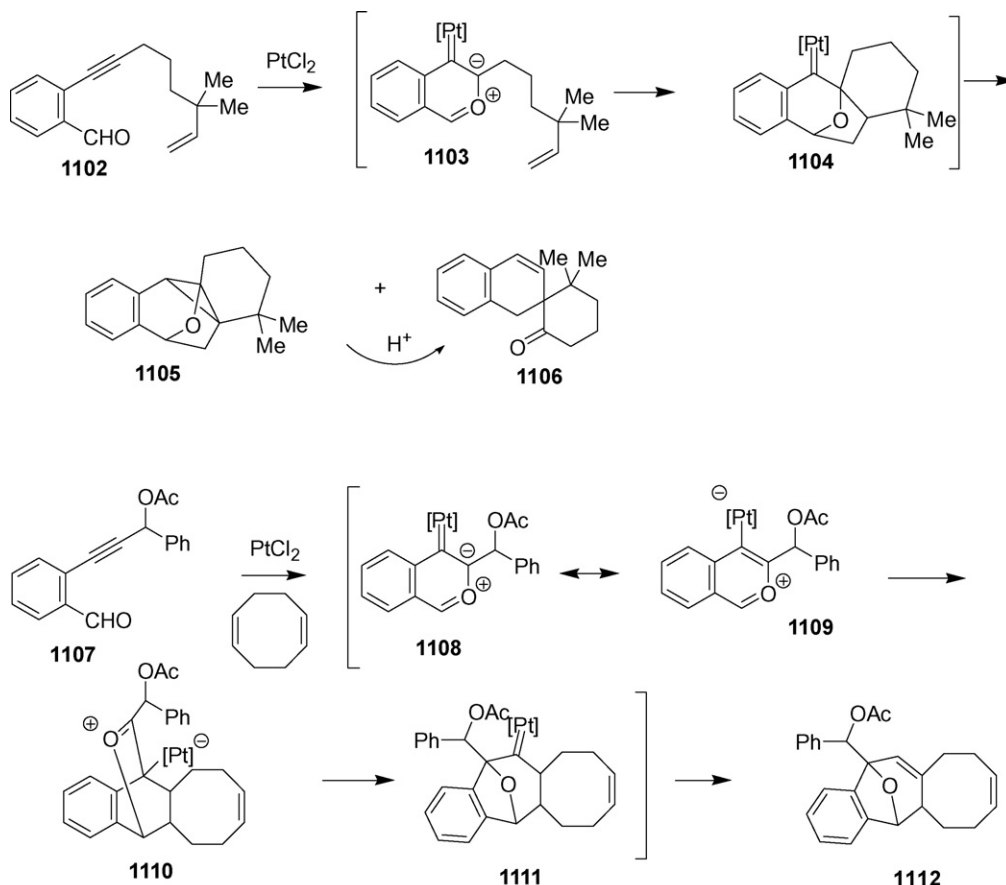


Scheme 117.

derivative **1147** upon treatment with a gold complex [1102]. In this case, after formation of the initial carbene complex (**1146**) a second 1,2-shift of an acetate group occurs to afford the final product. Treatment of propargyl esters that contain allylic alcohol groups (e.g. **1148**) with gold complexes resulted in the formation of cyclized compounds (e.g. **1151**) in a sequence involving tandem carboxylate shift, followed by oxonium ylide (**1150**) formation, followed by ylide rearrangement [1103]. A net [3+3]-cycloaddition was observed when propargyl carboxylates (e.g. **1152**) and azomethine imines (e.g. **1153**) were treated with a gold catalyst [1104]. Carbene complexes were also generated through 1,2-shift of sulfur

using propargylic sulfides. Generation of carbene complexes (e.g. **1157**) through shift of sulfur was proposed as a key step in the conversion of alkyne-dithioacetal **1156** to the cyclic allene **1158** [1105]. In this case the carbene complex intermediate undergoes an alkyl shift process. Gold carbene complexes were considered as likely intermediates in gold-catalyzed formation of naphthalenes from 5-aryl-3-acyloxy-1-pentyne derivatives, however ruled out based on deuterium labeling studies [1106].

Several examples employing gold or platinum catalysts in cyclopropane ring expansion reactions involving cyclopropylacetylenes were reported in 2009. Representative examples are depicted in



Scheme 118.

Scheme 122. Reaction of 2-ketocyclopropyl acetylenes (e.g. **1159**) with gold complexes led to the ring expansion/cyclization products (e.g. **1162**) [1107]. One of the proposed mechanisms involves formation of a gold carbene complex (**1161**) through heterocyclization and ring opening followed by ring closure through aromatic substitution. The conversion of alkynylcyclopropylketones (e.g. **1163**) to furans (e.g. **1165**) was studied by DFT calculations [1108]. Gold carbene complexes (e.g. **1164**) however were not part of the favored reaction pathway.

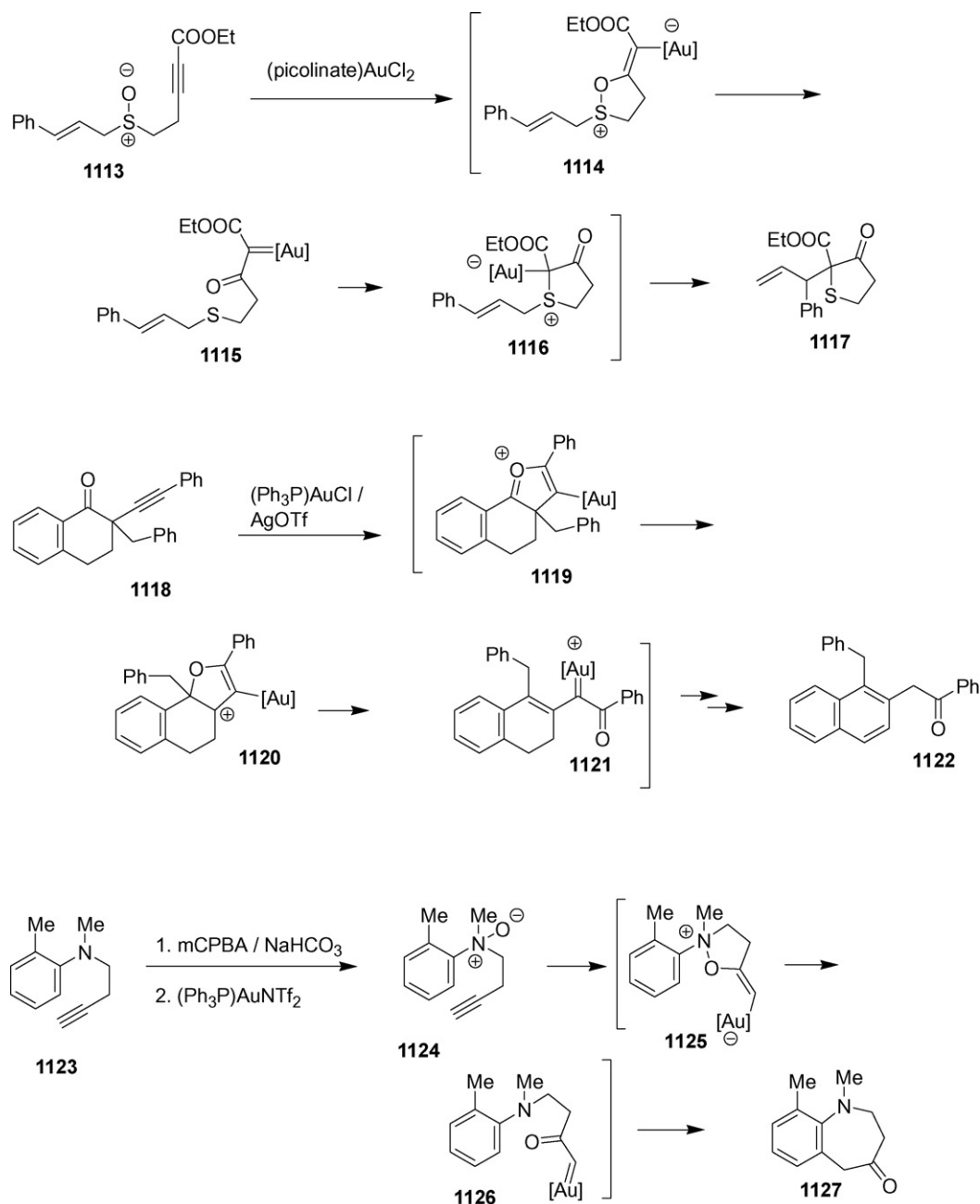
Several examples involve the generation of gold or platinum carbene complexes through reactions with allenes. Representative examples are depicted in Scheme 123. An intramolecular [4+3]-cycloaddition process was observed in the reaction of diene–allenes (e.g. **1167**) with gold complexes [1109,1110]. The proposed mechanism involves generation of an aurylated allyl cation (**1168**), followed by concerted cycloaddition and conversion of the resulting gold carbene complex (**1169**) to an alkene. In some cases, the intermediate carbene complex undergoes rearrangement to afford the net Diels–Alder product. The proportion of six- and seven-membered ring products was highly dependent on the catalyst. [1111]. A transannular version of this cycloaddition (conversion of **1171** to **1173**) was also demonstrated [1112]. The mechanism of the [4+3]-cycloaddition process was studied computationally [1113]. A mechanistically-related platinum-catalyzed intermolecular [3+2]-cycloaddition reaction (e.g. conversion of allene **1174** and enol ether **1175** to cyclopentenone **1177** via platinum carbene complex **1176**) was also reported [1114]. Platinum carbene complexes (e.g. **1180**) were suggested as intermediates in the cyclization of indole–allene derivatives (e.g. **1178**) [1115]. Platinum carbene complexes were also suggested as intermediates in platinum-catalyzed cyclizations

of allenyl ketones [1116]. In several cases platinum or gold complexes initiate Nazarov-like processes of vinylallene derivatives. Representative examples are depicted in Scheme 124. Nazarov-like processes were initiated through reaction of vinylallenes (e.g. **1182**) with gold complexes [1117]. The formation of tricyclic compound **1185** involves generation of an aurylated pentadienyl cation (**1183**), which undergoes electrocyclic ring closure to provide a carbene complex (**1184**). Subsequent cyclopropanation then affords the observed product. In situ generation of similar allenic systems (e.g. **1187**) through rearrangement of propargyl acetates (e.g. **1186**) and subsequent cyclization was also reported.

2.3.9. Group 11 carbene complexes

Relatively stable copper carbene complexes (e.g. **1192**, Scheme 125) were generated from the reaction of copper complex **1190** with α -diazo esters (e.g. **1191**) [1118]. The reaction afforded carbene complexes (**1192**) at very high steady state concentrations which could be observed by NMR spectroscopy. The barriers to rotation were determined by variable temperature NMR. One of the complexes was stable enough for isolation. This complex reacted with styrene to afford the cyclopropanation product **1194** and the alkene complex **1193**. A gold carbene complex (**1196**) was generated through gold-induced ring opening of cyclopropanone ketal derivative **1194** [1119]. The obtained resonance-stabilized carbene complex was actually more consistent with the vinylcarbene resonance form (**1197**). Significant carbene character (structure **1199**) was attributed to copper–iron complex **1198** [1120].

Processes that invoke Group 11 metal–carbene complexes as intermediates are depicted in Scheme 126. Copper carbene complexes (e.g. **1204**) were proposed as intermediates in the oxida-



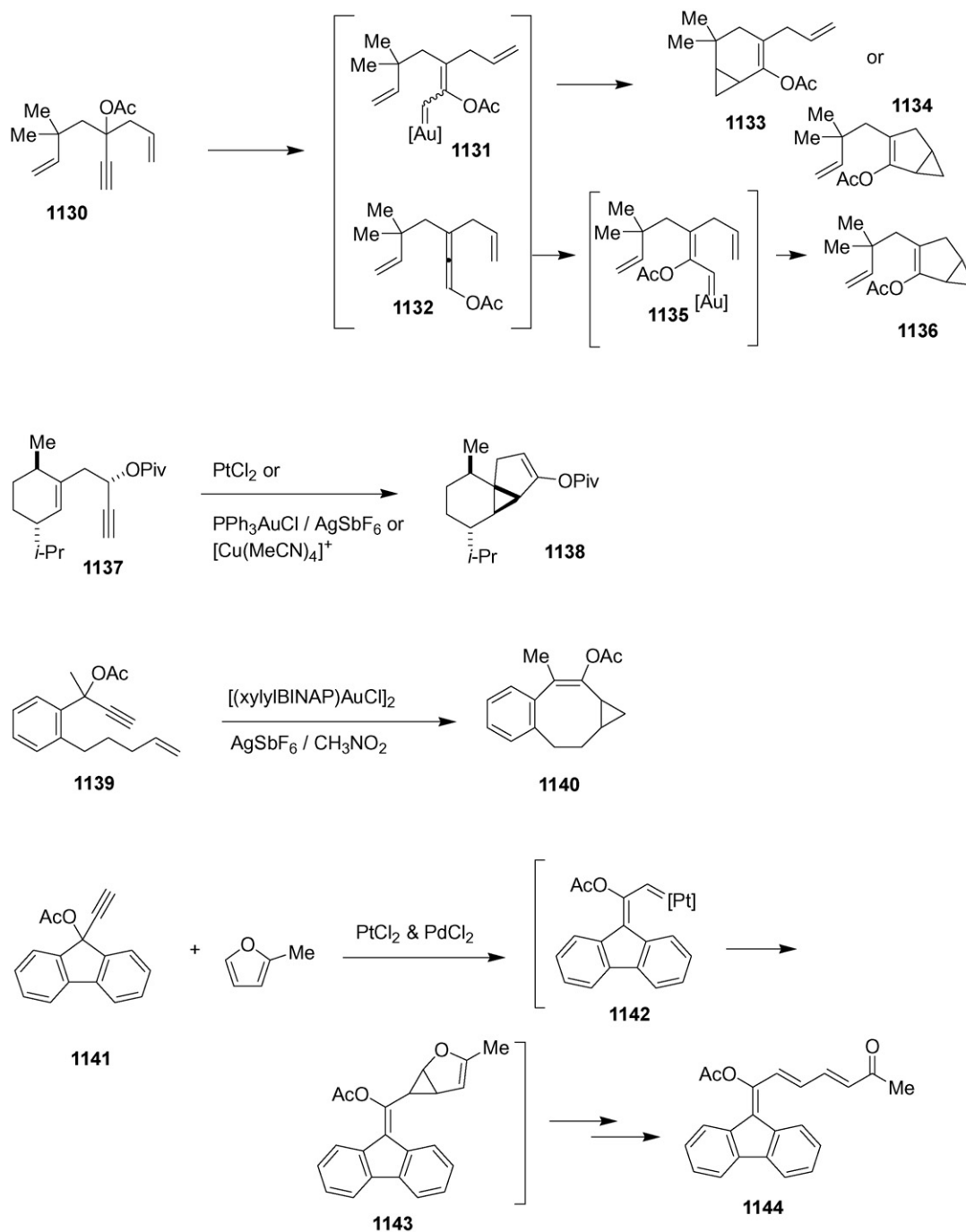
Scheme 119.

tive cycloaddition of propargyl alcohols and electron-deficient acetylenes (e.g. DMAD, **1200**) [1121]. Copper carbene complexes (e.g. **1209**) were suggested as intermediates in the copper-catalyzed coupling of vinyl diazo compounds (e.g. **1207**) with dialkynylmethyl esters (e.g. **1206**) [1122]. A complex mechanism involving copper catalyzed rearrangement to a furylcarbene complex (**1209**), followed by cyclopropanation of the alkene group of the diazo compound to afford diazo compound **1210**, followed by formation of a carbene and C–C insertion/ring expansion was proposed. Enantioselective propargyl substitution of racemic ethynylepoxides (e.g. **1212**) using copper catalysts and chiral ligands likely involves copper allenylidene complexes (e.g. **1213**) [1123].

The gas-phase generation and reactivity of gold carbene complexes was reported (see Scheme 127) [1124]. Benzylidene-gold complexes (e.g. **1220** and **1221**) were generated through gas-phase electrospray ionization of gold-phosphonium salt **1215** and were

subsequently converted to the cis-1,2-dimethoxyethylene adduct (**1217**) through ion-molecule reactions in the rf ion guide. The adducts then undergo either olefin dissociation, metathesis, or cyclopropanation reactions. Linear free energy relationships were established for each of these processes. Cyclopropanation was enhanced by electron-deficient X groups whereas electron-rich groups strongly favor dissociation and weakly favor metathesis processes.

Several papers report on mechanistic aspects of copper carbene complex intermediates generated from organic diazo compounds. Catalytic cyclopropanation (diazo compounds plus alkenes) employing copper(I) diphosphinoamine complexes was reported; a mechanism involving an alkene-copper complex as the catalytically active species was proposed [1125]. The silver-catalyzed reaction of ethyl diazoacetate and halogenated compounds was studied experimentally and computationally



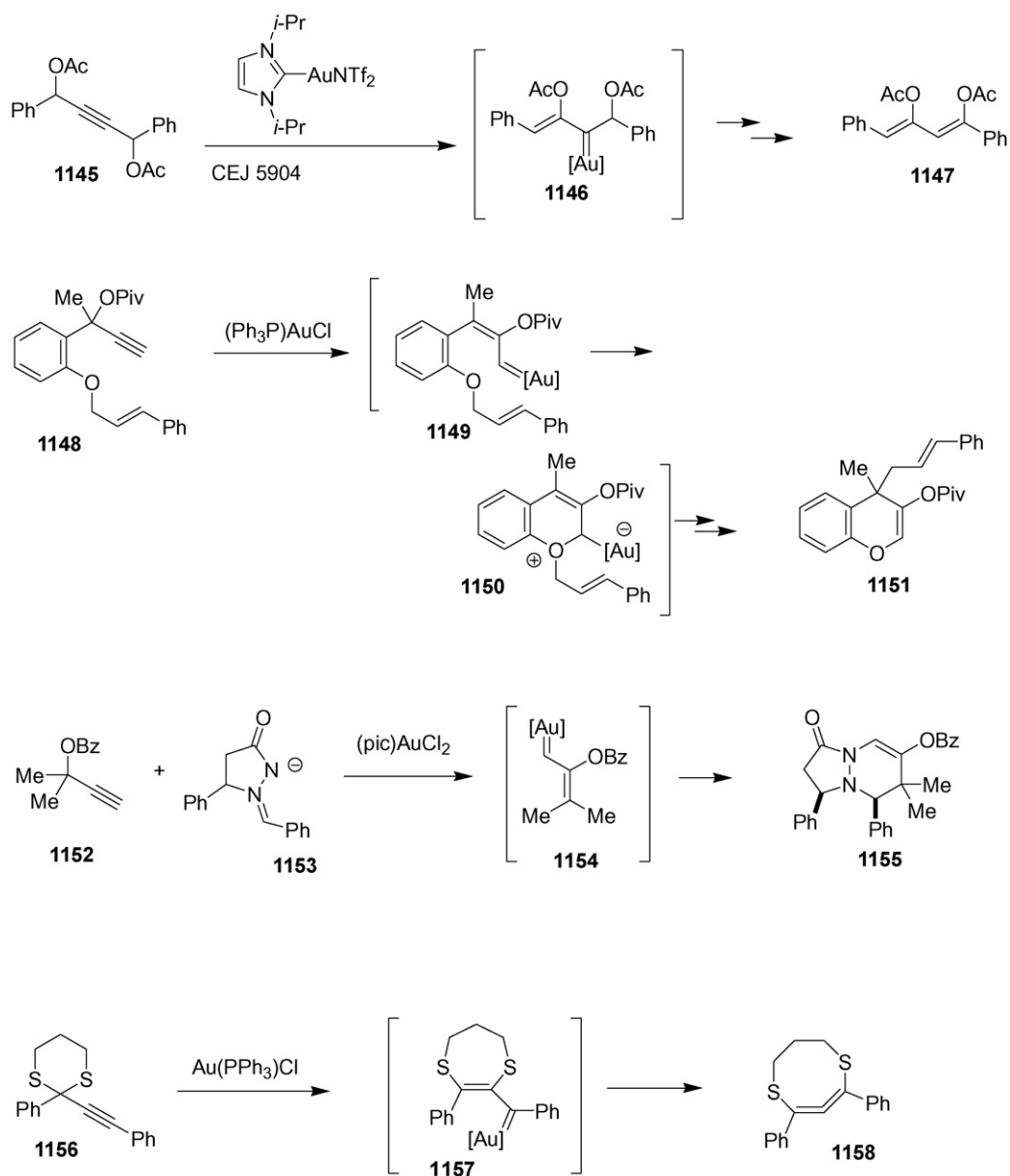
Scheme 120.

[1126]. Use of bulk gold powder to catalyze the decomposition of diazo compounds and subsequent carbene-like reactions was demonstrated [1127]. The reaction affords C–X insertion products from dihalides and carbene halogenation products using mono-halides. A novel mechanism where the metal is bound to all of the reactive intermediates after metal–carbene–halide contact was proposed and evaluated by DFT calculation. A DFT study of copper catalyzed diazomalonate–alkene cycloadditions was reported (see Scheme 128) [1128]. An emphasis of these studies is the cyclopropanation (e.g. formation of **1228**) vs. the [3 + 2]–cycloaddition (e.g. formation of **1227**) pathway. The carbene complex was at a relatively low energy point during the reaction process. Conversion of a diazo compound into a gold–carbene complex was determined to

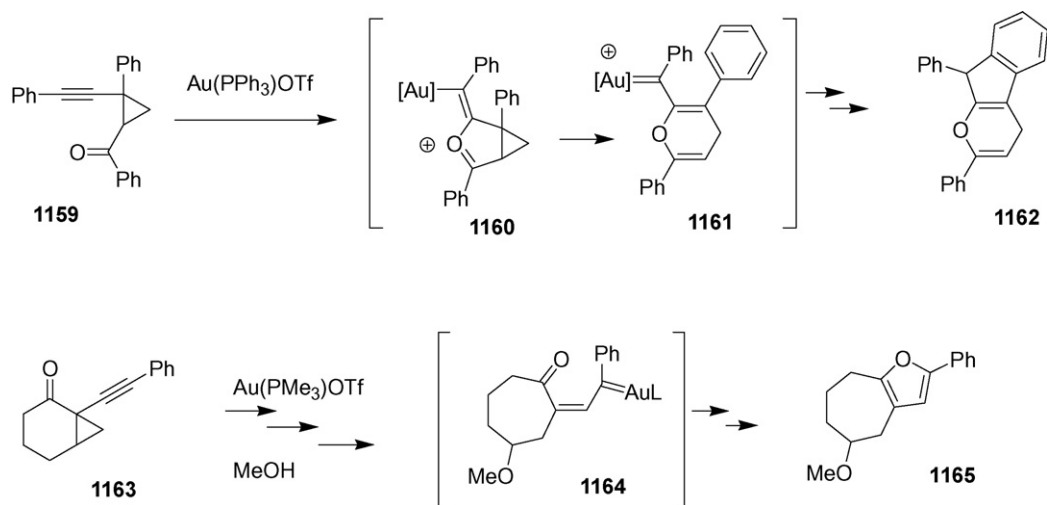
be the rate determining step for gold-catalyzed cyclopropanation reactions, which was also a key factor in the superiority of NHC ligands for this process [1129].

2.3.10. Lanthanide/actinide carbene complexes

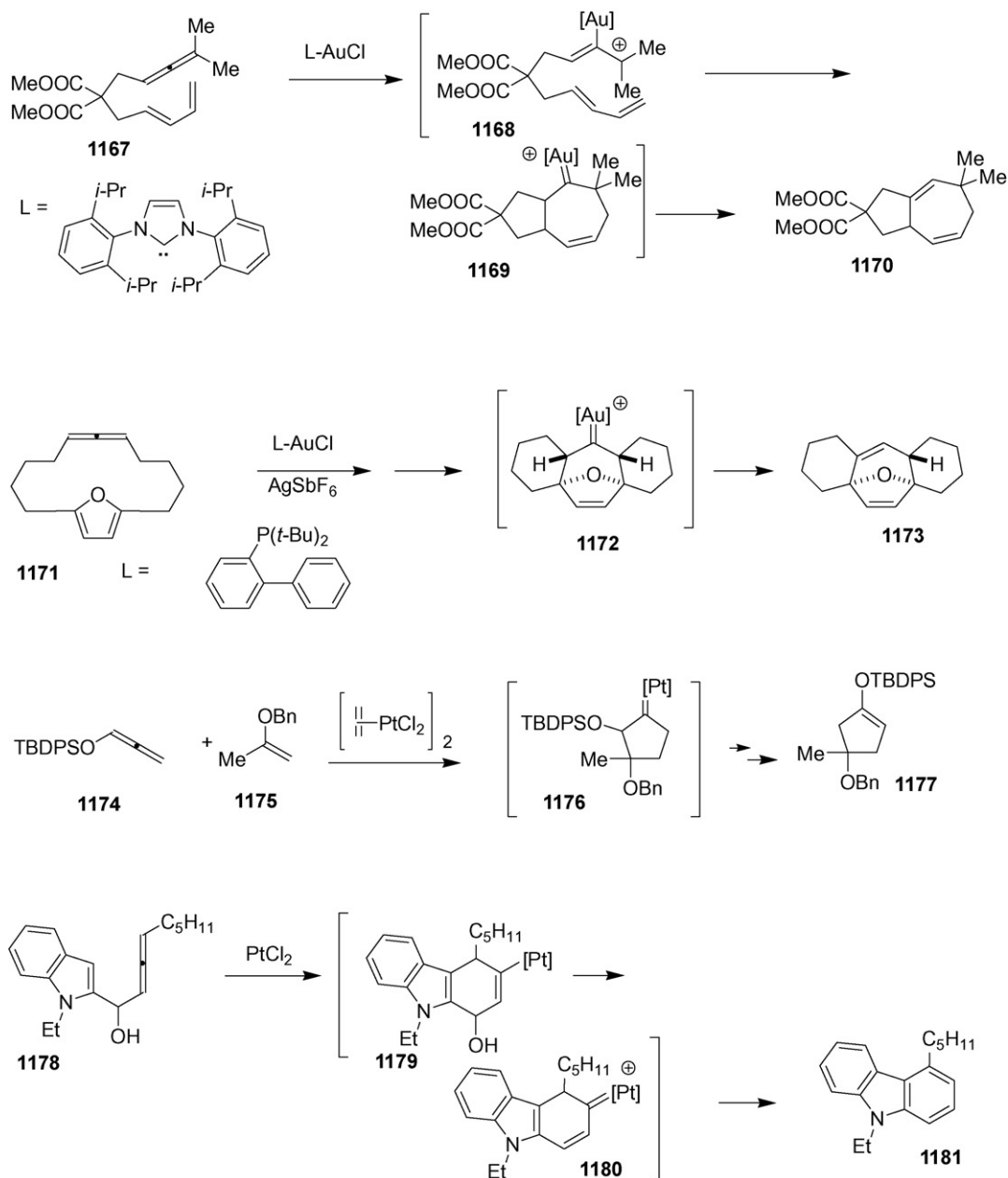
The formation of pincer carbene complexes of ytterbium (e.g. **1232**, Scheme 129) was reported [1130]. In this case, the carbene ligand is inert to reactions with electrophiles. The complex acts as a benzyl anion equivalent, and affords benzylation products (e.g. **1233**) upon treatment with the electrophile benzophenone. The benzophenone adduct **1233** disproportionates to the bridging carbene complex **1234** upon thermolysis. The benzylation product **1235** was obtained upon treatment of complex **1232** with azoben-



Scheme 121.



Scheme 122.



Scheme 123.

zene. Related yttrium and erbium carbene complexes (e.g. **1238**) were similarly prepared [1131].

A uranium tris(carbene) complex (e.g. **1240**, Scheme 130) was prepared from the reaction of dianion **1239** with uranium(IV) borohydride [1132]. A monocarbene complex (**1242**) could be generated through reaction in toluene followed by treatment with THF. The complexes react with ketones to form carbonyl olefination products (e.g. **1241**). The U–C double bond arises from σ - and π -donation from the ligand to the metal according to DFT calculations.

The degree of backbonding in uranium–CO complexes of general structure $(Cp-R)_3U-CO$ was evaluated computationally and spectroscopically [1133]. The backbonding was attributed to interaction of the π^* MO of the CO ligand with bonding orbital at U of mixed d and f character. The degree of U–C double bond character however could not be determined since calculated bond dissociation energies did not correlate with the degree of backbonding.

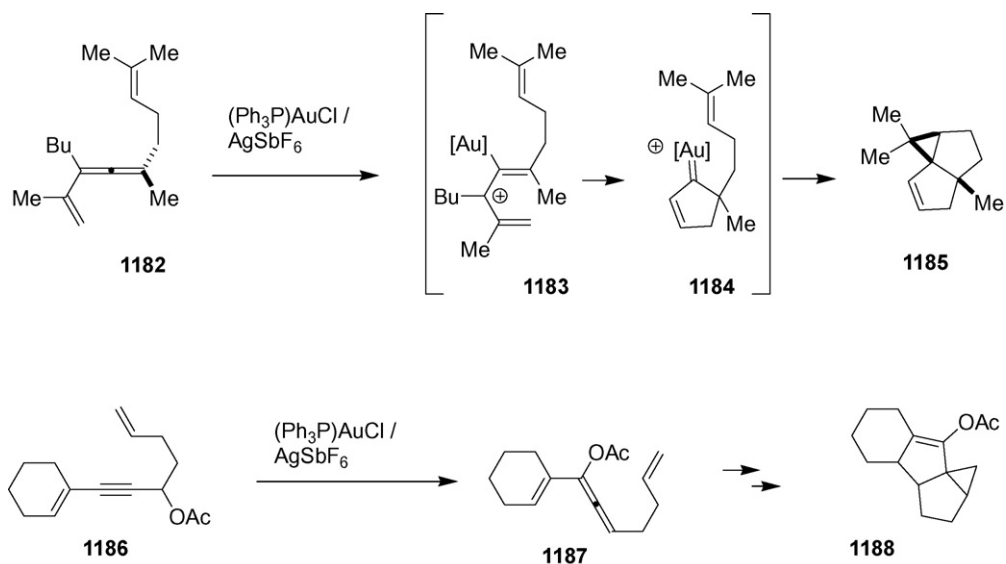
3. Metal–carbyne or metal–alkylidyne complexes

3.1. Synthesis and/or generation

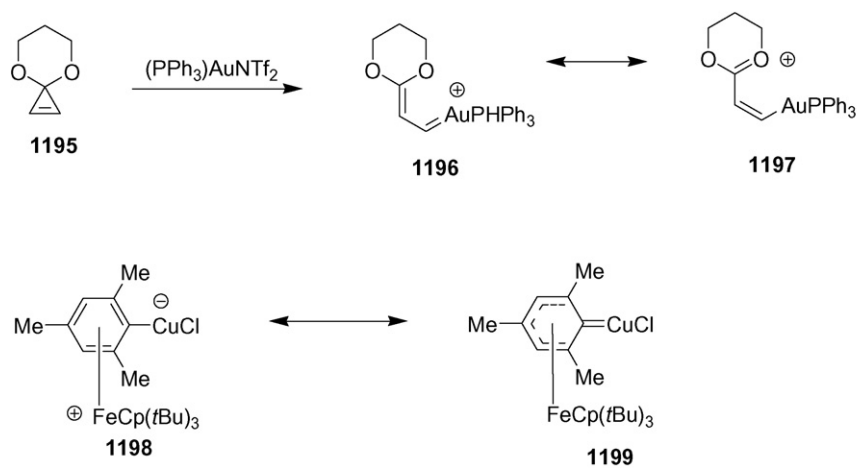
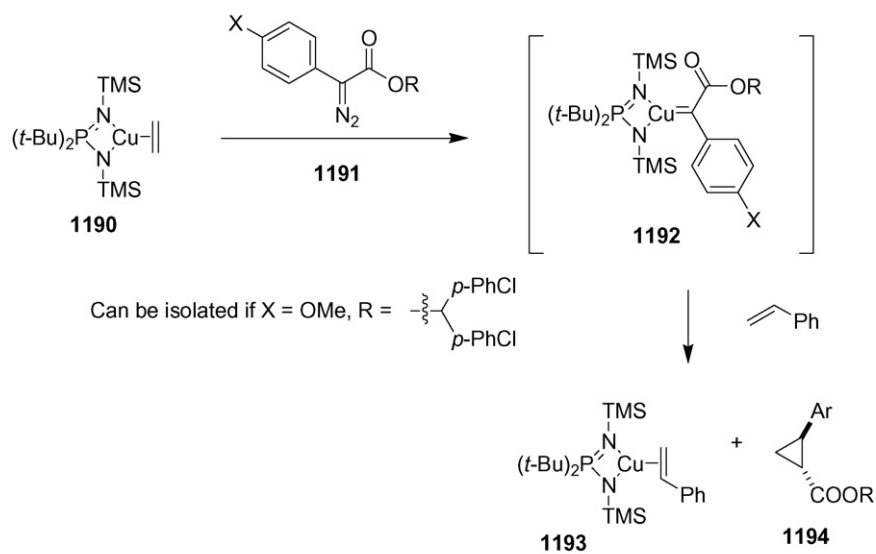
Some papers in the carbene section feature minor segments on carbyne chemistry. These studies include Refs. [842,985,992,996].

The metathesis of molybdenum and tungsten bimetallics featuring metal–metal multiple bonds (e.g. **1245**, Scheme 131) with nitriles was studied computationally [1134]. The tungsten reaction affords a nitride complex (**1247**) and a carbyne complex (**1248**). The key intermediate in this process is the ditungsta-azametallacyclobutadiene complex **1246**. The analogous dimolybdenum reaction was not thermodynamically favorable, however a similar pathway was observed using Me_2NCN in place of acetonitrile.

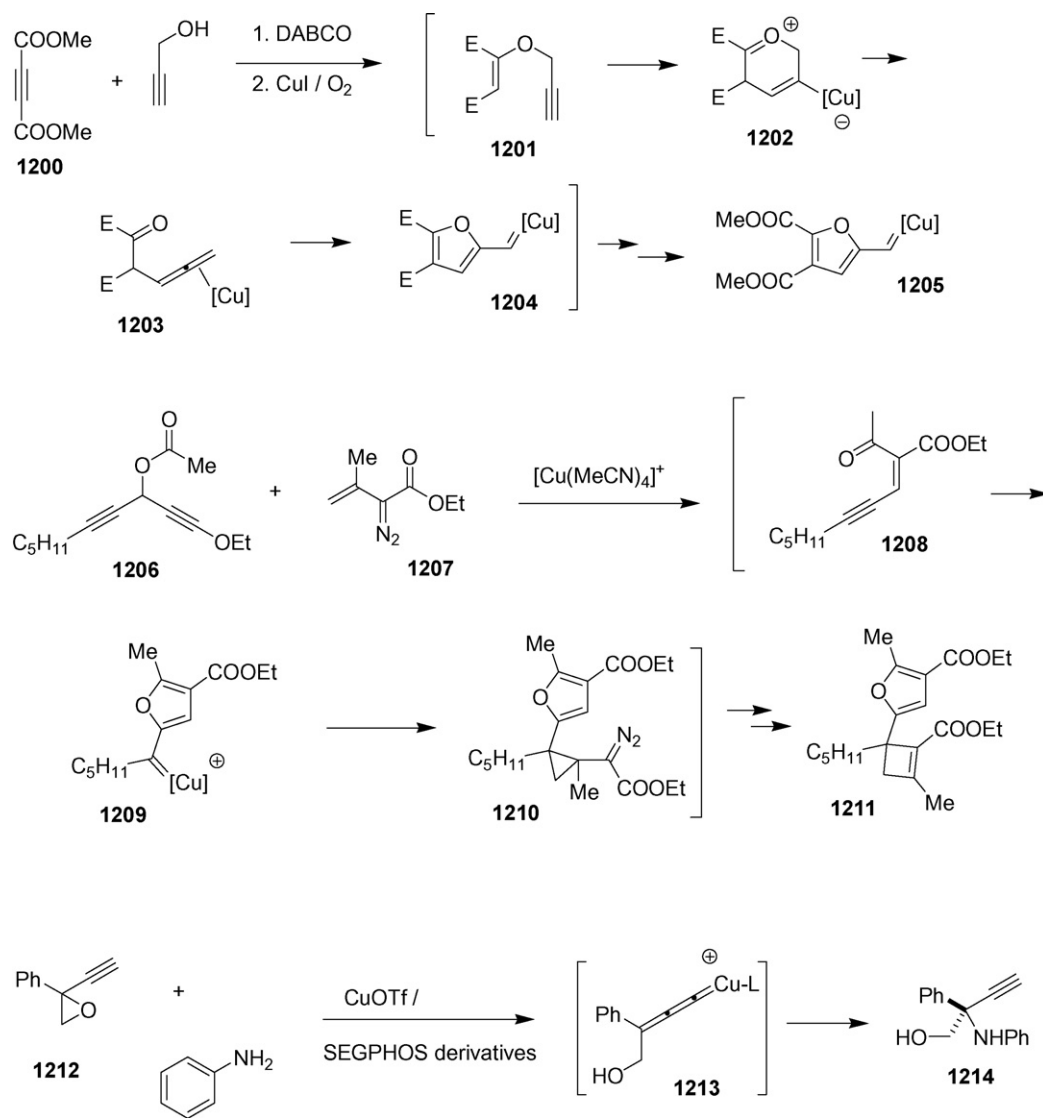
Osmium–alkenylcarbyne complexes (e.g. **1250**, Scheme 132) were prepared through reaction of osmium complex **665** with



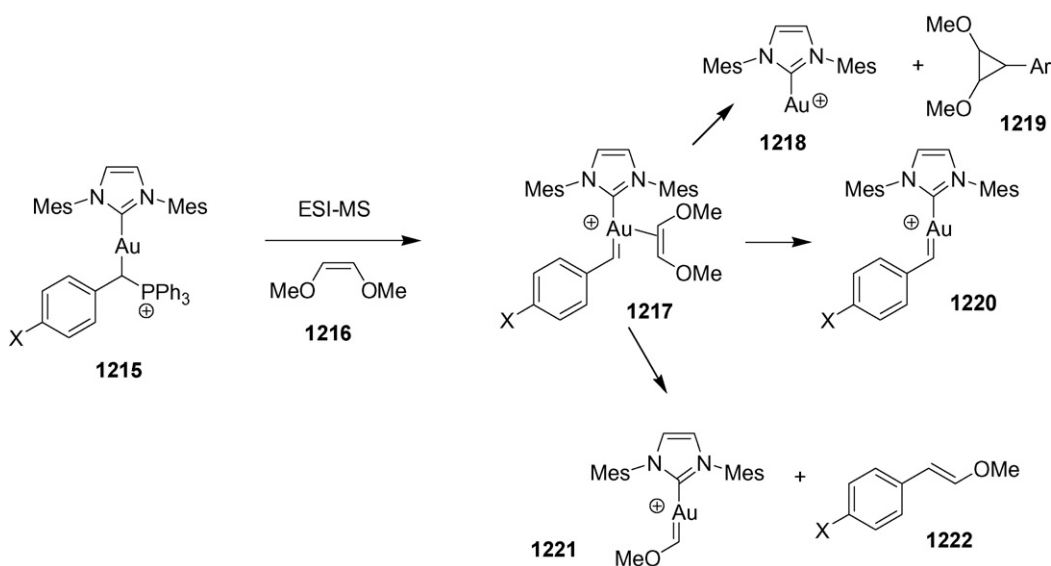
Scheme 124.



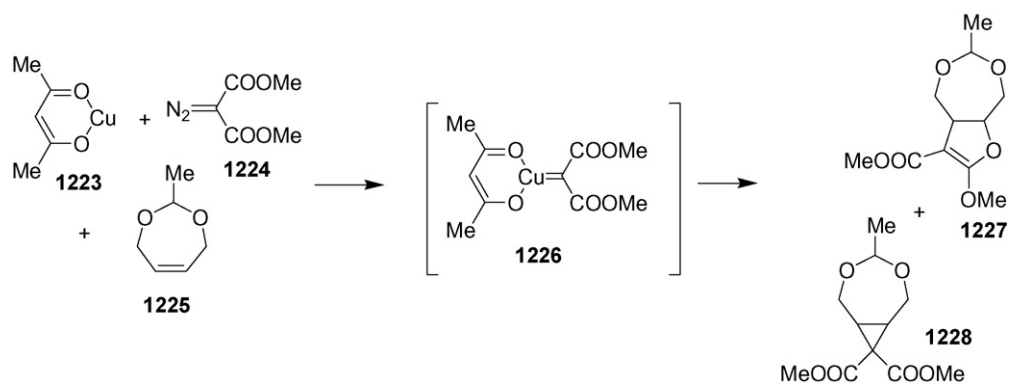
Scheme 125.



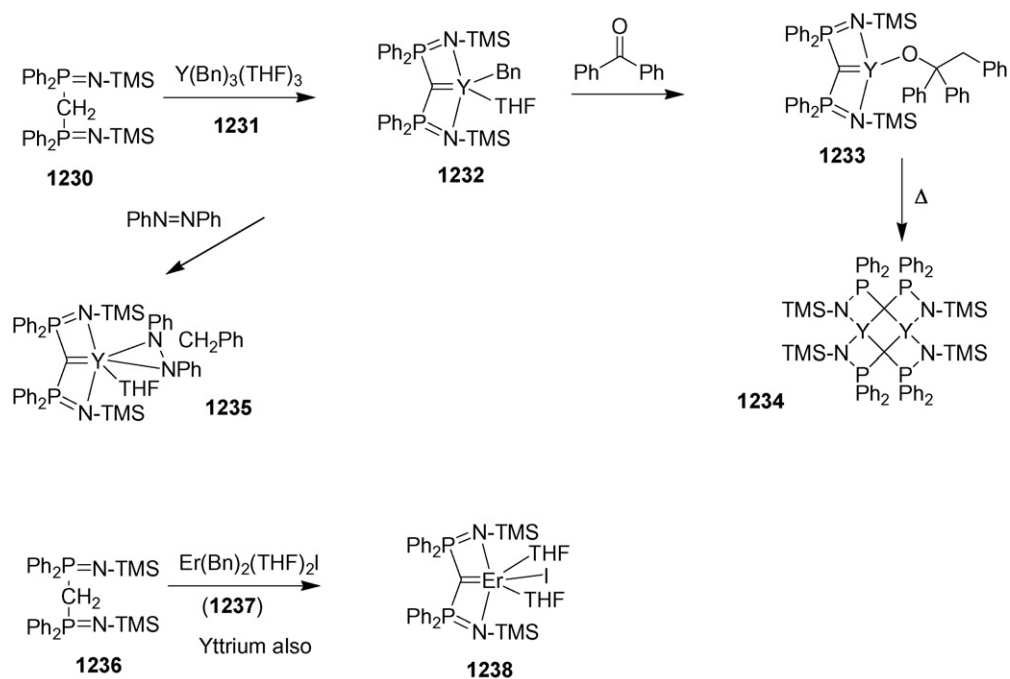
Scheme 126.



Scheme 127.



Scheme 128.

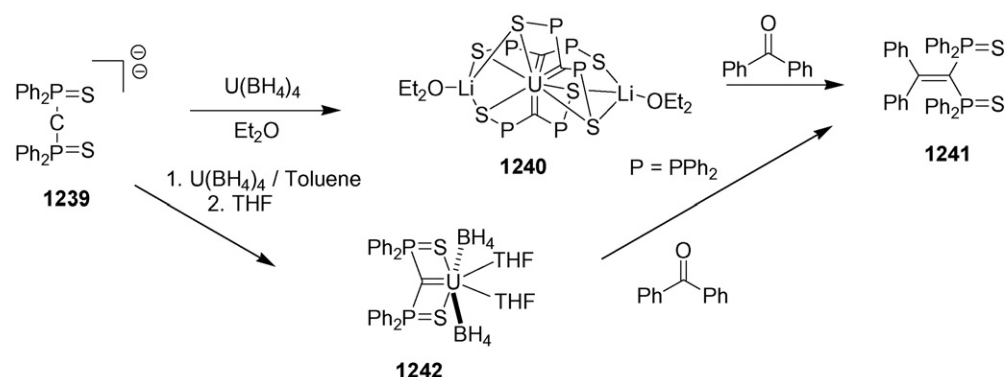


Scheme 129.

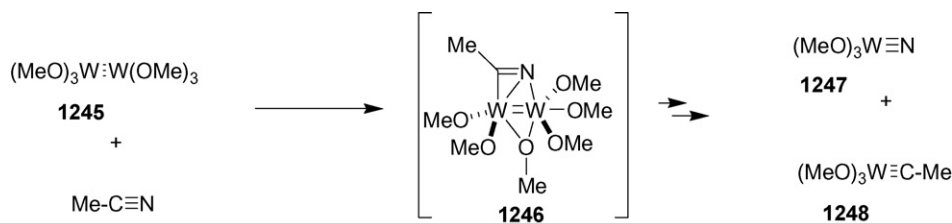
propargyl alcohols (e.g. **843**) followed by acid [1135]. Reaction of the carbyne complex with acetonitrile and triphenylphosphine led to the osmapyridine derivative **1251**. A mechanism involving hydrogenation migration, nitrile complexation, followed by rearrangement, followed by deprotonation was proposed. Thermolysis

of carbyne complex **1250** led to the bis(osmanaphthalene) derivative **1253**, which transforms to the osmanaphthalene **1254** upon treatment with acidic triphenylphosphine [1136].

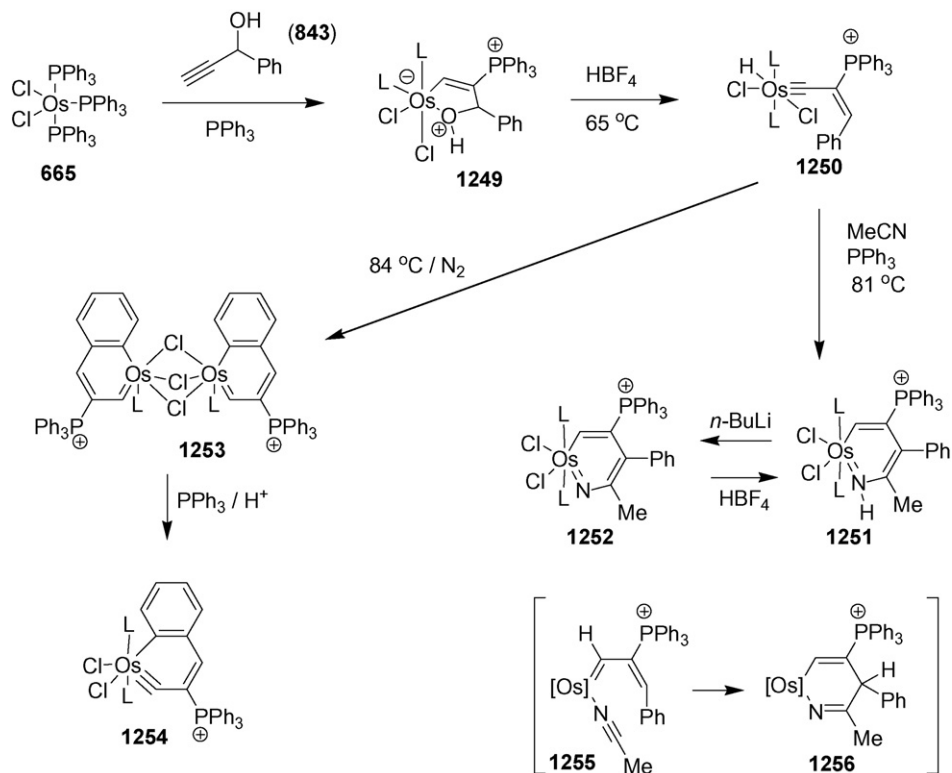
An osmium carbyne complex (**1258**, Scheme 133) was generated through the reaction of osmium bis(alkylidene) complex **1257** with



Scheme 130.



Scheme 131.



Scheme 132.

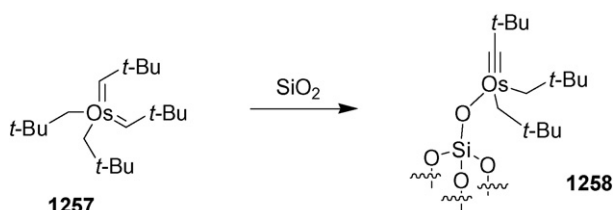
partially dehydroxylated silica [1137]. DFT calculations show that the bis(alkylidene) is the energetically favored structure when the auxiliary ligands are alkyls, however the carbyne complex is the more stable structure when one of the alkyl ligands is replaced by a silyloxy ligand. DFT calculations also suggest that the ligand replacement is via σ -bond metathesis and not through addition of the OH group to the alkylidene group.

Osmium carbyne complexes (e.g. **1262** and **1263**, Scheme 134) were produced in the reaction of propargylic alcohols with osmium phosphine complex **1260** [1138]. The initially-formed hydroxyvinylidene complex **1261** affords the alkenylcarbyne complex **1262** instead of the allenylidene complex upon treatment with

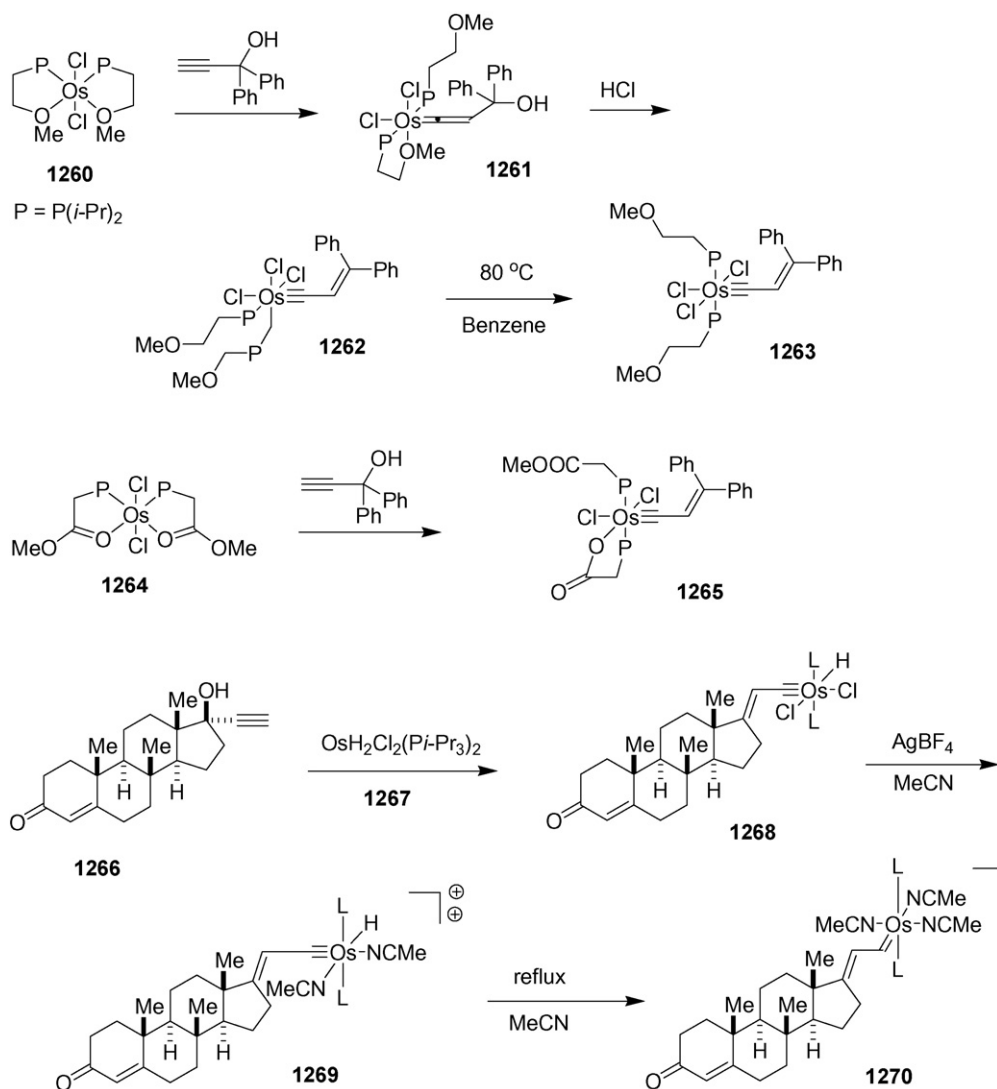
HCl. Further heating leads to isomerization of the cis diphosphine complex **1262** to the trans diphosphine complex **1263**. The acyl oxygen coordinated osmium complex **1264** and amino analogs lead directly to the carbyne complexes (e.g. **1265**). In the case of ester-coordinated compound **1264**, the methyl group was lost to afford the chelated carboxylate compound **1265**. Alkenylcarbyne–osmium complexes (e.g. **1268**) were generated directly from stereoidal propargyl alcohol **1266** [1139]. Additional ligand exchange studies were reported for this complex. Conversion to an alkenylcarbene complex (**1270**) involves ionization in acetonitrile followed by refluxing in acetonitrile.

The generation of osmium carbyne complexes from the reaction of laser ablation-generated osmium atoms and halomethane derivatives was reported [1140]. Compounds of general structure CH_2X_2 , CHX_3 , and CX_4 ($\text{X} = \text{Cl}$, F , or combinations) and their deuterium analogs were employed. The carbyne complexes were trapped in an argon matrix and were characterized through comparison of the experimental and DFT-calculated IR spectra. DFT studies were also reported for iron and ruthenium complex analogs.

The bond energies for methyl, methylene, and methane groups to a nickel surface were evaluated computationally [1141]. All species prefer to bind in the μ -3 mode and not as mono- or dinuclear complexes.



Scheme 133.



Scheme 134.

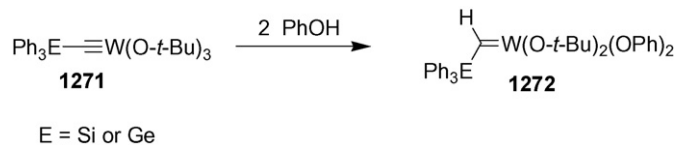
3.2. Reactivity

3.2.1. Addition reactions of metal–carbyne complexes

The reaction of tungsten carbyne complex **1271** with two equivalents of phenol led to the corresponding carbene complexes (**1272**) [1142]. The carbene complexes catalyze the ROMP of cyclooctene if they are activated with AlBr₃ (Scheme 135).

3.2.2. Alkyne metathesis

Alkyne metathesis, which involves metal carbyne complexes as intermediates, has been covered comprehensively regardless of whether the initiator is a carbyne complex. General equations describing the mechanism and preceded modes are presented in Scheme 136.



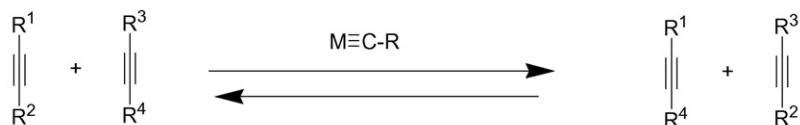
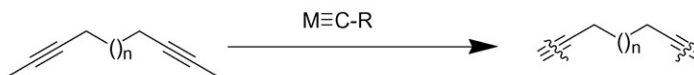
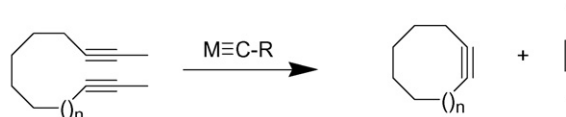
Scheme 135.

Several tungsten alkyne metathesis catalysts (e.g. **1277**, Scheme 137) were prepared [1143]. The catalyst **1277** was found to be the best of this series, and can catalyze the cross metathesis of 1-phenylpropyne at 30 °C. Development of a new user friendly catalyst for alkyne metathesis, nitrido complex **1278**, was reported [1144]. Examples of ring closing alkyne metathesis are depicted in Scheme 138. Conversion of diyne **1279** to cyclic alkyne **1281** using molybdenum catalyst **1280** was a key event in the total synthesis of amphidinolide V [1145]. Conversion of diyne **1282** to cyclic alkyne **1283** using catalyst **1280** was a key step in the total synthesis of cruentarin A [1146]. Compound **1283** was subsequently subjected to enyne metathesis. Patents were awarded for the development of tungsten carbyne complex olefin metathesis catalysts [1147,1148].

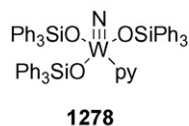
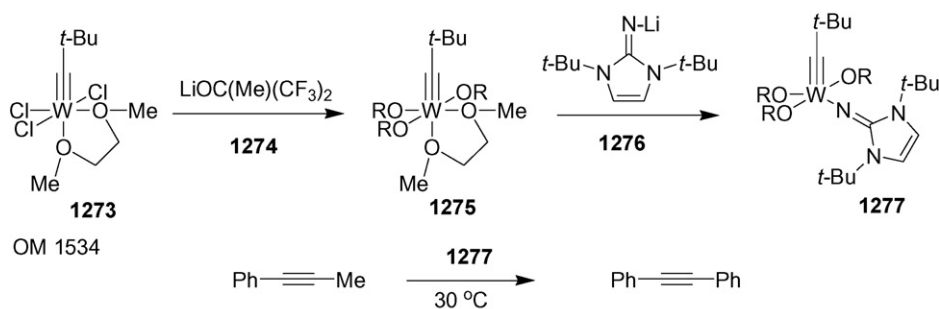
3.2.3. Other processes involving metal–carbyne complexes

The reaction of strong bases with vanadium carbyne complexes (e.g. **1285**, Scheme 139) and isoelectronic titanium alkylidene complexes (e.g. **1289**) was reported [1149]. The reaction of carbyne complex **1285** with benzylpotassium in ether solvent led to the deprotonation product (**1286**), which reacted with triarylboranes to afford the zwitterionic complexes (e.g. **1287**). Protonation of complex **1286** with [HNMMe₂Ph]⁺ [BAr'₄][−] led to the cationic carbyne complex (**1288**). The titanium alkylidene complex **1289** reacted similarly with benzylpotassium followed by triarylboranes.

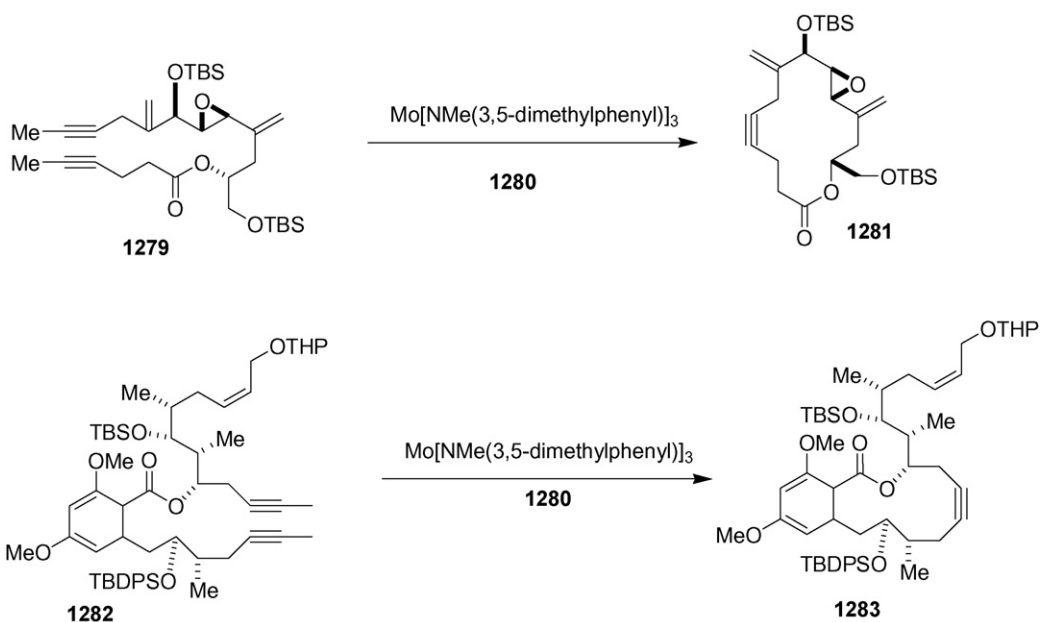
Alkyne Cross Metathesis

ADIMET Polymerization
(Acyclic Diyne Metathesis
Polymerization)RCAM
(Ring Closing Alkyne Metathesis)

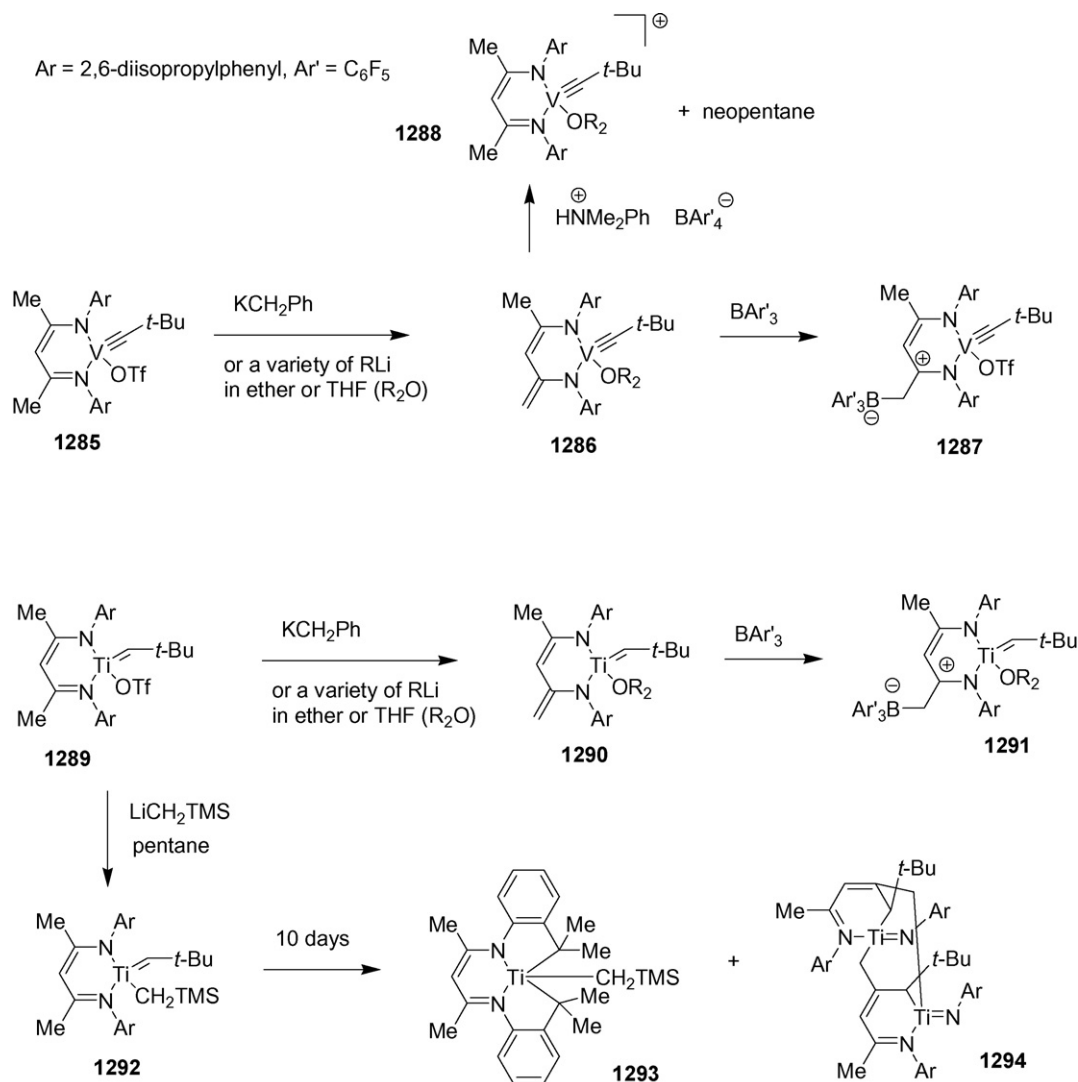
Scheme 136.



Scheme 137.



Scheme 138.



Scheme 139.

The substitution product (**1292**) was obtained from the reaction of **1289** with TMSCH₂Li in pentane solvent, however it decomposed to C–H activation product **1293** and the dimeric intramolecular metathesis/C–H activation product **1294** upon extended reaction time at room temperature.

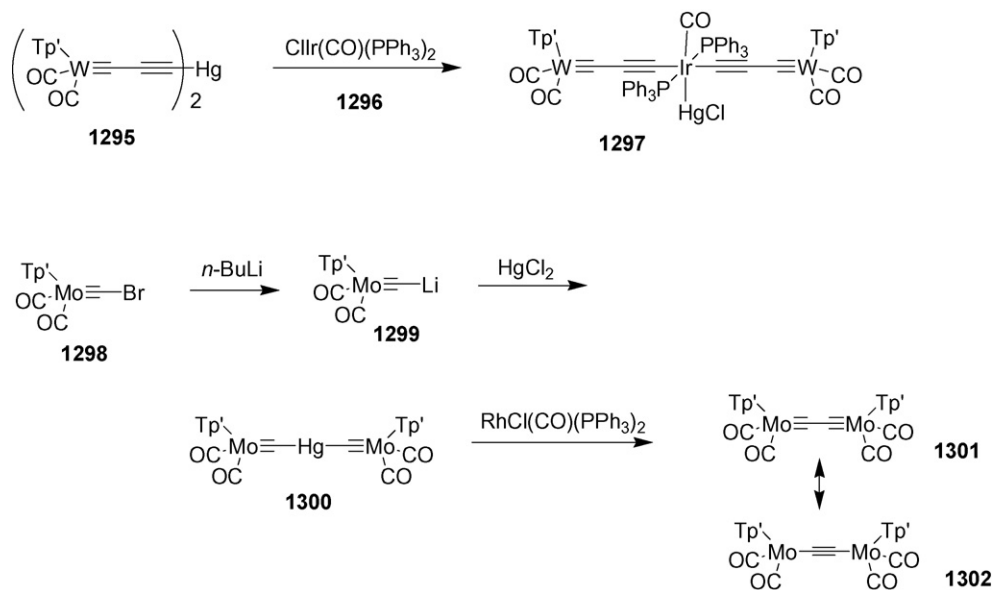
Several examples of bimetallic and trimetallic (bis)carbyne complexes were reported in 2009 (see Scheme 140). Transmetalation reactions were reported for tungsten carbyne complexes that contain alkynylmercury groups (e.g. **1295**) [1150]. The reaction of complex **1295** with Vaska's complex (**1296**) led to the bis(alkynyl)iridium bis(carbyne) complex **1297**. The mercury chemistry was also useful in the preparation of an ethanediylidene complex (**1301**) [1151]. The mercury complex **1300** was made through lithiation of bromcarbyne complex **1298**, followed by treatment with HgCl₂. Reaction of mercury complex **1300** with RhCl(CO)(PPh₃)₂ led to the bis(carbyne) complex **1301**. The X-ray structure revealed the bis(carbyne) resonance form to be a better representation compared with the bis(molybdenum)–alkyne complex form (**1302**) based on the Mo–C and C–C bond lengths. Attempted preparation of platinum-bridged analogs led to the reductive elimination products (C₆-bridged bimetallics) [1152].

The reaction of organolithium reagent **1299** (Scheme 141) with chlorophosphines led to the phosphinocarbyne complexes (e.g.

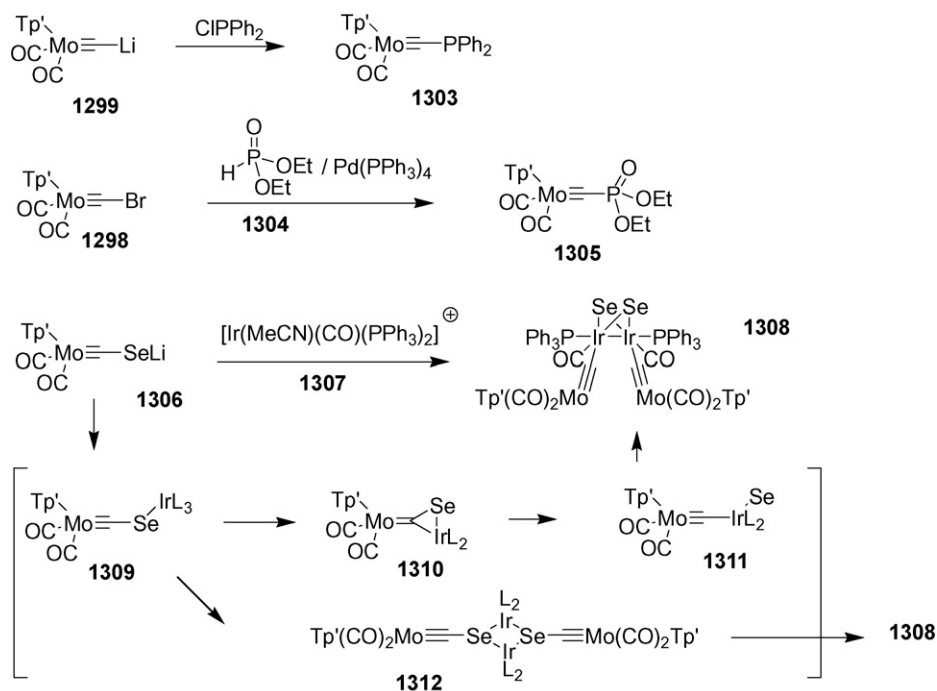
1303) [1153]. A phosphatocarbyne complex (**1305**) was prepared directly from bromocarbyne complex **1298**. The selenolato complex **1306** reacted with iridium complex **1307** to afford the dimeric complex **1308** [1154]. Two mechanistic pathways were proposed, both of which involve initial coordination of iridium to selenium, however they differ in the timing of the C–Se oxidative addition step.

Fluorocarbyne–manganese complexes (e.g. **1313**) were studied computationally [1155]. Complexes studied include those of general structure Mn(CF)(CO)_n (*n* = 3, 4) and Mn₂(CF)₂(CO)_n (*n* = 4–7). The CF ligand in mononuclear complexes was determined to have a π-acceptor strength comparable to the NO⁺ ligand. In most of the dinuclear complexes, the energy minimum structure is a bridging difluoroacetylene and not a bis(fluorocarbyne) complex. For example, the binuclear complex **1314** minimized as a bridging fluoroalkyne complex (**1315**). In one case (**1316**) the energy minimum structure was a bis(fluorocarbyne) complex (Scheme 142).

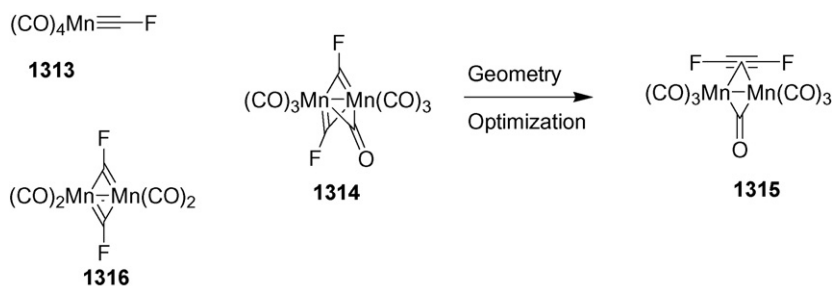
The reaction of TMS≡W(CH₂TMS)₃ (**1317**, Scheme 143) with bis(dimethylphosphino)ethane (dmpe) was examined [1156]. The reaction initially produces the mono-coordinated complex (**1318**) at low temperature, which converts to an equilibrium mixture of tautomeric compounds **1318** and **1319** at higher temperatures. When the equilibrium mixture was heated in toluene, conversion to the mixed carbene–carbyne complex **1320** was observed. Ther-



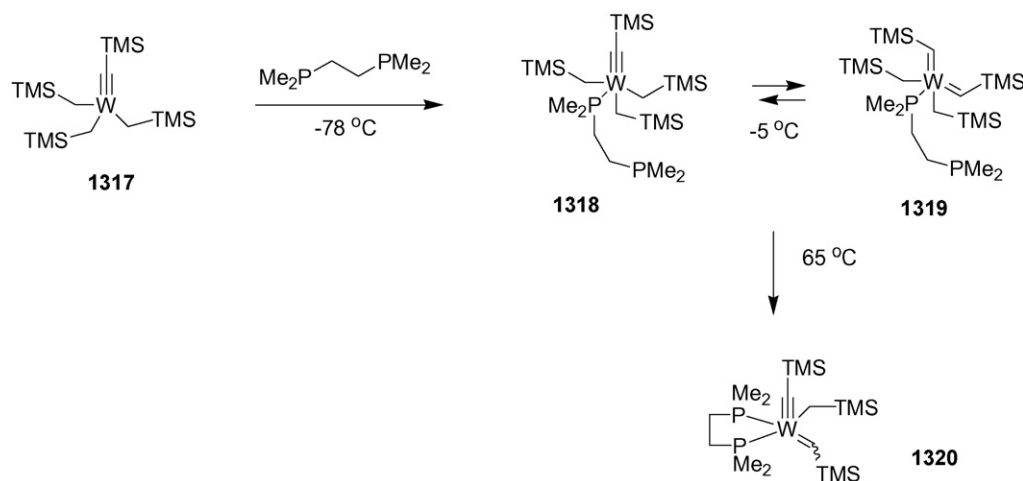
Scheme 140.



Scheme 141.



Scheme 142.



Scheme 143.

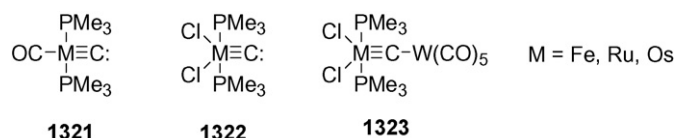


Fig. 14. Metal carbide complexes and their corresponding transition metal complexes.

modynamic and kinetic studies were conducted for the equilibrium process.

The use of metal carbide complexes (e.g. **1321** and **1322**, Fig. 14) (and silicon, germanium, and tin analogs) as ligands for transition metals was studied computationally [1157,1158]. Carbide complexes **1321** and **1322** were determined to be isolobal to carbon monoxide. The actual tungsten pentacarbonyl complexes (e.g. **1323**) exhibit computed spectral and structural properties similar to tungsten hexacarbonyl.

Acknowledgements

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