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Review

The chemistry of the carbon-transition metal double and triple bond: Annual survey covering the year 2008☆

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Contents

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ABSTRACT

This is a review of papers published in the year 2008 that focus on the synthesis, reactivity, or properties of compounds containing a carbon-transition metal double or triple bond.

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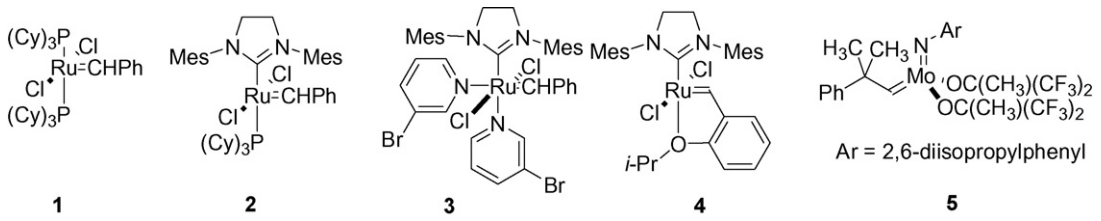


Fig. 1. Structures of alkene metathesis catalysts **1**–**5**.

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 are not covered, unless there is some effort to characterize the carbene complex intermediate. Several reviews in this area appeared in 2008 [1–6]. Although a determined effort has been made to include patents, in general only patents listed in the section "Organometallics and Organometallic Compounds" have been included. Patents that appear in 2008 editions of *Chemical Abstracts* 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 with transition metals have not been included; since the π-donation component of these complexes is usually minimal, there is no formal carbon–metal multiple bond [7,8]. This area was reviewed several times in 2008 [9–22]. 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 [23] 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 47, 14, 64, and 49 respectively, which covers the period of December 2007–December 2008 according to some search engines.

Abbreviations (see also the instructions for authors in the *Journal of Organic Chemistry* [24])

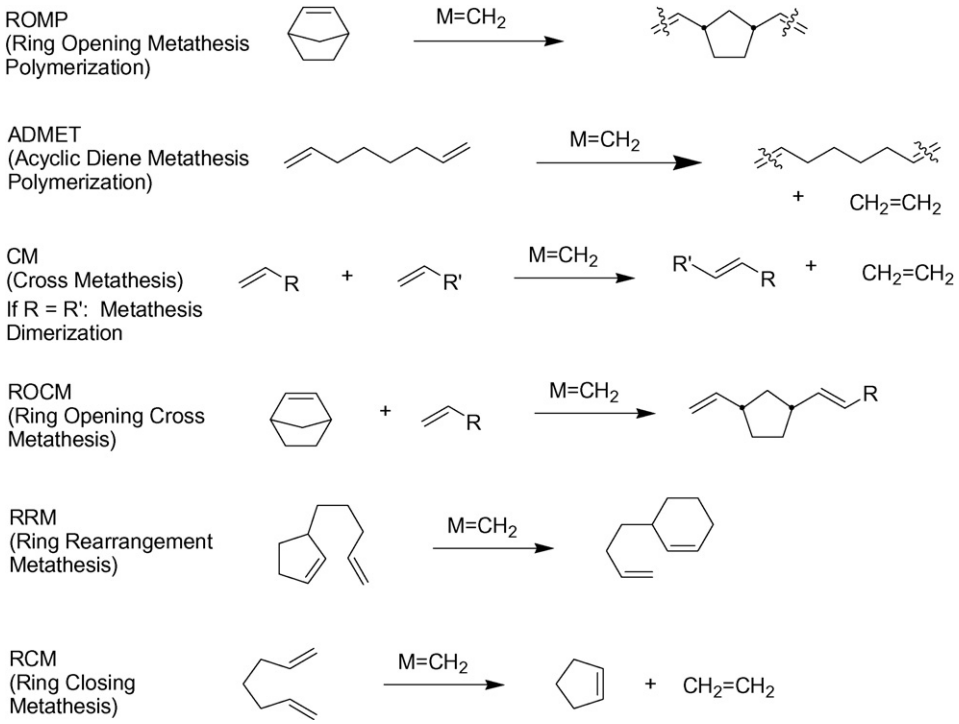
NHC	N-heterocyclic carbene
Grubbs catalyst I	Structure **1** (Fig. 1)
Grubbs catalyst II	Structure **2** (Fig. 1)
Grubbs catalyst III	Structure **3** (Fig. 1)
Hoveyda–Grubbs catalyst	Structure **4** (Fig. 1)
Schrock catalyst	Structure **5** (Fig. 1)
Tp′	Hydridotris(3,5-dimethylpyrazolyl)borate

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

2. Metal–carbene or metal–alkylidene complexes

2.1. Review articles, highlights, and comments

Several reviews/highlights/comments covering aspects of metal–carbene complex chemistry appeared in 2008. Many articles



Scheme 1.

focusing on some aspect of carbene complex-initiated olefin metathesis were published, including the following specific subjects: (1) alkene metathesis [25]; (2) comparisons of common ruthenium–carbene complex metathesis catalysts [26]; (3) highly efficient olefin metathesis catalysts [27]; (4) the quest for the ideal olefin metathesis catalyst [28]; (5) metathesis activity of fluoromethylene–ruthenium complexes [29]; (6) indenylidene complexes for olefin metathesis [30,31]; (7) ruthenium allenylidenes and indenylidenes as olefin metathesis catalysts [32];(8) immobilization of ruthenium alkylidene complex metathesis catalysts [33,34]; (9) heterogeneous olefin metathesis catalysts [35]; (10) controlling catalytic activity using ruthenium metathesis catalysts—use of latent metathesis catalysts [36–38]; (11) olefin metathesis catalysts [39]; (12) olefin metathesis in organic synthesis [40]; (13) use of metathesis for the synthesis of macrocycles [41–43]; (14) use of ring-closing metathesis in the direct formation of aromatic heterocycles [44]; (15) small ring metatheses [45]; (16) bridged ferrocenes through enantioselective metathesis [46]; (17) stereochemistry in ROMP of bicyclic and polycyclic alkenes [47]; (18) ROMP polymers containing main-group metals [48]; (19) synthesis of metal-containing polymers by metathesis [49]; (20) formation of helical ROMP polymers from bis(norbornene) derivatives [50]; (21) polyethylene derivatives through ADMET polymerization followed by hydrogenation [51]; (22) microwave assisted olefin metathesis [52]; (23) olefin metathesis in ionic liquids [53]; (24) fluorous phase olefin metathesis [54]; (25) olefin metathesis in chemical biology [55]; (26) olefin metathesis in protein modification [56]; (27) complex libraries of chiral molecules via cross-metathesis [57]; (28) olefin metathesis in the pharmaceutical industry [58]; (29) cross-metathesis of fatty acid derivatives [59]; (30) olefin metathesis in oleochemistry [60]; (31) olefin metathesis in sunflower-based feedstocks [61]; (32) tandem metathesis/hydrogenation reactions [62]; (33) tandem Bayliss–Hillman–RCM sequences [63]; (34) carbonyl group-olefin metathesis [64]; (35) electrochemically generated molybdenum and tungsten olefin metathesis catalysts [65]; and (36) computational modeling of Mo- and Ru-catalyzed metathesis reactions [66].

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 compounds or compound classes represented include: (1) taxol analogs [67]; (2) furan and pyrrole rings [68]; (3) guaianolides [69]; (4) C2–C11 cyclized cembranoids [70]; (5) lignans [71]; (6) cyclic amino acids [72]; (7) piperidin-3-oles [73]; (8) aeruginosins [74]; (9) platensimycin [75]; (10) manzamine alkaloids [76]; (11) brevitoxins [77]; (12) oxasqualenoids [78]; (13) pyranonaphthoquinones [79]; (14) spiroaminals [80]; (15) spirastrellolide A [81]; (16) papulacandin D [82]; (17) leucascandrolide and migrastatin [83]; (18) sarain A [84]; (19) apogalanthamine analogs [85]; (20) aminocyclitols [86]; (21) constrained nucleosides [87,88]; (22) cycloheptyne cobalt complexes [89]; (23) peptides and peptidomimetics [90–92]; (24) macrocycle-bridged peptides [93]; (25) peptide–polymer conjugates [94]; (26) glucose polymers [95]; (27) polymeric bio-conjugates [96]; (28) topological molecules (*i.e.* molecular knots, catenanes, rotaxanes, etc) [97]; and (29) cyclic macromolecules [98].

Additional review articles include some segments on carbene complex-initiated metathesis. Articles in this category focus on the following subjects: (1) ruthenium complexes featuring bidentate Schiff base ligands and use of these complexes to generate immobilized catalysts [99]; (2) desilylative coupling of vinylsilanes [100]; (3) ionic liquids in organic synthesis promoted by microwave irradiation [101]; (4) microwave heating in synthetic organic chemistry [102]; (5) design of polymer-supported catalysts [103]; (6) the chemistry of metathesis-derived stationary phases for chromatography [104]; (7) chemistry strategies in early drug discovery [105]; (8) recent development in the polymerization of dienes [106]; (9) transition metals in organic synthesis [107]; (10) the chemistry of allylic and propargylic fluorides [108]; (11) solid phase C–C coupling reactions [109]; (12) catalytic processes for the technical use of natural fats and oils [110]; (13) the chemistry of porphyrazines [111]; (14) polymer synthesis in ionic liquids [112]; (15) Fischer–Tropsch technology selection [113]; and (15) density functionals with broad applicability in chemistry [114].

Several reviews on carbene complex chemistry featuring some aspect other that metathesis appeared in 2008. These reviews include the following subjects: (1) synthesis of phenols and quinones via Fischer carbene complexes [115]; (2) titanium alkylidene complexes [116]; (3) titanium–carbene complexes in organic synthesis [117]; (4) titanacycles in organic synthesis [118]; (5) Group 6 metal–vinylidene complexes in catalysis [119]; (6) cationic bridging carbyne-diiron complexes [120];(7) catalyst design for enantioselective cyclopropanation using cobalt(II) complexes [121]; (8) phosphorus-stabilized carbene complexes [122]; (9) 4-pyridinylidene transition metal complexes [123]; (10) DFT and physical organic studies in solving mechanistic problems in Fischer carbene complex reactions [124]; (11) metal vinylidenes in organic synthesis [125]; (12) metal–vinylidene complexes in catalysis [126]; (13) cascade reactions involving vinylidene and/or allenylidene complex intermediates [127];(14) ruthenium vinylidene complexes as catalysts for carbocyclization [128]; (15) Group 9–11 metal–vinylidene complexes in catalysis [129]; and (16) coordination of tetrakis(dialkylamino)allenes as carbene ligands for transition metals [130].

Although not specifically focusing on metal–carbene complexes, some review articles place some emphasis on this subject. Subjects reviewed in this category include: (1) transition metals in organic synthesis for 2005 [131]; (2) benzannulation [132]; (3) β-lactam synthesis via ketenes and ketenimines [133]; (4) [8+2]-cycloadditions [134]; (5) recent advance in the chemistry of silicon- and boron-substituted 1,3-dienes [135]; (6) intermediacy of carbene complexes vs. carbocations in gold-catalyzed reactions [136]; (7) metal-catalyzed enyne cycloisomerization [137–141]; (8) gold-catalyzed cyclizations involving alkynes or allenes [142]; (9) gold catalysis of organic reactions [143]; (10) stereoselective gold catalysis [144,145]; (11) isomerization of allylic and propargylic alcohols [146]; (12) gold-catalyzed reactions involving alcohols [147]; (13) NHC–gold complexes in catalysis [148]; (14) perfluoroalkyl gold complexes [149]; (15) 1,2-alkyl migrations in cascade reactions catalyzed by π-acids [150]; (16) syntheses of cortistatin A [151]; (17) six-membered ring C–O bond-forming reactions [152]; (18) functionalization of N-heterocycles by rhodium-catalyzed C–H activation processes [153]; (19) reactions of α-diazocarbonyl compounds [154]; (20) asymmetric cyclopropanation [155]; (21) carbonylation of diazoalkanes [156]; (22) the organometallic chemistry of Group 8 tris(pyrazolyl)borate complexes [157]; (23) metal ylide complexes [158]; (24) high oxidation state organomolybdenum and tungsten chemistry in protic environments [159]; (25) vanadium NMR of organovanadium complexes [160]; (26) identification of reactive intermediates by electrospray MS (including titanium–carbenes from Petassis and Tebbe reactions) [161]; (27) molecular wires [162]; (28) magnetic perturbation of redox potential in carbon-bridged bimetallic compounds [163]; (29) gas phase activation of methane by transition metal cations [164]; (30) quantum mechanical methods in inorganic chemistry [165]; and (31) DFT-based mechanistic studies of ruthenium-mediated C–C coupling reactions [166].

2.2. Alkene metathesis

Alkene metathesis was the most common reaction process reported for metal–carbene complexes in 2008, 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 2008; some representative examples are depicted in Fig. 2. Several derivatives of the Grubbs and Schrock catalysts (see Fig. 1) were synthesized and tested in their ability to undergo various metathesis processes, including: (1) analogs of Grubbs catalyst I that feature 9-phosphabicyclo[3.3.1]nonane ligands in place of tricyclohexylphosphine ligands [167]; (2) analogs of Grubbs catalyst I that feature chelating imidodiphosphinate ligands [168]; (3) an alumina-bound analog of Grubbs catalyst I useful for metathesis reactions in ionic liquids [169]; (4) analogs of Grubbs catalyst II that feature chiral NHC ligands (*e.g.* **6**) [170]; (5) an analog of Grubbs catalyst II where a phenethyl group replaces one of the mesityl groups, which is especially effective for catalysis of the alternating copolymerization of norbornene and cyclooctene [171]; (6) analogs of Grubbs catalyst II that feature naphthyl groups in place of mesityl groups [172,173]; (7) analogs of Grubbs catalyst II featuring chelating carboxylate ligands (*e.g.* **7**), which become active catalysts upon treatment with CuCl [174]; (8) analogs of Grubbs catalyst II tethered to imidazolium rings, which are useful for metathesis reactions in ionic liquid solvents [175]; (9) tethered analogs of Grubbs catalyst II that are useful for formation of cyclic polymers (ring expanding metathesis polymerization, or REMP) (*e.g.* **8**) [176]; (10) analogs of Grubbs catalyst II immobilized on MCF [177]; (11) an analog of Grubbs catalyst II that transforms to a polymeric analog of the Hoveyda–Grubbs catalyst as the metathesis reaction progresses (**9**) [178]; (12) indenylidene–ruthenium complexes (*e.g.* **10**) [179–182]; (13) analogs of Grubbs catalyst II and the Hoveyda–Grubbs catalyst where the NHC ligand has been replaced by a thiazolium ring (*e.g.* **11**) [183]; (14) analogs of Grubbs catalyst II and the Hoveyda–Grubbs catalyst where the mesityl groups of the NHC ligand have been replaced with non-identical aryl groups [184]; (15) analogs of Grubbs catalyst II and the Hoveyda–Grubbs catalysts featuring chiral NHC ligands (*e.g.* **12**) and their use in asymmetric RCM [185]; (16) dimeric analogs of Grubbs catalyst II and the Hoveyda–Grubbs catalyst (*e.g.* **13**), which are effective in promoting cyclic dimerization over ADMET or RCM reactions using 1,12-tridecadiene [186]; (17) analogs of the Hoveyda–Grubbs catalyst where the mesityl groups have been replaced by aminated aryl groups, where the acidic form catalyzes metathesis reactions in water [187] or features easy removal during purification of metathesis products [188]; (18) analogs of the Hoveyda–Grubbs catalyst where the carbene phenyl group contains a quaternary ammonium salt group [189]; (19) an analog of the Hoveyda–Grubbs catalyst where *o*-tolyl groups replace the mesityl group of the NHC ligand, which are efficient for the formation of sterically hindered

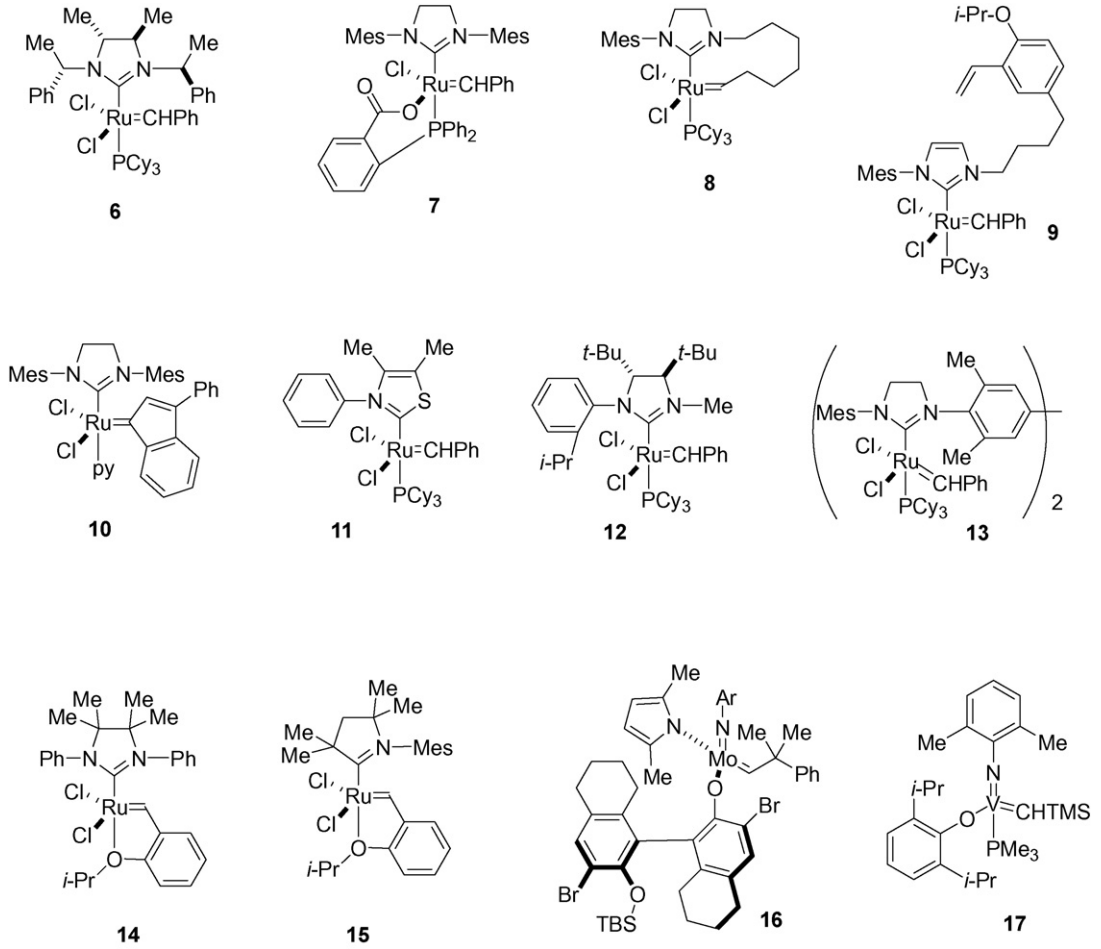
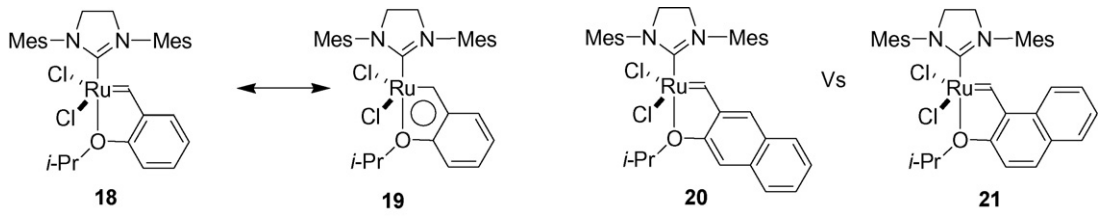
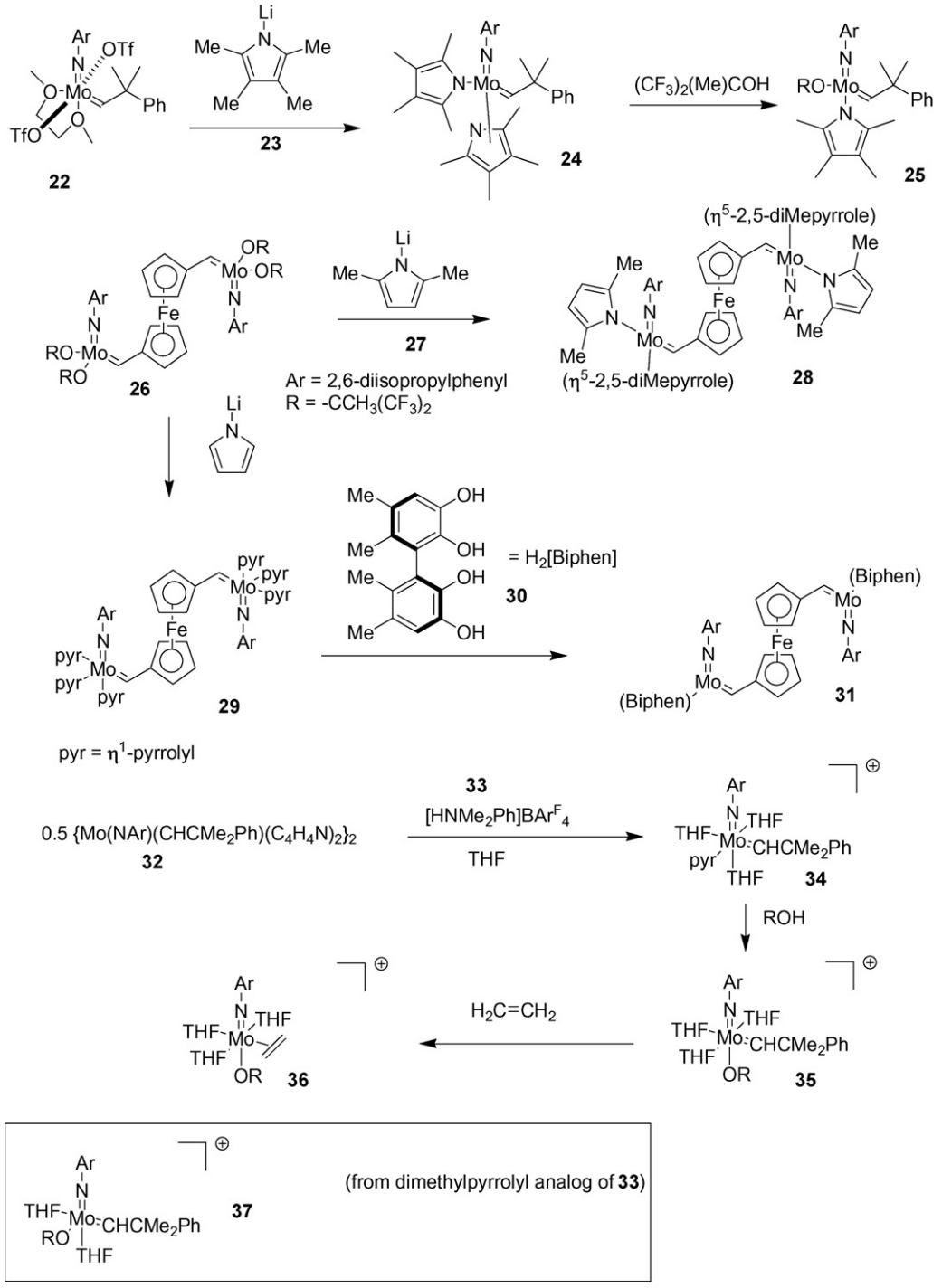


Fig. 2. Representative examples of new catalysts for alkene metathesis.

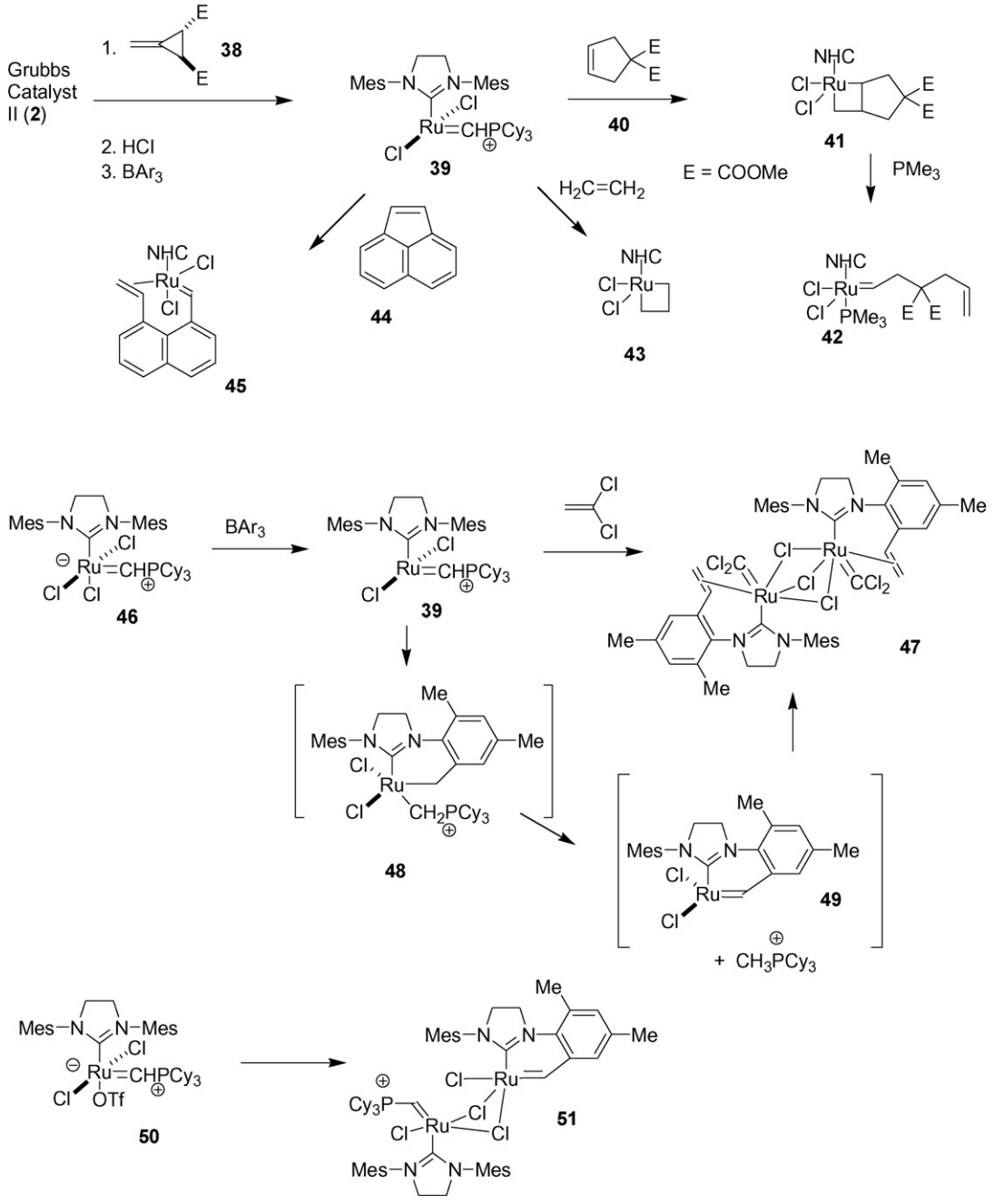


Scheme 2.

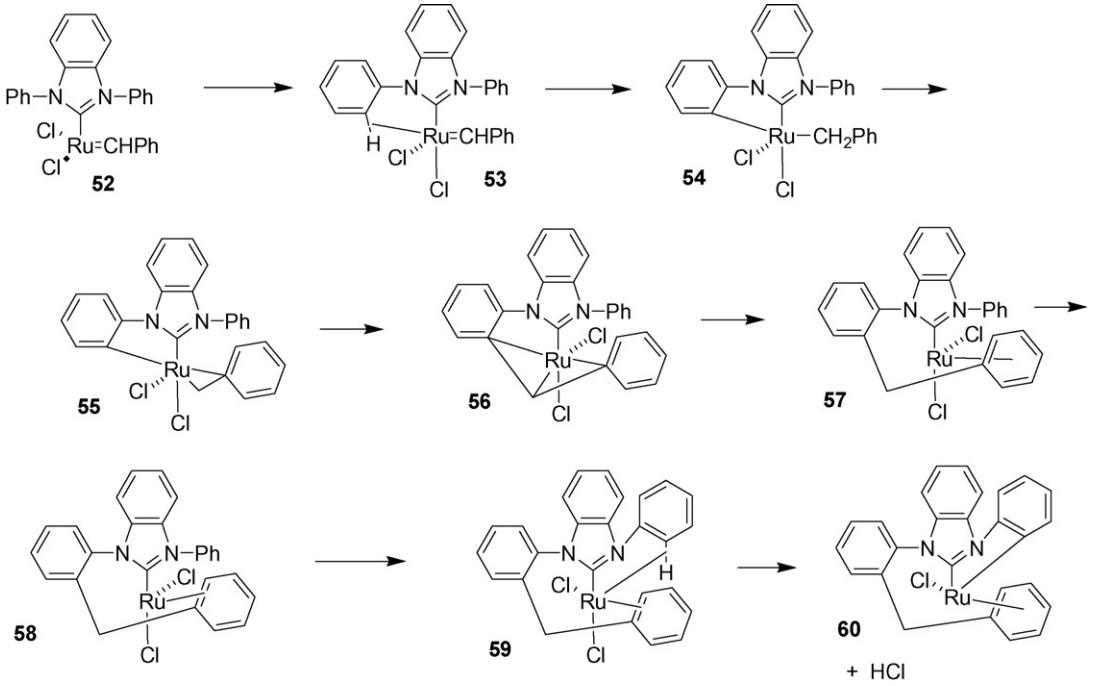


Scheme 3.

alkenes [190]; (20) an analog of the Hoveyda–Grubbs catalyst where the NHC backbone is methylated (*e.g.* **14**), which is highly active for the synthesis of tetrasubstituted alkenes [191]; (21) analogs of the Hoveyda–Grubbs catalyst where the carbene phenyl ring contains a trifluoroacetamide group [192] or a nitro group [193]; (22) a highly fluorinated analog of the Hoveyda–Grubbs catalyst for use in fluorous phase RCM reactions [194]; (23) analogs of the Hoveyda–Grubbs catalyst where a Bertrand carbene ligand replaces the H_2IMes_2 ligand (*e.g.* **15**) [195]; (24) the thioether analog of the Hoveyda–Grubbs catalyst [196,197]; (25) the phenyl–$Cr(CO)_3$ complex of the Hoveyda–Grubbs catalyst [198]; (26) analogs of the Hoveyda–Grubbs catalyst that contain an imidazolium salt for metathesis in ionic liquids [199,200]; (27) silica-bound recyclable analogs of the Hoveyda–Grubbs catalyst [201]; (28) analogs of the Hoveyda–Grubbs catalyst bound to silaceous mesocellular foam through the arylcarbene ligand [202]; (29) analogs of the Hoveyda–Grubbs catalyst bound to silica pellets [203]; (30) water soluble analogs of Grubbs catalyst III [204]; (31) analogs of Grubbs catalyst III bound to imidazolium cations for metathesis reactions in ionic liquids [205]; (32) analogs of the Schrock catalyst bound to dehydroxylated silica [206,207]; (33) chiral analogs of the Schrock catalyst useful for asymmetric metathesis reactions (*e.g.* **16**) [208]; (34) vanadium alkylidene complexes (*e.g.* **17**) [209,210]; and (35) non-carbene molybdenum and tungsten oxo complexes featuring aminophenolate ligands [211]. A DFT study predicted that analogs of Grubbs catalyst II where P-heterocyclic carbenes replace NHC ligands will show enhanced metathesis activity [212]. Several patents were issued for the synthesis and development of metal–carbene containing olefin metathesis catalysts [213–227].



Scheme 4.

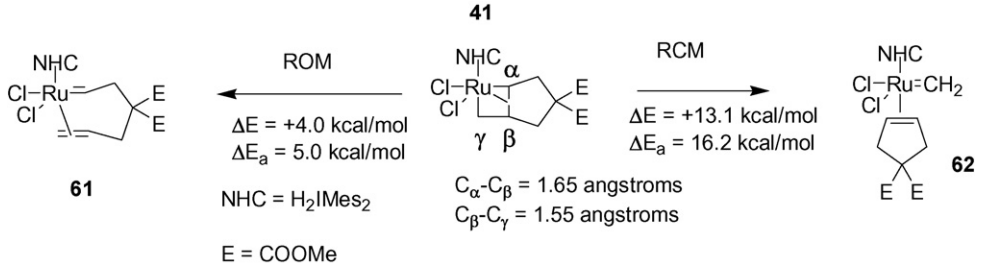


Scheme 5.

Efforts to control the reactivity of Hoveyda–Grubbs systems through aromaticity concerns were reported (Scheme 2) [228]. Stabilization of these catalysts through the metallafuran resonance form (*e.g.* structure **19**) is thought to stabilize the catalyst and thus reduce its reactivity. Disruption of this aromaticity, as in naphthylcarbene complex **20**, results in a more active catalyst. The alternative ring fusion exhibited by complex **21** increases the metallafuran contribution and results in a less active catalyst. Various other aromatic fused analogs of the Hoveyda–Grubbs catalyst were prepared and tested in the ability to catalyze RCM reactions.

The synthesis and reactivity of analogs of the Shrock catalyst and both stoichiometric and catalytic metatheses reactions were reported in 2008. The synthesis and reactivity of pyrrolyl-substituted molybdenum–carbene complexes (*e.g.* **24**, Scheme 3) was reported [229]. Reaction of complex **22** with lithiopyrrole derivatives led to N-pyrrolyl-η^5-pyrrolylmolybdenum complex **24**. Indole analogs led to a bis(N-indolyl) complex. Reaction of complex **24** with alcohols led to alkoxy-N-pyrrolyl complexes (*e.g.* **25**). The synthesis and metathesis reactivity of bis(molybdenum-alkylidene) ROMP initiators (*e.g.* **28**, **29**, **31**) was reported [230]. The reaction of bis(carbene complex) **26** with pyrrole anion **27** led to the mixed π-bound and σ-bound pyrrole complex **28**. Reaction with simple lithiopyrrole led to the complex **29**, which underwent reaction with phenols to afford the corresponding biphenoxide-ligated bis(carbene complex) derivatives (*e.g.* **31**). The biphenoxide complexes were evaluated as ROMP initiators. The formation and metathesis activity of cationic analogs of the Schrock complex (*e.g.* **35**) were reported [231]. The octahedral complex **35** reacted with ethylene to afford the cationic ethylene complex **36**. If the reaction was followed by ^{1}H NMR spectroscopy, a metallacyclopentane complex could be observed. The pentacoordinate complex **37** was an RCM catalyst however most of the cationic complexes appeared to be too unstable to efficiently catalyze metathesis reactions at low catalyst loadings.

The generation and reactivity of four-coordinate ruthenium–carbene complex **39** (Scheme 4) was reported [232]. Complex **39** was prepared by the reaction of Grubbs catalyst II (**2**) with methylenecyclopropane **38**, followed by reaction with HCl and then reaction with a triarylborane. Reaction with various alkenes resulted in phosphine-free metallacyclobutanes (*e.g.* **41**, **43**) or carbenes (*e.g.* **45**) via an intermediate methyleneruthenium complex. The metallacyclobutane **41** is stable and only opens upon treatment with trimethylphosphine to afford carbene complex **42**. Thermal decomposition modes for four-coordinate ruthenium–carbene complexes, likely intermediates in metathesis catalyst decomposition, were examined [233]. Decomposition of carbene complex **39** in the presence of 1,1-dichloroethylene led to the dimeric complex **47**. A mechanism was proposed involving benzylic C–H activation to afford intermediate complex **48** followed by α-hydride elimination and reductive elimination of the phosphonium salt to generate intermediate chelating carbene complex **49**, which undergoes metathesis with dichloroethylene and dimerization to afford



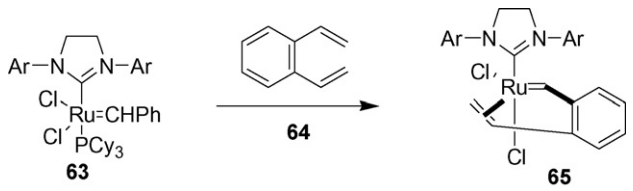
Scheme 6.

the observed product. A mixed dimer containing the chelating carbene group (**51**) was observed during decomposition of the triflate complex **50**.

The decomposition of Grubbs catalyst II derivative **52** (Scheme 5) was evaluated computationally [234]. The decomposition reaction proceeds through several intermediates to ultimately afford carbene complex **60** and HCl. Key steps involve C–H activation processes at the N-phenyl groups of the NHC ligand to afford benzylruthenium complex **54**. It was suggested that catalyst lifetime could be increased by controlling the flexibility of NHC substituents.

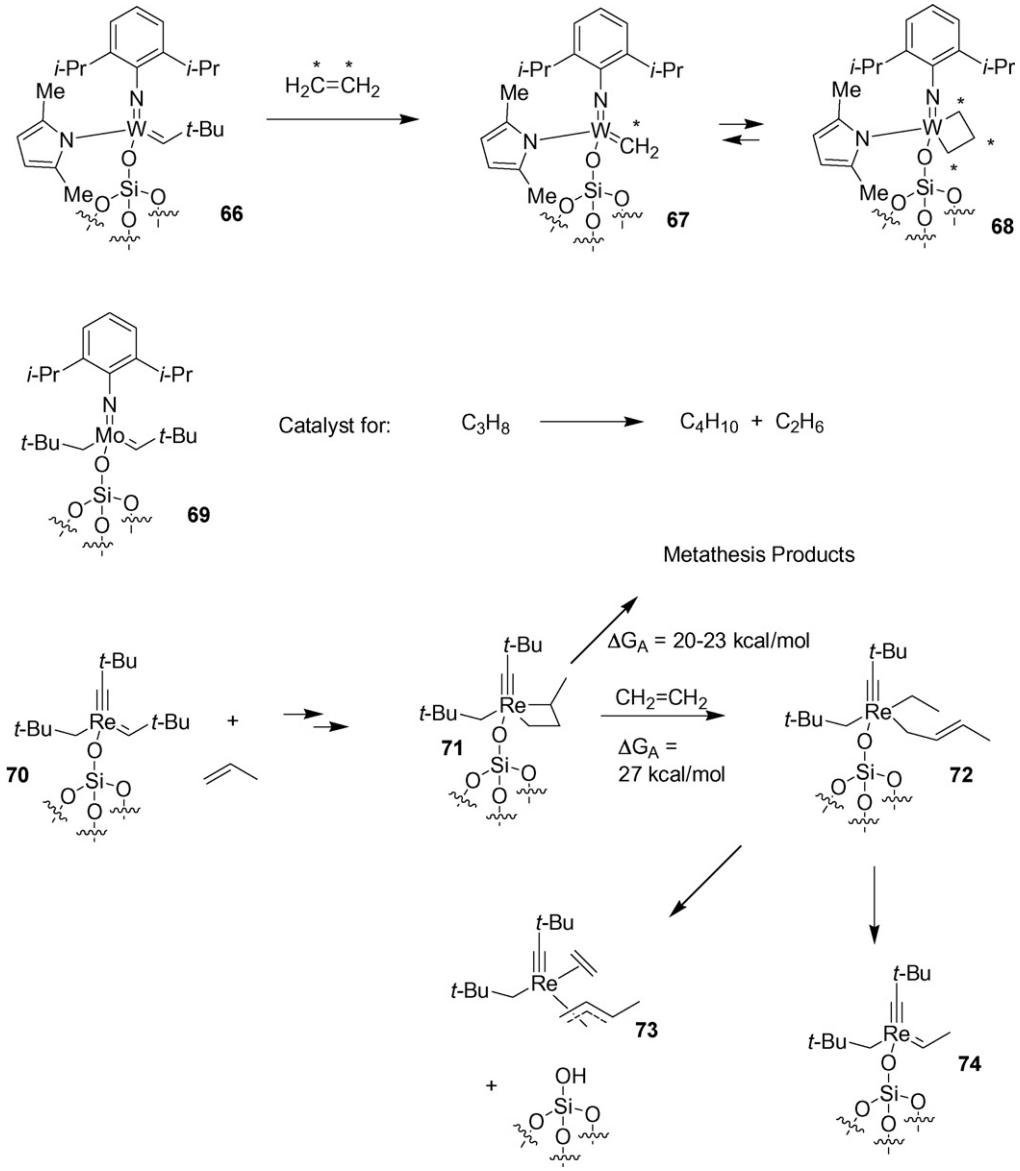
Ruthenacyclobutanes (*e.g.* **41**, Scheme 6) were studied computationally (DFT) [235]. Significant agostic interaction with the C_α–C_β bond was noted. In the unsymmetrical ruthenacyclobutane derivative **41**, the ring opening metathesis (ROM) precursor bond (C_α–C_β) was elongated relative to that giving rise to the RCM product (C_β-C_γ). The conversion of the substituted ruthenacyclobutane to either the ROM or the RCM products was also evaluated computationally. The ROM route was kinetically and thermodynamically favored.

Formation of alkene-coordinated ruthenium–carbene complexes (*e.g.* **65**, Scheme 7) was achieved through the reaction of various derivatives of Grubbs catalyst II (replacement of the mesityl groups with other arenes) with *o*-divinylbenzene (**64**) [236]. Conformational effects of the various ligands were studied by X-ray crystallography and ^{1}H NMR-NOE experiments. Dynamic processes and electrochemistry were reported for Grubbs catalyst II and analogs [237]. Significant rotational barriers were noted for the NHC ligands. For unsymmetrical complexes, the electrochemical oxidation processes occurred in two waves reflecting the different oxidation potentials of the different conformers.



Scheme 7.

Several reports on the structure, synthesis, and reactivity of silicon-bound molybdenum, tungsten, tantalum, and rhenium alkylidene complexes were reported in 2008 (see Scheme 8).



Scheme 8.

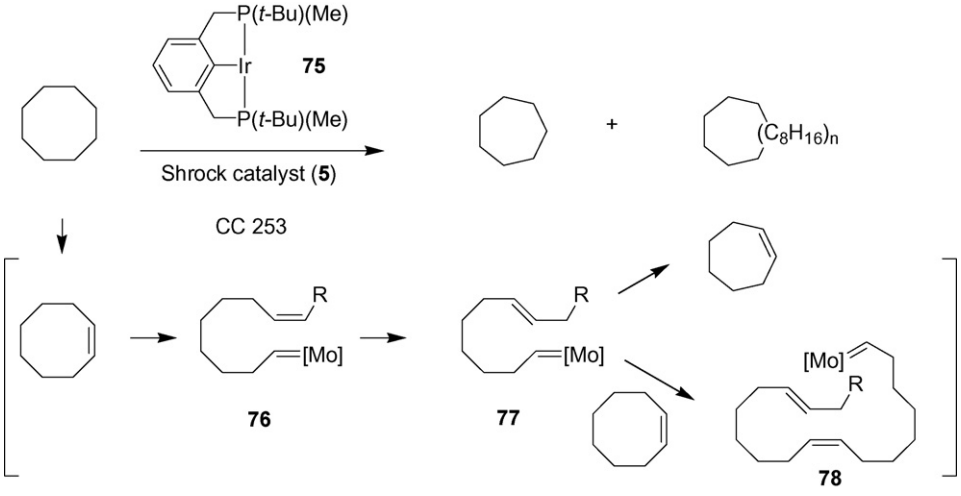
These complexes function as catalysts for alkene metathesis and for alkane metathesis. Carbon–carbon bond formation occurs via similar mechanistic events in alkane metathesis and in alkene metathesis. The reaction of silica-bound tungsten complex **66** with ^{13}C-labelled ethylene was reported [238]. From this process both the methylene complex **67** and the metallacyclobutane **68** could be observed by solid-state NMR. The metallacyclobutane exists in both the trigonal bipyramidal and the square pyramid forms. A key step in alkane metathesis reactions is C–H activation of the alkane by the catalyst (*e.g.* **69**) followed olefin metathesis steps [239]. Detailed studies of the structure and relative reactivity of these complexes using DFT calculations and solid-state NMR was reported [240]. An experimental and DFT-based study of the decomposition of the rhenium catalysts (*e.g.* **70**) was reported [241]. An energetically reasonable pathway identified involves ring opening of the metallacyclobutane (**71**) to form the *o*-allyl complex (**72**), which undergoes conversion to the π-allyl complex (**73**) and loss of the ligation to the solid support. Tantalum-catalyzed alkane metathesis was studied computationally [242].

Reaction of cyclooctane with the Schrock catalyst (**5**, Scheme 9) and a C–H activation catalyst (**75**) to afford cycloheptane and larger cycloalkanes was reported [243]. A mechanism involving iridium-catalyzed conversion to cyclooctene and an iridium hydride, followed by olefin metathesis and hydrogenation was proposed. Cycloheptane was proposed to arise from ring opening metathesis to alkene–carbene complex **76**, followed by alkene migration to afford **77**, followed by RCM and hydrogenation.

Other general studies of carbene complex-initiated alkene metathesis include: (1) use of high throughput screening to identify highly active metathesis catalysts using [244]; (2) a low-temperature NMR study of Grubbs catalysts I and II [245]; (3) studies of various ruthenium–carbene complex metathesis catalysts and ruthenium carbides via K-edge X-ray absorption spectroscopy [246]; (4) determination of the binding energies of the phosphine ligands in Grubbs catalysts I and II by ESI-mass spectrometry [247]; (5) observation of propagating carbene complex species by ^{1}H NMR in ROMP reactions of oxygen-containing norbornenes initiated by ruthenium–carbene complexes [248]; (6) observation of intermediates of ruthenium-catalyzed metathesis by MALDI-TOF mass spectrometry [249]; (7) solid-state NMR studies of the $MeReO_3$–alumina systems; no mononuclear carbene signals were detected suggesting that the catalyst resting states are μ-carbene complexes [250]; (8) a study of olefin metathesis using electrospray ionization mass spectrometry [251]; (9) a kinetic study of olefin metathesis using a $MoCl_5$–SiO_2–Me_4Sn system and proposal of mechanisms for formation, deactivation, and reactivation of the carbene complex metal catalyst [252]; (10) a mechanistic study of metathesis reactions catalyzed by Mo–Al alloy [253]; (11) olefin metathesis reactions in water [254,255]; (12) cross-metathesis and RCM in water using in the presence of a vitamin-E-based amphiphile [256,257]; (13) enhanced activity of Grubbs catalyst II in acetic acid solvent due to inhibition of phosphine return [258]; (14) encapsulation of Grubbs catalyst II in a polydimethyl siloxane thimble and subsequent metathesis reactions using the encapsulated catalyst [259]; (15) use of dimethyl carbonate as a solvent for metathesis reactions [260]; (16) improvement in the efficiency of tetrasubstituted alkene formation using perfluorinated aromatic solvents [261]; (17) removal of ruthenium impurities from metathesis reactions using $Fe@Fe_xO_y$ nanoparticles [262] or nanofiltration [263]; (18) generation of carbene complexes in the catalysis of olefin metathesis by tungsten–tungsten double bonded species [264]; and (19) formation of carbene complexes in Group 5—trialkylaluminum-based catalyst systems [265]. Several DFT studies of olefin metathesis were reported in 2008, including: (1) a study of phosphine ligand dissociation in metathesis reactions initiated by pyridinecarboxylate-ligated ruthenium–carbene complexes [266]; (2) a study evaluating conformation effects in ROMP of norbornene [267]; (3) a study of the formation of metal–carbene complexes using a Mo–β-zeolite catalyst system [268]; (4) a study of the generation of carbene complex intermediates in ROMP reactions catalyzed by [$MoCl(GeCl_3)(CO)_3$(norbornadiene)] [269]; (5) a study of the mechanism of WCl_6/atomic C catalyzed metathesis of 1-octene [270]; (6) a study evaluating the hypothetical formation of tungsten carbene complexes from norbornadiene via (η^2-norbornadiene)$W(CO)_3I_2$ [271]; (7) a study evaluating the formation of a carbene complex in ROMP reactions initiated by tetrakis(acetonitrile)(NHC)ruthenium(II) complexes [272]; (8) a study of ethylene metathesis using a Mo–Al catalyst system [273]; and (9) a study focusing on location of methylene binding sites for metathesis reactions catalyzed by molybdenum on alumina [274]. A mechanistic study of propene metathesis using methyltrioxorhenium grafted onto alumina comments on the failure to observe carbene complex intermediates [275]. A comparison of DFT vs. MO6 methods for the computational study of ruthenium-catalyzed metathesis revealed that MO6 was superior for prediction of E/Z selectivity [276].

2.2.2. Polymerization reactions

Initiation of the ring opening metathesis polymerization (ROMP) (see Scheme 1) reaction using carbene complexes remains a



Scheme 9.

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 2008; 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) phenethylnorbornenes [277]; (2) N-hydroxysuccinyl esters (*e.g.* **80**) followed by enol ether termination [278] or cross-metathesis with alkene-azides [279]; (3) norbornenecarboxylate esters [280–284] including polymer end-capping through cross-metathesis with 2-butene-1,4-diol [285]; (4) norbornenecarboxylic acid [286]; (5) norbornenyl ketones (*e.g.* **81**) [287]; (6) a protonated aminomethylnorbornene [288]; (7) norbornenes connected to polyethers [289,290]; (8) norbornenes connected to α-bromo ester groups for eventual radical polymerization [291,292]; (9) norbornene (and/or oxanorbornenes) fused to succinimides (*e.g.* **82**, **83**) [293–301] and subsequent end-capping of the living polymer chains with either cyclic enol ethers [302], alkenes connected to α-bromopropionate esters for eventual radical polymerizations [303], or 1,4-dichloro-2-butene (includes extensive DFT studies) [304]; (10) benzo-fused norbornenes [305]; (11) tetracyclo[4.4.0.12,5.17,10]dodecene derivatives (*e.g.* **84**) [306–308]; (12) norbornene esters containing photoactive groups (*e.g.* **85**) [309]; (13) norbornenes connected to photochromic spiropyrans and linkage of the resulting living polymer

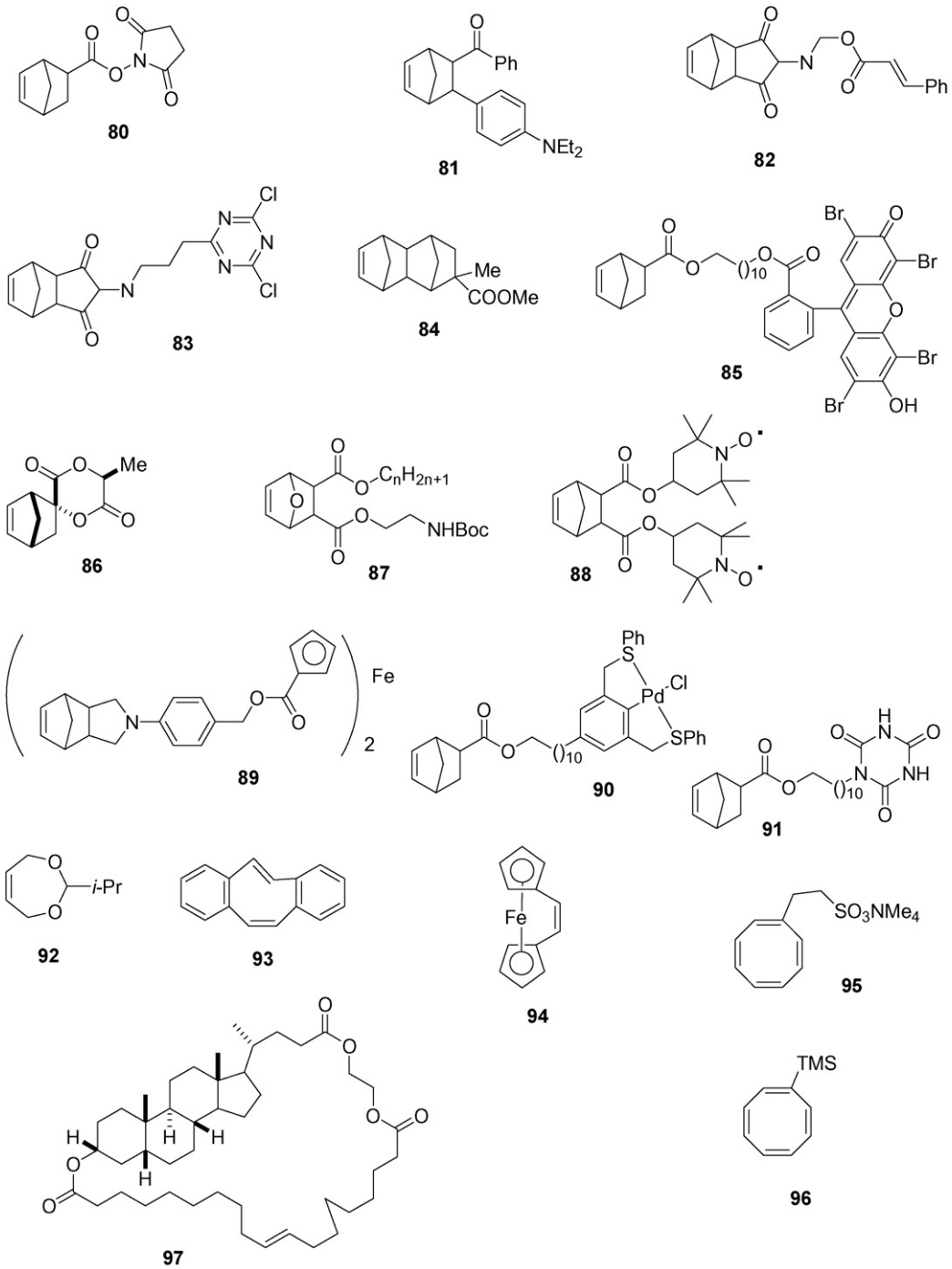


Fig. 3. Representative substrates for the ROMP reaction.

to a silicon surface via cross-metathesis [310]; (14) norbornenes connected to cyanuric acid-linked hydrogen-bonded assemblies [311]; (15) norbornenes that contain groups capable of ring opening polymerization (*e.g.* **86**) [312]; (16) norbornenes linked to tripeptide chains [313]; (17) norbornene diesters or oxanorbornene diesters (*e.g.* **87**) [314–317]; (18) norbornene bis(trimethylsilyl) esters [318]; (19) norbornene diketones [319]; (20) norbornene nitrile-esters [320]; (21) norbornene PEG ester-amides [321]; (22) norbornenedicarboxamides where the N-substituent is derived from amino acids [322]; (23) norbornenes containing nitroxyl groups (*e.g.* **88**) [323]; (24) aromatic ring-linked bis(norbornenes) [324]; (25) 5-trimethylsilynorbornene [325]; (26) 5,5-bis(trimethylsilyl)norbornene [326]; (27) sulfur-containing norbornenes [327]; (28) ferrocene-linked bis(norbornenes) (*e.g.* **89**) [328,329]; (29) a norbornene derivative fused to a triacylglyceride [330,331]; (30) gold surface-bound norbornenedicarboxylate esters [332]; (31) homo- and heteropolymers from various norbornenes functionalized with either a pincer palladium complex (*e.g.* **90**), a uracil group, or a cyanuric acid group (*e.g.* **91**) [333]; (32) norbornenes connected to carbon nanotubes in the presence of dicyclopentadiene [334]; (33) norbornene polymerization in the presence of dicyclopentadiene followed by end-capping through cross-metathesis [335]; and (34) dicyclopentadiene as a component in a self-healing epoxy resin [336,337]. Other ring systems that have been subjected to ROMP reactions include: (1) cyclopropenes [338]; (2) cyclopentene [339–341]; (3) acetal-protected 2-butene-1,4-diol derivatives (*e.g.* **92**) [342,343]; (4) benzo-fused trans cyclooctenes (*e.g.* **93**) [344]; (5) vinyl-bridged ferrocenes (*e.g.* **94**) [345]; (6) cyclooctenes bound to imidazolium salts [346]; (7) esters of 5-cyclooctene-1,2-diol [347]; (8) 1,5-cyclooctadiene [348]; (9) cyclooctatetraenes (*e.g.* **95**, **96**) [349,350]; (10) macrocycle-bridged steroids (*e.g.* **97**) [351]; (11) catenanes, which afford polypseudorotaxanes after polymerization [352]; and (12) pseudorotaxanes [353]. ROMP reactions in micelles [354] and in a water-in-oil-in-water emulsion were reported [355]. Simultaneous ROMP and radical polymerization in a microemulsion [356] and in bamboo stalks were reported [357]. Varying degrees of tacticity and stereoselectivity were noted in the ROMP of 3-methyl-3-phenylcyclopropene using chiral and optically pure molybdenum–carbene complexes as initiators [358]. An isolated silylnorbornene–tungsten complex was observed to undergo ROMP polymerization [359]. In some cases, the $Mo(CO)_6$-catalyzed hydrosilylation of norbornadiene is accompanied by ROMP products [360]. A patent was

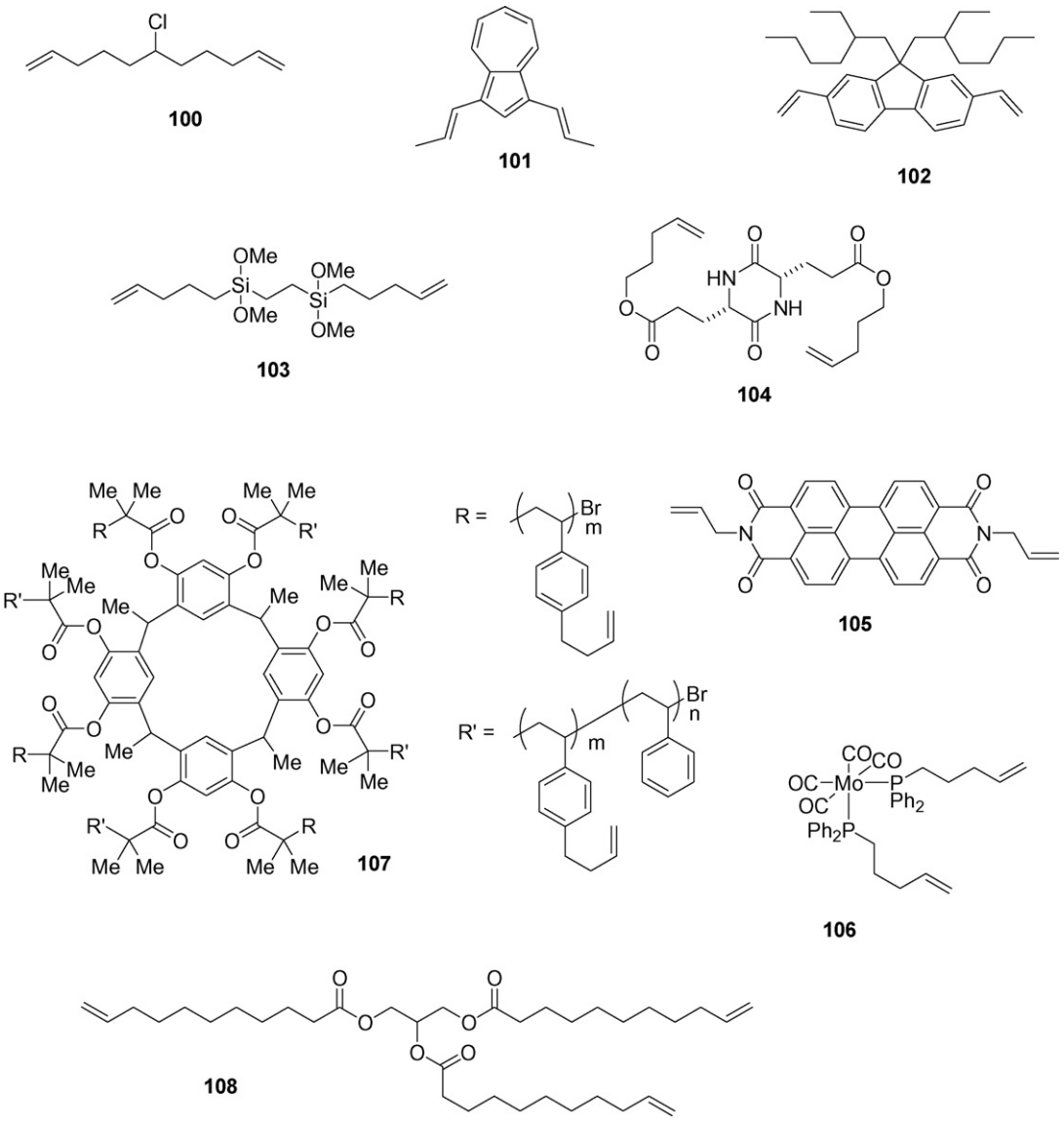


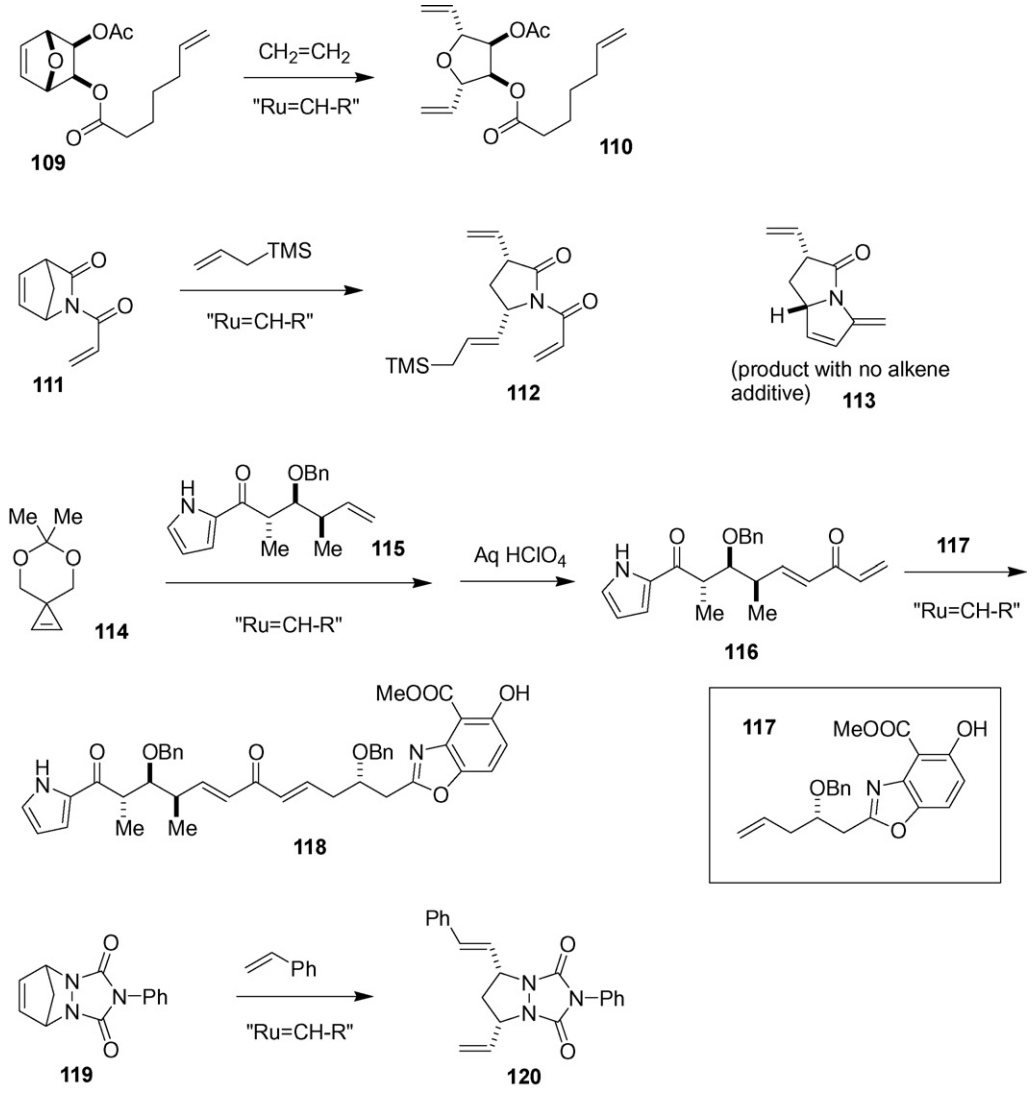
Fig. 4. Representative substrates for ADMET polymerization.

awarded for development of tandem ROMP-hydrogenation reactions [361].

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. Several α,ω-dienes possessing the followed structural features were subjected to ADMET polymerization: (1) free OH groups or PEG groups [362]; (2) amide substituents [363]; (3) halogens (*e.g.* **100**) [364]; (4) bis(alkenyl)azulenes (*e.g.* **101**) [365]; (5) linkage of the alkenes through an fluorene ring (*e.g.* **102**) [366–368]; (6) linkage of the alkene groups through an ester group [369]; (7) linkage of the alkenes groups through either silanes (*e.g.* **103**) or ethers [370]; (8) linkage of the alkene groups through diarylsilane and diarylsiloxane groups [371]; (9) linkage of the alkene groups through a diketopiperazine ring system (*e.g.* **104**) [372]; (10) linkage of the alkenes through perylenediimide group (*e.g.* **105**) [373,374]; (11) linkage of the alkene groups through a molybdenum bis(phosphine) complex (*e.g.* **106**) [375,376]; and (12) alkene-capped THF polymers [377]. Formation of an ADMET star polymer from a calixarene-templated alkene derivative (*e.g.* **107**) [378] and formation a tribranched polymer (ATMET) from a triene derivative (*e.g.* **108**) were also reported [379]. Poly(acrylate)-linked α,ω-dienes were converted to the corresponding cyclic polymers through high dilution RCM [380]. Two polyene-containing polymers could be equilibrated through a segment interchange process catalyzed by ruthenium–carbene complexes [381]. Higher trans alkene selectivity was noticed in the secondary ADMET-like metathesis of polybutadiene compared to ROMP of 1,5-cyclooctadiene [382].

2.2.3. Nonpolymer-forming ring opening metathesis reactions

Several examples of RO-CM (see Scheme 1) were reported in 2008. Representative examples are depicted in Scheme 10. Co-metathesis of ethylene and substituted norbornenes led to divinylcyclopentane derivatives [383]. The RO-CM of ethylene and various norbornenes that contain additional alkene groups was demonstrated (*e.g.* conversion of **109** to **110**) [384]; similar substrates were also subjected to RRM or ring-rearranging enyne metathesis. Regioselective RO-CM was observed in the reaction of azanorbornene **111** with monosubstituted alkenes [385]. The regioselectivity was attributed to coordination of the amide carbonyl. Compound **111** would undergo RRM to afford pyrrolidinone **113** if treated with carbene complex **3** in an argon atmosphere. The tandem RO-CM and CM of cyclopropenes (*e.g.* **114**) and alkenes **116** and **117** was a key step in the total synthesis of routiennocin [386]. The RO-CM of diazanorbornenes (*e.g.* **119**) with various monosubstituted alkenes was also reported [387]. A patent was awarded



Scheme 10.

for development of conditions for RO-CM of cyclohexene and 1,4-dihalo-2-butenes [388].

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 2008. Representative examples are depicted in Fig. 5. Regretfully it is very difficult to rationally organize these reactions. Several papers report on the cross-metathesis of inexpensive low molecular weight α,β-unsaturated carbonyl compounds with other more precious alkenes, including cross-metatheses of: (1) allylbenzene derivatives and methyl acrylate [389]; (2) a 1,1-dibromo-1,5-hexadiene derivative and methyl acrylate [390]; (3) various homoallylic alcohols and methyl acrylate [391]; (4) oleyl alcohol and methyl acrylate [392]; (5) allylporphyrins and ethyl acrylate [393]; (6) ferrocene-containing rotaxanes and acrylate esters [394]; (7) an allylic alcohol and ethyl acrylate for total synthesis of dodoneine [395]; (8) a steroidal allylic alcohol and methyl acrylate for total synthesis of drospirenone [396]; (9) double cross-metathesis of a 1,10-undecadiene derivative and methyl acrylate for total synthesis of hippodamine [397]; (10) a complex alkene-alcohol and methyl acrylate for synthesis of the pyran ring of bistramide A [398]; (11) an alkene-alcohol and methyl acrylate for total synthesis of bistramide A [399]; (12) a homoallylic alcohol and methyl acrylate and in a later step two dissimilar allylic alcohol derivatives for total synthesis of decarestrictine D [400]; (13) a homoallylic ether derivative and *t*-butyl acrylate for total synthesis of negamycin [401]; (14) a butenylpyran derivative and methyl acrylate for synthesis of halichondrin segments [402]; (15) a complex homoallylic alcohol and ethyl acrylate for preparation of the macrolactone core of leucasandrolide (synthesis also includes on O-heterocycle synthesis via RCM and another cross-metathesis event) [403]; (16) a complex homoallylic alcohol (**125**) and ethyl acrylate for total synthesis

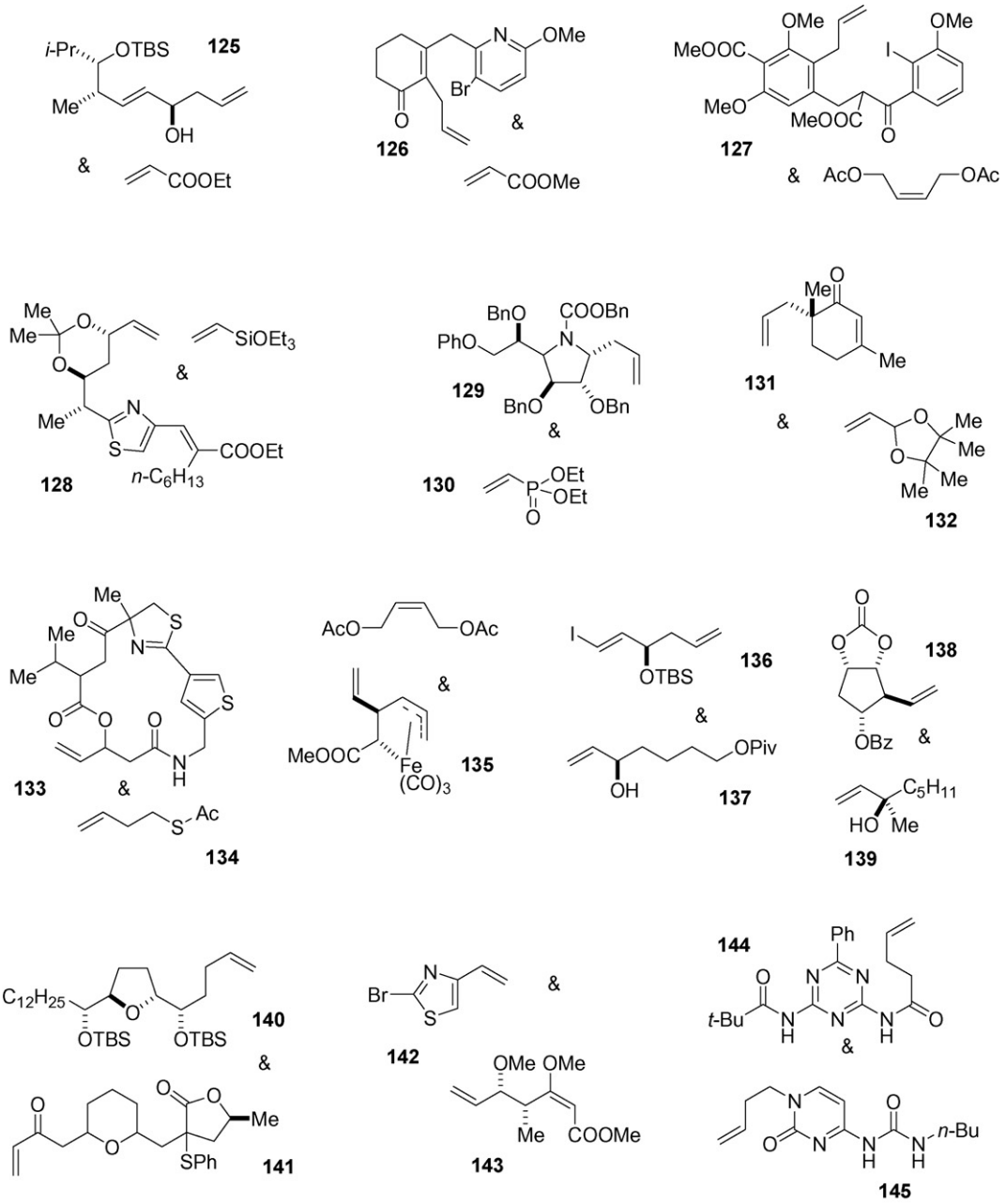
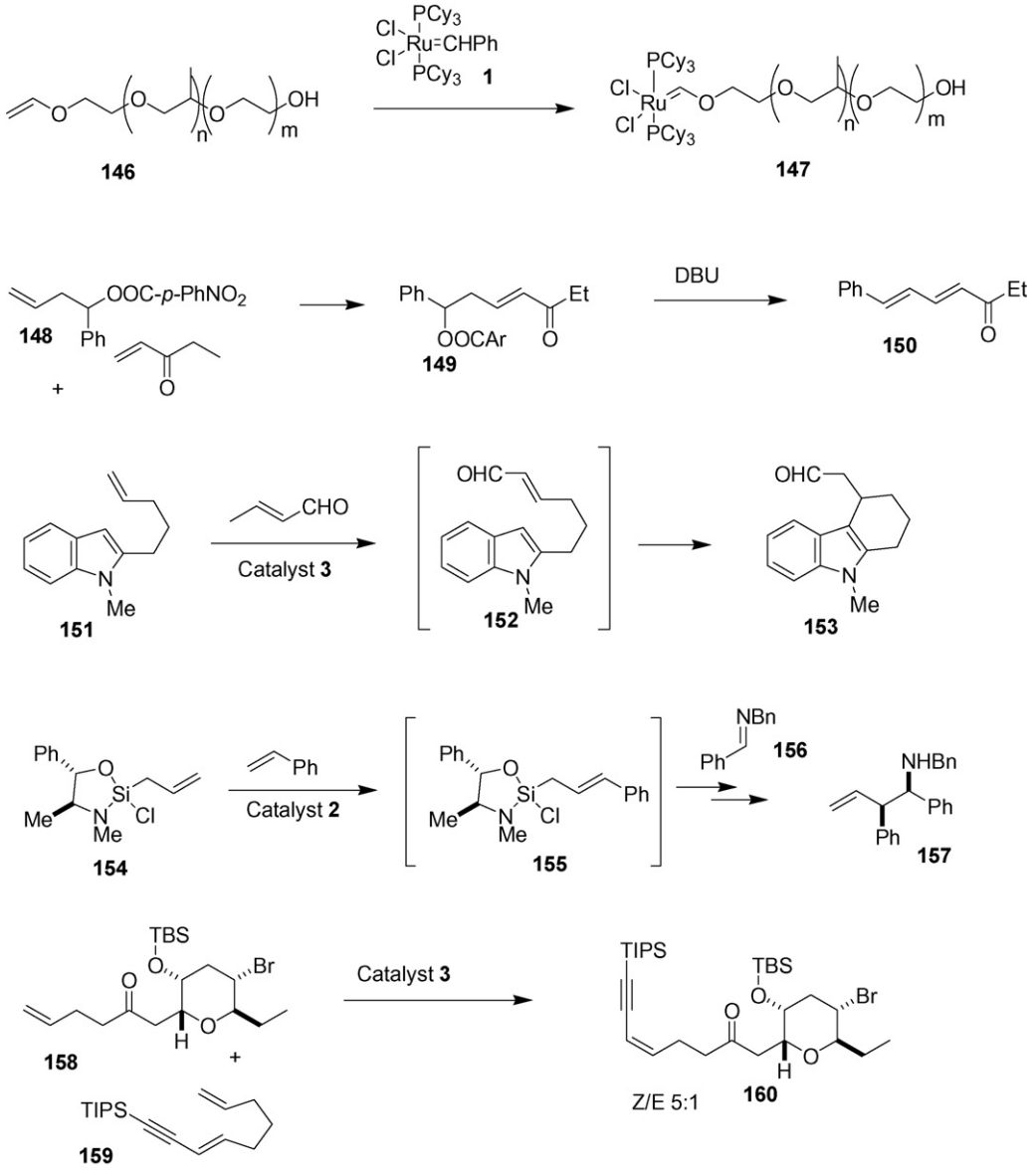


Fig. 5. Represent pairs of alkenes subjected to cross-metathesis.

of RK-397 [404]; (17) a complex pyridine–cyclohexene derivative (**126**) and methyl acrylate for total synthesis of lycopodium alkaloids [405]; (18) C-allylglycosides and methyl acrylate for synthesis of maitotoxin fragments [406]; (19) monosubstituted alkenes and various acrylate thioesters [407]; (20) gold surface-bound vinylated polystyrenes and PEG methacrylate esters [408]; (21) a homoallylic ester derivative and acrolein [409]; (22) an ether of 3-buten-1-ol and acrolein for total synthesis of amphidinolide J [410]; (23) an allyltetrahydropyran derivative and acrolein for synthesis of the macrocyclic core of zampanolide [411]; (24) a homoallylic alcohol and acrolein as part of a synthetic approach to octalactins [412]; (25) amine–alkenes and acrolein for total synthesis of angustureine [413]; (26) a complex bicyclic diether and acrolein for total synthesis of laurefucin [414]; (27) a homoallylic ether and methacrolein for total synthesis of GEX1A [415]; (28) an acrylamide featuring a chiral sultam group and various monosubstituted alkenes [416]; (29) steroids that contain vinyl groups and acrylamide derivatives [417]; (30) a 4-penenoate ester and Weinreb acrylamide for total synthesis of azaspiracid-1 [418]; (31) an allylic amine derivative with methyl vinyl ketone [419]; (32) an alkenyl epoxide and methyl vinyl ketone for total synthesis of synerazol and pseurotin E [420]; (33) a vinylcycloheptene derivative and methyl vinyl ketone for total synthesis of xanthatin [421]; (34) a homoallylic ether derivative with methyl vinyl ketone for total synthesis of leucasandrolide A [422]; (35) an azide–alkene with 3-penten-2-one for total synthesis of lycopodine [423]; and (36) acrylic acid and 6-chloro-1-hexene for total synthesis of allosedridine [424]. Several examples employing the cross-metathesis of simple inexpensive low molecular weight functionalized allyl derivatives with more precious alkenes were reported in 2008, including: (1) allylproline derivatives and monosubstituted alkenes [425]; (2) N-allyluracil derivatives and allylphosphonates [426]; (3) allylpyrimidines and allylphosphonates [427]; (4) allylic amine derivatives and vinyl sulfones [428]; (5) solid phase-immobilized allylsulfones and allylic amine derivatives [429]; (5) styrenes and allylic alcohol derivatives [430]; (6) alkenes containing O-alkyl hydroxylamine groups and electron-deficient alkenes [431]; (7) an allylic alcohol/phosphate derivative and Boc-protected 10-undecenylamine [432]; (8) an allylic phosphate ester and alkenes for synthesis of dolabelide segments [433,434]; (9) porphyrins and O-allyl carbohydrate derivatives [435]; (10) 1,4-diacetoxy-2-butene with a complex trisubstituted alkene with for total synthesis of biyouyanagin A [436] and with a complex allylbenzene derivative (**127**) for preparation of tetracycline skeleton [437]; (11) an allyltetrahydrofuran derivative and the dicarbonate ester of 2-butene-1,4-diol for total synthesis of spirastrellolide methyl ester [438]; and (12) cross-metatheses using allylic thioethers for cysteine incorporation [439]. Several examples employing the cross-metathesis of simple allylsilane derivatives with more precious alkenes were reported in 2008, including: (1) allylsilanes with various monosubstituted alkenes [440]; (2) a fluorous-tagged allylsilane with various monosubstituted alkenes [441]; (3) N-4-pentenylsuccinimde and allyltrimethylsilane for total synthesis of tashiromine [442]; (4) octavinylsilsesquioxane with *p*-bromostyrene for synthesis of star polymers or dendrimers [443,444]; (5) surface-bound allylsilanes and styrene derivatives [445]; and (6) 1,5-hexadiene-3,4-diol and allyltrimethylsilane, followed by fluorination/desilylation and RCM to afford fluorination cyclohexenes [446]. Several examples employing relatively abundant natural products in cross-metathesis reactions were reported in 2008, including (1) ethylene and oleate esters [447,448]; (2) self-metathesis of oleates [449]; and (3) ethylene and β-carotene [450]. Other examples of cross-metatheses where one of the partners is simple and readily available include: (1) alkene-β-ketoesters and various monosubstituted alkenes [451]; (2) remote end-of-chain alkene-esters and 5-bromo-1-pentene [452]; (3) pentenol derivatives and various electron-deficient alkenes [453]; (4) dienylnitriles and various monosubstituted alkenes (selective metathesis at the distal alkene group) [454]; (5) alkene-containing α-diazo-β-keto esters and various alkenes [455]; (6) polymer-bound alkene-esters and various monosubstituted alkenes [456]; (7) oxygenated allylphosphonate esters and monosubstituted alkenes [457]; (8) vinyltetrahydrofuran derivatives and alkene-containing amino acid derivatives [458] or styrenes [459]; (9) 1-heptadecene and a complex vinylacetal derivative [460]; (10) a vinyltetrahydrofuran derivative and 1-tetradecene for total synthesis of pachastrissamine [461]; (11) an allylic alcohol derivative with 1-pentadecene for preparation of sphingomyelin analogs [462]; (12) vinyloxazoles with various monosubstituted alkenes [463]; (13) N-homoallylamide derivatives and various monosubstituted alkenes [464]; (14) alkene–phenols (cardanols) with various monosubstituted alkenes [465]; (15) allyltetrahydrofurans and 10-undecen-1-ol [466]; (16) methyl 10-undecenoate and a vinyltetrahydrothiazole derivative for total synthesis of somocystinamide A [467]; (17) 2-methyl-1,4-pentadiene and a vinylboronate ester for total synthesis of amphidinolide E [468]; (18) an allylpyrrole derivative and 1-octen-3-ol for total synthesis of indolizidine 167B [469]; (19) an allylic ether derivative (**128**) with vinyltriethoxysilane for total synthesis of thuggacin [470]; (20) an allylpyrrolidine derivative (**129**) with a vinylphosphonate (**130**) for synthesis of a UDP-Gal mutase inhibitor [471]; (21) a complex polyene–ester and a vinylborane for total synthesis of apoptolidinones A and D [472]; (22) an allylcyclohexene derivative (**131**) with a vinylboronate ester (**132**) for preparation of platensimycin [473] and platensimycin analogs [474]; (23) a complex allylic ester (**133**) and a homoallylic thioester (**134**) for total synthesis of largazole and largazole analogs [475–479]; (24) π-allyliron complexes that contain vinyl groups (*e.g.* **135**) with various alkenes [480]; (25) vinylporphyrins with various simple alkenes and formation of macrocycle-bridged porphyrins via RCM [481]; (26) vinylferrocene and fluorinated alkenes [482]; (27) oligosiloxanes that feature vinyl groups with styrene derivatives [483]; (28) binding of cucurbituril [6] to a gold surface [484]; and (29) a failed attempt to cross-metathesise a vinyltetrahydrofuran derivative with 1,3-pentadiene [485]. Many examples of successful cross-metatheses involving two different and structurally complex alkenes were reported in 2008, including: (1) structurally similar but different complex alkene-alcohol derivatives [486,487]; (2) non-identical alkene-containing carbohydrate derivatives [488]; (3) a C-allylcarbohydrate derivative and either styrene or various alkene–carbohydrates [489]; (4) a homoallylic alcohol derivatives and an allyldihydropyran for total synthesis of ethyl deoxymonate B [490]; (5) a 1-iodo-1,5-hexadiene derivative (**136**) and an allylic alcohol (**137**) for total synthesis of amphidinol 3 [491]; (6) a vinyltetrahydrofuran derivative and a styrene derivative for total synthesis of varitriole [492]; (7) a styrene derivative with a γ,δ-unsaturated ketone for total synthesis of ovalifoliolatin B [493]; (8) an allylic alcohol and a vinyl ester for total synthesis of pentadecyl 6-hydroxydodecanoate [494]; (9) an allylic alcohol derivative (**139**) with a vinylcyclopentene derivative (**138**) for total synthesis of 15(R)-Me-PGD [495]; (10) an allylic alcohol–tetrahydrofuran derivative and a 2-(9-decenyl)furanone for total synthesis of solamin A [496]; (11) an undecenylfurnanone and an allylic alcohol–bis(tetrahydrofuran) for total synthesis of 27-hydroxybullatacin [497]; (12) an allylic alcohol–tetrahydropyrrole and a vinyl ketone for total synthesis of an ant-derived hydroxyindolizidine alkaloid [498]; (13) a vinyloxepin derivative and an allylic alcohol derivative for preparation of ciguatoxin segments [499]; (14) a vinyl epoxide and a vinyllactone (ten-membered) for total synthesis of mueggelone-I [500]; (15) a homoallylic alcohol derivative and a vinyl ketone derivative for preparation of a lyngobouloside segment [501]; (16) a homoallylic alcohol derivative and an allylamine for total synthesis of febrifugine [502]; (17) a homoallylic ether derivative and a complex allylic ether

for synthesis of the core of pladienolide B [503]; (18) a pentenol derivative and an alkene–ketophosphonate for preparation of dictyostatin analogs [504]; (19) a complex acrylate ester and an allyltetrahydropyran derivative in an approach to rhizoxin D [505]; (20) an alkene–tetrahydrofuran derivative (**140**) and a complex enone (**141**) for total synthesis of montanacin [506]; (21) a vinyl thiazole (**142**) and an allylic alcohol derivative (**143**) for total synthesis of cystothiazole A [507]; (22) a conjugated diene and a 4-vinyl-β-lactam for total synthesis of N-Boc-ADDA [508]; (23) preparation of analogs of laulimalide by a late stage cross-metathesis [509]; and (24) cross-metathesis assisted by preorganization based on hydrogen bonding interactions (*e.g.* **144** + **145**) [510]. Polymer-bound oligosaccharides linked to the polymer through an alkene group were cleaved from the resin through cross-metathesis with ethylene [511–513]. Cross-metathesis of the complex macrocyclic lactone etnangien with ethylene provided smaller fragments that were useful in the structure determination of the natural product [514].

Several more complex examples of cross-metathesis were reported in 2008; representative examples are depicted in Scheme 11. Stoichiometric cross-metathesis reaction of Grubbs catalyst I and polymer-bound vinyl ethers (*e.g.* **146**) led to polymer-bound alkoxycarbene–ruthenium complexes (*e.g.* **147**) [515]. A one-pot cross-metathesis of homoallylic *p*-nitrophenyl esters (*e.g.* **148**) and α,β-unsaturated carbonyl compounds followed by base-catalyzed elimination led to the direct synthesis of pentadienoyl systems (*e.g.* **150**) [516]. Several reaction processes reported in 2008 involve cross-metathesis followed by capture of the resultant alkene. The cross-metathesis of pentenylindoles (*e.g.* **151**) and 2-butenal was accompanied by cyclization to afford the fused-ring indoles (*e.g.* **153**) [517]. A tandem cross-metathesis/intramolecular Michael addition process was also observed in the cross-metathesis of Cbz-protected alkene–amines and acrolein [518]. Tandem cross-metathesis and allylation was observed in the coupling of allylsilane chlorides (*e.g.* **154**) with styrenes in the presence of imines (*e.g.* **156**) to afford homoallylamines (*e.g.* **157**) of high enantiomeric and diastereomeric purity [519]. A relay cross-metathesis (**158** + **159**) was employed for the total synthesis of scanlonenyne [520]. A patent was awarded for the preparation of alkenes via cross-metathesis [521].



Scheme 11.

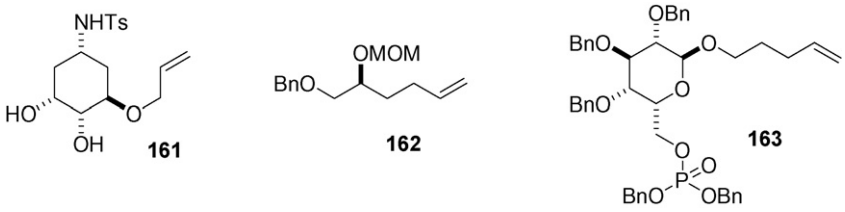
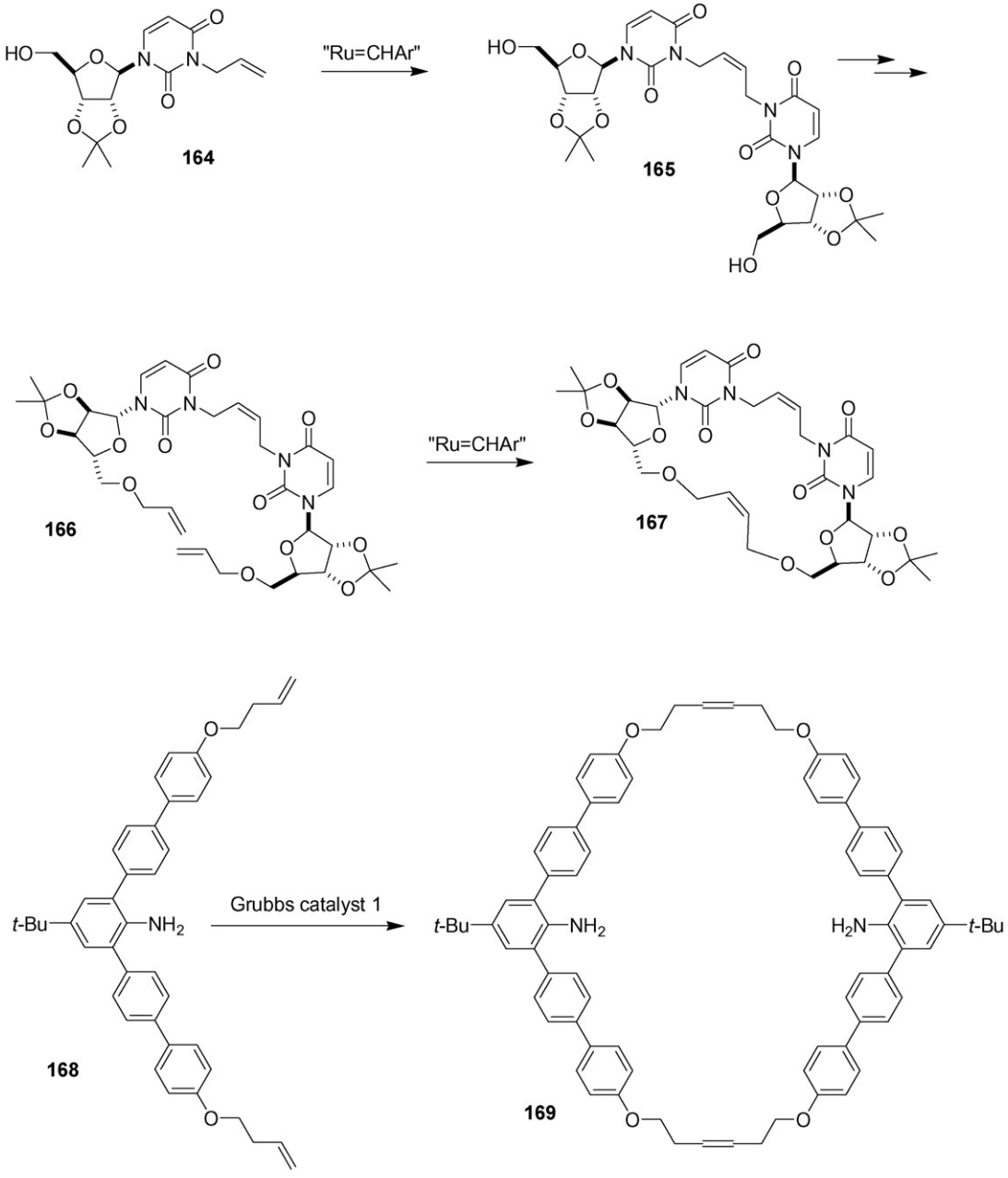


Fig. 6. Representative alkenes subjected to metathesis dimerization.

Several examples of dimerization via metathesis (see Scheme 1) were reported in 2008. Compounds subjected to carbene complex-catalyzed metathesis dimerization are depicted in Fig. 6, and include: (1) allyloxycyclohexane derivatives (*e.g.* **161**) [522]; (2) alkene-diol derivatives (*e.g.* **162**) [523]; (3) metathesis dimerization and cross-metathesis of alkene-containing carbohydrate derivatives (*e.g.* **163**) [524]; and (4) metathesis dimerization of an allyloxyporphyrin followed by cross-metathesis of the dimer with an alkene–peptide [525].

Additional examples feature cross-metathesis in tandem with some other metathesis mode. Examples are depicted in Scheme 12. Tandem dimerization-RCM (metathesis cyclodimerization) leading to macrocycles (*e.g.* **167**) was observed for diallyl nucleosides [526]. Both head-to-head and head-to-tail dimers were reported. A more



Scheme 12.

selective process involves metathesis dimerization of a mono-allyl species (*e.g.* **164**), followed by incorporation of the second alkene and subsequent macrocyclic RCM. A similar metathesis cyclodimerization was employed for the synthesis of rigid macrocycles (*e.g.* **169**) [527]. Preorganized alternatives based on the RCM reaction were also tested. Formation of a cyclic trimer from a bis(porphyrin) tetraalkene was reported [528].

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 2008, 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) cyclopentenes [529–533], including total syntheses of estrone [534], fommanosin (see **172**) [535], otteliones A and B [536], spirotenuipesines A and B (see **173**) [537], cephalotaxine [538], a lactam analog of brefeldin A [539], 4′-epi-formycin [540], cyclopentane core of viridenomycin [541], herbertenones [542], carbocyclic nucleosides [543–548], and conversion of a xylose octadienyl ether to cyclopentene and the corresponding allyl ether [549]; (2) fluorinated cyclopentenes [550]; (3) α,β-unsaturated cyclopentenones (*e.g.* **174**) for total synthesis of carbovir and prostaglandin TEI-9826 [551]; (4) spiro-fused cyclopentenes [552–554], including total synthesis of acorenone [555]; (5) preparation of five- and six-membered ring carbocycles in a one-pot boronation/allylation/RCM sequence [556]; (6) preparation of five- to seven-membered rings in a one-pot allylation-RCM sequence using both a palladium catalyst and Grubbs catalyst II [557]; (7) five- to seven-membered rings fused to γ-lactones and macrocyclic ethers fused to γ-lactones [558]; (8) five- to seven-membered rings spiro fused to various heterocyclic ring systems [559]; (9) cyclohexenes [560–563], including total syntheses of perrottetinene [564], the core structure of branimycin (see **175**) [565], durhamycin A [566], platencin (see **176**) [567,568], quinic acid [569], allosecurinine (see **177**) [570], gusanlung D (see **178**) [571], 6a-carba-β-D-fructose [572], quercitol [573],valienamine [574], and highly oxygenated cyclohexene rings for preparation of various conditurols [575,576]; (10) six-membered ring fluorinated cyclic amino acids [577]; (11) a spiro-fused chlorocyclohexene (**179**) for total synthesis of elatol [578]; (12) tetrahydrophenanthrenes [579]; (13) the carbocyclic ring of hexahydroquinolones [580]; (14) asymmetric synthesis of helicenes (*e.g.* **180**) using chiral

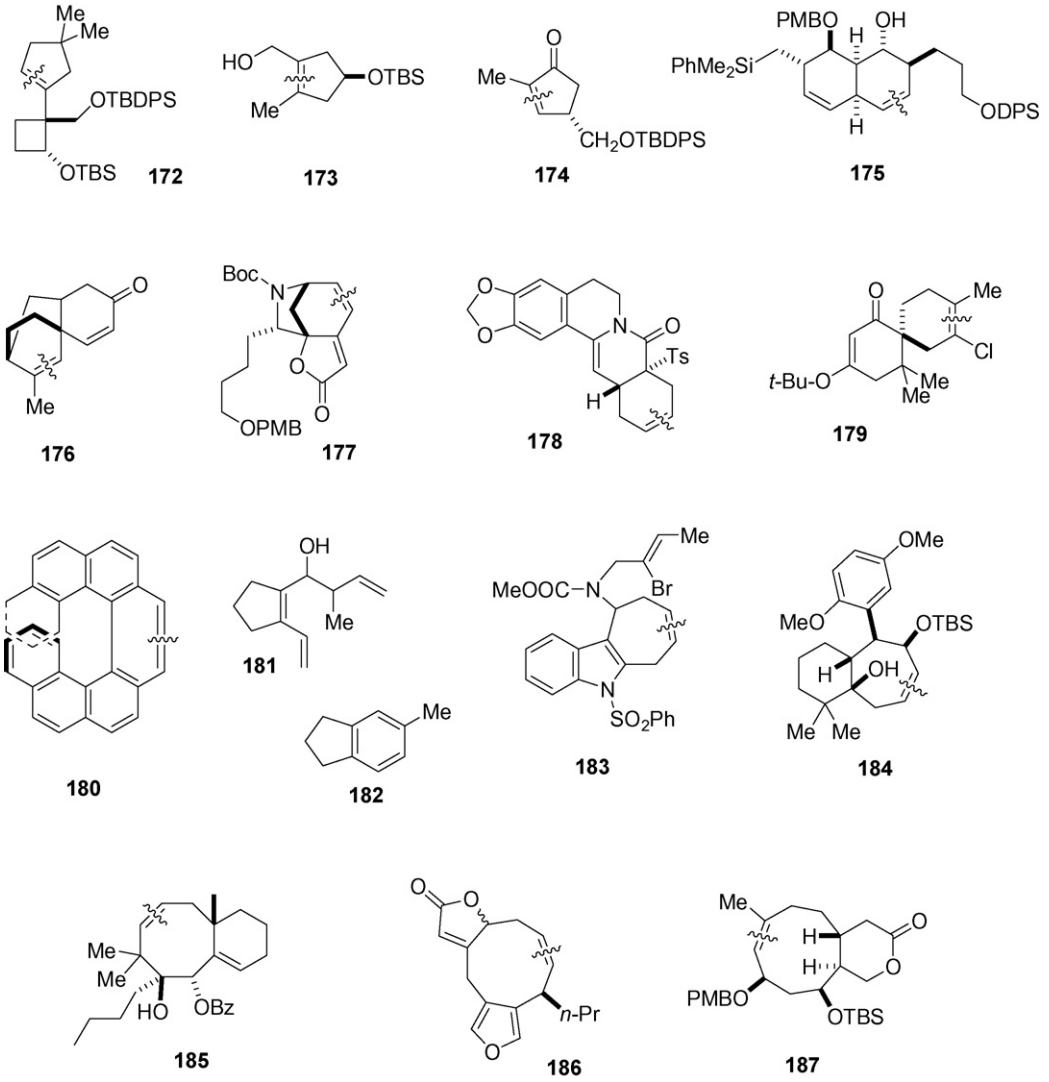
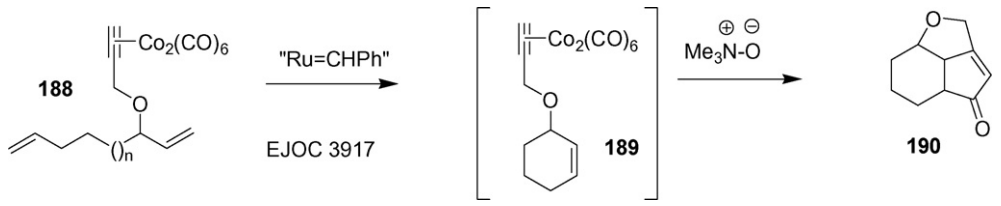


Fig. 7. Representative carbocycles produced through an RCM reaction (bond constructed through RCM indicated).



Scheme 13.

ruthenium–carbene complexes [581]; (15) direct formation of benzene rings (*e.g.* **182**) through RCM of trienols (*e.g.* **181**) [582,583]; (16) α-alkylidenecyclohexenones, which afford phenols upon base treatment [584]; (17) nitrogen-bridged seven-membered rings [585]; (18) an aminocycloheptene derivative for total synthesis of anatoxin A [586]; (19) a highly functionalized cycloheptene for total synthesis of calystegine A [587]; (20) cycloheptenes fused to indole rings (*e.g.* **183**) for synthesis of evistine alkaloids [588]; (21) cycloheptenes spiro fused to tetrahydroisoquinoline rings for synthesis of thiaerythrinanes [589]; (22) hydroazulenes for synthesis of the core of guanacastepenes [590] and confertin [591]; (23) a cyclohexane-fused seven-membered ring (**184**) for total synthesis of frondosins A and B [592]; (24) a tricyclic cycloheptene for synthesis of the core ring system of erinacine E [593]; (25) a cycloheptene ring for total synthesis of cyanthiwigin F [594]; (26) fluorinated eight-membered rings [595]; (27) the eight-membered ring of the taxol B,C ring system (*e.g.* **185**) [596]; (28) carbonyl group bridged cyclooctenes [597]; (29) carbohydrate-bound cyclooctenes [598]; (30) a nine-membered ring (**186**) for total synthesis of 5-epi-hydroxycorneistin [599]; and (31) a nine-membered ring (**187**) for total synthesis of blumiolode C [600]. One-pot tandem RCM–Pauson–Khand reactions were observed in the reaction of diene-cobalt–alkyne complex **188** (Scheme 13) with ruthenium–carbene complexes followed by treatment with amine oxides [601].

Numerous examples of the formation of nitrogen heterocycles using the RCM reaction (Fig. 8) were reported in 2008, including: (1) dihydropyrroles (*e.g.* **191**) [602–606], including stereoselective preparation from precursors that contain diastereotopic vinyl groups [607], and those employed in total syntheses of lentiginosine [608], uniflorine A (see **192**) [609], aphanorphine [610], castanospermine [611], and swainsonine [612]; (2) an α,β-unsaturated five-membered ring lactone (**193**) for synthesis of pyrrolizidine alkaloids [613]; (3) five- and six-membered ring amines for total synthesis of isofagomaine or a five-membered ring analog [614]; (4) five- and six-membered ring cyclic amino acid derivatives

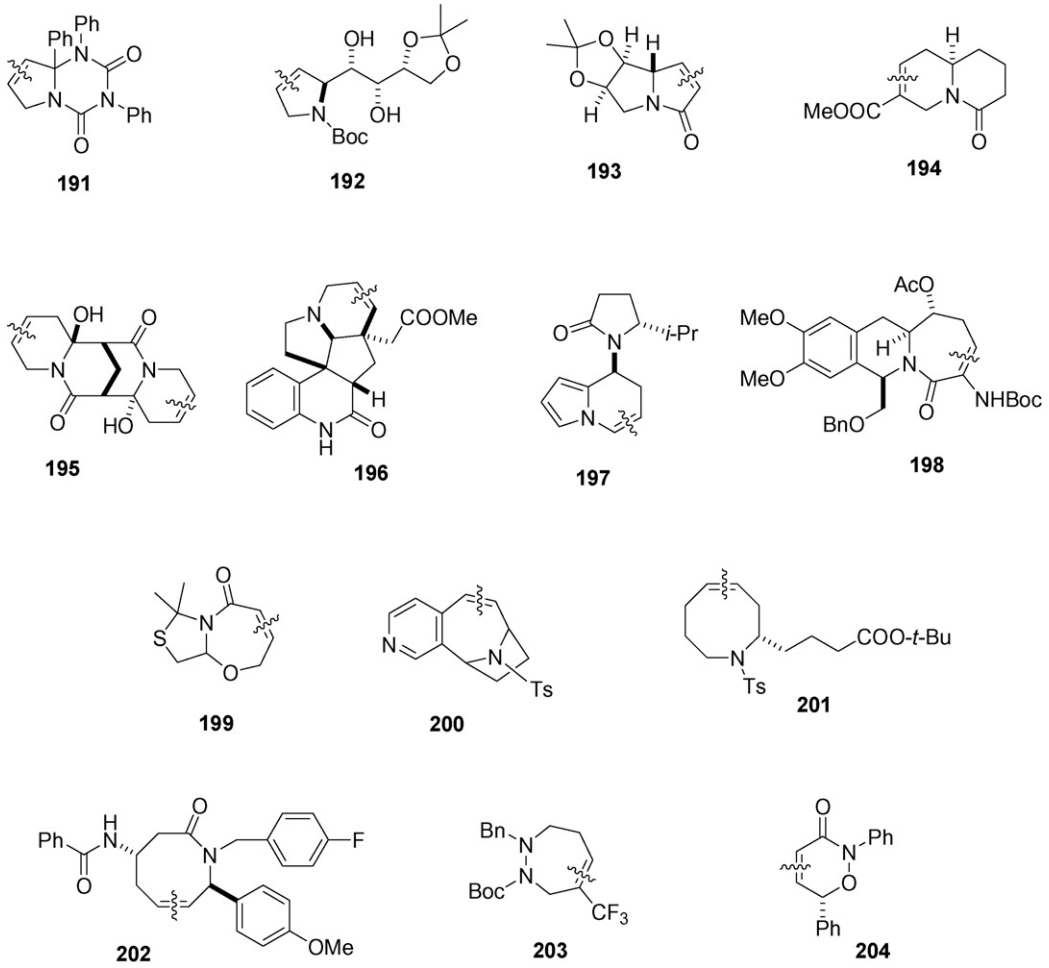
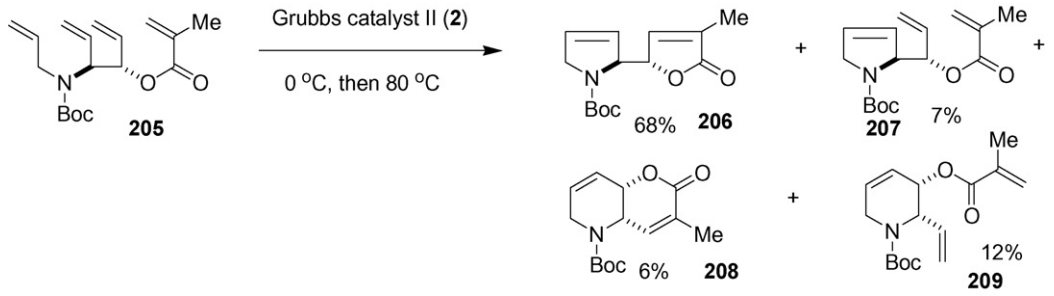


Fig. 8. Representative N-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).



Scheme 14.

[615,616]; (5) five- to eight-membered ring cyclic α,β-unsaturated lactams fused to dihydroindole rings [617]; (6) tetrahydropyridines [618–623], including those employed in total syntheses of dideoxynojirimycin [624] and analogs [625], an approach to halichlorines (see **194**) [626], isospartine (see **195**) [627], naouamine A [628], meloseine (see **196**) [629], hydroxypipecolic acid derivatives [630–632], conhydrine [633,634], epiquinamide [635], and N-tosysedridine [636]; (7) a pyridone ring system using an N-alkoxyamide starting material [637,638]; (8) α,β-unsaturated six-membered ring lactams for total synthesis of coniceine [639] and for azasugars or oxygen analogs [640]; (9) a dihydroindolizine (**197**) for total synthesis of 8-aminoindolizidine [641]; (10) six- to seven-membered ring amines where one of the reactant alkenes contains a perfluorinated alkyl chain [642]; (11) six- to eight-membered ring amides fused to pyrrolidinone rings through either RCM or intramolecular enyne metathesis [643]; (12) seven-membered ring amines [644–646], including those for total synthesis of balanol [647,648] and SB-462795 [649]; (13)

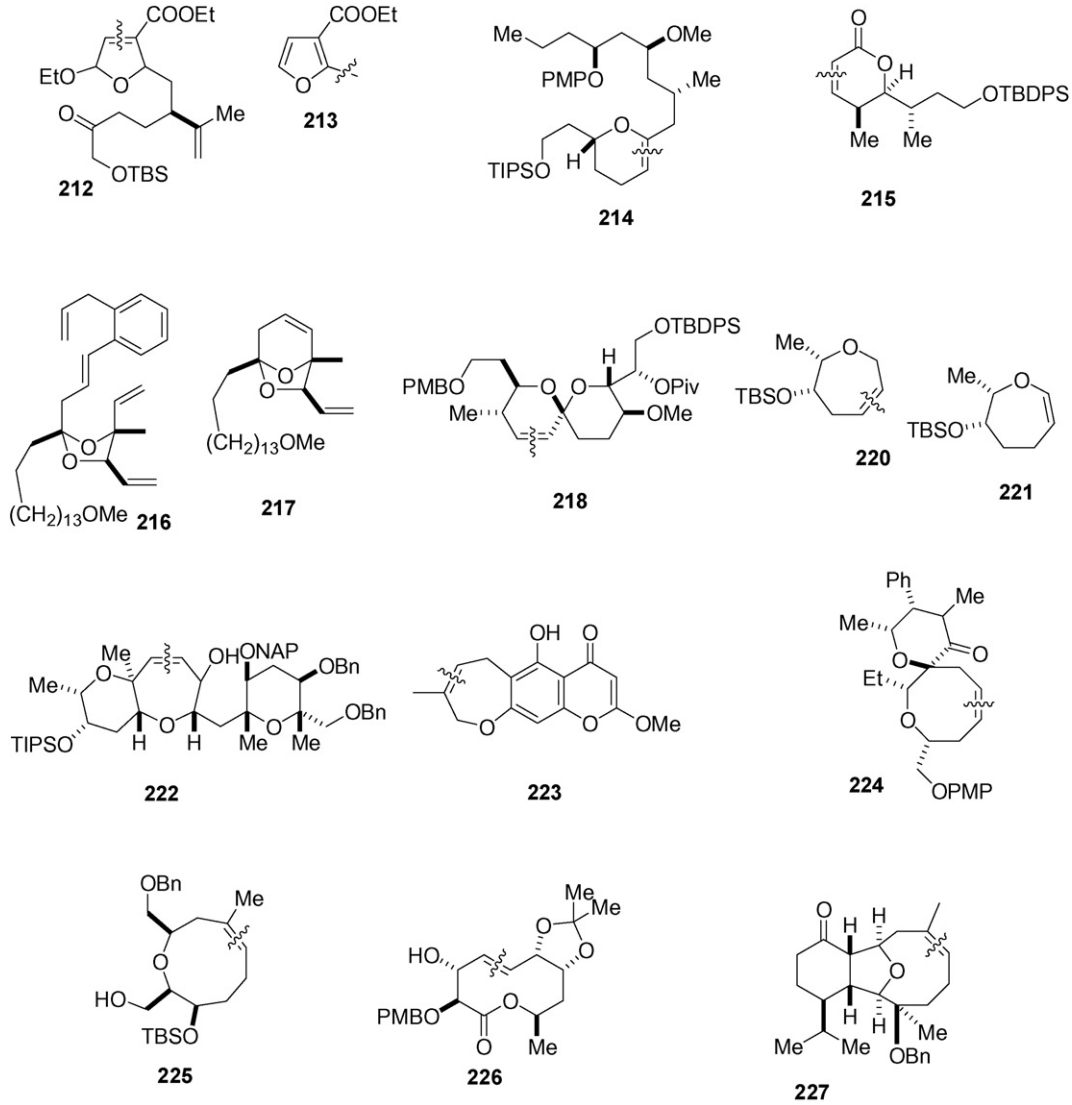
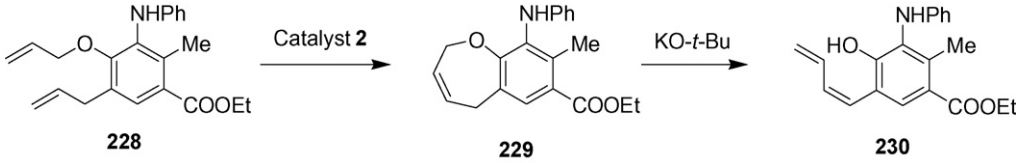


Fig. 9. Representative oxygen heterocycles produced through an RCM reaction (bond constructed through RCM indicated).



Scheme 15.

synthesis of benzazepines [650–652], including preparation of muscarinic receptors [653] and a failed RCM-based approach to this ring system [654]; (14) seven-membered ring-fused tetrahydroisoquinolines (*e.g.* **198**) [655]; (15) seven-membered ring lactams [656,657], including total synthesis of telecagepant [658]; (16) seven-membered ring cyclic amino acids [659]; (17) seven-membered ring O,N-heterocycles (*e.g.* **199**) [660]; (18) seven-membered ring cyclic ureas [661]; (19) an azabicyclo[4.2.1]nonane ring (**200**) for total synthesis of pyrido[3,4-b]homotropane [662]; (20) eight-membered ring cyclic amines [663]; (21) an eight-membered ring cyclic amine (**201**) for total synthesis of epohelmin B [664]; (22) eight-membered rings fused to benzene rings and subsequent alkene isomerization via addition of ruthenium hydrides [665]; (23) eight-membered ring cyclic lactams (*e.g.* **202**) [666]; (24) cyclic hydrazines (*e.g.* **203**) through either RCM or intramolecular enyne metathesis [667,668]; and (25) cyclic N-acylhydroxylamines (*e.g.* **204**) [669]. A double RCM reaction resulting in predominately the dumbbell-shaped two-ring systems (*e.g.* conversion of **205** to **206**, Scheme 14) was employed for the total synthesis of pandamarilactonines [670].

Many diverse oxygen heterocycles were synthesized via the RCM reaction in 2008 (Fig. 9), including: (1) direct formation of a furan (*e.g.* **213**) or pyrrole rings through *in situ* elimination from RCM products (*e.g.* **212**) [671] and total synthesis of Z-deoxypukalide (a later step employs macrocyclic RCM) [672]; (2) α,β-unsaturated five-membered ring lactones for synthesis of bioactive marine butenolides [673] and muracatacin [674]; (3) five-membered ring ethers for total synthesis of furanomycin derivatives [675]; (4) five- and six-membered ring ethers attached to pyrrolidine rings [676]; (5) five- and six-membered ring ethers spiro fused to oxazolidinone rings [677] or cyclopentene rings [678]; (6) six-membered ring ethers [679,680], including those employed in total syntheses of laulimalide [681], neopeltolide (see **214**) [682], kavain and jerangolide D segments [683], and the dihydropyran subunit of sorangicin A [684]; (7) six-membered ring α,β-unsaturated lactones for total synthesis of bitungolide [685], phoslactomycin B [686], pironetin (in a one-pot alkyne trans reduction and RCM) [687], dodoneine [688], biselide E segments [689], a bryostatin segment [690], leustroducsin [691], passifloricin A [692], hyptolide [693], butterfly pheromones [694], cytostatin (see **215**) [695], tanikolide [696], asperlin [697], goniothalamin [698] and analogs [699], the C3–C9 segment of soraphen A1α [700], noviose [701], and various unnamed polyol natural products [702]; (8) benzene-fused six-membered ring esters [703]; (9) diastereoselective formation of a bridged six-membered ring (**217**) in a relay process from tetraene **216** for total synthesis of didemniserinolipid B [704]; (10) spiroketals (*e.g.* **218**) [705]; (11) glycals via an RCM-isomerization sequence [706]; (12) six- and seven-membered ring spiro-fused carbocycles and oxygen heterocycles [707]; (13) six-and seven-membered ring lactones via a domino metathesis route [708]; (14) six- to eight-membered ring allylic ethers (*e.g.* **220**), which isomerize to enol ethers (*e.g.* **221**) upon addition of NaOH/isopropyl alcohol [709,710]; (15) formation of six- to eight-membered ring ethers using relay metathesis [711]; (16) six- to nine-membered ring cyclic ethers of brevitoxin, ciguatoxin, and related marine toxins (see **222**) [712–717]; (17) seven-membered ring ethers and various carbocyclic ring systems [718]; (18) benzoxepins [719–721] and naphthoxepines [722]; (19) a benzo-fused seven-membered ring ether (**223**) for total synthesis of ptaeroxylin [723]; (20) a seven-membered ring lactone for total synthesis of laurallene [724]; (21) a seven-membered ring lactam using Grubbs catalyst II and a hindered aluminum phenoxide additive [725]; (22) seven- and eight-membered rings fused to quinoline rings [726,727]; (23) seven- or ten-membered ring lactones for synthesis of glucopyranosyl serines [728]; (24) eight-membered ring ethers for total synthesis of laurencin (see **224**) [729] and heliannuol A [730]; (25) an eight-membered ring lactone for total synthesis of the octalactin ring system [731]; (26) a nine-membered ring ether (**225**) for total synthesis of 11-acetoxy-4-deoxyasbestinin D [732]; (27) nine-membered ring lactones [733]; (28) ten-membered ring lactones [734], including those used in total syntheses of multipolide A (see **226**) [735], herbarumin III [736,737], vigulariol (see **227**) [738]; and nonenolide [739]; and (30) lactonic amino acid analogs featuring two oxygens in the ring [740]. One-pot RCM and elimination/ring opening was successfully demonstrated (*e.g.* conversion of **228** to diene **230**, Scheme 15) [741].

Heterocyclic compounds involving elements other than N and O were also constructed via the RCM reaction (Fig. 10). Examples include: (1) formation cyclic siloxanes for total synthesis of cis

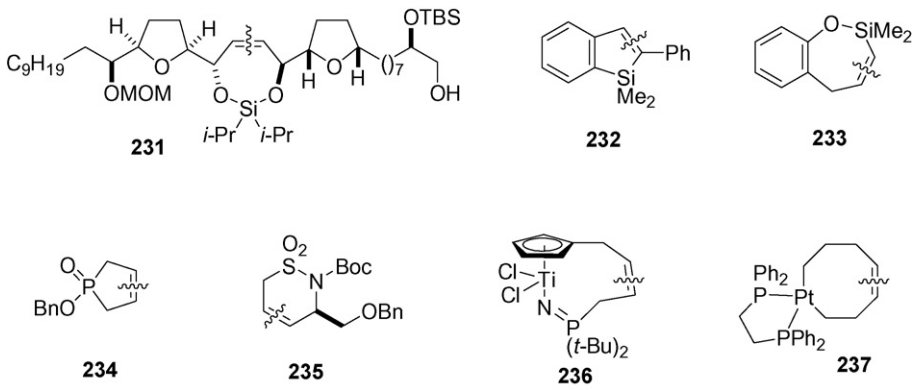
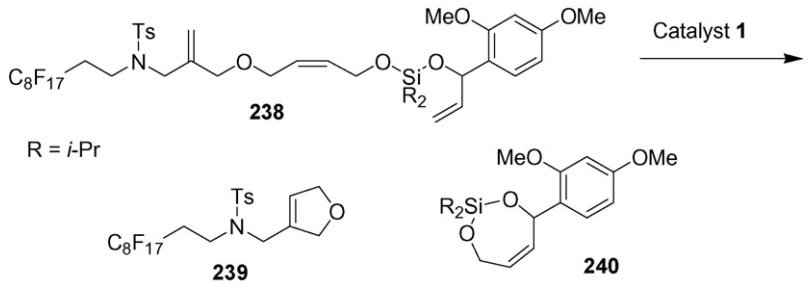


Fig. 10. Representative examples of other heterocycles prepared via the RCM reaction (bond constructed through RCM indicated).



Scheme 16.

sylvaticin (see **231**) [742], amphidinolide X [743], discodermolide segments [744], and a peluroside A fragment [745]; (2) siloles (*e.g.* **232**) [746]; (3) seven- to ten-membered ring benzene-fused silacycles featuring additional O or N atoms in the ring (*e.g.* **233**) [747]; (4) cyclic phosphinic acid esters (*e.g.* **234**) [748]; (5) cyclic sultams (*e.g.* **235**) [749,750]; (6) a chelating titanium–phosphinidine complex (*e.g.* **236**) [751]; (7) platinacycles (*e.g.* **237**) [752]; and (8) failure to form an eight-membered ring cyclic phosphonate due to competing metathesis dimerization [753]. Treatment of fluorous-tagged triene **238** (Scheme 16) with Grubbs catalyst I resulted in simultaneous formation of a fluorous nitrogen heterocycle (**239**) and a cyclic siloxane (**240**) [754].

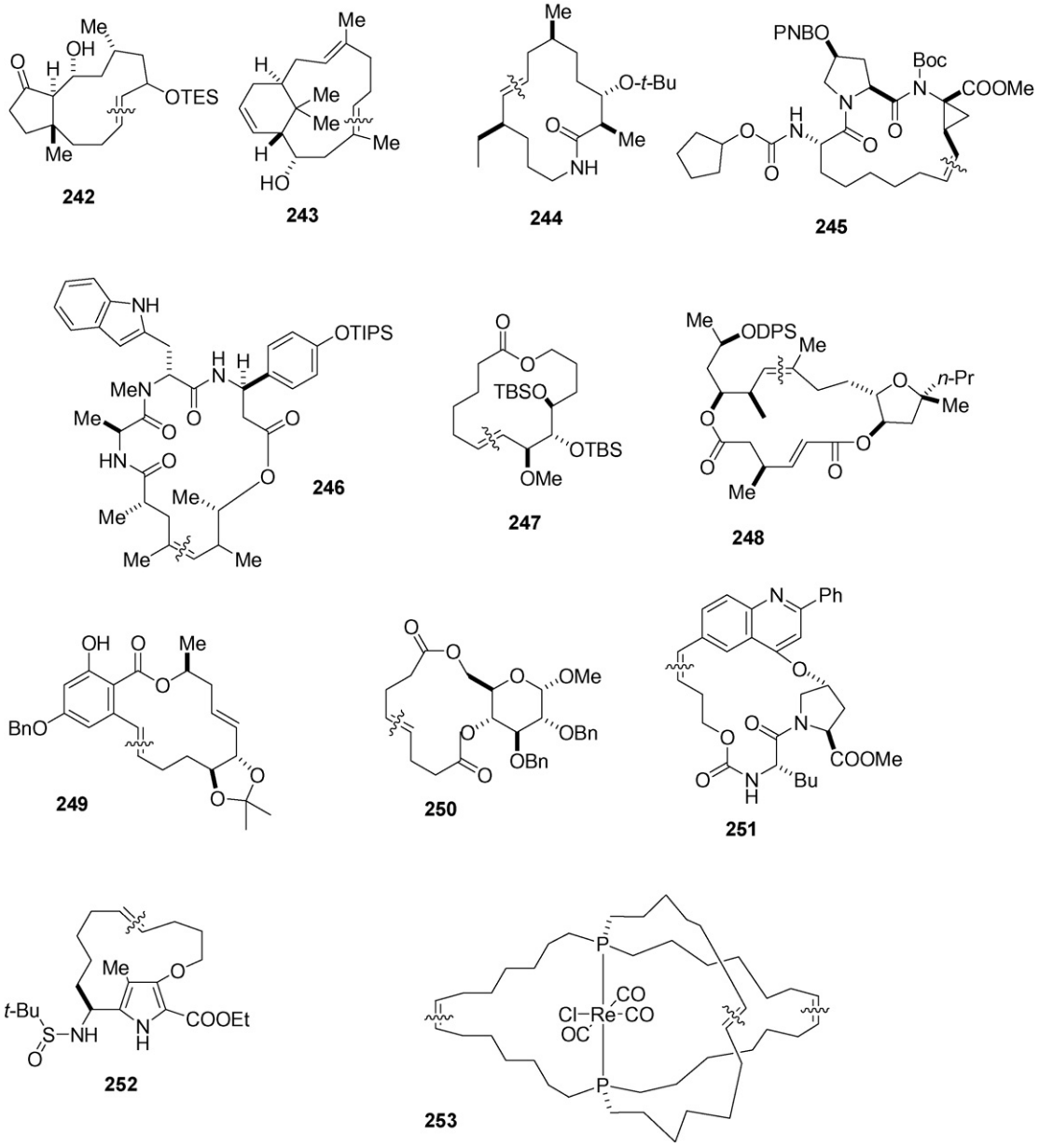
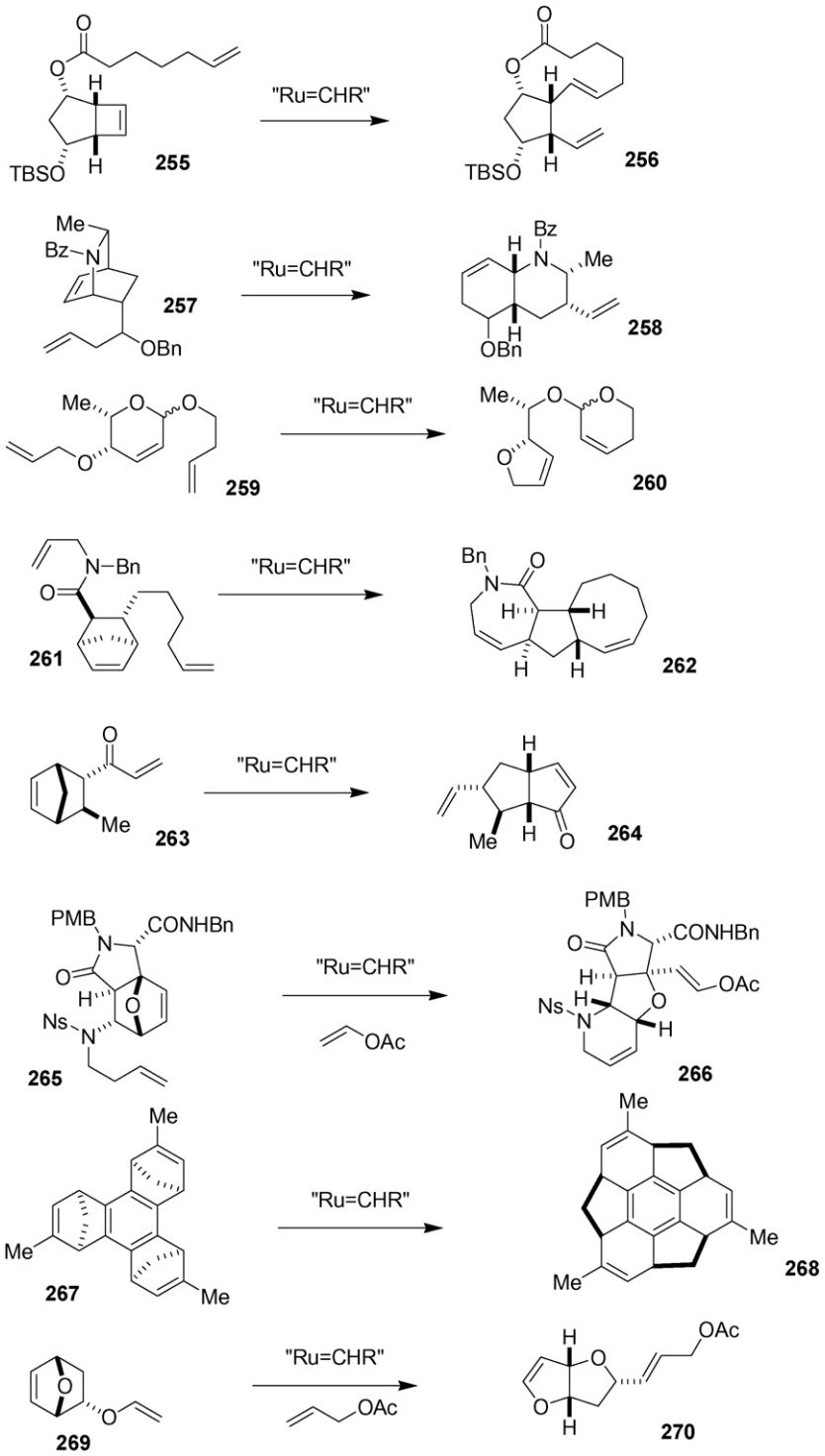


Fig. 11. Representative macrocycles (ring size >10) prepared using the RCM reaction (bond constructed through RCM indicated).

Numerous macrocyclic compounds (rings with ≥11 atoms) were synthesized using the RCM reaction in 2008 (Fig. 11), including: (1) macrocyclic alkenes for total syntheses of clavirolide C (see **242**) [755], a cembranolide [756], and a relay approach to an inside-out meta-bridged cyclohexene derivative (**243**) [757]; (2) crown bis(sulfonamides) [758]; (3) a furan-bridged macrocyclic ketone ring for total synthesis of tonatzitlolone B [759]; (4) a macrocyclic diketone for total synthesis of pinnatoxin A [760]; (5) macrocyclic lactams for total syntheses of fluviricine A1 (see **244**) [761] and excentricine [762]; (6) macrocyclic bis(lactam)s for total syntheses of paliurine E [763], BILN 2061 (see **245**) [764], cytotrienin A [765], TMC435350 [766], various HCV protease inhibitors [767–769], and failure to close the macrocyclic lactone ring of SCH 351448 using RCM [770]; (7) macrocycle-bridged β-lactams [771]; (8) both of the macrocyclic rings of a bis(macrocyclic lactam) inhibitor of hepatitis C protease [772]; (9) a macrocyclic tris(lactam) (**246**) for total synthesis of chondramine C [773]; (10) macrocyclic amino acid derivatives [774,775]; (11) macrocyclic peptide derivatives [776–785]; (12) macrocyclic bis(hydantoin)s [786]; (13) macrocyclic lactones for total synthe-



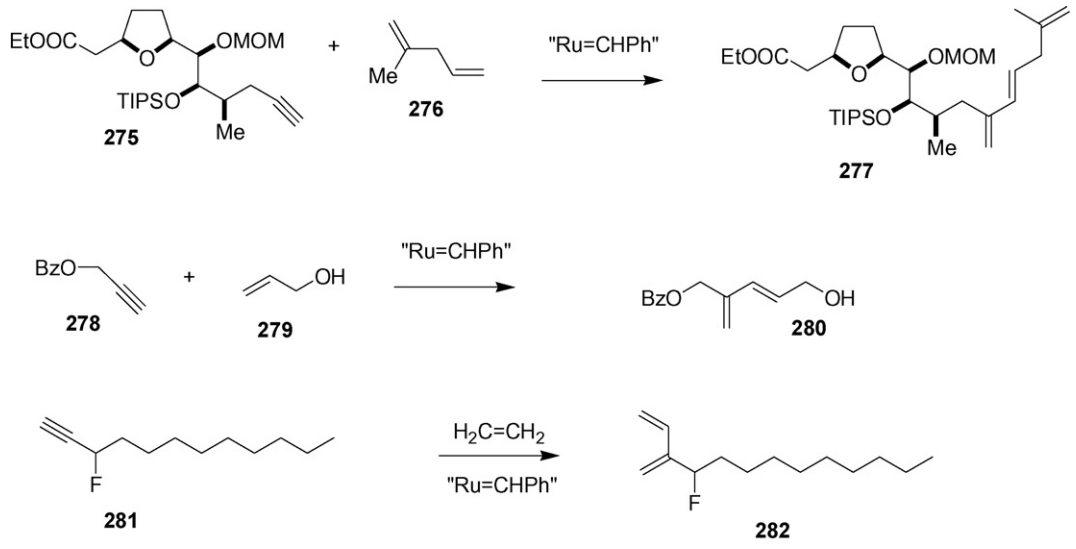
Scheme 17.

ses of migrastatin (see **247**) [787], migrastatin analogs [788], the core ring system of migrastatin [789], palmerolide A (syntheses of both the correct structure and the incorrect originally proposed structure) [790], palmerolide A analogs [791], norbonolide [792], dactyolide (the synthesis also employs CM and six-membered ring lactone-forming RCM) [793], epothilone analogs [794–796], a failed synthetic route to epothilone analogs [797], azaepothilones [798], exigulide [799], pochonin derivatives [800], amphidinolide X (see **248**) [801], the macrocyclic core of amphidinolide H1 [802], curvularin [803,804], salicylhalamide analogs [805]; zearalenone [806], epi-aigialomycin D (the RCM event is stereoselective due to kinetic resolution) (see **249**) [807], cladospolide B [808], an unnamed 12-membered ring keto-lactone natural product [809], patulolide C [810], and various salicylic macrolides [811]; (14) a macrocyclic bis(lactone) for synthesis of the tricyclic core of dictyosphaeric acids [812]; (15) macrocyclic bis- and tris(lactones) [813]; (16) a macrocyclic lactone/lactam for total synthesis of spongidepsin [814]; (17) carbohydrate-fused macrodilactones (*e.g.* **250**) [815,816]; (18) macrocyclic urethanes (*e.g.* **251**) for synthesis of hepatitis C inhibitors [817]; (19) macrocycle-bridged taxol analogs [818]; (20) a *p*-cyclophane using a relay approach [819]; (21) a *p*-cyclophane synthesis that exploits π-stacking interaction to introduce favorable conformational bias in the RCM event [820]; (22) a furan bridged by a macrocyclic allene as an approach to cortistatins [821]; (23) ansa-pyrroles (*e.g.* **252**) [822]; (24) a failed attempt to form a bridged pyrazole through RCM [823]; (25) a macrocyclic hexa-oxazole [824]; (26) a macrocycle bridging two 1,4-phenylenediamine-linked monosacharrides [825]; (27) macrocycle-linked bis(porphyrins) [826,827]; (28) a macrocycle-bridged tetraaza-anthracene derivative for eventual catenane synthesis [828]; (29) calixarenes bridged through a macrocyclic bis(urea) [829,830]; (30) pseudorotaxanes [831]; (31) catenanes [832,833]; (32) tris(catenane)s [834]; (33) gold-templated catenanes and rotaxanes [835]; (34) molecular knots [836]; (35) formation of a covalent linkage between self-assembled porphyrin molecules [837]; and (36) formation of a two-ring polymer from a four-alkene-capped polymer [838]. Threefold RCM was demonstrated for bis(phosphine) complexes where the phosphine ligands contain remote alkene groups, resulting in interligand-bridged metal–phosphine complexes (*e.g.* **253**) [839,840]. A patent was awarded for synthesis of macrocyclic HCV protease inhibitors via RCM [841].

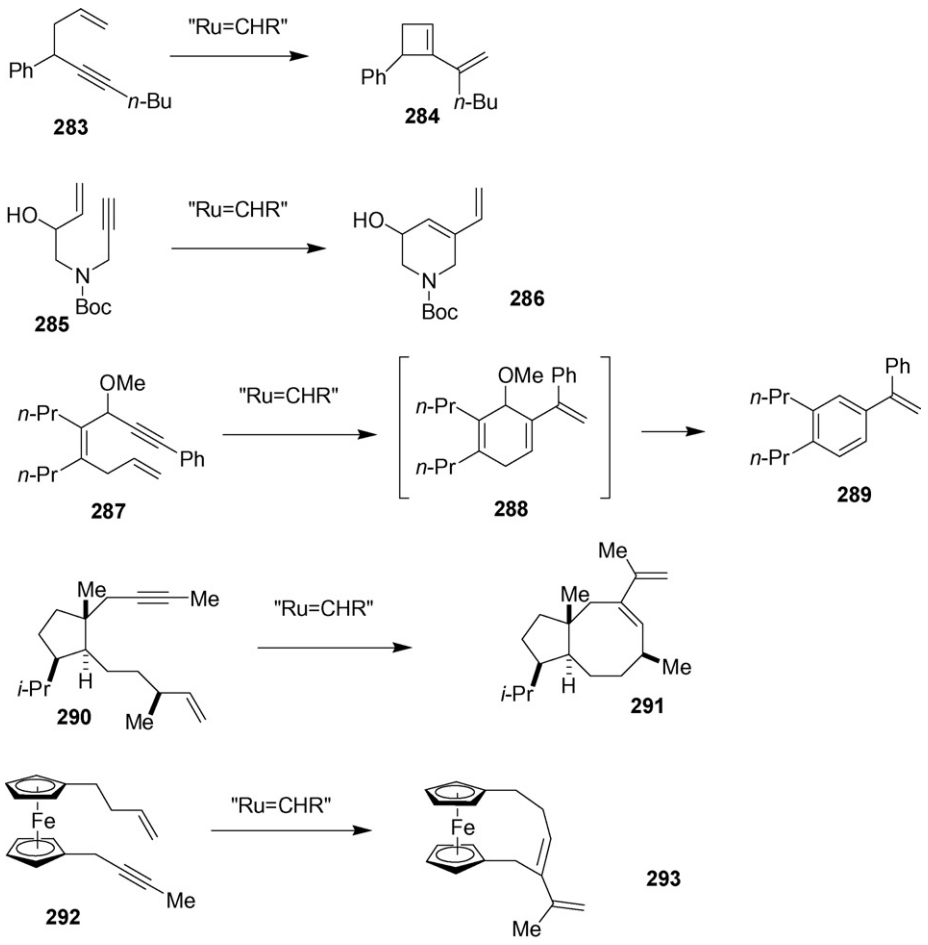
Several examples of ring rearrangement metathesis (RRM) were reported in 2008 (see Scheme 17). These examples include: (1) preparation of prostaglandin derivatives through either RRM or RO-CM (*e.g.* conversion of **255** to **256**) [842]; (2) preparation of lepadin B using RRM of an azabicyclo[2.2.2]heptene derivative (conversion of **257** to **258**) as a key step [843]; (3) rearrangement of alloxydihydropyran derivatives (*e.g.* **259**) to isomeric dihydropyrans (*e.g.* **260**) [844] and a tandem simultaneous preparation of a tris(dihypropyran) system from a bis(dihydropyan) precursor [845]; (4) preparation of cyclooctene-fused cyclopentenes (*e.g.* **262**) as part of a diversity-oriented approach to antibodies (RO-CM reactions were also investigated) [846]; (5) preparation of bicyclo[4.3.1]decadien-10-one derivatives [847]; (6) preparation of the umbellalactol core through RRM of spirolactone-fused norbornenes [848]; (7) preparation of a bicyclo[3.3.0]octenone derivative (**264**) for total synthesis of aburatubolactam A through RRM and a later stage cross-metathesis event [849]; (8) rearrangement of oxanorbornenes to six- to eight-membered ring heterocycles fused to a dihydrofuran ring [850]; (9) double RRM reactions of oxanorbornenes containing two allyloxy groups or two acrylate ester groups [851]; (10) preparation of the hydroazulene ring system through RRM of a pentenylnorbornene derivative [852]; and (11) preparation of tricyclic compounds (*e.g.* **266**) from ring-fused oxanorbornenes (*e.g.* **265**) [853]. The threefold RRM of benzonorbornene derivative **267** was a key step in the asymmetric preparation of chiral buckeyballs [854]. Tandem RRM/CM of vinyloxy-oxynorbornenes (*e.g.* **269**) was employed for synthesis of furanofuran derivatives (*e.g.* **270**) [855].

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 using carbene complexes were reported in 2008. Examples of enyne CM are depicted in Scheme 18 and include the following coupling partners: (1) alkyne **275** and diene **276** for total synthesis of amphidinolide E [856]; (2) terminal alkynes (*e.g.* **278**) and alkene-alcohols (*e.g.* **279**) [857]; (3) terminal alkynes and methylenecyclobutanes [858]; and (4) propargylic fluorides (*e.g.* **281**) with ethylene [859]. Examples of intramolecular enyne metathesis are depicted in Scheme 19 and include formation of the following ring systems: (1) four-membered rings (*e.g.* **284**) [860]; (2) five-membered ring α,β-unsaturated lactams [861];

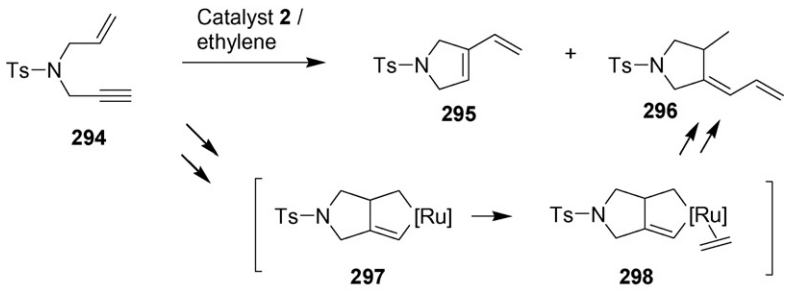


Scheme 18.

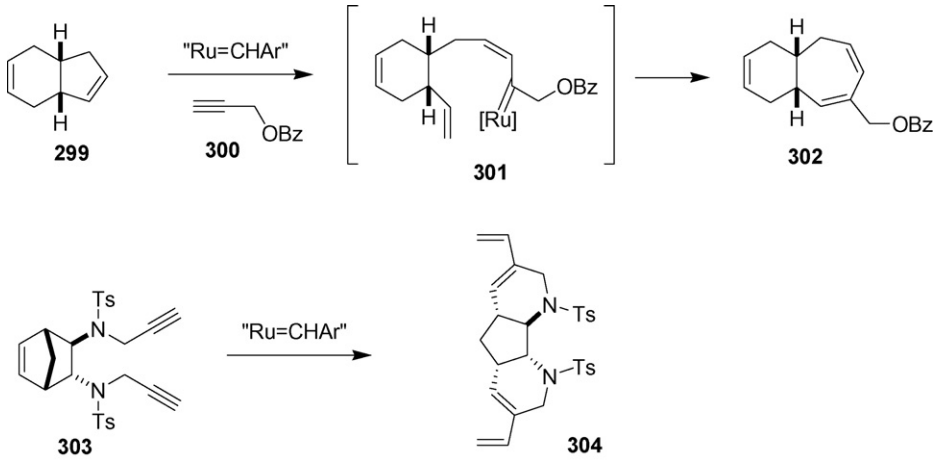


Scheme 19.

(3) five- and six-membered ring cyclic amines [862]; (4) fluorinated dihydropyridines [863]; (5) a six-membered ring (**286**) for total synthesis of isofagomine and evaluation of the acceleration effect of allylic hydroxyl groups on the efficiency of intramolecular enyne metatheses in the absence of an ethylene atmosphere [864]; (6) a highly oxygenated six-membered ring for preparation of angelmicin B segments [865]; (7) a six-membered ring using a conjugated alkynone starting material as part of a synthetic approach to rhodexin A [866]; (8) cyclic N-acetoxyamines [867]; (9) direct formation of a benzene ring (*e.g.* **289**) through a one-pot enyne metathesis (forming **288**) and methanol elimination sequence [868]; (10) vinyl-tetrahydropyridines [869]; (11) a seven-membered ring as part of a synthetic approach to lancifodilactone G (a later step employs cross-metathesis) [870]; (12) seven-membered ring cyclic amino acids [871]; (13) seven- and eight-membered rings fused to a benzene ring for total synthesis of allocolchicine and analogs [872]; (14) an eight-membered ring (**291**) for synthesis of the fusicoccane A–B ring system [873]; and (15) bridged ferrocenes (*e.g.* **293**) [874]. The enyne metathesis of compound **294** (Scheme 20) in ethylene using Grubbs catalyst II led to the enyne metathesis product **295** accompanied by a minor amount of the ethylenation product **296**, obtained through a metallacyclopentene intermediate (**297**) [875]. Non-carbene complex ruthenium catalysts (*e.g.* Cp*Ru(cod)Cl) were more effective catalysts for forming the ethylenation product **296**. Competitive binding of monosubstituted alkene and terminal alkyne groups to Grubbs catalyst I was studied through fluorescent resonance energy transfer [876]. It was determined that binding to the alkene is faster, thus supporting the "alkene-first" mechanism for enyne metathesis. Ring expanding enyne metathesis was reported for various cyclopentene derivatives (*e.g.* conversion of **299** + **300** to **302**, Scheme 21) [877]. Ring-rearranging enyne metathesis reactions



Scheme 20.



Scheme 21.

(*e.g.* conversion of **303** to **304**, Scheme 21) were demonstrated for alkynylnorbornenes [878].

Representative examples of tandem enyne metathesis-RCM are depicted in Scheme 22. Tandem enyne metathesis-RCM was employed for the synthesis of a hexahydroisoquinoline (**306**) used in the total synthesis of ent-lepadins F and G [879]. Tandem enyne metathesis-RCM was employed for the preparation of taxol-like eight-membered rings (*e.g.* **308**) [880]. A tandem enyne metathesis-intramolecular Diels–Alder reaction involving enyne **309** and ethylene was employed for synthesis of the taxane skeleton (**311**) [881]. Tandem ring opening enyne metathesis-RCM was also reported for norbornene–alkyne derivatives (*e.g.* **312**) [882]. Failure to form a bicyclo[6.5.0]tridecadiene ring system through tandem enyne metathesis-RCM was noted [883]. A relay metathesis-1,5-carbene rearrangement-cross-metathesis sequence using diene–diyne **314** was employed as a key step in the total synthesis of panaxytriol [884].

Several examples of dialkyne cyclopolymerization (conversion of **322** to **323**, see Scheme 23) were reported in 2008. New molybdenum carboxylate initiators (*e.g.* **321**) were developed [885]. Molybdenum dicarboxylate complexes afford predominantly the β-polymerization products (six-membered rings). Attempts to manually synthesize dialkyne-derived oligomers (*e.g.* **327**, **328**) were also reported [886]. In the coupling of molybdenum–carbene complexes and dienals (*e.g.* **326**), a major side reaction to the desired carbonyl olefination process is competing metathesis, which results in dimers as well as trimers. Addition of trimethylphosphine or quinuclidine to the dimolybdenum species minimized the competing metathesis reaction pathway.

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

Several publications in 2008 report on processes unrelated to metathesis that are initiated by ruthenium–carbene complex catalysts **1**–**5** 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 isomerization of a terminal alkene group of bis(tetrahydropyran) derivative **330** (Scheme 24) using Grubbs catalyst II in methanol was a key step in the total synthesis of clavosilide D [887]. Deliberate alkene isomerization is also often performed in tandem with RCM (see Refs. [709,710]).

Several examples employing traditional metathesis catalysts as cyclopropanation catalysts were reported in 2008 (see Scheme 25). The one-pot RCM-alkene isomerization–cyclopropanation employing diene **332**, ethyl diazoacetate, and Grubbs catalyst II led to cyclopropane-fused heterocycles (*e.g.* **335**) [888]. In this case the active ruthenium species from the metathesis and isomerization events serves as the cyclopropanation catalyst. The one-pot tandem enyne metathesis/cyclopropanation leading to alkenylcyclopropanecarboxylate esters (*e.g.* **338**) was also demonstrated [889].

The reaction of ruthenium–carbene metathesis catalysts with isocyanides (*e.g.* **340**, Scheme 26) was reported [890]. The reaction leads to the tris(isocyanide) complex **340** and the ylide **341**. The analogous reaction with CO led to the ion pair **342**/**343** accompanied by neutral ruthenium carbonyl complexes (*e.g.* **344**). The key step in both reactions is reversible 1,2-shift of the phosphine ligand to form the 14-electron ylide complex (*e.g.* **345**), which reacts with multiple equivalents of the added ligand.

The use of ruthenium metathesis catalysts for the initiation of Karasch-type reactions (see Scheme 27) was reported [891]. Treatment of trichloroacetate ester **346** with various transition metals, including ruthenium–carbene complexes **1**–**2**, leads to the naphthalene derivative **349**. The reaction proceeds by C–Cl bond cleavage and radical cyclization to afford an eight-membered ring lactone (**348**), followed by elimination and decarboxylation.

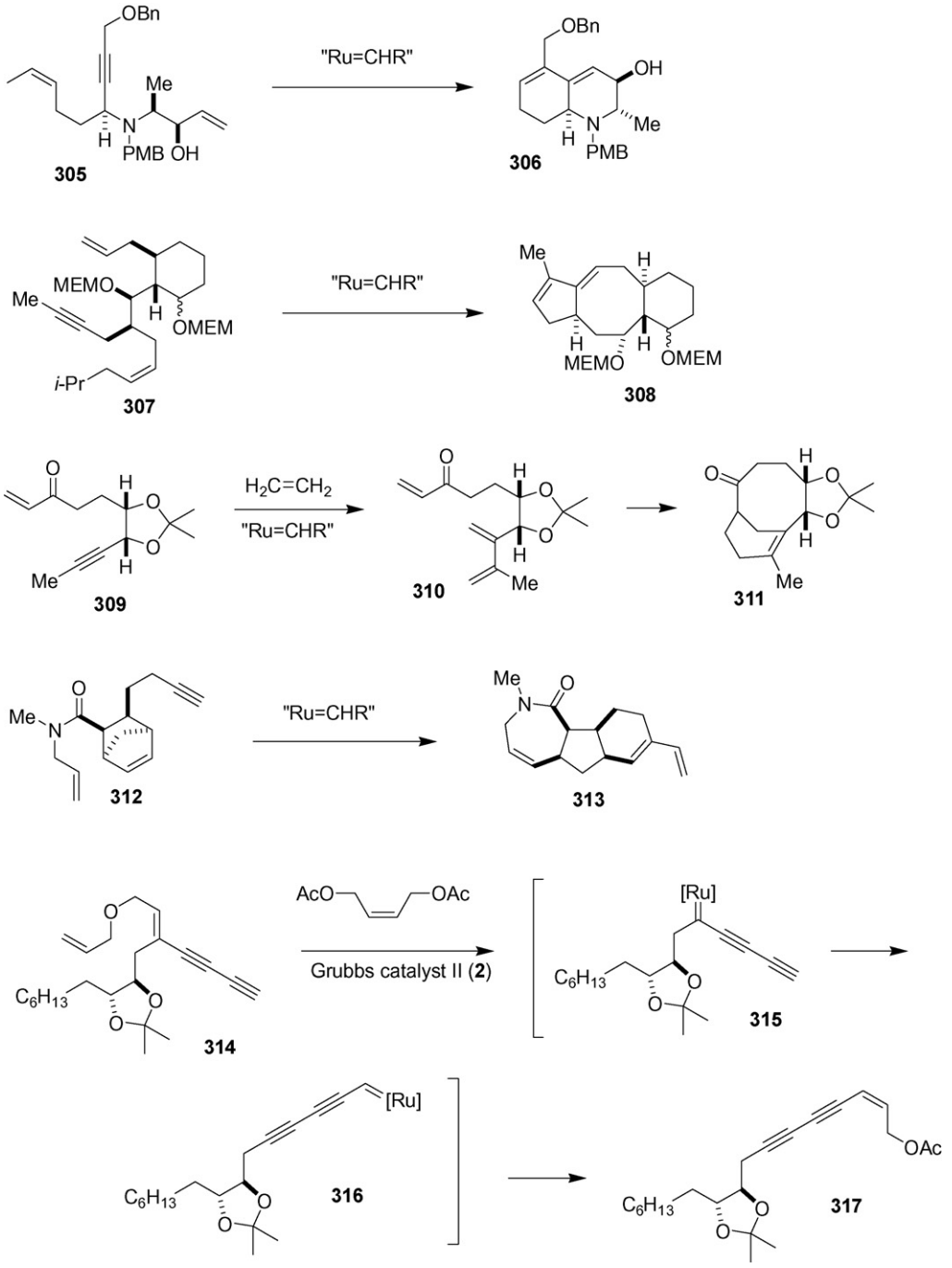
Other alternative uses for ruthenium metathesis catalysts are depicted in (Scheme 28). Grubbs catalyst II is an effective catalyst for the formation of quinolines (*e.g.* **352**) via condensation of 2-aminobenzyl alcohols (*e.g.* **350**) and phenyl ketones (*e.g.* **351**) [892]. The role of the catalyst was attributed to assisting in the redistribution of hydrogen atoms. Grubbs catalyst II and oxygen can initiate diol cleavage reactions (*e.g.* conversion of **353** to **354**) [893].

2.3. Individual carbene or alkylidene complexes classified according to metal

Several studies were reported in 2008 that report on carbene complexes of a variety of groups. A DFT study of co-arctate reactions (conversion of **355** to **356**, Scheme 29) of carbenes and carbene–metal complexes was reported [894]. A DFT study of the bonding in MCH_2 systems was also reported [895]. The degree of aromatic character in several metallabenzenes (resonance form **358** vs. **359**) was determined through analysis of DFT and NMR chemical shift analysis [896]. In general, the 18-electron complexes appear to exhibit the highest degree of aromatic character.

2.3.1. Group 4 metal–carbene complexes

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

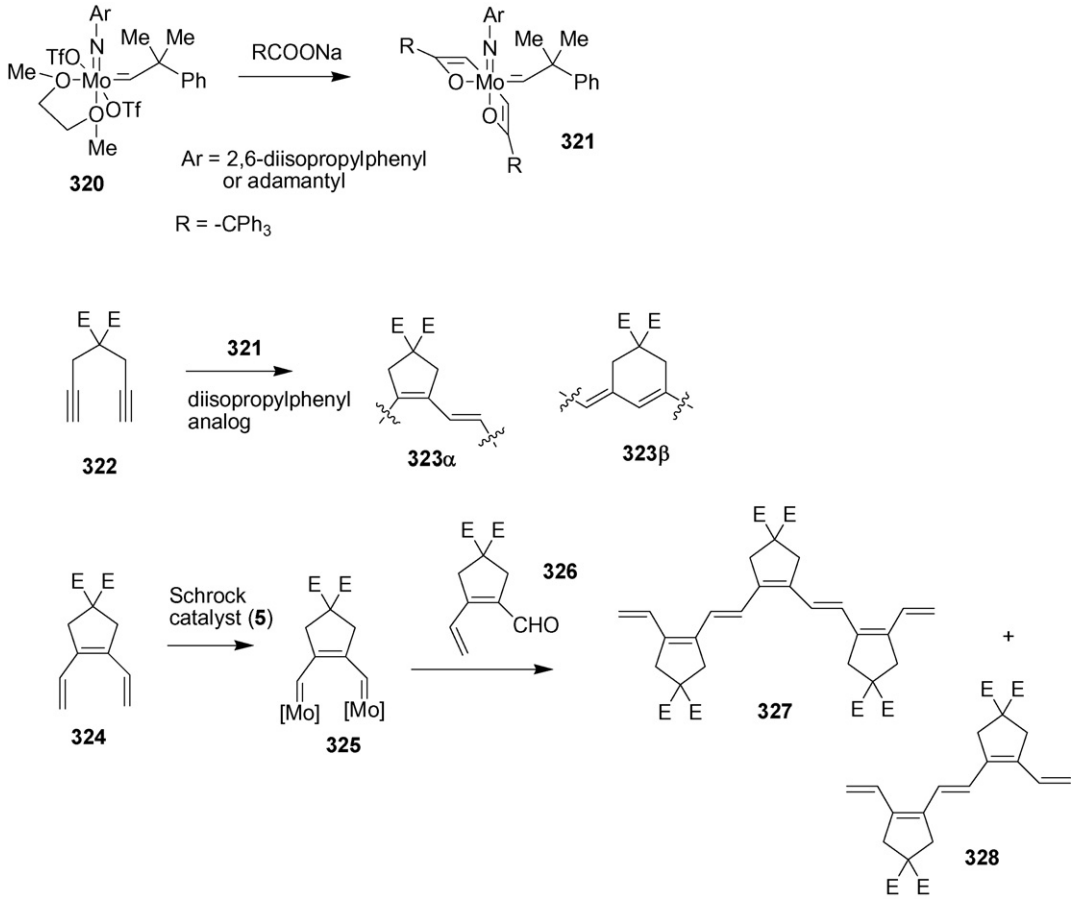


Scheme 22.

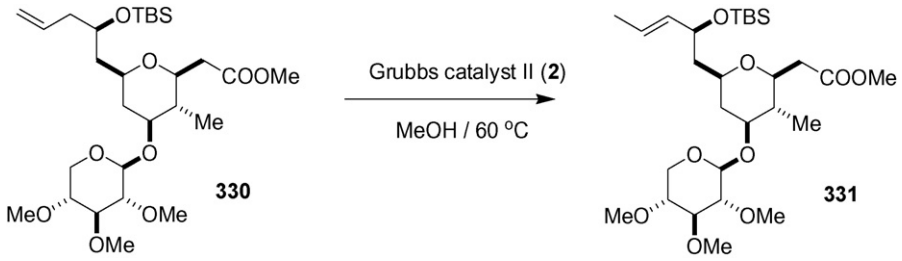
The formation of bis(aluminum)carbene–titanium complexes (*e.g.* **361**, Scheme 30) was reported [897]. These complexes were generated through the reaction of dimethyltitanium complex **360** with trimethylaluminum.

Several examples employing *in situ*-generated titanium–carbene complexes in synthetic organic chemistry were demonstrated in 2008; representative examples are depicted in Scheme 31. Reaction of gem dichlorocyclopropanes (*e.g.* **365**) with titanium(II) complex **366** led to *in situ* formation of the cyclopropylidene complex (**367**), which subsequently coupled with alkynes to provide vinylcyclopropanes (*e.g.* **369**) [898]. Reaction of cyclobutanone dithioacetals (*e.g.* **370**), alkynes and complex **366** led to dienes (*e.g.* **374**) in a process involving carbene complex (**371**) formation, followed by metallacyclobutene formation (**372**), followed by β-hydride elimination and reductive elimination [899]. Complex **366** was also used for carbonyl olefination of lactones (*e.g.* **377**) using dithioacetals that contain a latent hydroxyl group (*e.g.* **375**) [900]. The initially produced enol ether (*e.g.* **378**) was subsequently transformed to a spiroketal derivative (*e.g.* **379**). Similarly generated titanium–carbene complexes (*e.g.* **381**) were instrumental in a solid phase stereoselective synthesis of indole derivatives (*e.g.* **384**) [901].

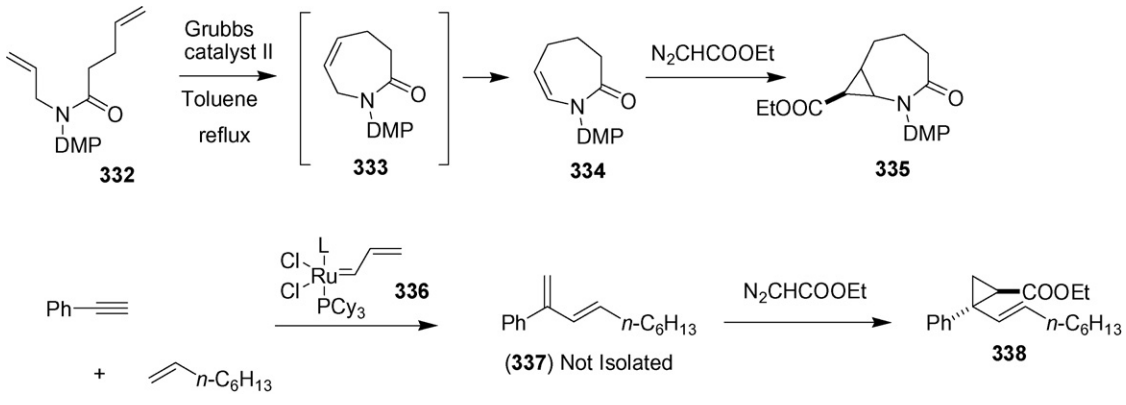
Various carbonyl olefination reactions were accomplished using *in situ*-generated titanium–carbene complexes (see Scheme 32). Methylene enol ether **386** was prepared from the corresponding lactone and the Tebbe reagent (**389**) [902]. Enol ether **386** was immediately subjected to the Claisen rearrangement. Tandem carbonyl group olefination/Claisen rearrangement employing **386** was



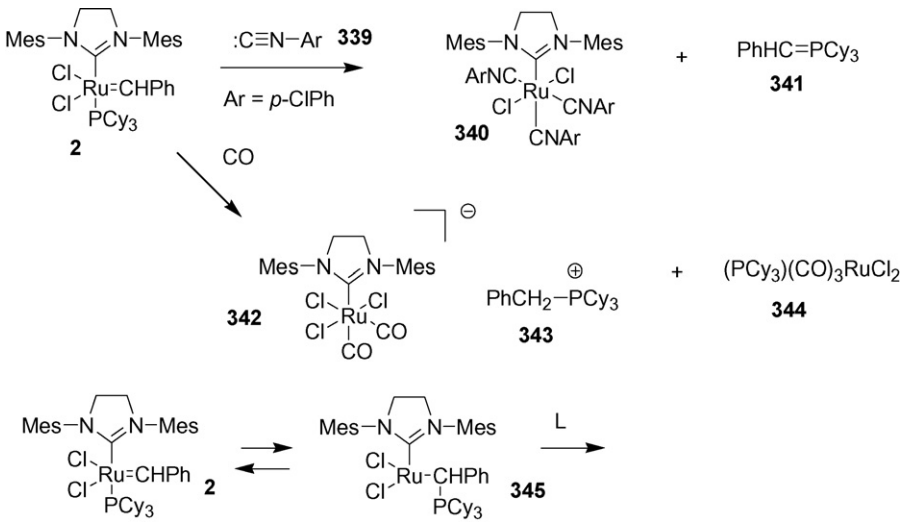
Scheme 23.



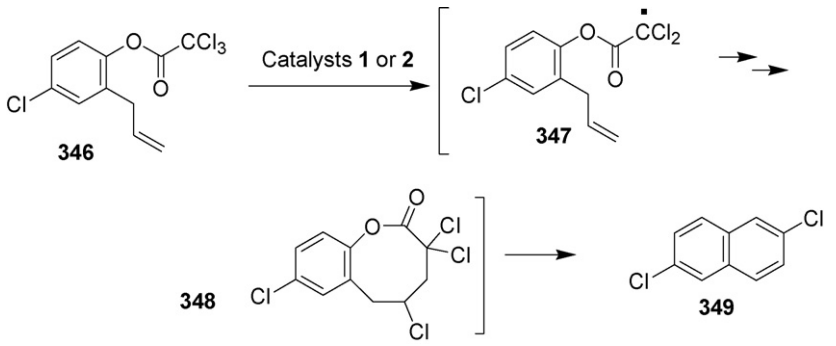
Scheme 24.



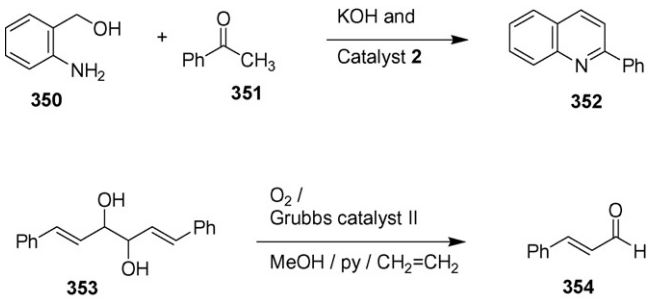
Scheme 25.



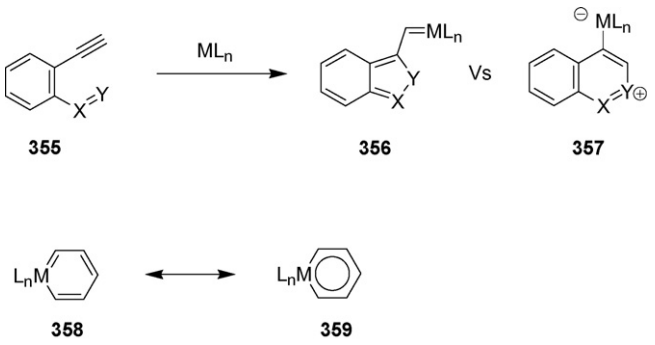
Scheme 26.



Scheme 27.



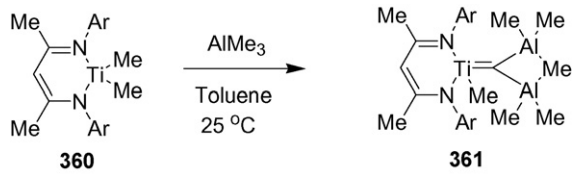
Scheme 28.



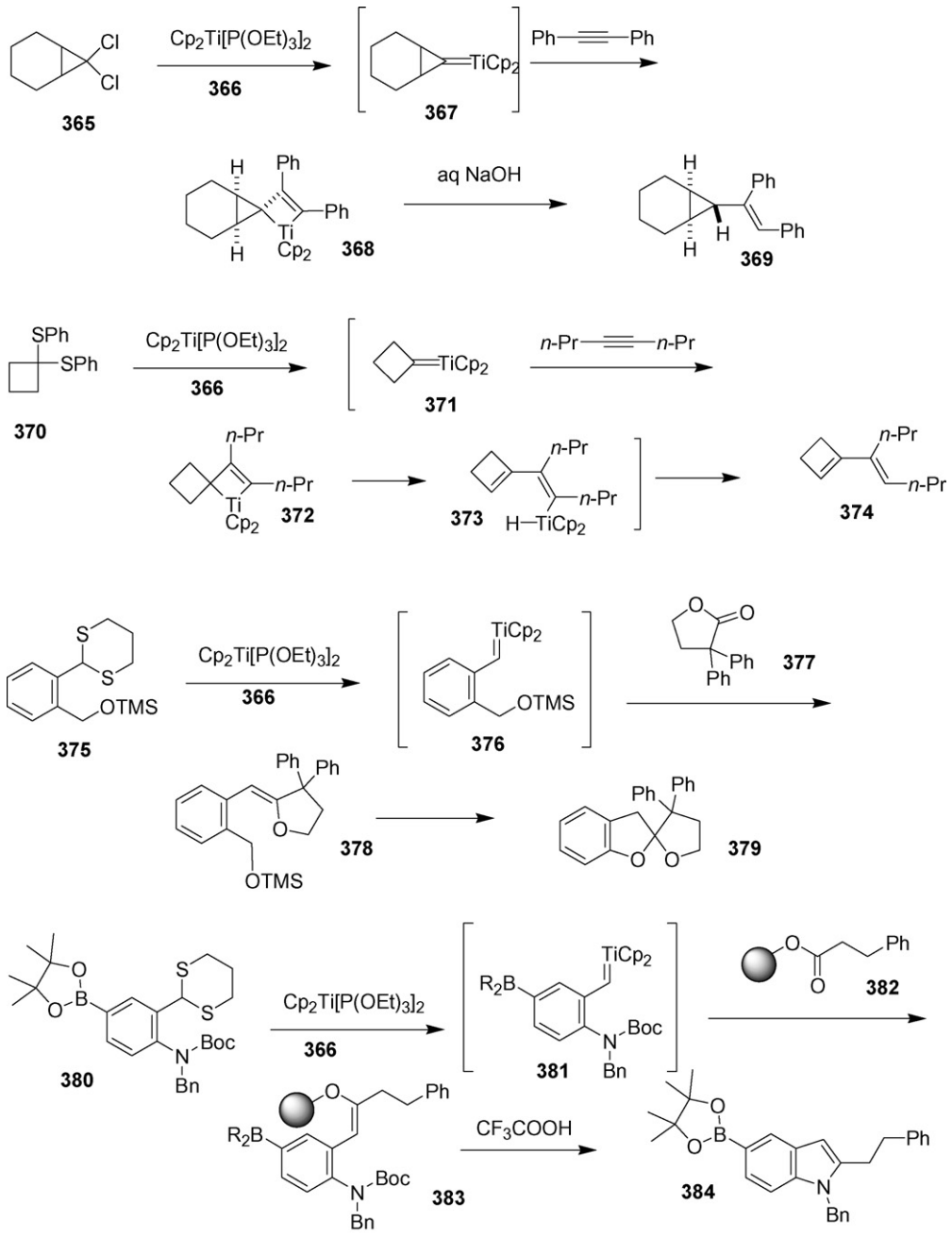
Scheme 29.

a key step part of a total synthesis of obtusenyne. The scope and limit of tandem carbonyl olefination/Claisen rearrangement was examined [903]. Olefination of a ketone or keto-thioester to afford **387** using the Petassis reagent (**390**) is a key early event in the total synthesis of phorboxazole A [904]. Carbonyl olefination of a carbonate derivative to afford ketene acetal derivative **388** was a key step in the total synthesis of various solandelactones [905]. A double carbonyl olefination was employed for the conversion of γ-alkoxy-γ-lactones (*e.g.* **391**) to methylenecyclopentanes (*e.g.* **394**) [906]. In this process, the initially produced enol ether (**392**) undergoes a Ferrier rearrangement under the reaction conditions to afford the cyclopentenone (**393**), which undergoes further carbonyl olefination. Additional examples of employing the Tebbe or Petassis reagents for carbonyl olefination were also reported [402,460,743,883,907–913]. An *in situ*-generated titanium–carbene complex serves as a reagent for the RCM of a ketone and an alkene (*e.g.* substrate **395**) and affords a key intermediate (**396**) for the synthesis of adriatoxin [914].

Titanium–carbene complexes were suggested as intermediates in the epimerization of cationic alkyltitanium complexes obtained



Scheme 30.



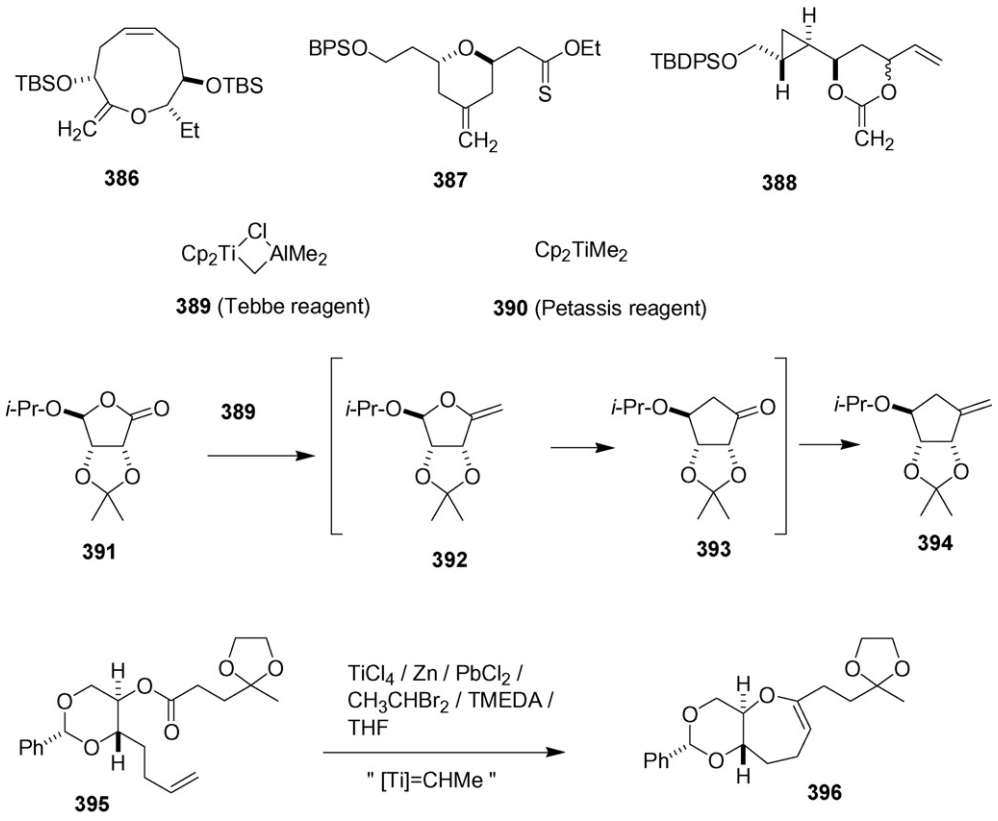
Scheme 31.

through alkene dimerization [915]. An attempt to generate a titanium(III) NHC complex through reduction of a titanium(IV) NHC complex and compare the degree of back bonding was thwarted by the unanticipated formation of a complex polynuclear species where the degree of back bonding is unclear [916]. Matrix effects for $H_2C{=}MH_2$ complexes (M = Ti, Zr, Hf) were evaluated computationally and afforded better agreement with experimentally derived vibrational frequencies than values based on gas phase models [917]. The reaction of laser ablation-generated Group 4 metal atoms with acetylene afforded η^2-alkyne complexes and not vinylidene complexes [918].

2.3.2. Group 5 metal–carbene complexes

Vanadium–carbene complexes (*e.g.* **402**–**405**, Scheme 33) were isolated from the reaction of dialkylvanadium complex **400** with various ligand additives [919]. The higher oxidation state carbene complexes **403** and **404** were isolated from the reaction of the dialkyl complex with diazo compounds, phosphine oxides, nitrous oxide, or sulfur. The dimeric compound **405** was isolated from the reaction with nitrogen or with phosphine imines in the presence of nitrogen. These reactions were proposed to proceed through ligation and α-hydride elimination/reductive elimination to afford a simple carbene complex–ligand adduct (**401**) followed by transformation to the observed products. This initial complex however could not be isolated. Reaction with bipyridyl did result in a stable vanadium(III) alkylidene complex (**402**).

The coupling of vanadium–carbene complexes (*e.g.* **406**, Scheme 34) with various unsaturated organic compounds was reported [920]. Reaction with nitriles led to the nitrile insertion products, vinylimido complexes (*e.g.* **407**). Reaction with alkynes led to stable metallacyclobutenes (*e.g.* **408**). Reaction with styrene led to the metallacyclopropane complex **409**. A

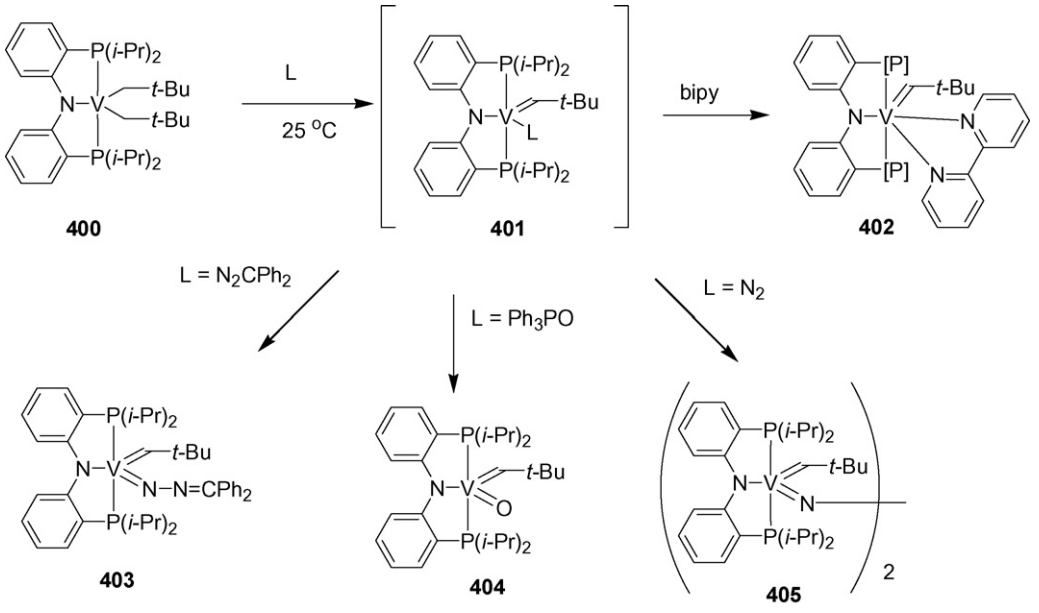


Scheme 32.

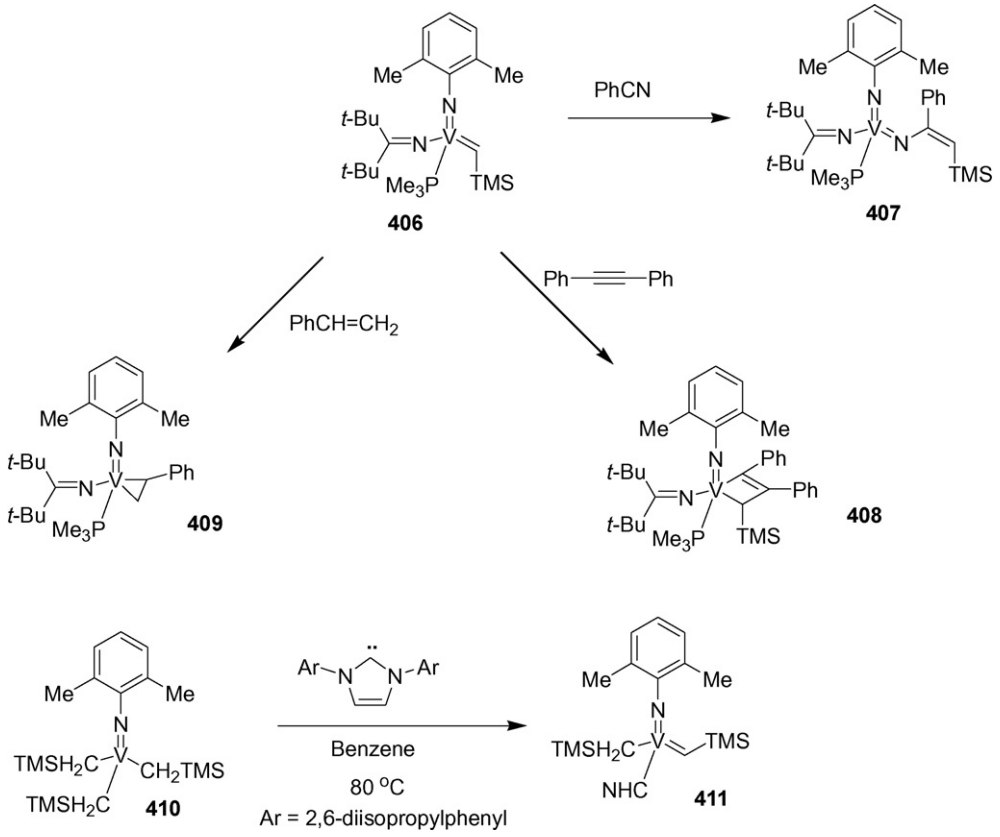
vanadium–carbene–NHC complex (**411**) was generated through thermolysis of complex **410** in the presence of a NHC ligand [921]. Related complexes were used as olefin metathesis catalysts [209,210].

A vanadium–bis(carbene) complex (**413**, Scheme 35) was prepared through reaction of divanadium complex **412** with magnesium in the presence of phenylacetylene [922]. The bis(carbene) complex depicted was determined to be a better representation than the metallacyclopentadiene based on the X-ray structure.

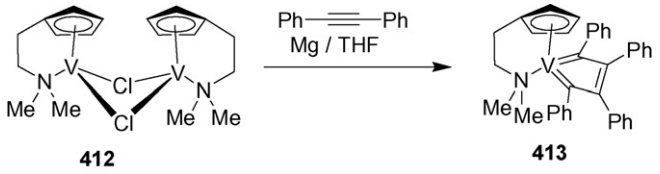
Experimental and computational studies of olefin exchange in (t-$Bu_3SiO)_sM$(alkene) (M = Nb or Ta) complexes and the corresponding rearrangement to carbene complexes (t-$Bu_3SiO)_sM$=CHR were reported (see Scheme 36) [923]. The lowest energy pathway for rearrangement of niobium–alkene complexes (*e.g.* **414**) to the corresponding niobium–carbene complex (*e.g.* **417**) involves dirad-



Scheme 33.



Scheme 34.



Scheme 35.

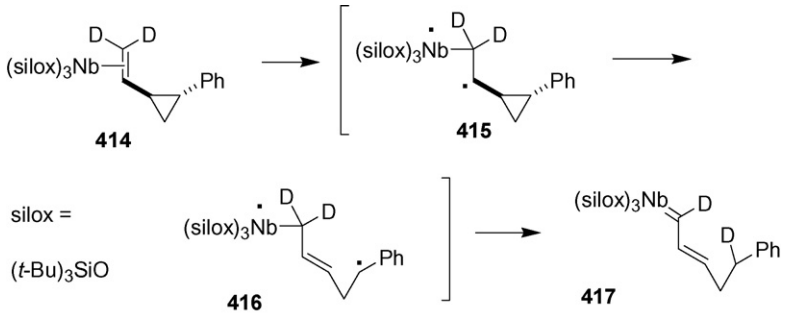
ical intermediates (*e.g.* **415**, **416**). The vinylcyclopropane complex **414** rearranges to the alkenylcarbene complex **417** via the labeling scheme depicted, and is consistent with the computationally predicted mechanism.

A tantalum–benzylidene complex (**419**, Scheme 37) was formed through elimination of toluene from tribenzyltantalum complex **418** [924]. A mechanism involving elimination of toluene followed by coordination of the phosphine ligand was proposed. A spectroscopically observable tantalum–carbene complex (**421**) was generated through deprotonation of phosphinomethyltantalum complex **420** [925]. Niobium–carbene complexes (*e.g.* **423**) were suggested as possible intermediates in the reductive cyclization of 1-trifluromethylbiphenyls (*e.g.* **422**) using niobium metal [926]. Dicationic tantalum–carbene complexes and carbide complexes were identified in the reaction of dicationic tantalum with methane in a guided ion beam mass spectrometer [927]. The structure of the complexes and the mechanism for their formation were also studied by DFT calculations. The argon matrix effect on the geometry and IR spectra of $H_2C{=}MH_2$ (M = Nb or Ta) was evaluated computationally [928].

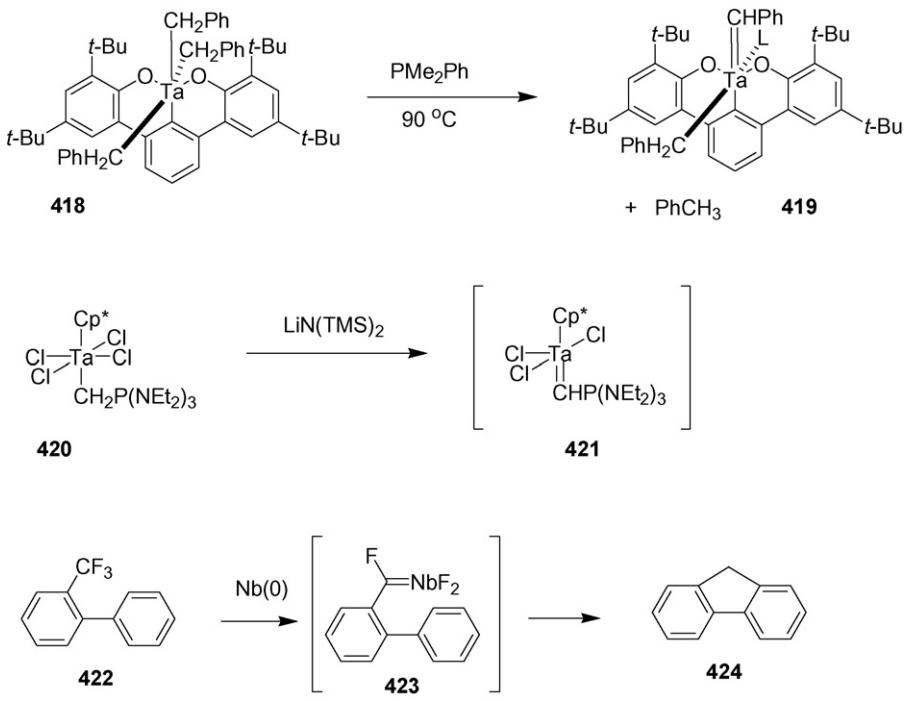
2.3.3. Group 6 metal–carbene complexes (further classified according to structure and reaction type)

2.3.3.1. Schrock-type carbene complexes. A significant portion of this subject material has already been presented in Section 2.2; the Schrock catalyst (**5**) belongs to this class of compounds.

Tethered analogs of the Schrock carbene complex (*e.g.* **430**, **431**, Scheme 38) were reported [929]. Reaction of the



Scheme 36.



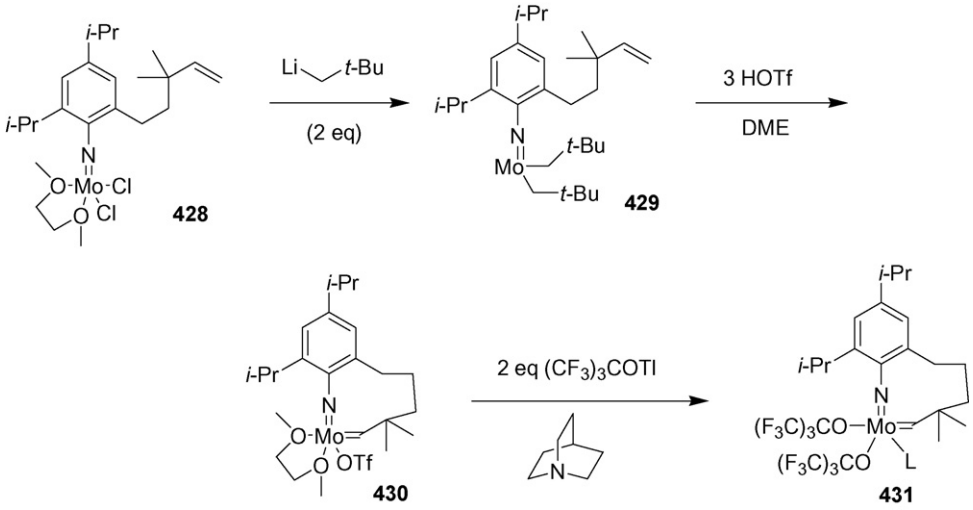
Scheme 37.

bis(neopentylidene)molybdenum complex **429** with triflic acid led to the bridged carbene complex **430**. Various ligand modifications were reported for initially formed carbene complex **430**. Similar complexes devoid of the tether were also prepared for comparison purposes.

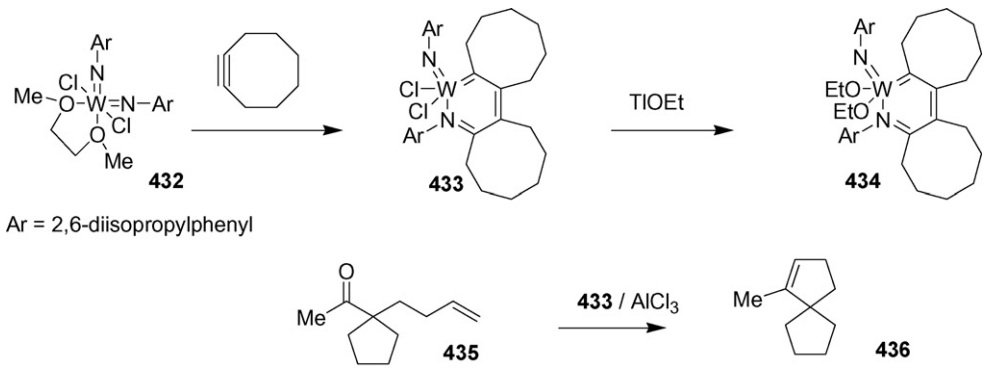
The synthesis and reactivity of tungstapyridine derivatives (*e.g.* **433**, Scheme 39) was reported [930]. Tungstapyridine **433** was prepared from reaction of bis(imido)tungsten complex **432** with strained alkynes (*e.g.* cyclooctyne). Various ligand exchange processes were reported for complex **433**. The ability of the complex to mediate carbonyl-alkene metathesis (*e.g.* conversion of alkene ketone **435** to cycloalkene **436**) in the presence of various Lewis acids and salts was also noted.

Hypothetical reaction pathways for carbene complexes of general structure **437** (Scheme 40) were explored computationally [931]. Potential rearrangements and their addition reactions to ethylene were considered. For molybdenum complexes, the kinetically and thermodynamically most favorable reaction pathway with ethylene is [2+2]-cycloaddition of ethylene and Mo=CH_2 to afford the metallacyclobutane (**438**). The [3+2]-cycloaddition product, the Mo=O cycloaddition product, and cyclopropanation were disfavored both kinetically and thermodynamically. In the chromium case the reaction was far more complex. The most favored process is isomerization to the agostic complex **439** followed by complexation of an additional ethylene molecule, leading to complex **440**.

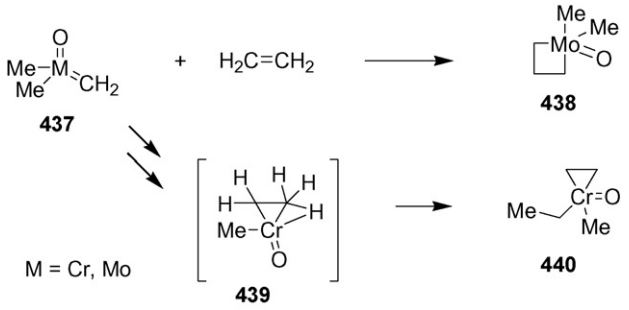
A chromium–carbene complex (**443**, Scheme 41) was generated during thermal decomposition of silica-bound dialkylchromium complex **441** [932]. Although the carbene complex could not be thoroughly characterized due to its paramagnetic nature, carbene-like reactions were successfully demonstrated. The complex reacts with styrene to afford a metathesis product, alkene **444**, and with HCl to form a chromium dichloride and neopentane.



Scheme 38.



Scheme 39.



Scheme 40.

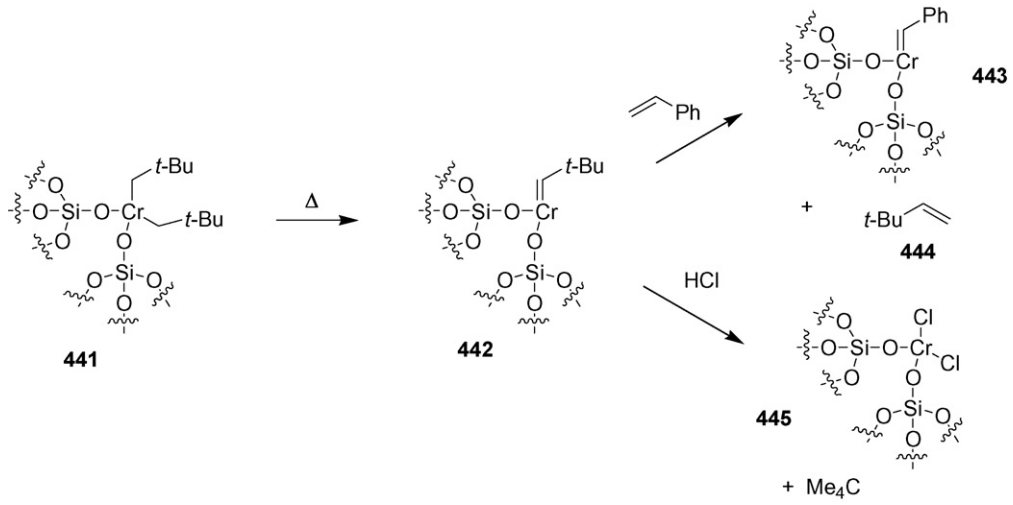
Additional reports involving high oxidation state Group 6 metal–carbene complexes appeared in 2008. Tungsten–carbene complexes were identified through an NMR study of alkyne polymerization reactions initiated by $Na_4[W_2Cl_8(THF)_x]$ and $[W_2Cl_4(PMe_3)_4]$ [933]. Chromium alkylidene complexes were noted as likely species formed upon thermolysis of silica-bound tetraneopentylchromium(IV) [934].

2.3.3.2. Publications focusing on synthesis, formation, or physical properties of Fischer carbene complexes of Group 6 metals. The most common procedure used for the synthesis of Group 6 metal–carbene complexes is the Fischer synthesis, which involves coupling of an organolithium reagent with a Group 6 metal carbonyl derivative, followed by alkylation of the resulting acylate. A variety of Fischer carbene complexes containing one or more additional other metal groups (*e.g.* **448**, **451**, Scheme 42) were prepared using the Fischer synthesis [935]. Various ferrocenylcarbene–Group 6 metal complexes were synthesized and subjected to electroplating conditions [936]. Group 6 metal–carbene complexes (*e.g.* **454**) were prepared using a Fischer-like route [937]. Treatment of 1-lithio-4-halobutadiene derivatives (*e.g.* **453**) and Group 6 metal hexacarbonyls directly affords the carbene complex. Several transformations were reported for the pyranylidene complexes, including oxidation to pyrones (*e.g.* **455**), reduction to afford cyclopentenones (*e.g.* **456**), or conversion to thiopyrones (*e.g.* **457**).

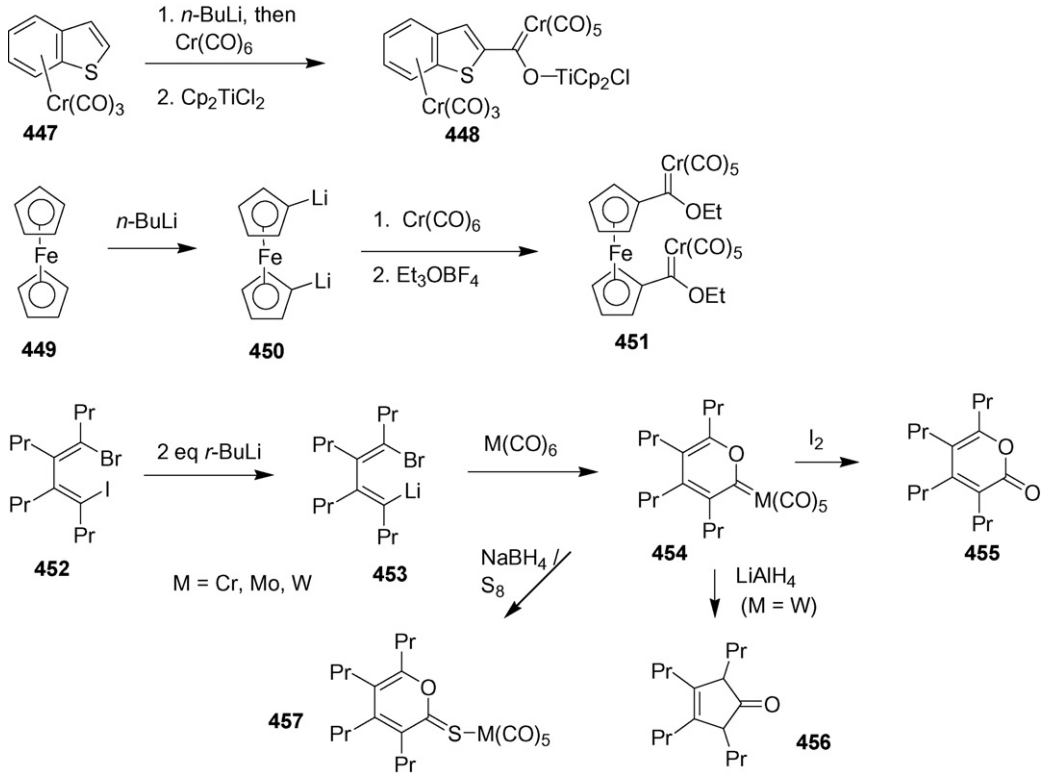
Synthesis of a variety of axially chiral aminocarbene complexes (*e.g.* **461**, **462**, Scheme 43) was reported [938]. These complexes were prepared from the correspond amide via the dianion route. Complex **461** was prepared as a single diastereomer (due to axial chirality) and even after removal of the chiral auxiliary (formation of **462**) configurational stability up to 70 °C was observed.

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).

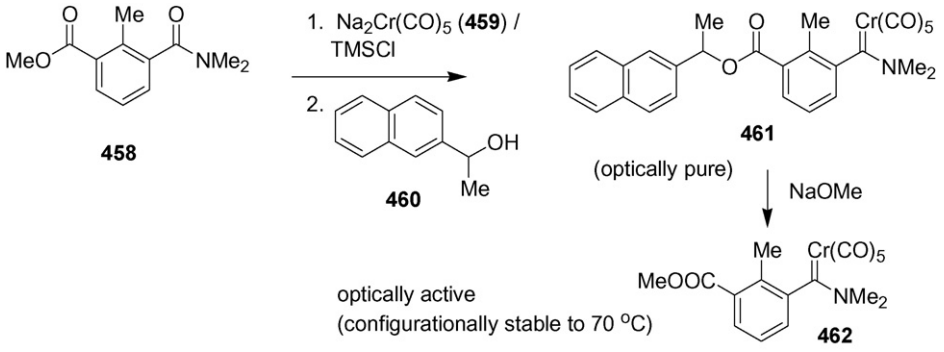
Cyclopropanation reactions involving glycal–carbene complexes (*e.g.* **465**, Scheme 44) and various electron-deficient alkenes were reported [939]. Varying degrees of diastereoselectivity were



Scheme 41.



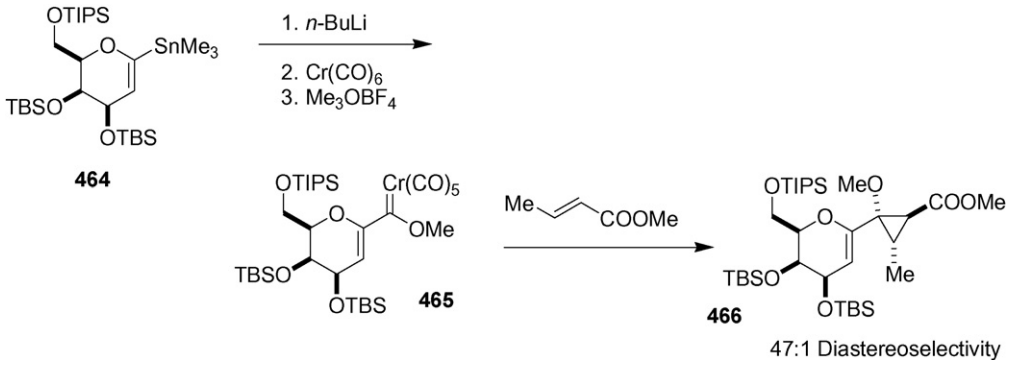
Scheme 42.



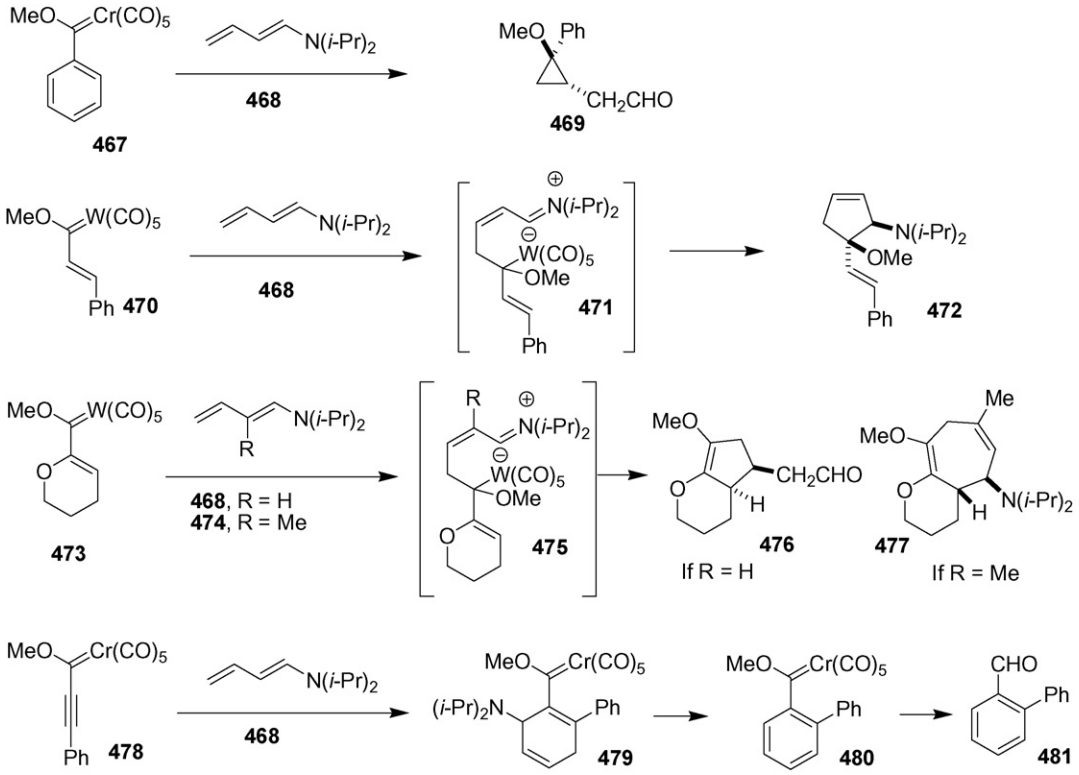
Scheme 43.

observed depending on the aldohexose and the alcohol protecting groups.

The reaction of various Fischer carbene complexes with 1-amino-1,3-dienes was reported (see Scheme 45) [940]. The reaction pathway was governed by the structure of the carbene complex. Arylcarbene complexes (*e.g.* **467**) cyclopropanate the distal alkene group, and afford cyclopropanecarboxaldehydes (*e.g.* **469**) after hydrolytic workup. Alkenylcarbene complexes (*e.g.* **470**)



Scheme 44.



Scheme 45.

lead to cyclopentene derivatives (*e.g.* **472**) in a process involving nucleophilic addition of the alkene to the carbene carbon followed by ring closure of the intermediate zwitterion (**471**). Cyclopentenes (*e.g.* **476**) or cycloheptadienes (*e.g.* **477**) were formed using the α-alkoxyalkenylcarbene complexes (*e.g.* **473**) due to more nucleophilic alkene groups and the greater potential for allylic rearrangements in the intermediate zwitterion (**475**). Alkynylcarbene complexes (*e.g.* **478**) led to benzaldehyde derivatives (*e.g.* **481**) in a process involving Diels–Alder reaction, followed by elimination of the amine group of the intermediate cyclohexadiene (**479**) followed by amine-induced hydrolysis of the carbene group of the intermediate arylcarbene complex (**480**).

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 and alkynes (commonly known as the Dötz reaction, see Scheme 46) were reported in 2008. Examples include: (1) benzannulations using 1,2-dichloroethane as solvent, which proceed at 30 °C [941]; (2) reaction of methoxyphenylcarbene complex **484** with homoallylic alcohol derivative **485** for total synthesis of eleutherin [942]; (3) benzannulation using dihydrofurylcarbene complex **487**, which was employed for the total synthesis of aflatoxin B_2 [943]; (4) atroposelective benzannulation of benzocycloheptenylcarbene complexes (*e.g.* **490**) as a model for total synthesis of allocolchicinoids [944]; (5) benzannulations involving alkynes that feature propargylic leaving groups and pendant alkene groups (*e.g.* **492**) and α,β-unsaturated carbene complexes (*e.g.* **493**) to provide tricyclic compounds (*e.g.* **495**) in a process involving benzannulation, followed by elimination of the leaving group and formation of the *o*-quinonemethide (**494**), followed by intramolecular Diels–Alder reaction [945].

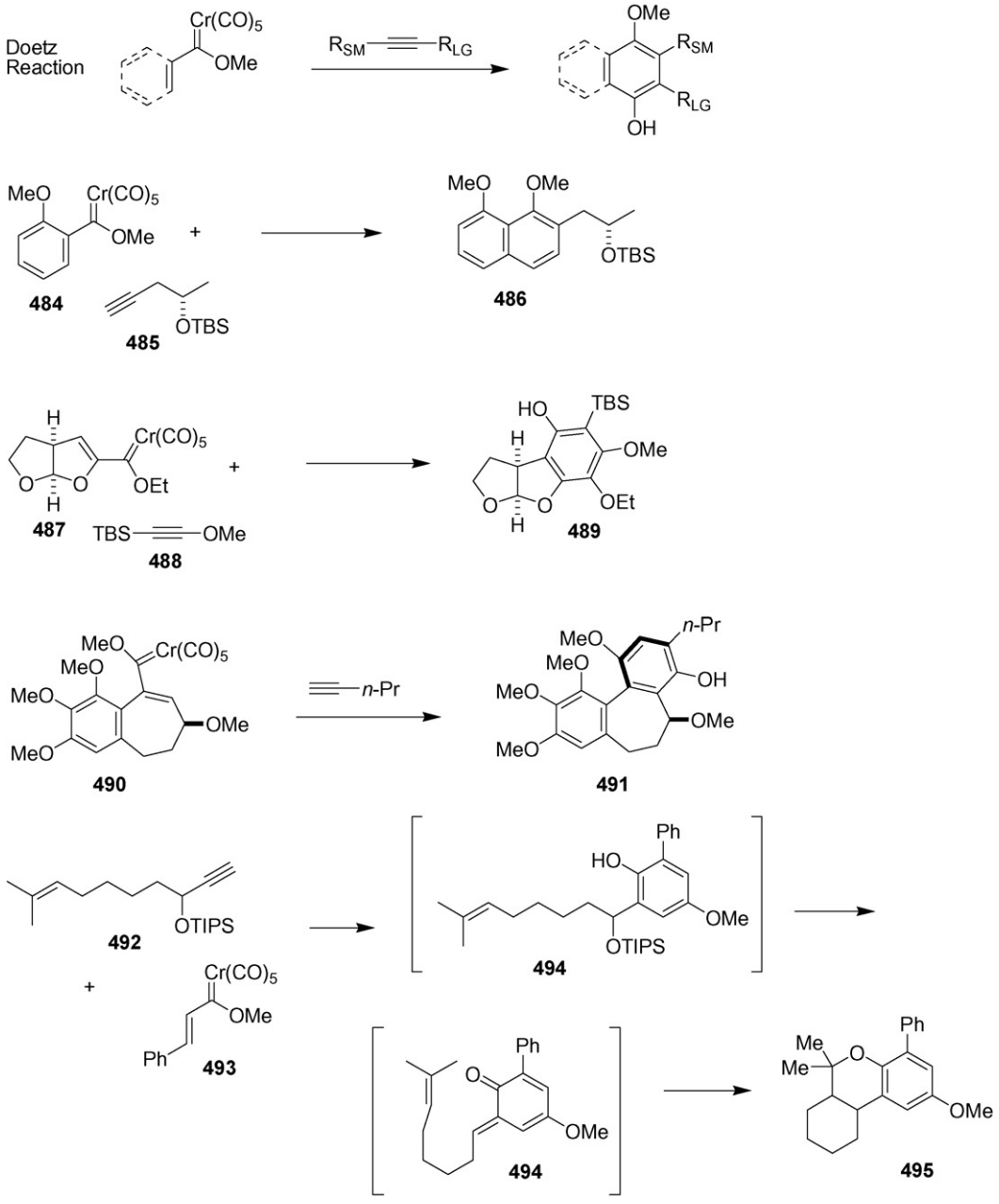
2.3.3.5. Nonbenzannulation reactions of Group 6 metal–carbene complexes with alkynes. Other processes involving the capture of vinylcarbene complexes generated from the coupling of carbene complexes and functionalized alkynes were reported in 2008.

The three-component coupling of 2-alkynylbenzaldehyde hydrazones (*e.g.* **498**, Scheme 47), carbene complexes (*e.g.* **499**), and electron-deficient alkynes (*e.g.* **500**) leads to substituted naphthalene derivatives (*e.g.* **503**) [946]. The carbene–alkyne coupling process results in isoindoles (**502**), which undergo Diels–Alder reaction with the alkyne followed by nitrene extrusion to provide the naphthalene derivatives.

The coupling of carbene complexes (*e.g.* **499**, Scheme 48) with conjugated enediynes that feature radical traps (*e.g.* **504**) was reported [947]. After formation of an enyne–ketene intermediate (**505**) from carbene–alkyne coupling and CO insertion, Moore cyclization followed by radical cyclization occurs. The radical addition occurs in preferentially in a 6-endo fashion to afford **508** after hydrogen transfer. The reaction process was also studied by DFT and the 6-endo process was favored both kinetically and thermodynamically, regardless of whether the diradical species was configured as a singlet or a triplet. DFT calculations also suggest that the intermediate from radical cyclization is best represented as the chromium(II) chelate complex depicted by structure **507** when the diradical is configured as a singlet.

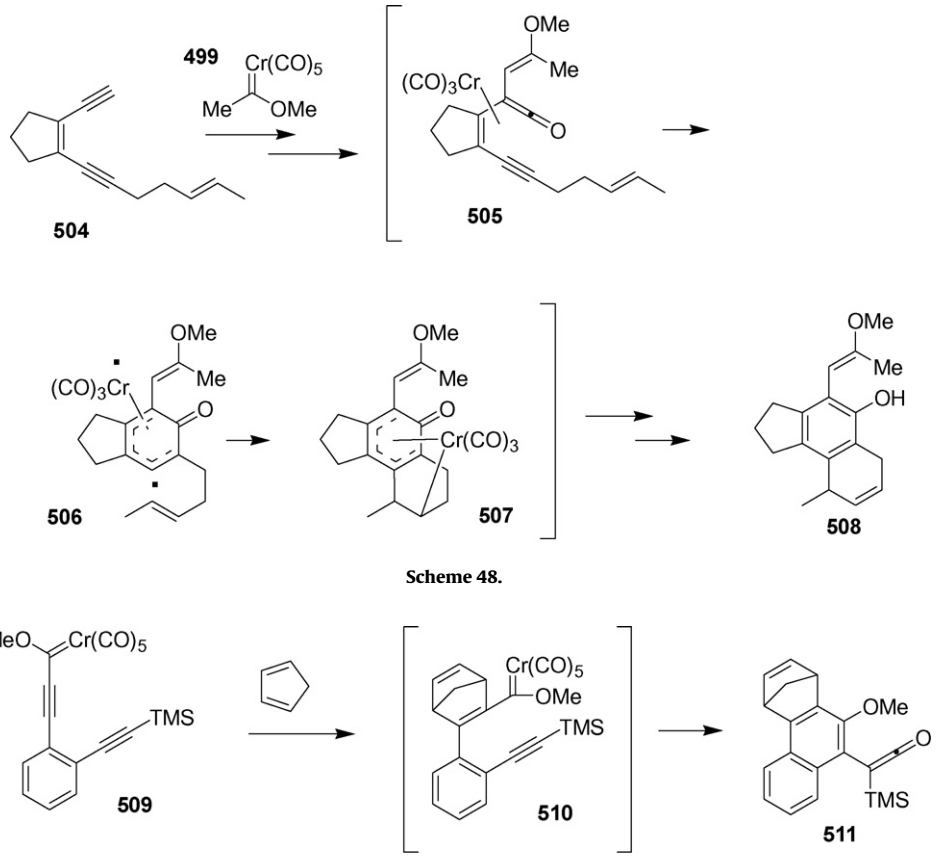
A tandem cycloaddition-alkyne insertion sequence was reported (Scheme 49) [948]. In this case, a conjugated enediyne–carbene complex (*e.g.* **509**) undergoes cycloaddition to afford an alkenylcarbene complex (*e.g.* **510**), which subsequently undergoes intramolecular alkyne–carbene insertion reaction. In cases of trimethylsilylalkynes, a stable ketene (*e.g.* **511**) could be isolated. In other cases ketene trapping products were obtained. The reaction process was successfully initiated by [2+2]-cycloadditions, Diels–Alder reactions (depicted), and 1,3-dipolar cycloadditions.

The formation of stable vinylketenes (*e.g.* **514**, Scheme 50) from the coupling of Fischer carbene complexes (*e.g.* **512**) with alkynes that contain a bulky silyl group (**513**) was reported [949]. In some cases, cyclobutenones (*e.g.* **515**) were obtained. Reactions involving



Scheme 46.

Scheme 47.

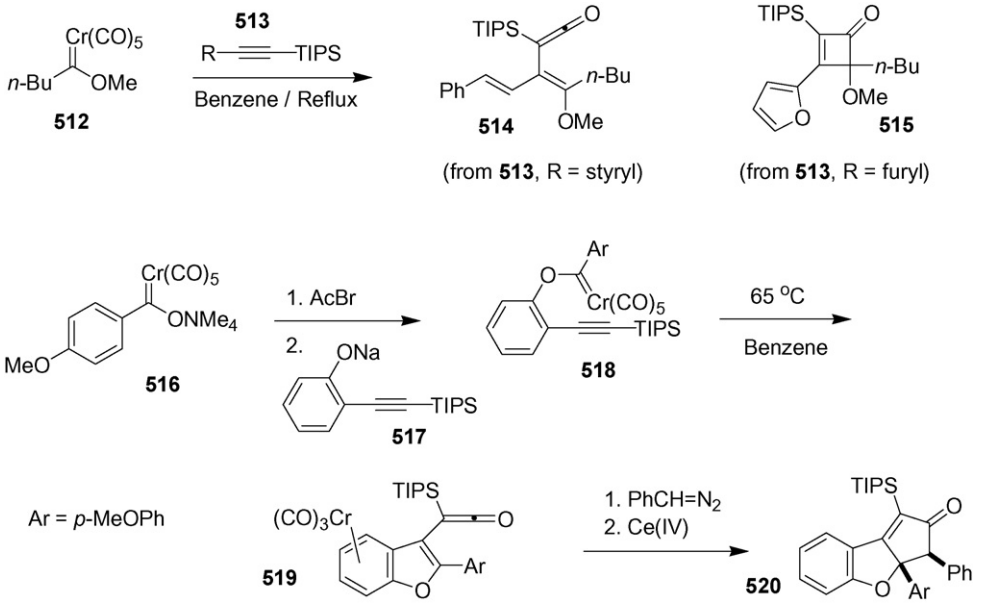


Scheme 48.

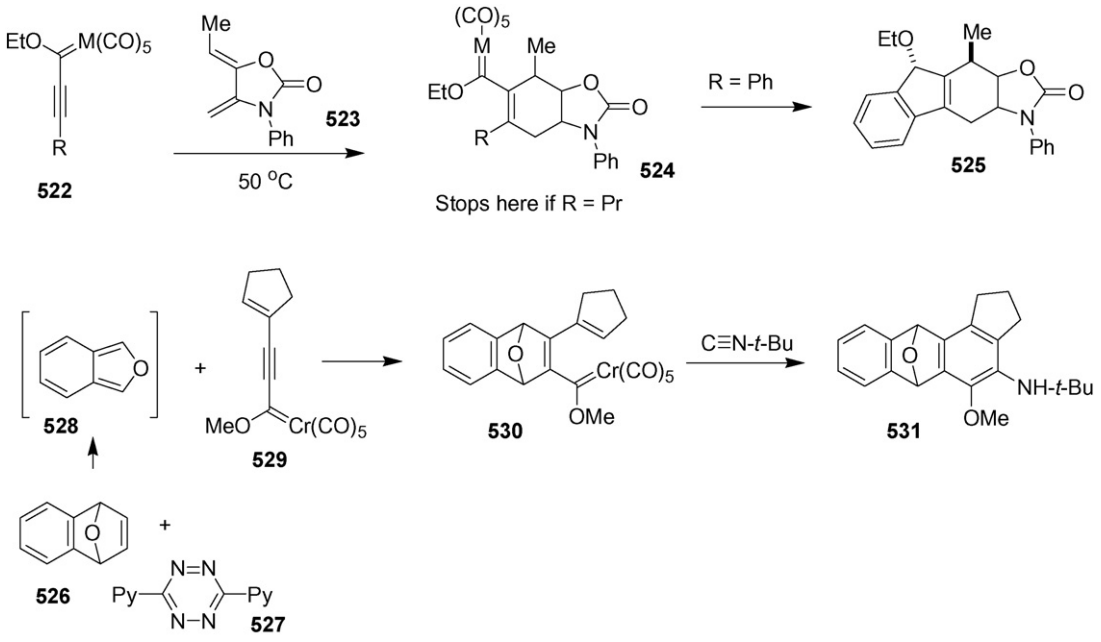
Scheme 49.

trimethylsilylacetylene provided cyclobutenones with the opposite regiochemistry using either thermal or photochemical conditions. The intramolecular coupling of silylated alkynes featuring sterically bulky silyl groups and Fischer arylcarbene complexes (*e.g.* alkyne–carbene complex **518**) followed by treatment with diazo compounds led to cyclopentenones (*e.g.* **520**) [950]. The aryloxycarbene starting materials were prepared through coupling of *in situ*-generated acetoxycarbene complexes with the sodium salts of the phenols (*e.g.* **517**). Subsequent thermolysis afforded the stable silylketene complexes (**519**), which subsequently react with diazo compounds to afford cyclopentenones (**520**).

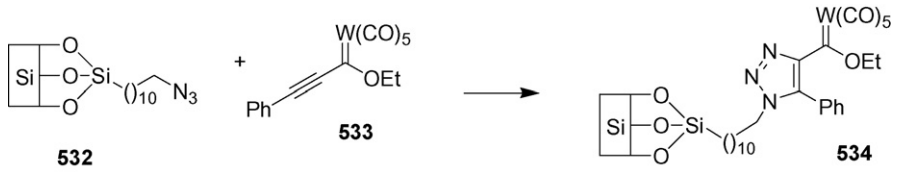
2.3.3.6. Reactions occurring at the conjugated C–C π-bond of α,β-unsaturated Group 6 metal–carbene complexes. Numerous reaction processes were reported in 2008 where a carbene complex activates a π-bond for nucleophilic addition or cycloaddition reac-



Scheme 50.



Scheme 51.

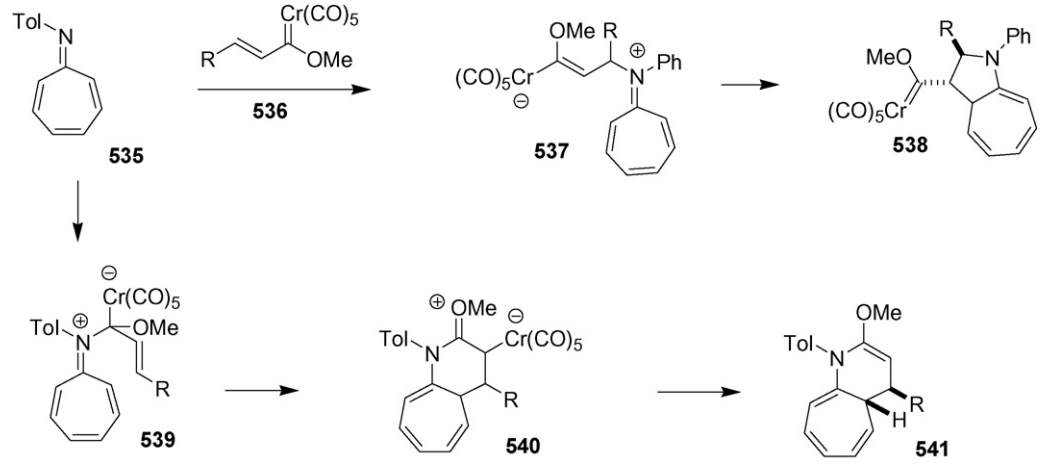


Scheme 52.

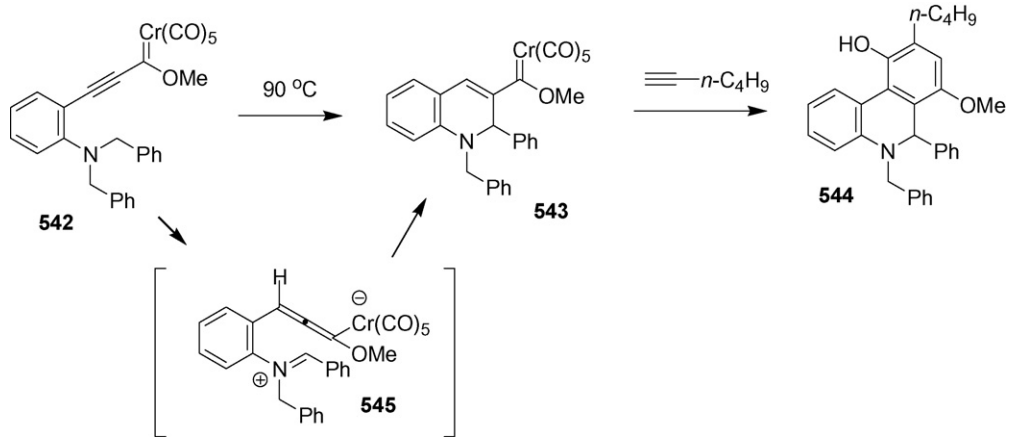
tions (*i.e.* the carbene complex is a surrogate for an "activated ester").

The reaction of alkynylcarbene complexes of general structure **522** (Scheme 51) with dienes (*e.g.* **523**) was reported [951]. The reaction proceeds through an initial Diels–Alder reaction to afford a carbene complex (*e.g.* **524**). In the case of arylethynylcarbene complexes, tetracyclic compounds (*e.g.* **525**) were produced in a process involving tandem Diels–Alder reaction followed by cyclization of the alkenylphenylcarbene complex. Diels–Alder reactions were reported for enynylcarbene complexes (*e.g.* **529**) and *in situ*-generated isobenzofuran derivatives (*e.g.* **528**) [952]. The reaction of adducts (**530**) with isocyanides or photolysis in the presence of CO leads to formation of oxygen-bridged anthracene derivatives (*e.g.* **531**). Diels–Alder reactions of α,β-unsaturated carbene complexes were studied computationally [953]. Endo selectivity was attributed to secondary orbital interactions and the reaction was more asynchronous than the analogous reactions involving esters.

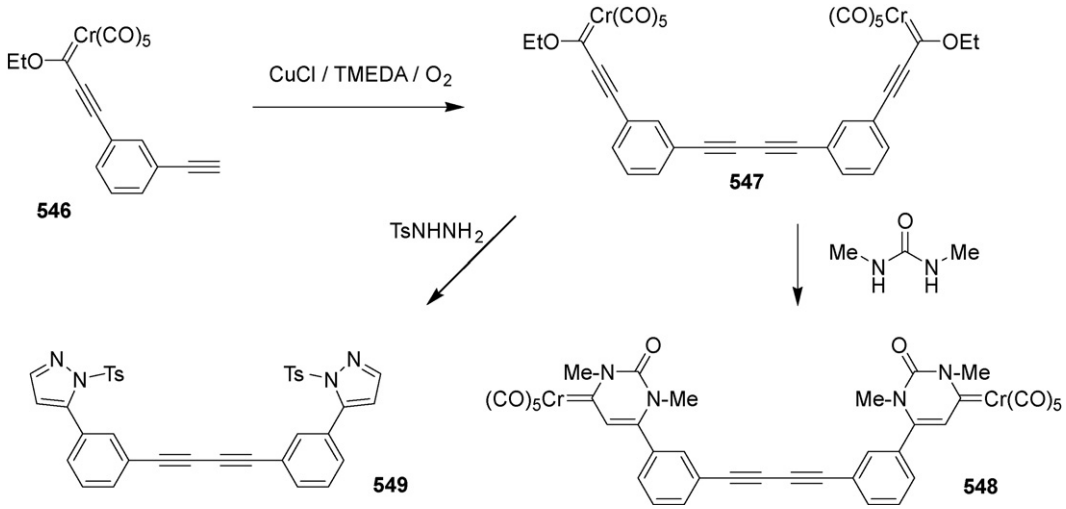
The reaction of alkynylcarbene–tungsten complexes (*e.g.* **533**, Scheme 52) with silicon monolayer-bound azides (*e.g.* **532**) led to the 1,3-dipolar addition product, polymer-bound triazolylcarbene complexes (*e.g.* **534**) [954]. Aminolysis of the solid-state carbene complexes with bovine serum albumin was also reported.



Scheme 53.



Scheme 54.



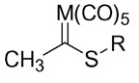
Scheme 55.

The reaction of alkenylcarbene complexes (*e.g.* **536**, Scheme 53) with azafulvenes (*e.g.* **535**) led to either [8+2]-cycloadducts (*e.g.* **538**) or [8+3]-cycloadducts (*e.g.* **541**), depending upon the structure of the carbene complex and/or the solvent employed [955]. Most β-arylcarbene complexes afforded [8+2]-cycloadducts exclusively when acetonitrile was the solvent and mixtures of [8+2]- and [8+3]-cycloadducts when hexane was the solvent. Carbene complexes that feature a five-membered heterocycle or an alkene group in the β-position afforded exclusively [8+3]-cycloadducts regardless of the solvent employed. The proposed mechanism for the [8+2]-cycloaddition involves Michael addition followed by ring closure of the zwitterion (**537**). In the case of [8+3]-cycloaddition, direct addition to the carbene carbon followed by intramolecular attack on the ring by the allylchromium intermediate establishes the ring system (**540**), and subsequent loss of chromium affords the final product (**541**). Both processes were highly stereoselective. A variety of carbene complex demetallation reactions were demonstrated for the [8+2]-cycloadducts.

Thermolysis of *o*-aminophenylethynylcarbene complexes (*e.g.* **542**, Scheme 54) led to dihydroquinoline–carbene complexes (*e.g.* **543**) [956]. A mechanism that involves intramolecular hydride addition to afford a zwitterion (**545**) followed by reaction of the resultant anion with the iminium salt was proposed. The products undergo the Dötz benzannulation to afford azaphenanthrene derivatives (*e.g.* **544**).

The synthesis of dialkyne-linked bis(alkynylcarbene) complexes (*e.g.* **547**, Scheme 55) and their subsequent cycloaddition reactions with hydrazines or ureas was reported [957]. Copper-induced dimerization of terminal alkyne–carbene complexes (*e.g.* **546**) afforded the dimeric species (*e.g.* **547**). Reaction with tosylhydrazine led to the pyrazole derivatives (*e.g.* **549**). Reaction with N,N′-dimethylurea led to the carbene complex/pyrimidone species (*e.g.* **548**).

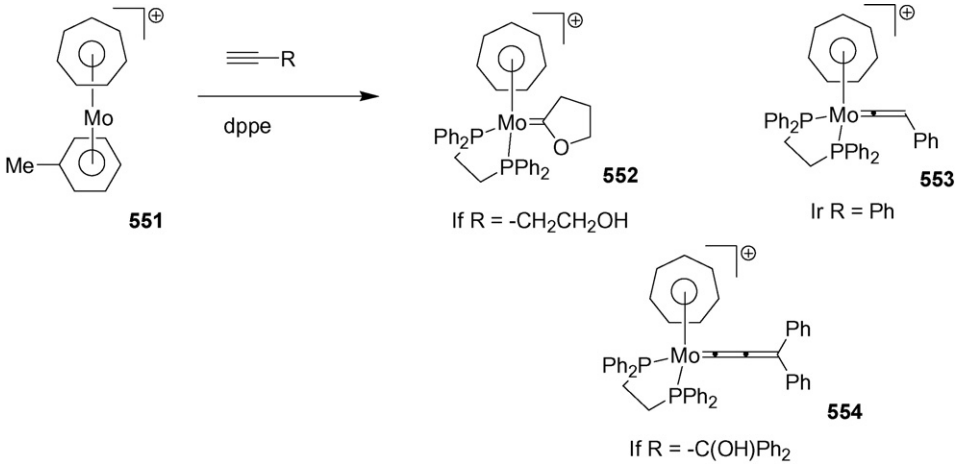
2.3.3.7. Physical organic chemistry of Group 6 Fischer carbene complexes. The kinetic and thermodynamic acidities of Group 6 thiocarbene complexes of general structure **550** (Scheme 56) in 50:50 acetonitrile:water was reported [958]. A very slight effect of the R group on the pK_a value was noted. The compounds featuring small alkyl groups (*e.g.* Me) were more acidic than those featuring large and/or bulky groups (*e.g.* cyclohexyl). Tungsten–carbene



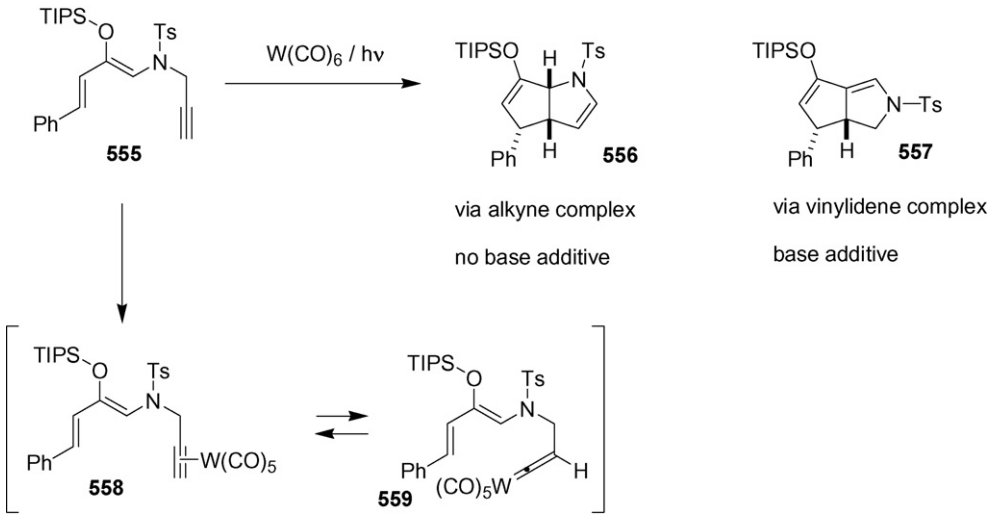
550

M	R	Them. pKa	Kin. pKa
Cr	Me	9.00	9.05
Cr	Cy	9.46	9.35
W	Me	N/A	8.37
W	Cy	N/A	8.81

Scheme 56.



Scheme 57.



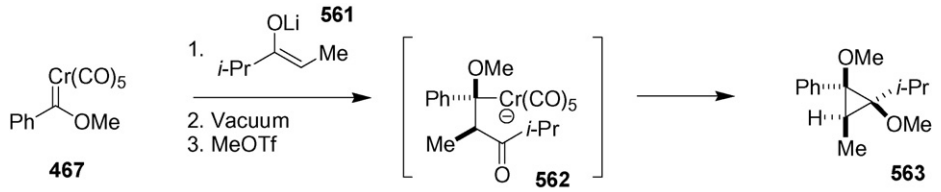
Scheme 58.

complexes were slightly more acidic than chromium–carbene complexes.

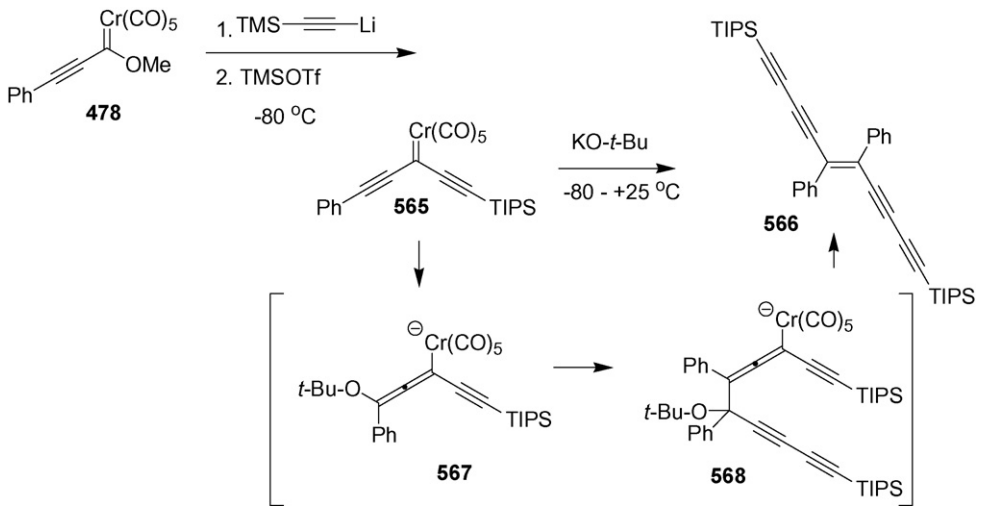
2.3.3.8. Synthesis and reactivity of Group 6 metal–vinylidene complexes, and reactions that involve vinylidene–metal complexes as intermediates; also includes other process that involve the formation of a carbene complex from an alkyne and a non–carbene metal complex. The synthesis of Group 6 vinylidene (*e.g.* **553**, Scheme 57), allenylidene (*e.g.* **554**), and Fischer carbene–molybdenum complexes (*e.g.* **552**) that feature cycloheptatrienyl ligands was reported [959]. These complexes were prepared through the reaction of complex **551** with various terminal alkynes in the presence of diphenylphosphinoethane. The barriers to rotation of the complexes were studied by variable temperature NMR and DFT.

Reaction of diene-terminal alkynes (*e.g.* **555**, Scheme 58) with tungsten pentacarbonyl sources led to two different classes of bicyclo[3.3.0]octane derivatives (*e.g.* **556** or **557**), depending upon the presence of reaction additives [960]. If no additive was present, π-alkyne complex-derived products of general structure **556** were obtained. If base was added, then vinylidene-derived products of general structure **557**were observed.

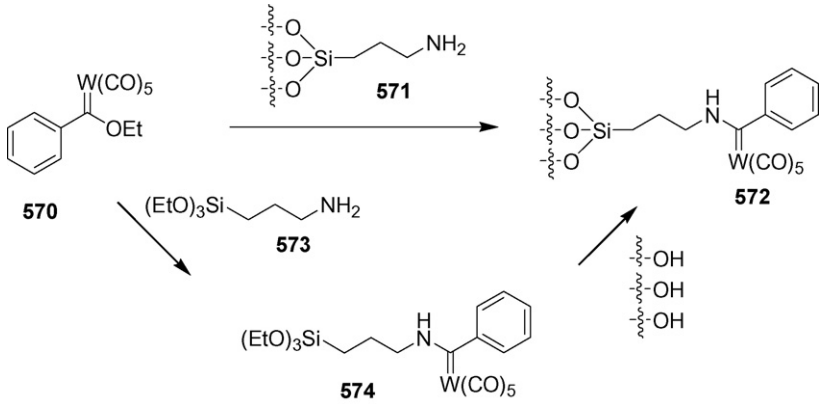
2.3.3.9. Reactions involving the addition of nucleophiles to the carbene carbon. The coupling of silylcarbene complexes (*e.g.* **467**, Scheme 59) with enolates (*e.g.* **561**) was reported [961]. The sequence of enolate addition, followed by removal of amines by vacuum, followed by reaction with methyl triflate or chlorotrimethylsilane leads to dialkoxycyclopropanes (*e.g.* **563**) with a very high degree of stereoselectivity. A mechanism involving



Scheme 59.



Scheme 60.



Scheme 61.

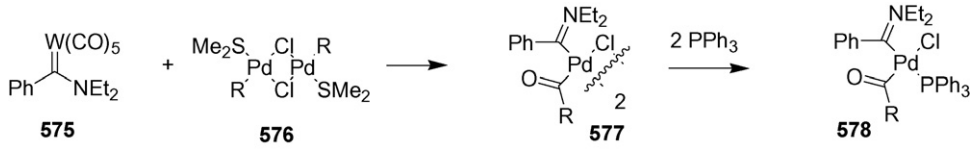
addition to the carbene carbon to afford a tetrahedral intermediate (**562**) followed activation of the ketone oxygen through alkylation/silylation and addition of the alkylchromium intermediate to the activated ketone was proposed.

The addition of alkynyllithium reagents to carbene complexes devoid of α-hydrogens (*e.g.* **478**, Scheme 60) followed by reaction with trimethylsilyl triflate led to alkynylcarbene complexes devoid of heteroatom stabilization (*e.g.* **565**) [962]. These carbene complexes are stable only at low temperature and form dimers (*e.g.* **566**) when warmed to room temperature in the presence of potassium *t*-butoxide. Head-to-head as well as head-to-tail dimers were produced, depending on the structure of the carbene complex. A mechanism was proposed that involves Michael addition of *t*-butoxide ion followed by propargylation of a virgin carbene complex resulting in dimeric anionic complex (**568**), followed by demetallation/alkoxide elimination.

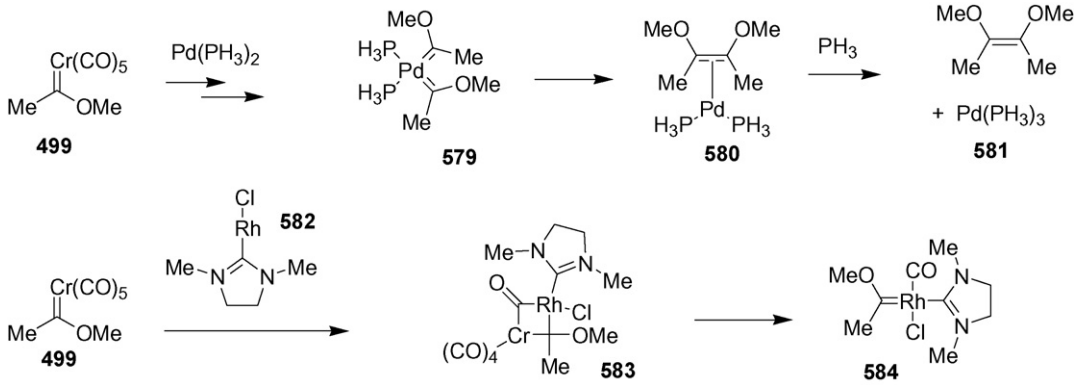
The binding of carbene complexes to a silica solid support and subsequent spectroscopic characterization was reported (see Scheme 61) [963–965]. Two methods were explored. In one method carbene complex **570** was treated with silica-bound primary amine **571**, resulting in silica-bound carbene complex **572**. The other method involves reaction of carbene complex **570** with amine-siloxane **573** followed by binding of resulting the aminocarbene complex **574** to silica.

2.3.3.10. Reactions that involve transfer of a Fischer carbene ligand to another metal. The reaction of aminocarbene-tungsten complexes (*e.g.* **575**, Scheme 62) with arylpalladium complexes (*e.g.* **576**) resulted in transfer of both a carbene ligand and a CO ligand to afford dimeric palladium carbene complexes (*e.g.* **577**) [966]. The dimeric complex **577** was converted to a monomeric complex (**578**) through treatment with triphenylphosphine.

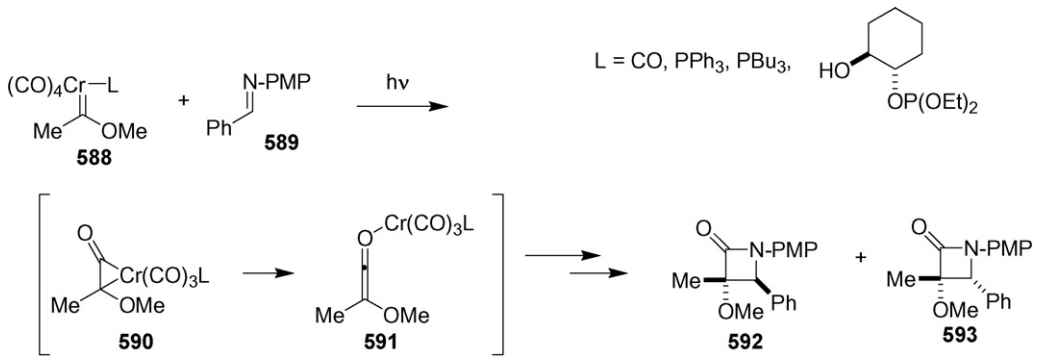
The transmetallation of Group 6 carbene complexes was studied computationally (see Scheme 63) [967]. The study emphasizes transfer to palladium(0), copper(I), and rhodium(I) and the resulting reaction pathways. In the case of palladium and copper, dimerization through formation of a bis(carbene) complex (*e.g.* **579**) followed by a C–C coupling of the carbene ligands was pro-



Scheme 62.



Scheme 63.

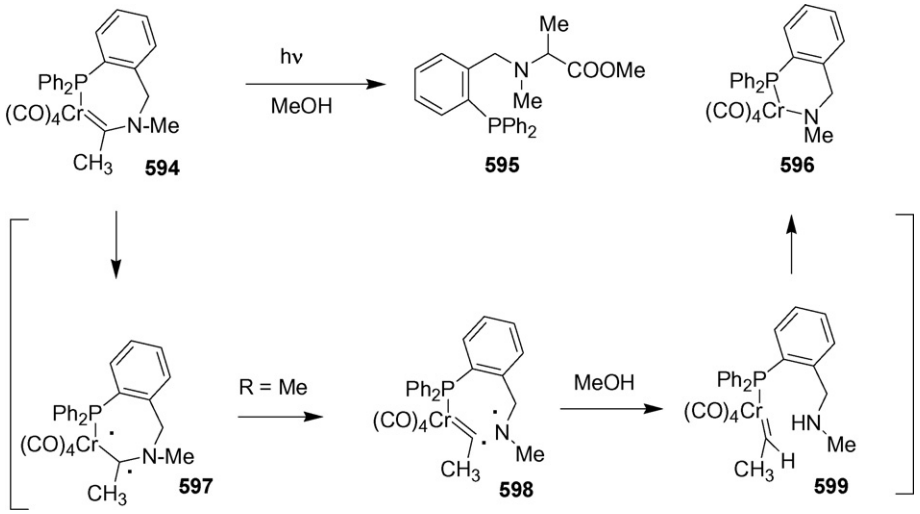


Scheme 64.

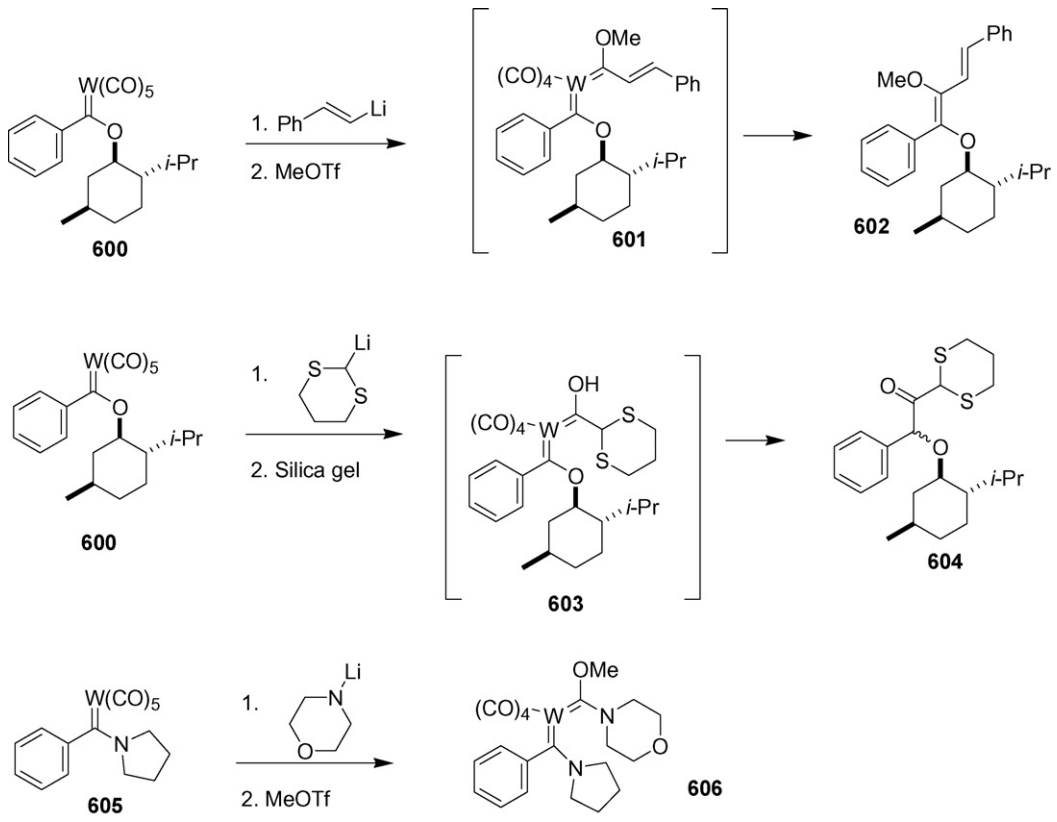
posed. The C–C bond-forming step is highly exothermic in both cases, however this step had the highest activation energy of any step. Rhodium systems react through transfer of a carbene and a CO ligand via a dimetallacyclobutane intermediate (**583**).

2.3.3.11. Other reactions of Group 6 metal–carbene complexes. Photochemically induced reaction of carbene complexes (*e.g.* **588**, Scheme 64) with imines to afford β-lactams (*e.g.* **592**, **593**) was studied experimentally and computationally [968]. Key events in this process include photoexcitation to generate an alkene-bound ketene complex (**590**), which isomerizes to an oxygen-bound ketene complex (**591**), which subsequently reacts with the imine to eventually afford the β-lactam species. The presence of the metal in the imine–ketene coupling event was supported by reactions with a chiral carbene complex, which led to low but detectable levels of enantioselectivity in the formation of β-lactams.

Photoinduced reactions of phosphine-chelated aminocarbene complexes (*e.g.* **594**, Scheme 65) were reported [969]. Both the chromium and tungsten complexes were found to be photochemically reactive. The reactions involving the chromium complexes afford either the carbene excision product (**596**) or the ketene-derived products (*e.g.* **595**), depending on the substitution at nitrogen. Various mechanistic pathways were evaluated compu-



Scheme 65.



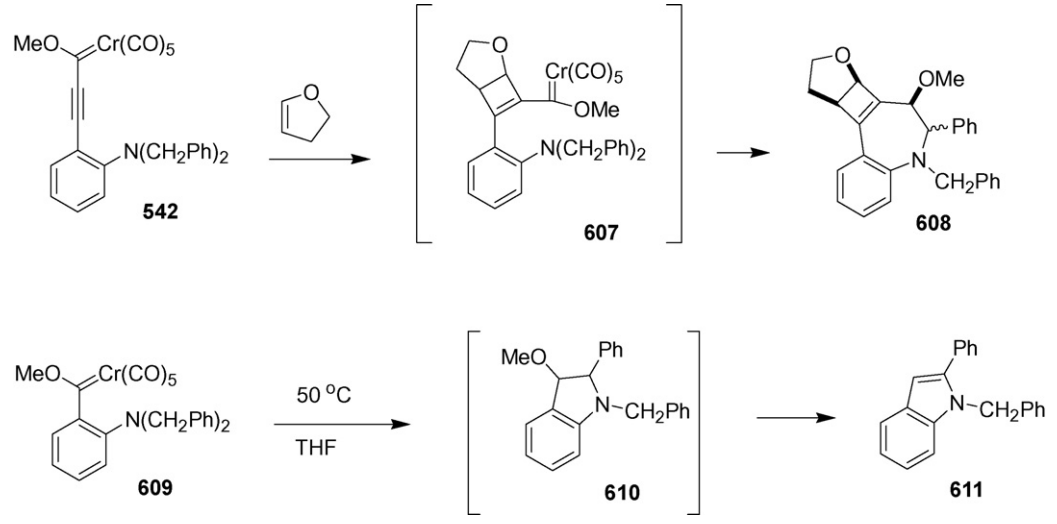
Scheme 66.

tationally. A free radical mechanism (depicted) was determined to be the most energetically reasonable reaction pathway. The carbene excision product arises through cleavage of the C–N bond to form diradical **598** followed by reduction of the diradical to form intermediate **599**, which then loses the carbene ligand to afford **596**.

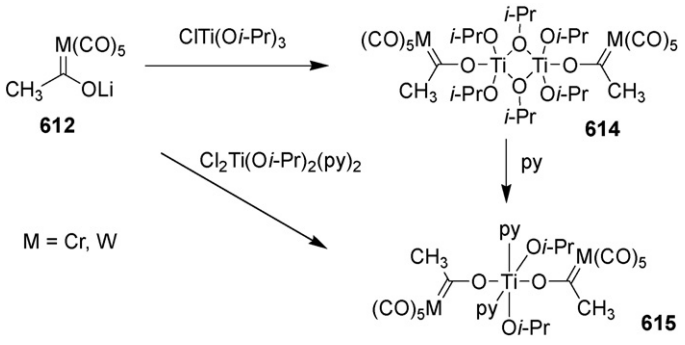
The addition of organolithium reagents to the carbonyl ligands of Fischer carbene complexes (*e.g.* **600**, Scheme 66) was reported [970]. Reaction of organolithium reagents with menthoxycarbene complexes devoid of α-hydrogens followed by methyl triflate leads to the 1,2-dialkoxyalkenes (*e.g.* **602**) in a process involving a mononuclear bis(carbene) complex (**601**). If the alkylating agent was omitted the α-alkoxyketone (*e.g.* **604**) could obtained. In one case, the bis(carbene) complex (**606**) could be isolated.

C–H activation reactions were reported for various anilinocarbene complexes (*e.g.* **542**, **609**, Scheme 67) [971]. Reaction of anilinoalkynylcarbene complexes (*e.g.* **542**) with reagents that cyclo-add to the triple bond (*e.g.* dihydrofuran) results in tandem cycloaddition-C–H activation products (*e.g.* **608**). Similar reactions were observed for simple anilinocarbene complexes (*e.g.* **609**), resulting in indoles (*e.g.* **611**) in a process involving hydride addition, cyclization, and elimination of methanol.

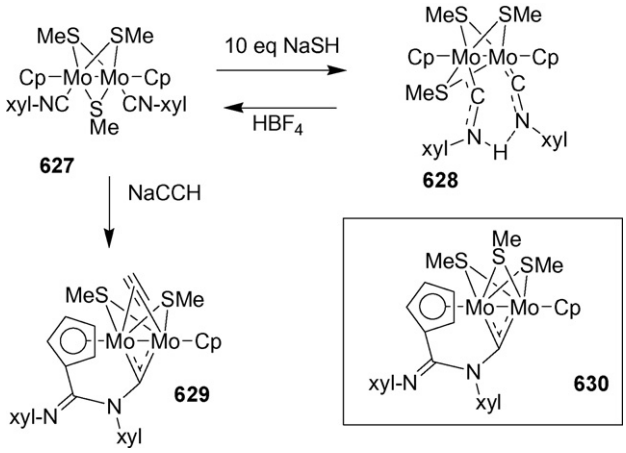
The preparation and structure determination of titanoxycarbene–Group 6 metal–carbene complexes (*e.g.* **614**,



Scheme 67.



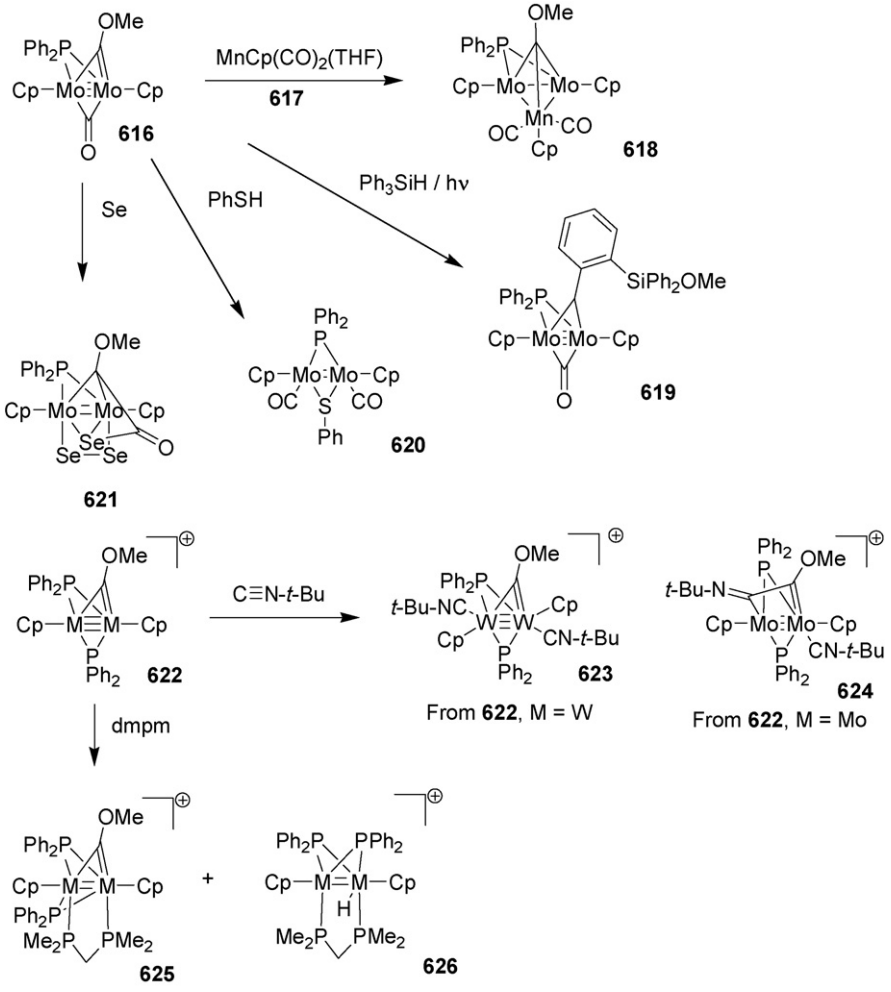
Scheme 68.



Scheme 70.

Scheme 68) was reported [972]. The complexes were prepared by reaction of the lithium acylates (*e.g.* **612**) with $ClTi(Oi\text{-}Pr)_3$. The complexes were dimeric and featured a four-membered Ti_2O_2 ring that was dynamic at room temperature, however could be observed as a static structure by low-temperature NMR. Reaction with pyridine led to an octahedral titanium bis(acyloxy) complex **615**, which could also be produced from reaction of the metal acylate with $Cl_2Ti(Oi\text{-}Pr)_2(py)_2$. Thermal decomposition of complex **615** led to nanoparticle formation.

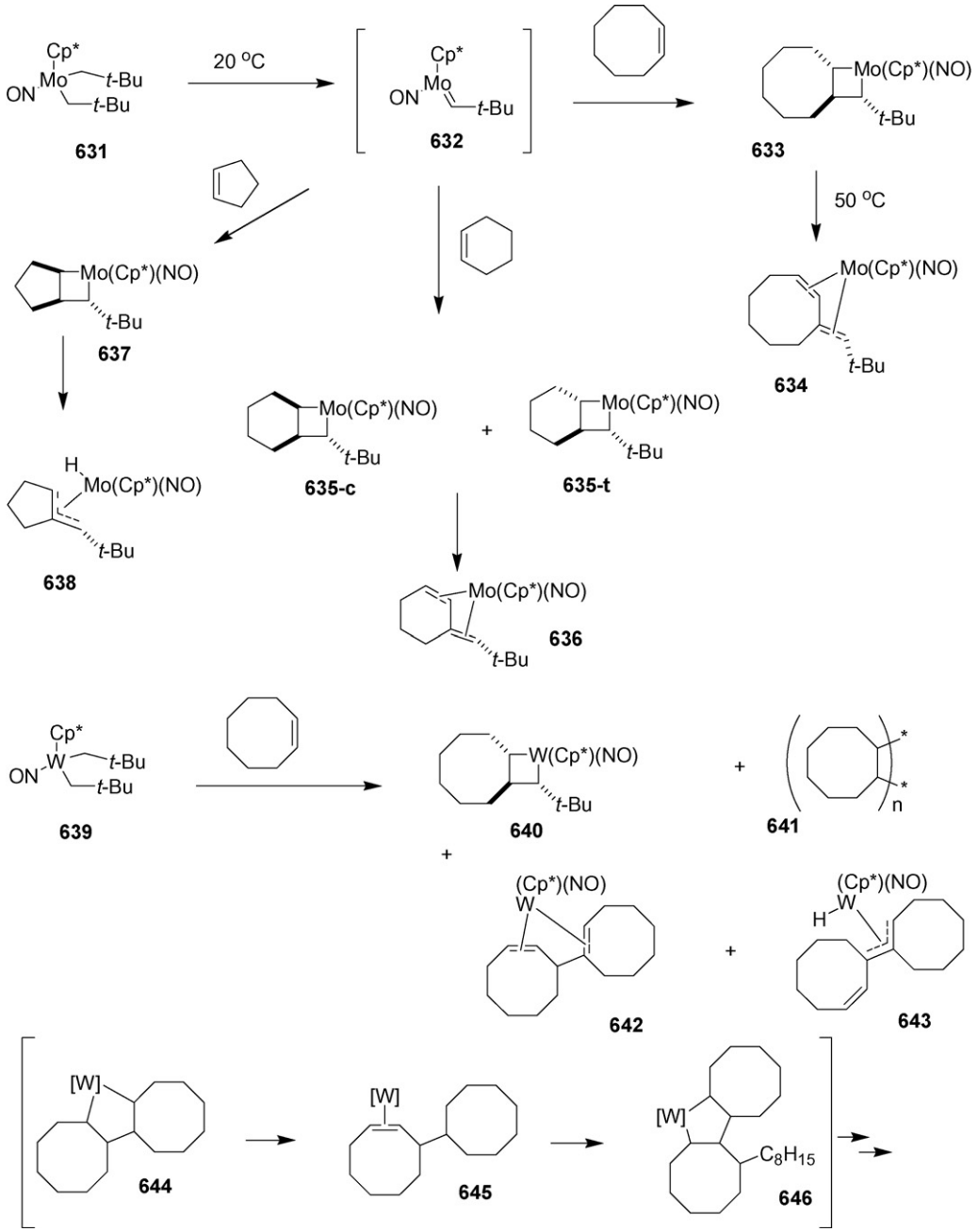
The synthesis and reactivity of bridging carbine–dimetal complexes (*e.g.* **616**, Scheme 69) was reported [973]. A μ^3-carbyne complex (**618**) was obtained upon treatment of dimolybdenum carbyne complex **616** with manganese complex **617**. Reaction of complex **616** with a variety of other reagents was also reported. Reaction with selenium leads to the molybdenum selenide **621**. Reaction with thiophenol results in displacement of the carbyne ligand and to afford the bridging thiophenyl complex **620**. Photochemical reaction with triphenylsilane led to the bridging carbene complex **619**. The reaction of diphosphide–tungsten complex **622** with isocyanides leads to the isocyanide-ligated complex **623** [974]. The



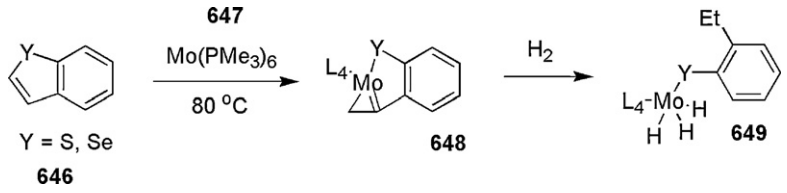
Scheme 69.

molybdenum analog led to the isonitrile insertion product **624**. Analogous reaction with CO leads to the CO analog of complex **623**, regardless of the metal. Reaction with DMPM led to the simple ligation product (**625**) and a minor amount of the hydride complex **626**. Similar studies were reported for bis(carbyne)phosphide complexes [975]. The reaction of sulfide-bridged dimolybdenum complex **627** (Scheme 70) with NaSH led to the reductively coupled carbenoid–dimolybdenum complex **628** [976]. Reaction of the carbenoid complex with HBF_4 restored the original complex. A carbenoid complex (**629**) was also obtained through reaction with sodium acetylide. Similar reactions were attempted for bridging carbenoid complex **630**.

Coupling of cyclic alkenes with dialkylmolybdenum (*e.g.* **631**, Scheme 71) or dialkyltungsten complexes (*e.g.* **639**) was reported. Reaction of the molybdenum complex with cycloalkenes led to metallacyclobutanes, which undergo further conversion to either the π-allylmolybdenum complex or the diene complex, depending upon the ring size of the cycloalkene [977]. The metallacyclobutanes were produced via [2+2]-cycloaddition with an intermediate carbene complex (**632**). Larger rings lead to the trans metallacyclobutanes (*e.g.* **633**, **635-t**). Further reaction of the cyclopentene-derived metallacyclobutane **637** led to the π-allyl complex (**638**). The diene complexes (*e.g.* **634**, **637**) were obtained from larger ring systems. Reaction of complex **631** with norbornene led to the stable metallacyclobutane. Reaction with a monosubstituted alkenes led to dienes. The reaction of tungsten analog **639** with cyclic alkenes led to similar metallacyclobutanes (**640**) accompanied by alkene oligomers (**641**) [978]. The oligomers appear to retain the original ring systems. In the reaction with cyclooctene, additional products incorporating two moles of cyclooctene were obtained (**642**, **643**). The oligomerization process was proposed to proceed via formation of a metallacyclopentane (**644**) followed



Scheme 71.



Scheme 72.

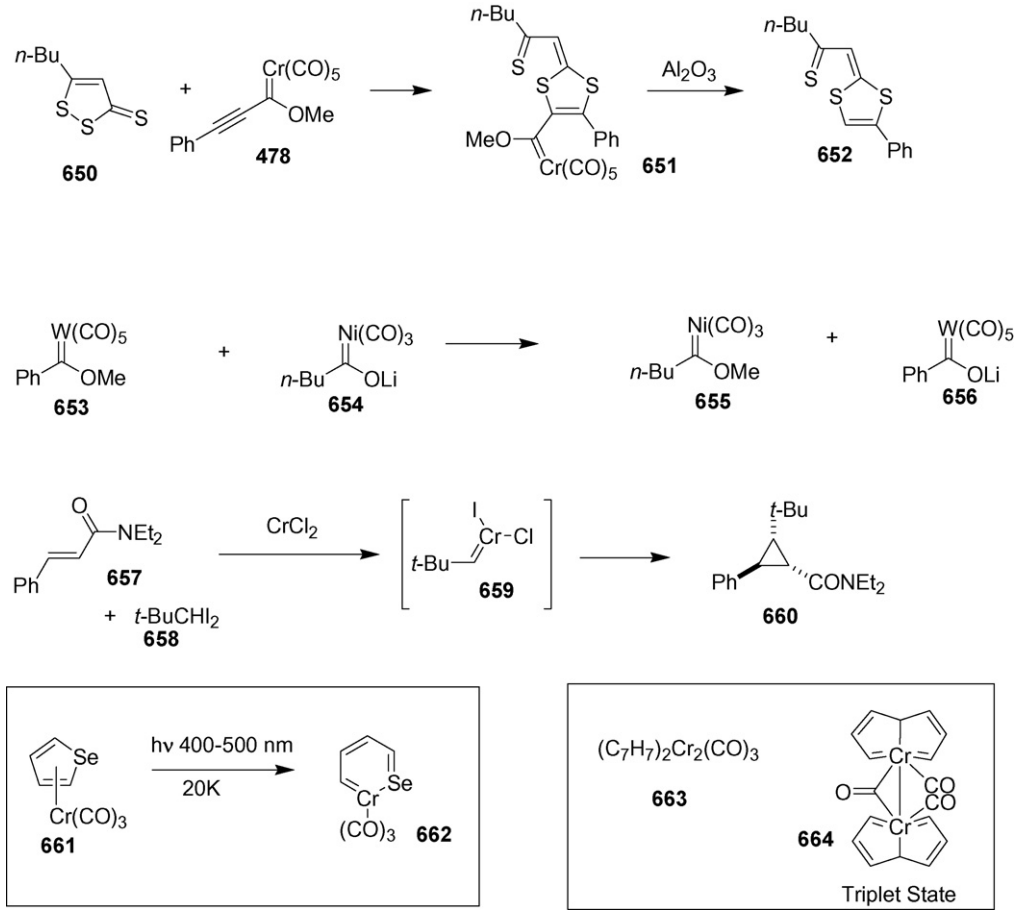
by rearrangement to the alkene complex, followed by formation of a higher metallacyclopentane through reaction with additional alkene. The structure of lower MW oligomers supports this mechanistic pathway.

Molybdenum η^2-alkenyl complexes (*e.g.* **648**, Scheme 72) were generated from the reaction of benzothiophene or benzoselenophene with $Mo(PMe_3)_6$ at elevated temperature [979]. Subsequent reaction with hydrogen led to the molybdenum sulfide/selenide derivative (**649**).

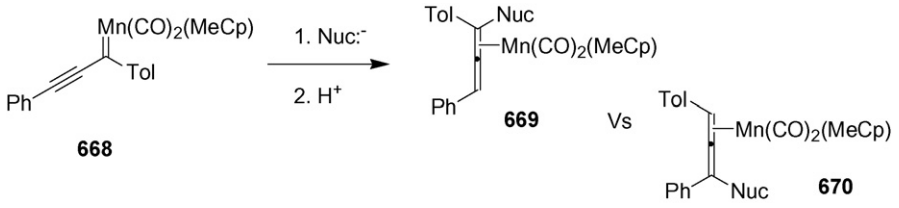
Other studies of Group 6 metal–carbene complexes are depicted in Scheme 73 and include: (1) reaction of alkynylcarbene complexes (*e.g.* **478)** with cyclic disulfides (*e.g.* **650**) to afford 2-alkylidene dithiolenes (*e.g.* **652**) in a process involving cycloaddition/ring opening, followed by removal of the carbene complex functionality using alumina [980]; (2) use of alkynylcarbene tungsten complexes as catalysts for the Pauson–Khand reaction (simple non-carbene tungsten carbonyls however proved to be better catalysts) [981]; (3) calculation of the MO energies of alkoxycarbene–chromium complexes and correlation with observed UV–visible spectra for these complexes [982]; (4) electron donation in cyclopropenylidene-chromium complexes [983]; (5) an alkyl group exchange process from the reaction of tungsten carbene complex **653** with nickel–carbene complex **654** [984]; (6) possible involvement of chromium–carbene complexes (*e.g.* **659**) in cyclopropanation reactions using gem diiodides (**658**) and chromium(II) chloride [985]; (7) formation of a selenachromabenzene (**662**) through photolysis of selenophene-$Cr(CO)_3$ (**661**) in an argon matrix at 20 K (the product was identified by difference IR spectroscopy) [986]; (8) comparison of mass spectral fragmentation modes and thermogravimetric analysis methods to study decomposition of chromium–carbene and allenylidene complexes [987]; (9) discussion of Cr–C multiple bond character in $(AcCp)(NO)_2Cr{-}C{\equiv}CPh$ [988]; (10) discussion of Cr–C bonding in ruthenium(II) and osmium(II) complexes of $[(CO)_5CrCN]^-$ [989], and (11) generation a bis(carbene) ring opened product during a DFT investigation of cycloheptatrienylchromium complexes (**663**) in the triplet state [990].

2.3.4. Group 7 metal–carbene complexes

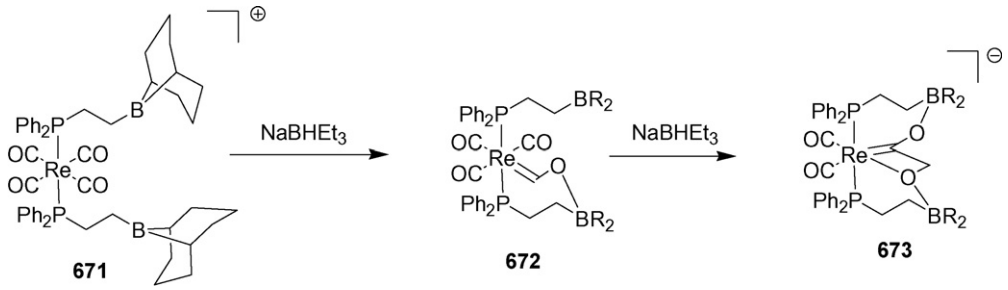
The addition of various nucleophiles to alkynylcarbene complexes (*e.g.* **668**, Scheme 74) was reported [991]. The reaction results in allenyl derivatives (*e.g.* **669**, **670**) obtained from either 1,2- or



Scheme 73.



Scheme 74.



Scheme 75.

Michael addition to the carbene complex. Nucleophiles examined include thiols, phosphides, and enolates.

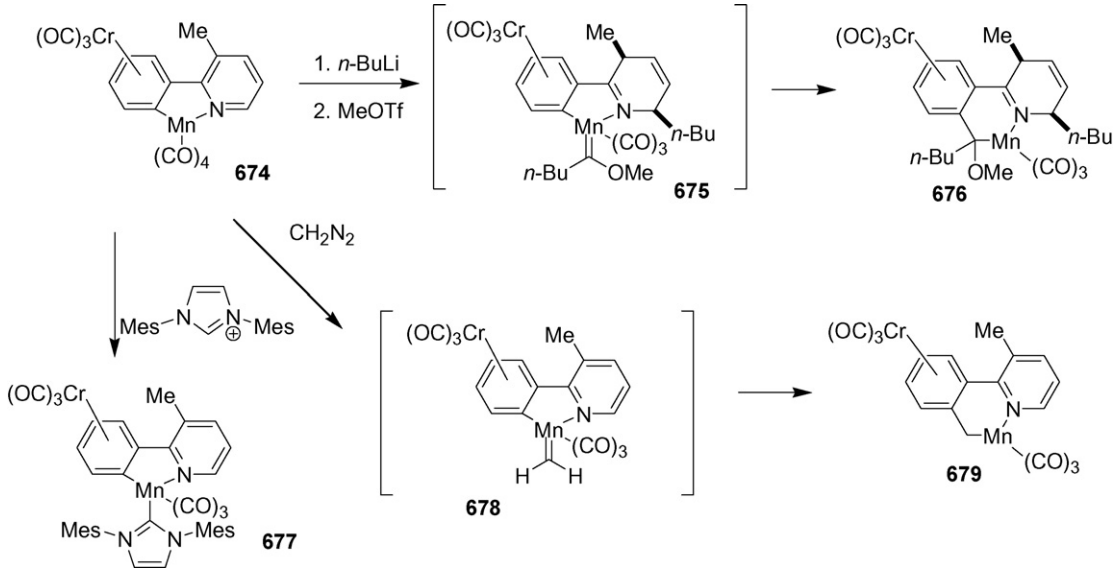
Boryloxycarbene complexes (*e.g.* **672**, **673**, Scheme 75) were prepared through reaction of boron-containing rhenium carbonyls (*e.g.* **671**) with hydride reagents [992]. Initially the boron-complexed metal formyl (**672**) was produced, which undergoes further hydride reduction to afford the boron-complexed alkoxymethylacrylate (**673**) after CO insertion from the alkoxymethylrhenium intermediate.

Manganese–carbene complexes (*e.g.* **675**, **678**, Scheme 76) were suggested as intermediates in the reaction of manganese–chromium complex **674** with organolithium reagents followed by methyl triflate, or the reaction of **674** with diazo compounds [993]. Organolithium reagents add to both the pyridine ring and to the manganese carbonyl ligand of **674**, and result in azametallacycles (*e.g.* **676**) after carbene insertion. Reaction of **674** with diazomethane also afforded an azametallacycle (**679**) from the carbene complex intermediate (**678**). Reaction with a stable carbene led to the simple carbene complex **677**, which was stable and could be isolated.

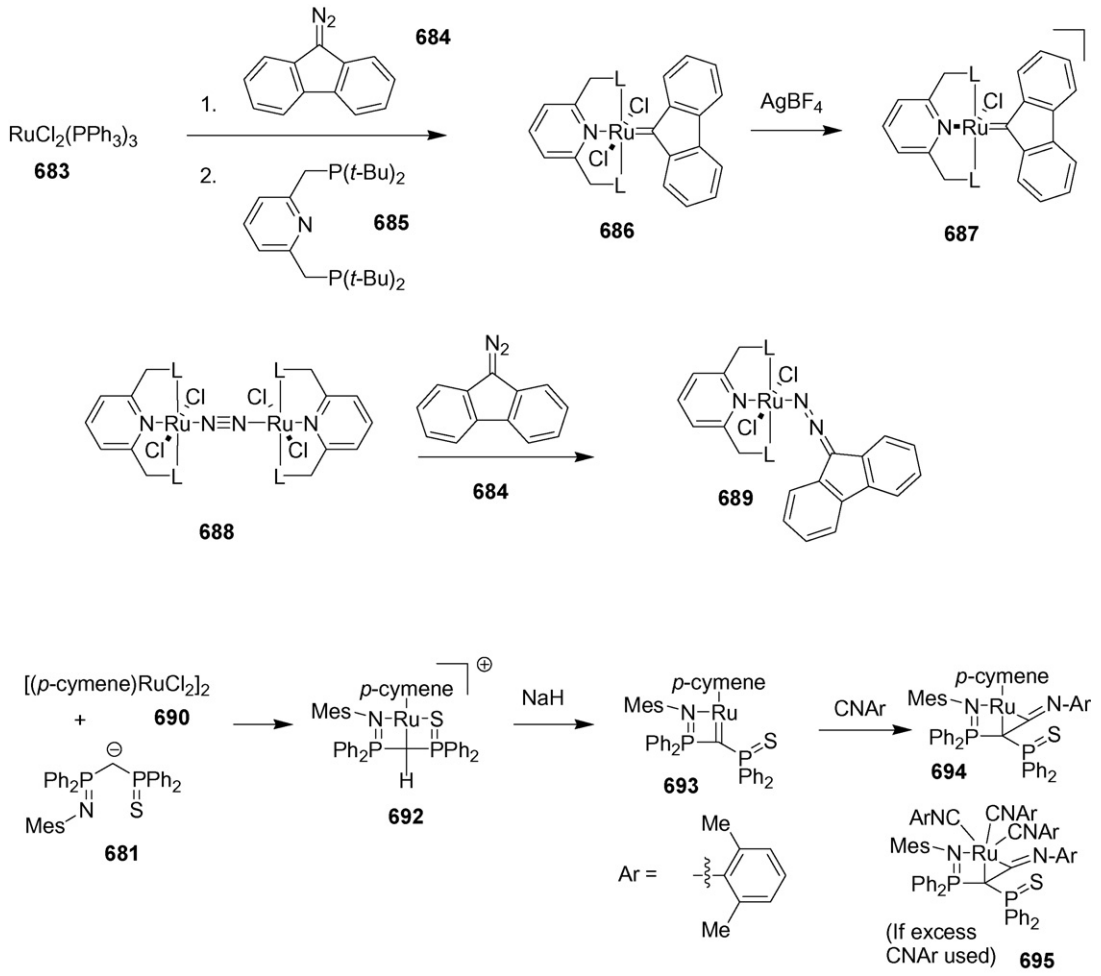
2.3.5. Group 8 metal–carbene complexes

2.3.5.1. Non-heteroatom-stabilized metal–carbene complexes that are not metallacumulenes. Numerous additional examples of the synthesis and reactivity of this class of compounds have been presented in the alkene metathesis section. The Grubbs catalysts fall into this classification.

Formation of pincer ligated carbene complexes (*e.g.* **686**, **687**, Scheme 77) from diazo compounds and ruthenium complex **683** was reported [994]. Sequential treatment of complex **683** with a diazofluorene (**684**) and pincer ligand **685** affords neutral carbene complex **686**, which converts to the cationic complex (**687**) upon treatment with silver ion. A stable diazo complex (**689**) was formed from the dinitrogen dimer complex **688** and diazofluorene. Pincer ligated ruthenium–carbene complexes **693** was prepared through deprotonation of bis(sulfidodiphenylposphinyl)methyl–ruthenium



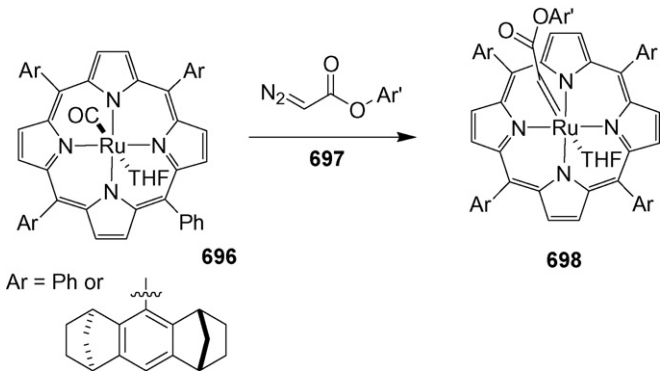
Scheme 76.



Scheme 77.

complexe **692** [995]. Coupling with isocyanides leads to the ketenimine complexes (**694**). Additional ligand substitution occurs when excess isocyanide ligands were added, resulting in bis(isocyanide) ketenimine complexes (**695**).

The reaction of ruthenium porphyrin carbonyl complex **696** (Scheme 78) with α-diazoesters leads to stable ruthenium–carbene complexes (*e.g.* **698**) [996]. The carbonyl complex **696** also serves as a catalyst for the cyclopropanation of styrene using α-diazoesters. Moderate degrees of asymmetric induction were observed when complexes featuring a chiral aromatic substituent were employed. Ruthenium porphyrin–carbene complexes derived from various



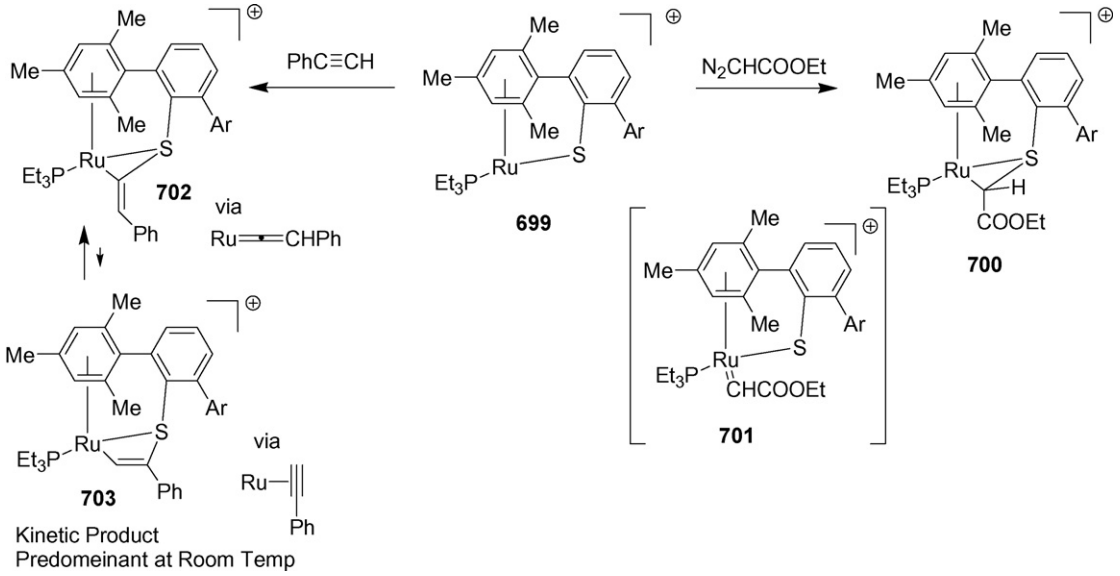
Scheme 78.

diazo compounds (diaryldiazo compounds or α-diazoketones) were similarly prepared and successfully used as cyclopropanation catalysts [997]. The use of iron–porphyrin complexes as carbene transfer agents was also reported [998]. Significant mechanistic discussion involving the role of iron–carbene complexes in C–H insertion processes was presented.

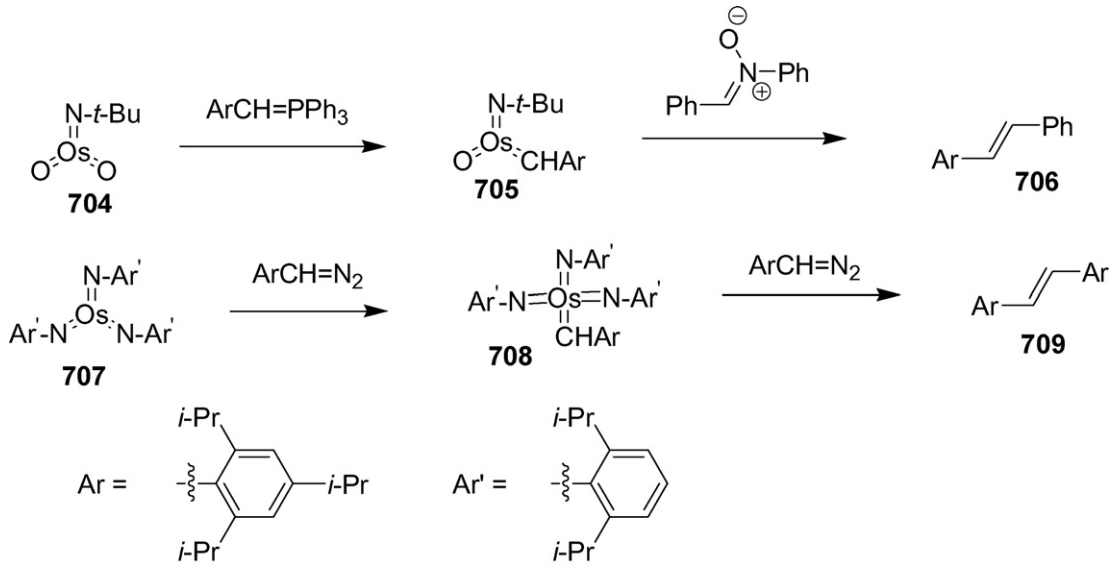
Carbene-derived products were obtained from the reaction of ruthenium complex **699** (Scheme 79) with various carbene sources [999]. Reaction with diazo compounds led to the Ru–S insertion product **700**, likely derived from addition of sulfur to carbene complex intermediate **701**. Reaction with phenylacetylene led to a related compound (**702**) likely derived from addition of sulfur to a vinylidene complex intermediate. Kinetically the four-membered ring product (**703**) was produced, which isomerized to the three-membered ring compound **702** at elevated temperatures.

Osmium–carbene complexes (*e.g.* **705**, Scheme 80) were generated through the reaction of Wittig reagents with osmium oxo complexes (*e.g.* **704**) [1000]. The complexes were stable in solution and were characterized by NMR and electrospray mass spectrometry. The carbene complexes react with diazo compounds or nitrones to afford alkenes. Alternatively similar carbene complexes could be generated by reaction of tris(imido)osmium complex **707** with diazo compounds. Subsequent reaction with additional diazo compound led to the alkene.

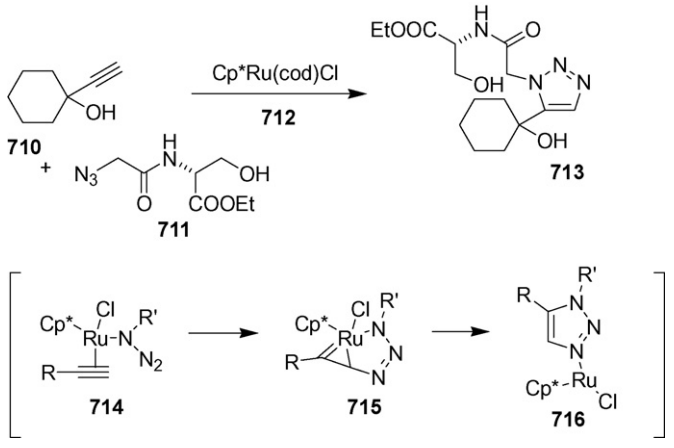
Ruthenium–carbene complexes (*e.g.* **715**, Scheme 81) were suggested as intermediates in the ruthenium-catalyzed 1,3-dipolar cycloaddition of azides with alkynes [1001]. A key step in the proposed mechanism is the conversion of the azido-alkyne complex



Scheme 79.



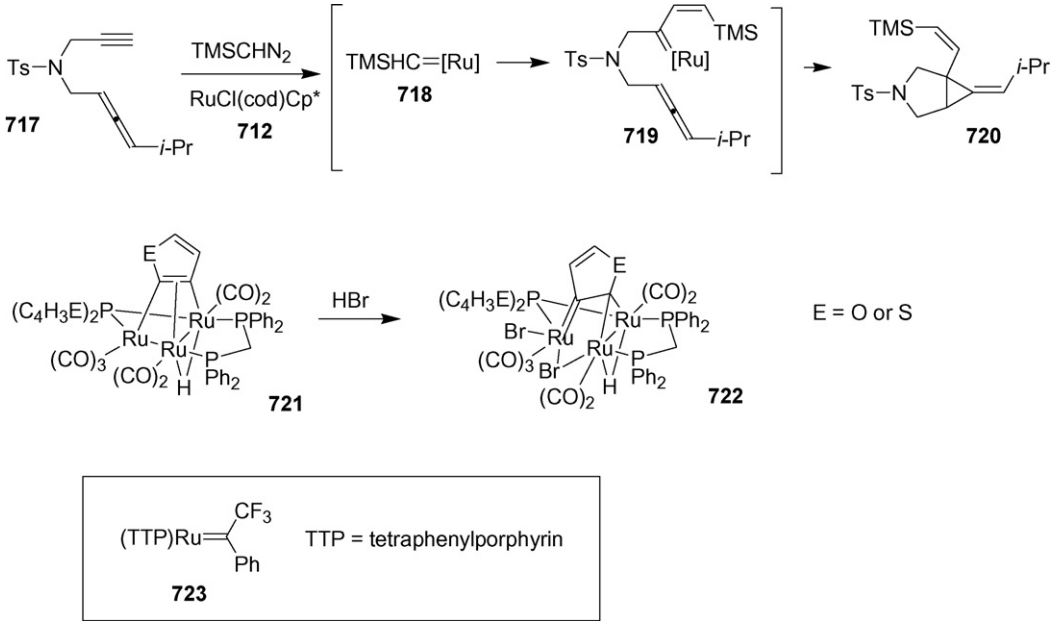
Scheme 80.



Scheme 81.

714 to the η^2-alkenyl complex **715** followed by migration of the nitrogen ligand to the carbene carbon. This mechanism was supported by DFT studies.

Other studies of carbene complexes in this category are depicted in Scheme 82, and include: (1) *in situ* generation of ruthenium–carbene complex (**718**), followed by its reaction with an allene-yne (*e.g.* **717**) to afford ring-fused alkylidenecyclopropanes (*e.g.* **720**) [1002]; (2) formation of a ruthenium–carbene complex (**722**) through reaction of tris(ruthenium) complex **721** with HBr [1003]; (3) a crystal structure report of ruthenium porphyrin–carbene complexes (*e.g.* **723**) and their synthesis [1004,1005]; (4) hypothetical involvement of mononuclear ruthenium–carbene and/or carbyne complexes as intermediates in the Fischer–Tropsch reaction (the C–O bond cleavage event required excessive activation energy in mononuclear systems) [1006]; (5) spectroscopic and DFT studies of carbenes generated from the reaction of surface-bond ruthenium nanoparticles and diazo compounds [1007]; and (6) a DFT study of the alkene cyclopropanation using Cp(CO)LFe=CHR cations [1008]. The gen-



Scheme 82.

eration of iron–carbene complexes through the interaction of laser ablation-produced iron atoms with halomethane derivatives was reported [1009]. The products were deposited in an argon matrix and identified through their IR spectra. Hypothetical products were studied by DFT and their predicted spectra compared with the observed spectra. Similar carbene complexes were generated through reaction of laser ablation-generated ruthenium atoms with methane and halogenated methane derivatives containing 1–4 halide atoms [1010].

2.3.5.2. Group 8 metalla-aromatics. The formation and reactivity of osmabenzene derivatives (*e.g.* **728**, Scheme 83) was reported [1011]. Protonation of chelating η^2-alkyne complex **725** leads to chelating enone complex **726**. Subsequent treatment with trimethylphosphine leads initially to chelating enone complex **727**, which affords the osmabenzene upon warming to room temperature. A similar osmabenzene (**733**) was also reported [1012]. Reaction of diethynylmethanol (**729**) with osmium complex **730** leads to the chelating allylic alcohol complex **731**. Heating to 60 °C leads to a mixture of four compounds, osmabenzene **733**, osmafuran **734**, and alkene-chelates **735** and **736**. Addition of sodium bicarbonate during the thermolysis maximizes the yield of osmabenzene **732**. The key event in formation of the osmabenzene complex is formation of the cationic π-allyl complex **732**. Addition of acetic acid led to the optimal amount of allene chelate **735**. Addition of acetic anhydride led to the optimal amount of enone chelate **736**. Extensive time of complex **731** at 25 °C led to the optimal amount of osmabenzofuran **734**. Protonation of ruthenabenzene derivative **737** led to the dialdehyde **738** [1013]. An iron metallabenzene complex (**740**) was proposed as an intermediate in the conversion of cyclic iron acylate complex **739** to the analogous Cp complex (**741**) [1014].

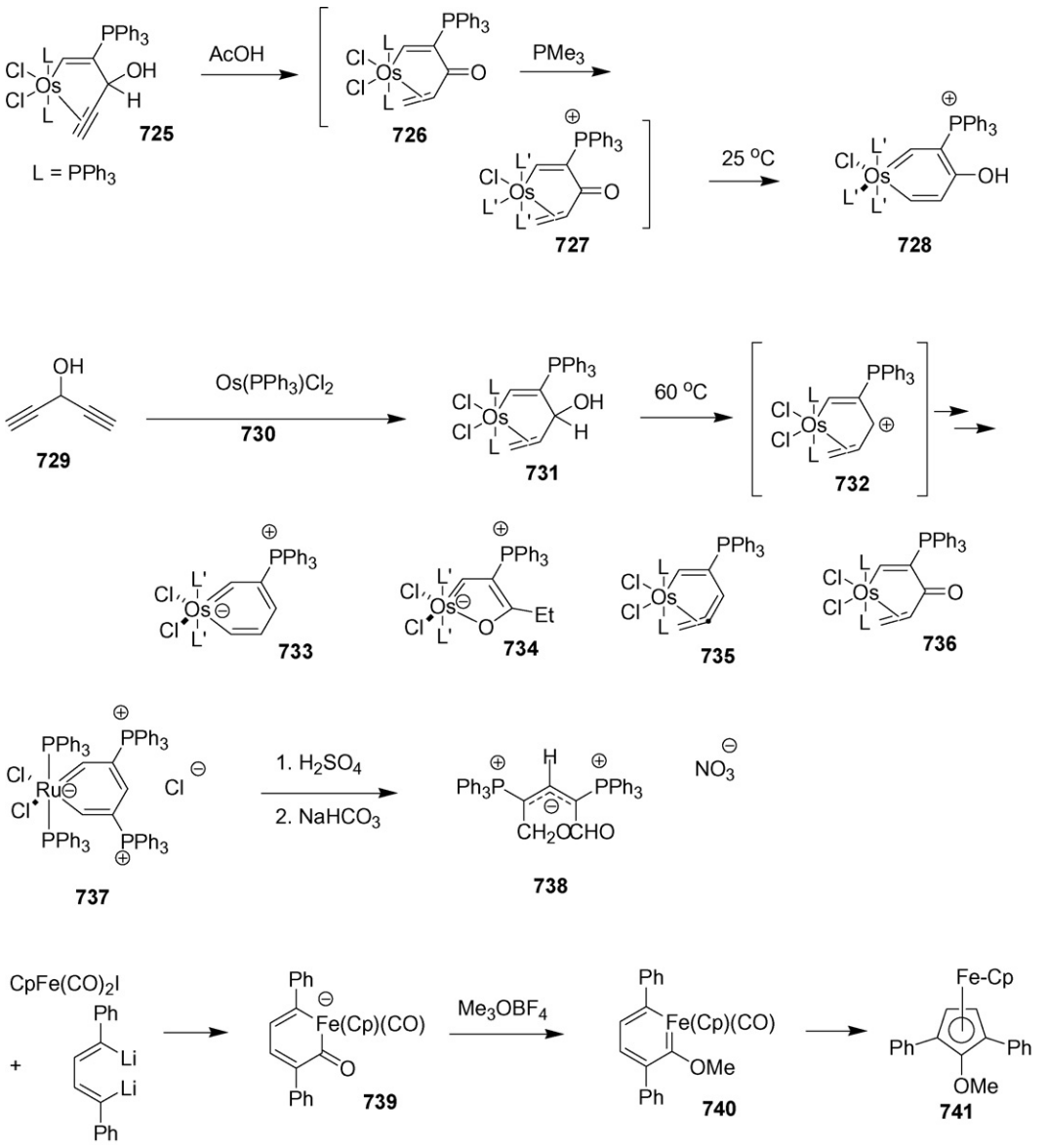
Osmafuran-hydrides (*e.g.* **743**, Scheme 84) were produced in the coupling of osmium–vinylpyridine complexes (*e.g.* **742**) with α,β-unsaturated ketones [1015]. The bis(osmafuran) **743** is an important resonance form of bis(β-osmaenone) complex **744**. Deprotonation afforded the neutral osmafuran **745**, which undergoes reprotonation at the osmafuran ring to afford observable carbene complex **746**, which converted to the carbyne complex **747** above room temperature. The formation of osmaisoindoles (*e.g.* **749**) was also reported [1016]. A double C–H activation process resulting in the bis(osmaindole) **749** was observed during the reaction bis(iminyl)benzene derivative **748** with $OsH_6(Pi\text{-}Pr_3)_2$.

2.3.5.3. Bis(carbene)ruthenium complexes from coupling of two alkynes and a ruthenium complex. Numerous studies have shown that ruthenacyclopentadienes obtained through the oxidative ligand coupling of ruthenium(II) complexes with two alkynes are better represented as metallacyclopentatrienes, thus they are included in this review [1017].

The cationic bis(carbene)ruthenium complex **750** was obtained through the reaction of complex **712** with phenylacetylene in benzene [1018]. When the reaction was conducted in methanol, the metallapentatriene diruthenium complex (**751**) was accompanied by COD-ligand–alkyne [2+2+2]-cycloaddition products (**752**, **753**). Related products were obtained using 3-hexyne or *t*-butylacetylene (Scheme 85).

Bis(carbene)–ruthenium complexes are likely intermediates in ruthenium-catalyzed alkyne trimerization reactions (see Scheme 86). Reaction of dialkyne **754** with terminal alkynes in the presence of either RuCl(cod)Cp* (**712**) or Grubbs catalyst I (**1**) led to the alkyne trimerization product (**755**) [1019]. The bis(iodoalkyne) **756** undergoes successful ruthenium-catalyzed cocyclization with alkynylboronates (*e.g.* **757**) [1020]. Ruthenium-catalyzed trimerization of ethynyl-deoxyribose derivatives was reported [1021]. Related bis(carbene) complexes (*e.g.* **761**) were suggested as intermediates in [2+2+2]-cycloadditions of dialkynes with alkenes. This coupling proceeds via formation of a hexatriene derivative (**763**), which subsequently undergoes electrocyclic ring closure to afford the apparent [2+2+2]-cycloaddition product (**764**) [1022,1023].

2.3.5.4. Heteroatom-substituted Group 8 metal–carbene complexes. Several papers in 2008 report on the chemistry of carbene- and carbyne complexes bridged by two iron atoms (see Schemes 87 and 88). The reaction of aminocarbene–diiron complexes (*e.g.* **767**, **769**, **771**, Scheme 87) with isocyanides followed by base (NaH) was reported [1024]. The direction of the reaction was highly dependent on the substitution pattern of the bridging ligand. The methyl-substituted compound (**767**) led to the aminocarbene complex **768**. The ester analog **769** led to the insertion product **770**. The bridge-unsubstituted compound **771** led to the bridging

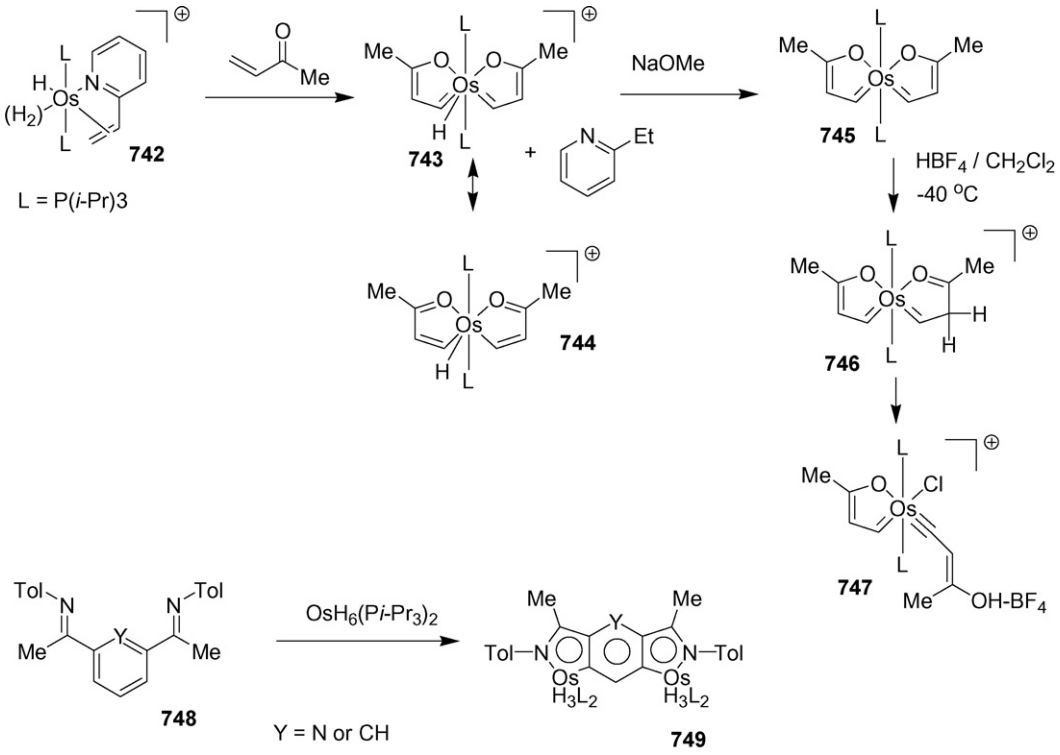


Scheme 83.

aminocarbyne complex **772** and the Cp-addition product **773**. The mechanism for the formation of **772** involves deprotonation and formation of the ethynyl complex (**774**), followed by replacement of the acetylide ligand with the isocyanide ligand. Sulfenylation from bridging carbene–diiron complexes was reported [1025]. Thiolate complexes (*e.g.* **776**) underwent alkylation reactions (*e.g.* formation of **777**) or oxidation to disulfides (*e.g.* **778**) [1026]. The reaction of cationic bridging aminocarbyne-diiron complexes (*e.g.* **779**, Scheme 88) with alkenes was reported [1027]. Reaction with alkenes in the presence of trimethylamine-N-oxide and sodium hydride let to the insertion product, bridging allenyl complexes (*e.g.* **780**). The proposed reaction mechanism involves coordination of the alkene to the vacant coordination side created by amine oxide induced CO removal followed by insertion and deprotonation. In the absence of base, a cationic nitrile complex (*e.g.* **781**) could be isolated from the reaction of complex **779** with acrylonitrile. The reaction of bridging aminocarbyne complexes with allenes (*e.g.* **782**) was also reported [1028]. The reaction of complex **779** with allene **782** followed by base and CO removal results in the bridged carbene-π-allyl complex **783**. Further treatment with triflic acid leads to the cationic binuclear carbene complex **784**. The mononuclear cyclic carbene complex **785** was formed upon treatment of the cationic complex with base.

The reactivity of fluxional chelating aminocarbene–ruthenium complexes (*e.g.* **786**–**787**, Scheme 89) was reported [1029]. The equilibrium mixture converted to the chloro complex **788** when dissolved in dichloromethane. Upon treatment with triethylamine the isomeric complex **789** was obtained. Treatment with the stronger base $NaNTMS_2$ led to the fluxional carbene complex **790**–**791**. Further reaction with ligands (*i.e.* CO or PPh_3, amines) led to the ligand-substituted nonfluxional carbene complex **792**. Reaction with secondary phosphines led to non-chelating aminocarbene complex **793**. Silicon–hydrogen insertion products were obtained upon treatment with hydrosilanes.

The synthesis of ruthenium– or osmium–carbene complexes (*e.g.* **796**/**797**, Scheme 90) through net C–H activation of pyridine derivatives was reported [1030]. Reactions with 2-methylpyridine led to the aminocarbene complexes, while reaction with simple pyridine led only to the simple N-ligated pyridine complexes. The carbene formation process has been denoted as capturing the minor carbene tautomeric form of a pyridine. The five-coordinate complex **797** was obtained in the ruthenium system, which converted to



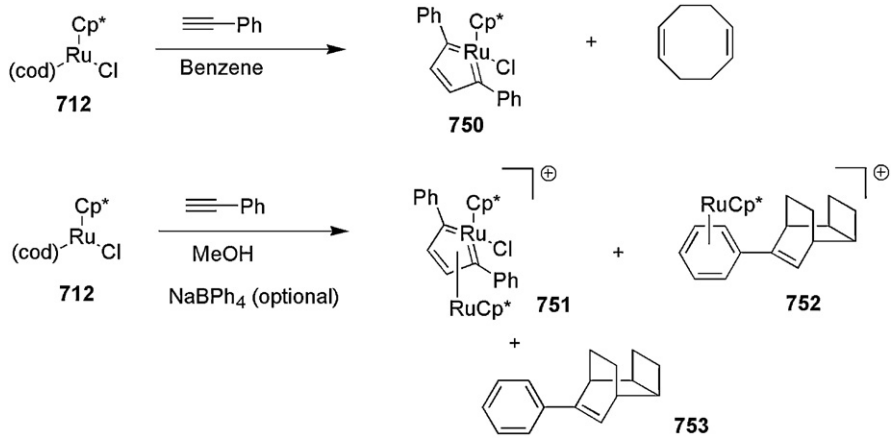
Scheme 84.

the six-coordinate complex **796-Ru** upon treatment with hydrogen. The reaction pathways leading to carbene complexes were studied by DFT calculations. The most reasonable mechanism involves intermolecular protonation of the pyridine nitrogen by the metal hydride, followed by C–H activation of the protonated heterocycle, and dihydride to dihydrogen ligand tautomerization. Reactions from the N-coordinated pyridine complexes were considerably higher in energy.

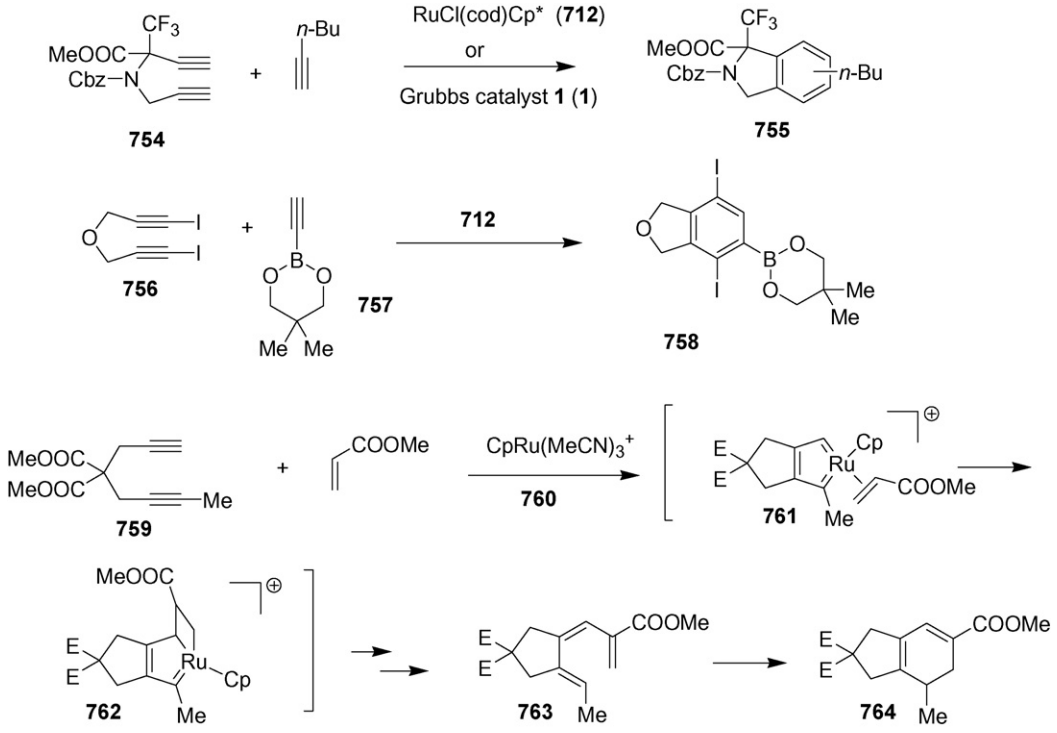
A polynuclear Fischer carbene–ruthenium complex (**799**, Scheme 91) was generated from the reaction of cobalt complex **798** with $Ru_3(CO)_{12}$ [1031]. The analogous cobalt–carbene complex **800** was generated through reaction of complex **798** with $Co_2(CO)_8$.

Alkoxycarbene–iron complexes (*e.g.* **802**, Scheme 92) are likely intermediates in the formation of chelating π-allyl iron complexes (*e.g.* **803)** from the reaction of η^4-diene–iron complex **801** with an organolithium reagent followed by alkylation [1032]. Hydroxycarbene–osmium complexes were suggested as intermediates in the osmium-mediated decarbonylation of aliphatic aldehydes [1033].

2.3.5.5. Group 8 metallacumulene complexes. Many examples of the formation of metal–vinylidene complexes (**805**, Scheme 93) via coupling of coordinatively unsaturated Group 8 metal complexes with terminal or silylated alkynes were reported in 2008. Representative examples are depicted in Fig. 12. Common reaction pathways for these complexes include reaction with nucleophiles to form vinylmetal species (**808**), reaction with alcohols (or amines) to form Fischer carbene complexes (**809**) or water to form metal acyls (**807**), and deprotonation at the β-position to form alkynylmetal complexes (**806**). Other common synthetic routes to metal vinylidenes included addition of electrophiles to metal acetylide complexes (*e.g.* the reverse of the reaction synthesizing **806**), and treatment of acylmetal complexes with dehydrating agents (*i.e.* the reverse of the reaction synthesizing **807**). Metal–higher allenyli-



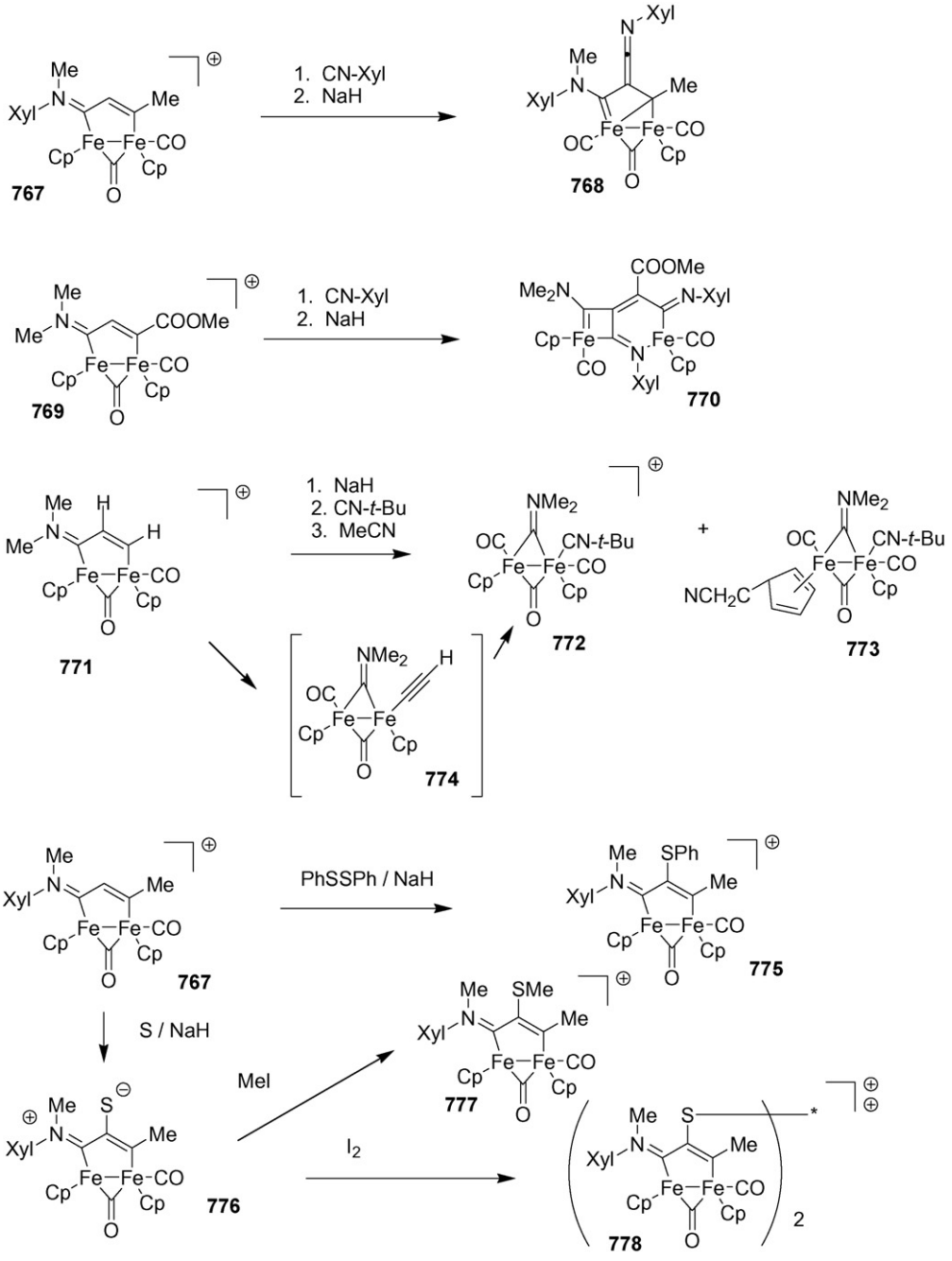
Scheme 85.



Scheme 86.

dene complexes (**811**, **816**) are produced from the coupling of coordinatively unsaturated Group 8 metal complexes with propargyl alcohols (usually those that contain no hydrogens β- to the OH group), or by addition of electrophiles to the δ-carbon of alkenylethynyl–metal complexes (**815**). Common reaction pathways for allenylidene complexes include reaction with nucleophiles at the γ-position, resulting in alkynylmetal complexes (**813**), or attack at the γ-position, resulting in allenylmetal complexes (**814**). Reaction with alcohols or amines can lead to α,β-unsaturated Fischer carbene complexes (**812**). Representative examples of this class of compounds are depicted in Fig. 12.

Specific reports which highlight the reaction pathways of Scheme 93 are depicted in Fig. 12 and include the following examples of vinylidene complexes: (1) formation of iron–alkynylpyridine complexes [1034]; (2) double protonation of 4-pyridylalkynylruthenium complexes to afford dicationic vinylidene complexes and protonation of complexes where the pyridine nitrogen is complexed to platinum [1035]; (3) electrophilic cyanation of neutral alkynylruthenium complexes (*e.g.* **818**) to afford cyanovinylidene complexes (*e.g.* **819**) [1036]; (4) formation of tetrametallic vinylidene complexes (*e.g.* **821**) through methylation of butadiynyl complex **820** [1037]; (5) formation of ruthenium vinylidene complexes (*e.g.* **822**) and subsequent hydrazine additions to form hydrazinocarbene complexes (*e.g.* **823**) [1038]; (6) formation of cationic ruthenium vinylidene complexes and studies of their decomposition in acetonitrile to afford terminal alkynes [1039]; (7) formation of a ruthenium vinylidene complex featuring a 2-(diphenylphosphinomethyl)pyridine ligand from a ruthenium dichloride and use of the starting dichloride complex and ethyl diazoacetate as a ROMP catalyst [1040]; (8) formation of dicationic vinylidene, allenylidene (*e.g.* **825**) and Fischer carbene–ruthenium complexes (*e.g.* **826**) featuring the tacn ligand [1041]; (9) formation and electrochemistry of ruthenium vinylidene complexes (*e.g.* **827**) and conversion to bis(alkynyl)ruthenium complexes (*e.g.* **828**) [1042]; (10) formation of neutral vinylidene, allenylidene and Fischer carbene complexes featuring N,N,O-heteroscorpionate ligands [1043]; (11) formation of cationic ruthenium–carbene complexes that contain remote flavinol groups and subsequent deprotonation to afford alkynylruthenium complexes [1044]; and (12) low-temperature NMR observation of metal–vinylidene complex intermediates involved in anti-Markovnikov alkyne hydration [1045]. Several processes likely involve metal–vinylidene intermediates synthesized by these pathways, including: (1) synthesis of alkynylruthenium bimetallic complexes from terminal alkynes connected to other metals [1046–1050]; (2) electrophilic iodination of ethynylruthenium complexes [1051]; (3) formation of alkynylmetal complexes from reaction of ruthenium, nickel, or gold halides with azo group-containing terminal alkynes in the presence of base [1052]; (4) synthesis of bis(alkynylruthenium) complexes through reaction of a bis(ruthenium) complex with terminal alkynes in the presence of diethylamine [1053,1054]; (5) ruthenium-catalyzed addition of thiourea derivatives to terminal alkynes [1055]; and (6) formation of a $Ru(C{\equiv}CPh)(CO)(PPh_3)Cp^*$ as a minor product from the reaction of $RuCl(PPh_3)_2Cp^*$ and $\{CuC{\equiv}CPh\}_n$ (the CO ligand arises from hydration of a vinylidene ligand followed by retro CO insertion) [1056]. Formation and electrochemistry of allenylidene complexes linked to a bipy system (*e.g.* **829**) from the corresponding propargylic alcohol was also reported [1057,1058]. Several processes reported in 2008 invoke metal–higher cumulene complexes as intermediates, including: (1) ruthenium-catalyzed alkyne hydration via spectroscopically observable metal–vinylidene complex intermediates [1059]; (2) formation of oxygen-bridged ruthenium alkenylcarbene complexes (*e.g.* **830**) from propargyl alcohols and subsequent use of the carbene complex as an alkene cyclopropanation catalyst [1060]; (2) alkyne dimerization [1061]; (3) ruthenium-catalyzed 1,3-rearrangement of propargyl carbamate derivatives [1062]; (4) reaction of a hydroxymethylethynylruthenium complex with HBF_4/PPh_3 to afford the 1-ruthena-allenylphosphonium salt [1063]; and (5) formation of an osmium allenylidene complex $\{[P(OEt)_3]_5Os{=}C{=}C{=}CPh_2\}^+$ from 1,1-diphenylpropargyl alcohol and $\{[P(OEt)_3]_4OsOTf\}$ [1064]. Electrochemical oxidation products of alkynylmetal complexes feature cumulenylidene resonance contributions [1065]. Electrochemical oxidation products



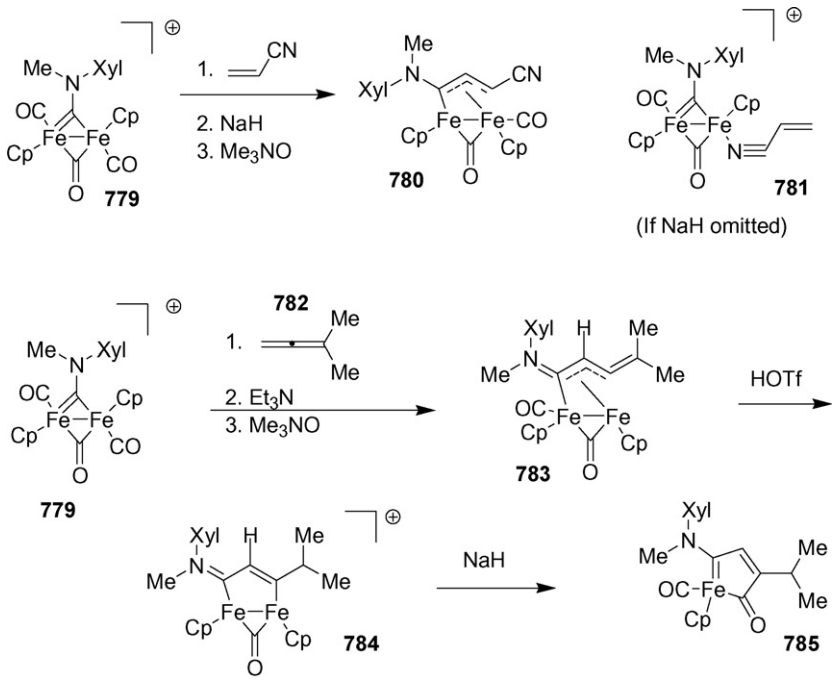
Scheme 87.

of alkyne-bridged bimetallics are likely bis(cumulenylidene) complexes [1066,1067].

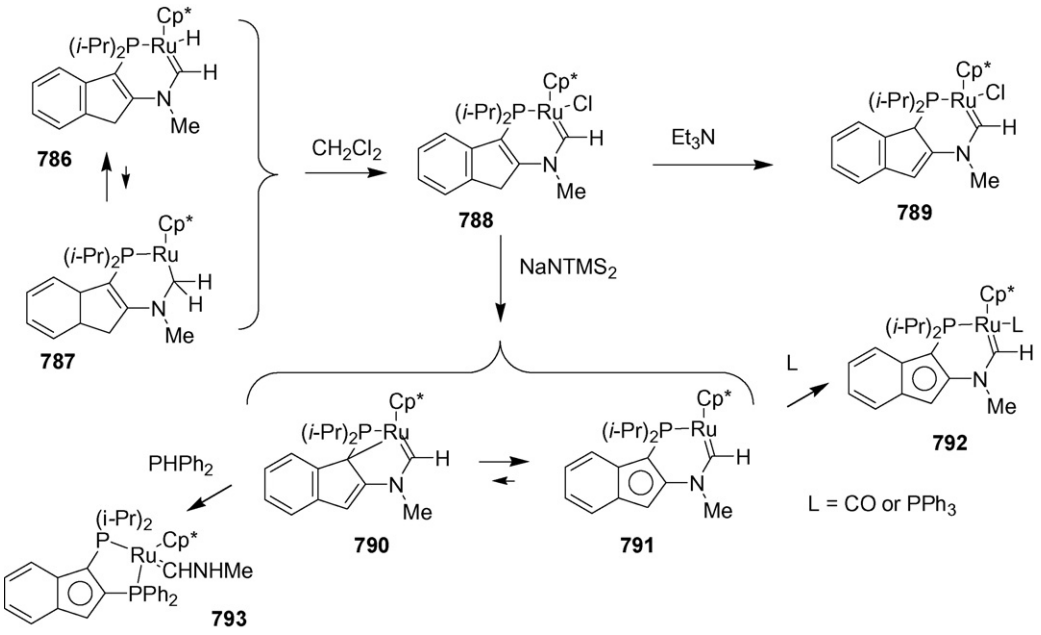
The formation and reactivity of pyridinylvinylidene complexes was reported (Scheme 94) [1068]. Reaction of ruthenium halide **831** with 2-ethynylpyridine led to either the alkynyluthenium complex **833** or the Fischer carbene complex **834**, depending on the solvent choice. Protonation and Lewis acid complexation were reported for the alkynylruthenium complex **833**. The acid–base adducts (*e.g.* **835**) feature substantial resonance contribution from the allenylidene complex form (**836**). Treatment with aqueous HBF_4 leads to the hydroxyborate complex **837**, which eventually evolves into the boryloxycarbene complex **838**. N-alkylation reactions were also reported for the alknylruthenium complexes.

Cationic ruthenium vinylidene complexes (*e.g.* **841**, Scheme 95) were suggested as intermediates in the reaction of cyclopropenylruthenium complexes (*e.g.* **839**) with trimethylsilyl azide to form tetrazolate complexes (*e.g.* **840**) [1069]. In the case where the L′ ligand is MeCN, the reaction afforded the vinylidene complex, which could be isolated at low temperature.

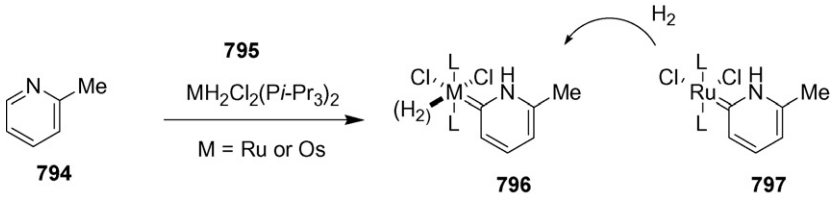
Several examples of stoichiometric and catalytic alkyne dimerization reactions were reported in 2008 (Scheme 96). Formation of cationic ruthenium vinylidene complexes (*e.g.* **843**) and their subsequent coupling with alkynes was reported [1070]. Formation of alkyne-coupled products (*e.g.* **844**) involves formation of the alkynyl(vinylidene) complex (**845**) followed by alkynyl migration and loss of chloride. Complex **844** was a catalyst for alkyne dimerization. Related cationic η^3-enynylruthenium complexes (*e.g.* **849**) were produced via treatment of bis(alkynyl)ruthenium complexes (*e.g.* **847**) with electrophiles [1071]. Dimerization of terminal alkynes by $[Ru(p\text{-cymene})Cl_2]_2$ to afford conju-



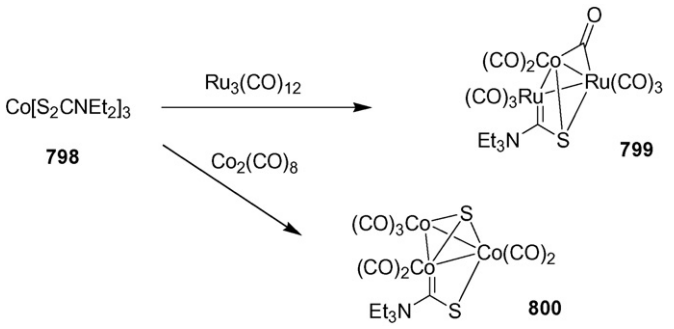
Scheme 88.



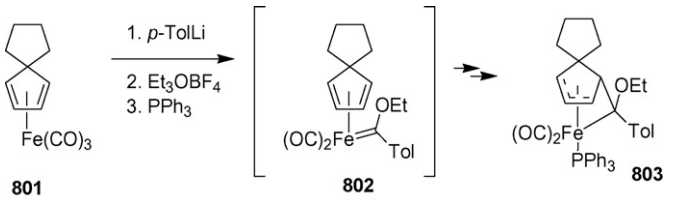
Scheme 89.



Scheme 90.



Scheme 91.



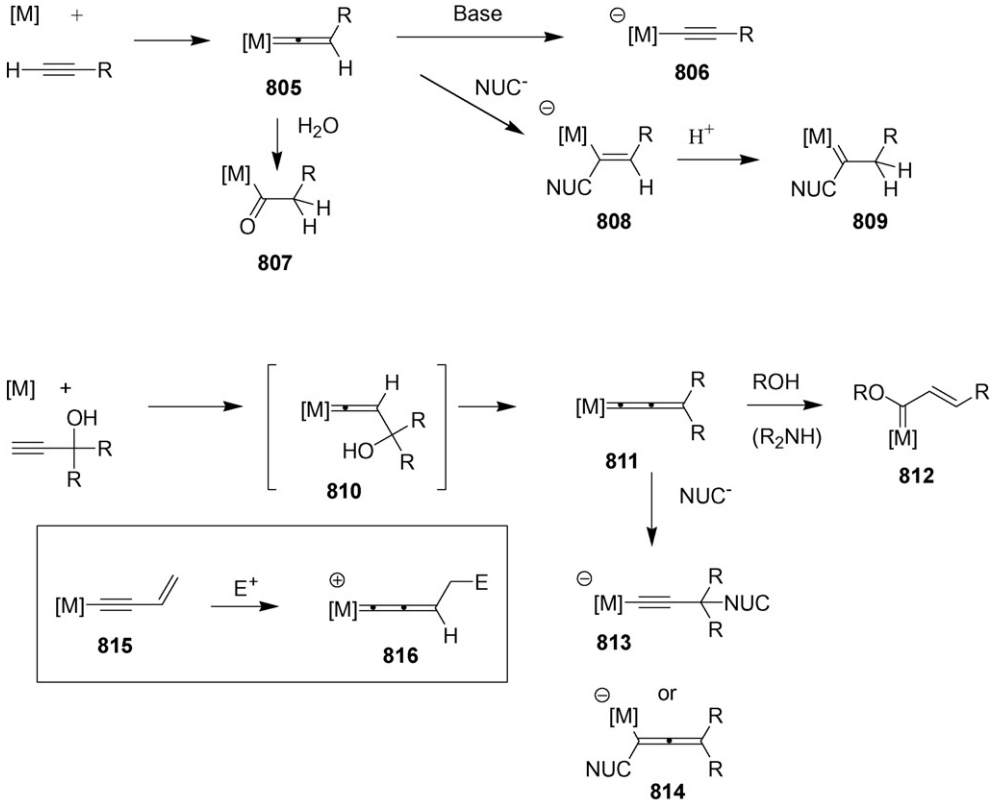
Scheme 92.

gated enynes was suggested to involve vinylidene intermediates [1072].

Formation of ruthenium vinylidene complexes (*e.g.* **851**, **853**, Scheme 97) from internal alkynes was reported [1073]. The η^2-alkyne complex **852** was obtained from the reaction of complex **850** with various electron-deficient alkynes at 50 °C. The vinylidene complex **851** was obtained directly from reaction of complex **850** with aryl-substituted alkynes at 70 °C. The η^2-alkyne complex **852** could be converted to the vinylidene complex **853** by irradiation. In the formation of the vinylidenes complex from 1-phenylpropyne, C-13 labeling studies revealed that the product forms predominantly from methyl group migration.

The reaction of bis(allene)s (*e.g.* **854**, Scheme 98) with $Fe_2(CO)_9$ led to cyclopentadiene–iron complex **857** [1074]. The proposed mechanism for this transformation involves reversible conversion of the silylallene to a propargylsilane (**855**), followed by transformation to the vinylidene–iron complex (**856**). Ring closure and CO insertion provides the product **857**.

Ruthenium–allenylidene complexes were proposed as intermediates in a variety of propargyl substitution reactions (see Scheme 99) [1075]. Ruthenium complex **860** catalyzes the coupling of propargyl alcohols (*e.g.* **858**) with conjugated dienes (*e.g.* **859**) to afford dienyne derivatives (*e.g.* **863**). In this case the nucleophilic diene attacks the allenylidene intermediate (**861**). The use of bridging phosphidodiruthenium complexes to catalyze propargyl substitution reactions via allenylidene–ruthenium intermediates was also reported [1076]. Diruthenium complex **860** catalyzes the isomerization of 3-butyne-1,2-diols (*e.g.* **864**) to furans (*e.g.* **867**) [1077]. Key steps in this transformation include formation of the allenylidene complex (**865**) followed by isomerization to the enol–vinylidene complex (**865**) followed by nucleophilic attack of oxygen at the carbene carbon to close the furan ring. A similar nucleophilic substitution of enyne triflates (*e.g.* **868)** via butatrienylidene intermediates (*e.g.* **870**) was also reported [1078]. The allenylidene complex intermediates (*e.g.* **873**) can be captured through an intramolecular ene reaction to afford cyclic enynes (*e.g.* **875**) [1079]. Further reaction with platinum chloride results in enyne cycloisomerization to afford cyclopropane **876**, another metal–carbene-based reaction process. Ethynyl ketones were also successfully employed in this transformation in place of enyne triflates. After generation of the butatrienylidene complex, nucleophilic addition to the γ-carbon leads to the observed substituted enyne.



Scheme 93.

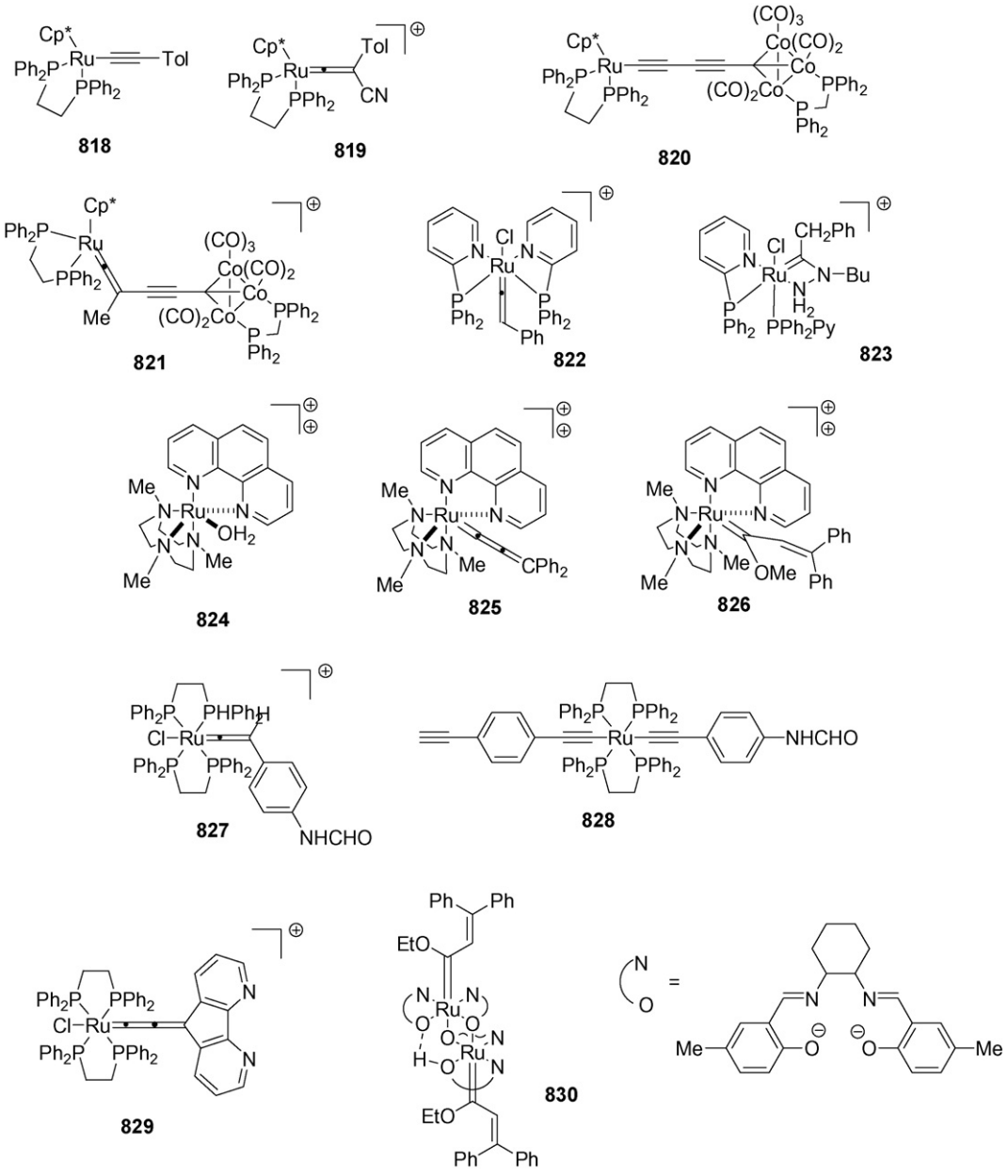


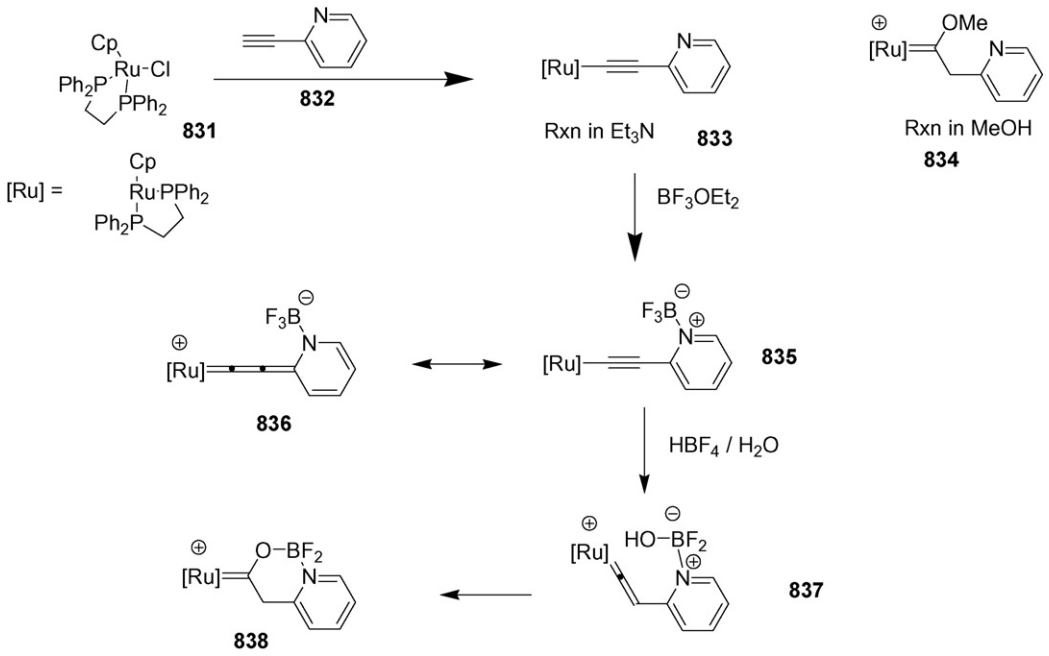
Fig. 12. Representative group 8 metallacumulene complexes, precusors, and reaction products reported in 2008.

The formation and reactivity of cationic allenylidene–osmium complexes (*e.g.* **879**, Scheme 100) was reported [1080]. Reaction of osmium complex **877** with 1,1-diphenylpropargyl alcohol leads to the observable hydroxyvinylidene complex **878**, which affords the allenylidene complex **879** slightly above room temperature. Chloride removal and ligand exchange processes were reported for allenylidene complex **879**. Treatment of the cationic complex **881** with isopropyl alcohol and sodium chloride led to the carbyne complex **882** and acetone in a net hydrogen atom transfer process.

2.3.6. Group 9 metal–carbene complexes

2.3.6.1. Simple carbene complexes. Several Group 9 metal–carbene complexes were prepared through C–H oxidative addition processes (see Scheme 101). Reaction of cyclic imine derivative **885** with rhodium complex **886** led to the carbene complex **887** [1081]. The reaction of complex **888** with anisole results in an equilibrium mixture of the starting materials and arene C–H oxidative addition product **889**, however upon heating to 90 °C carbene complex **890** is formed [1082].

Reaction of dihydridoiridium complex **891** (Scheme 102) with norbornene and methyl *t*-butyl ether led directly to carbene complex **892** [1083,1084]. A variety of reaction processes were demonstrated for carbene complex **892**. Thermolysis led to the net dealkylation product **895** and isobutylene. Conversion of the carbene complex to the carbonyl complex (see inset of Scheme 102) was proposed to involve hydrogen transfer with loss of isobutylene to afford the metal formyl followed by retro CO insertion. Reaction with carbon dioxide led to ethyl formate and the carbonyl derivative **898**. The proposed mechanism involves stepwise [2+2]-cycloaddition to afford the oxametallacyclobutanone (**897**) followed by retro [2+2]-cycloaddition. Similar reactions were observed using other heterocumulene species. Reaction of the carbene complex with azides led to the iminoether **894** and the iridium–dinitrogen complex **893** [1085]. A moderately catalytic



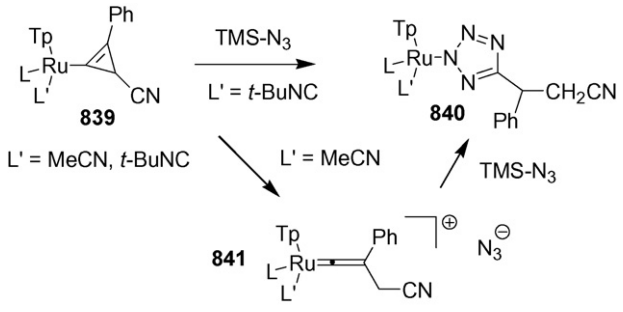
Scheme 94.

C–H functionalization reaction of methyl *t*-butyl ether based on this transformation was demonstrated. Similar formation and reactivity studies for analogous aminocarbene complexes were reported [1086].

Formation of iridium–carbene complexes (*e.g.* **902**–**904**, Scheme 103) through C–H oxidative addition of vinyl ethers was reported [1087]. The initial coupling of iridium complex **901** with ethyl vinyl ether affords the metallacyclic carbene complexes **902** and **903**. The metallacyclopentene–carbene **902** undergoes an elimination reaction to afford cyclic alkenylcarbene complex **904**. A mechanism involving C–H oxidative addition and benzene elimination, followed by complexation of a second alkene and C–C coupling was proposed to account for the formation of carbene complex **902**. Formation of a pyridinylidene complex (**906**) was observed when iridium complex **905** was treated with bipyridyl [1088].

The formation of iridium–carbene complexes (*e.g.* **910**, Scheme 104) through C–H activation reactions of pyridinium salts (*e.g.* **907**) was reported [1089,1090]. The reaction was ligand directed. A variety of bipy and phenanthroline type systems were reported to undergo the transformation. The mechanism for the formation of the carbene complexes was studied by DFT calculations. The carbene complexes were employed as catalysts for the transfer hydrogenation of acetophenone.

Several carbene-based coupling reactions were reported for cobaltacyclobutene complexes (*e.g.* **911**, Scheme 105) [1091].

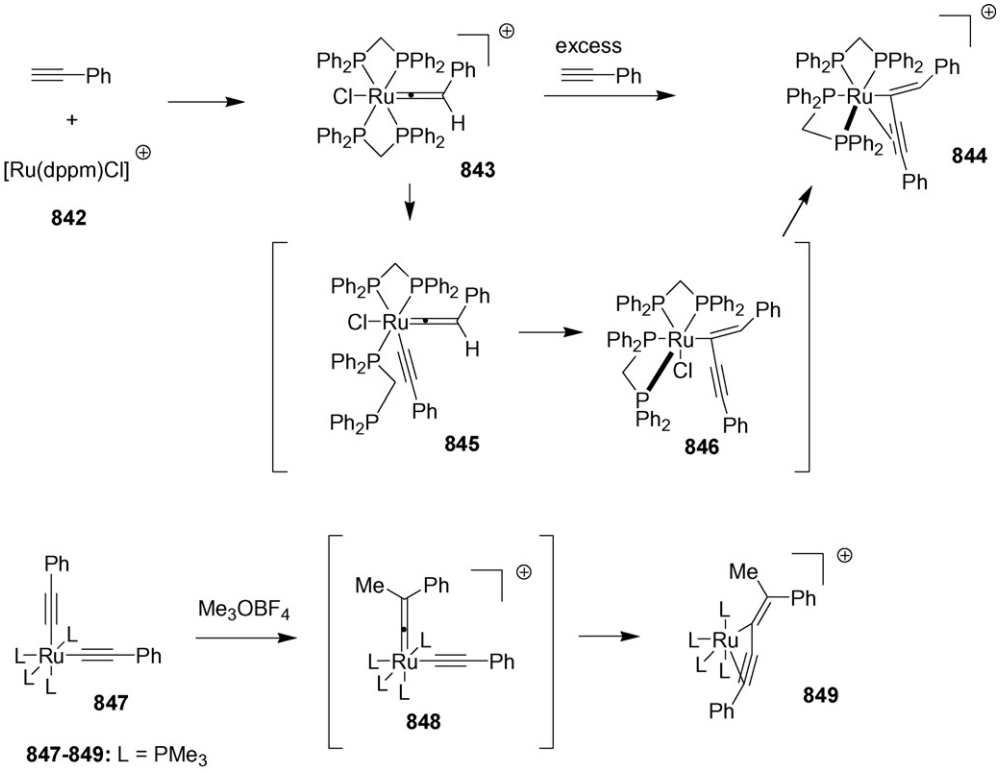


Scheme 95.

Reaction with fumarate esters (**914**) led to the 1,4-diene–cobalt complexes (*e.g.* **915**). Reaction with maleic anhydride (**916**) led to the oxametallbicyclo[2.2.2]octane derivative **917**. A mechanism involving reversible formation of a vinylcarbene complex (**912**) or oxametallabenzene complex (**913**) followed by metalla Diels–Alder reaction and isomerization was proposed.

Several reaction processes reported in 2008 involve Group 9 metal–carbene complexes generated from diazo compounds; representative examples are depicted in Scheme 106. Cobalt–carbene complexes were proposed as intermediates in the decomposition of N-ligated diazo compounds to symmetrical alkenes and diazadienes [1092]. Solid-state photolysis of a cobalt-ligated diazo compounds (*e.g.* **918**) or bis(diazo) complexes led to a single molecule magnetic material, presumably a cobalt–carbene complex [1093,1094]. Stereoselectivity models involving carbene complexes were proposed to explain the direction of asymmetric induction in various rhodium-catalyzed cyclopropanation reactions, including: (1) cyclopropanations employing α-diazophenylacetonitrile [1095]; (2) coupling of dienes with esters of α-diazovinylacetic acid, ultimately leading to [4+3]-cycloadducts after cyclopropanation and Cope rearrangement [1096]; (3) cyclopropanation of chiral enamides with α-diazoesters [1097]; (4) rhodium-catalyzed cyclopropanations using diazomalonates [1098]; and (5) iridium-catalyzed asymmetric cyclopropanation [1099]. Computational studies of Group 9 metal-catalyzed cyclopropanation include: (1) a DFT study of rhodium porphyrin-catalyzed C–H insertion reactions of diazoacetates with hexane [1100]; (2) bonding in hypothetical rhodium–carbene intermediates (**919**) in Rh_2OAc_4-catalyzed cyclopropanations, with emphasis on the degree of back bonding of various chelating ligands on the degree of back bonding in the carbene ligand [1101]. The coupling of the bridging methylene ligand in rhodium–osmium bimetallic complexes with diazo compounds likely involves carbene complexes [1102].

Additional reaction processes reported in 2008 involve Group 9 metal–carbene complex intermediates that were generated from precursors other than diazo compounds. Rhodium–carbene complexes (*e.g.* **921**) were proposed as intermediates in a nitrene initiated cascade reaction of enyne-sulfonamides (*e.g.* **920**) [1103].

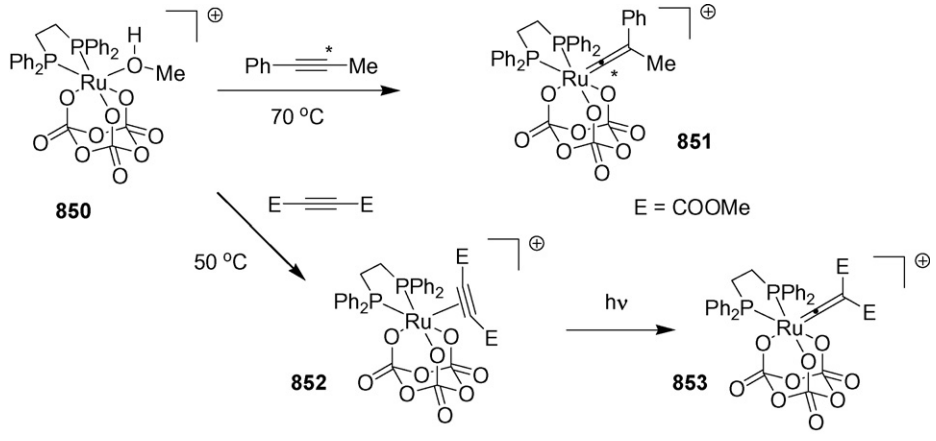


Scheme 96.

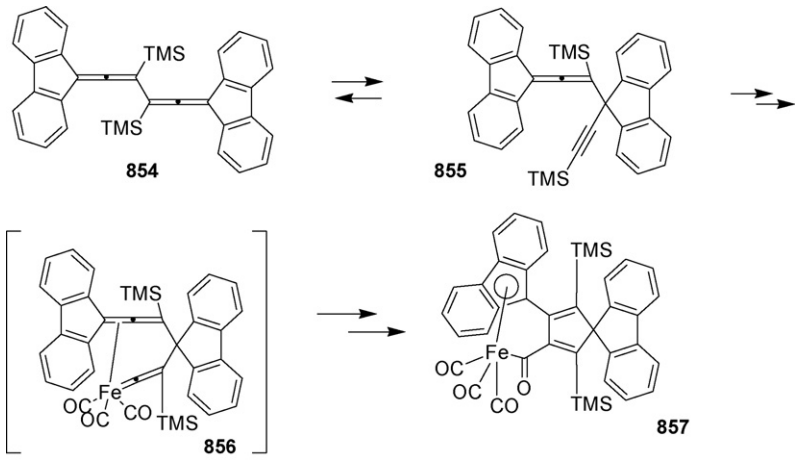
Rhodium–carbene complexes (*e.g.* **928**, **929**) were also suggested as intermediates in a novel rhodium-catalyzed cyclization of bicyclo[1.1.0]butane derivatives containing pendant alkene groups (*e.g.* **925**) [1104]. The proposed mechanism involves oxidative addition into the highly strained C–C bond followed by ring opening to either of two isomeric carbene complexes, **928** or **929**, which undergo intramolecular cyclopropanation. The Rh_2OAc_4-catalyzed cycloaddition of triazoles (*e.g.* **932**) and nitriles to afford N-sulfonated imidazoles (*e.g.* **934**) likely involves rhodium–carbene complex intermediates (*e.g.* **933**) [1105]. Cyclopropylcarbene–rhodium complexes (*e.g.* **936**) were proposed as intermediates in rhodium-catalyzed enyne metathesis [1106] (Scheme 107).

Iridium–carbene complexes (*e.g.* **939**, **941**, Scheme 108) were proposed as intermediates in the coupling of paramagnetic iridium–ethylene complex **938** with diazo compounds [1107]. A bimetallic compound (**940**) was produced from the reaction of complex **938** with ethyl diazoacetate. The analogous reaction with trimethylsilyldiazomethane led to the trimethylsilylmethyl complex **942**. In both cases, the key step in the proposed mechanism involves formation of a paramagnetic carbene/carbenoid complex. The mechanism and the structure of individual intermediates were evaluated by DFT calculations.

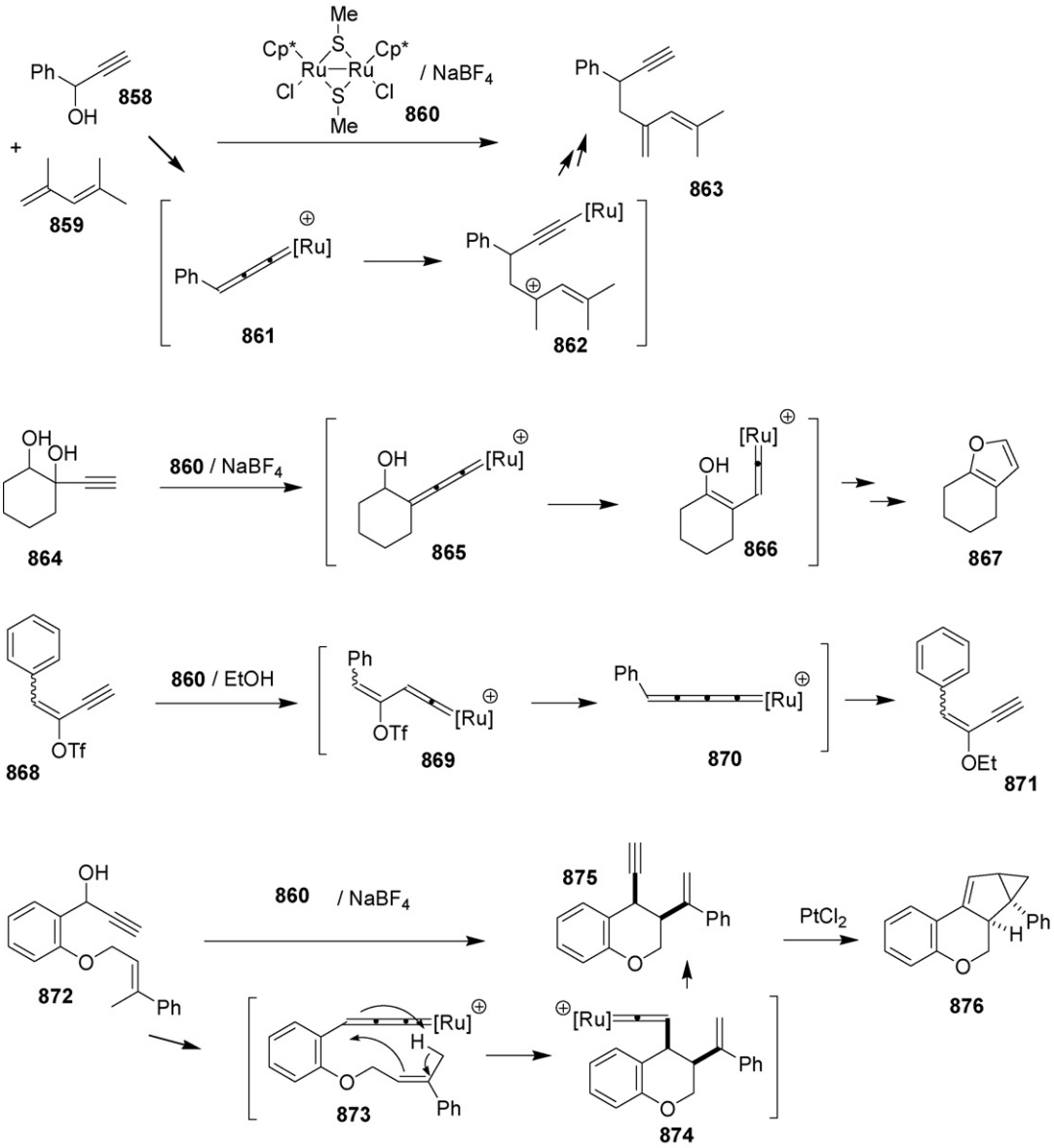
Other studies of Group 9 metal–carbene complexes (excluding metalla-aromatics and metallacumulenes) include: (1) preparation of cyclopropenylidene–rhodium (*e.g.* **943**, Scheme 109) iridium, nickel, and palladium complexes through either reaction of solutions of the free carbene **942** with coordinatively unsaturated transition metal complexes or carbene exchange processes [1108]; (2) formation of chelating Bertrand carbene–rhodium complexes [1109]; (3) possible involvement of cobalt carbene complexes in cobalt-catalyzed Fischer–Tropsch reactions [1110]; (4) involvement of iridium–carbene complexes in ROMP reactions initiated by an iridium(III) complex–methylaluminoxane system [1111]; (5) possible involvement of bridging carbyne–dirhodium



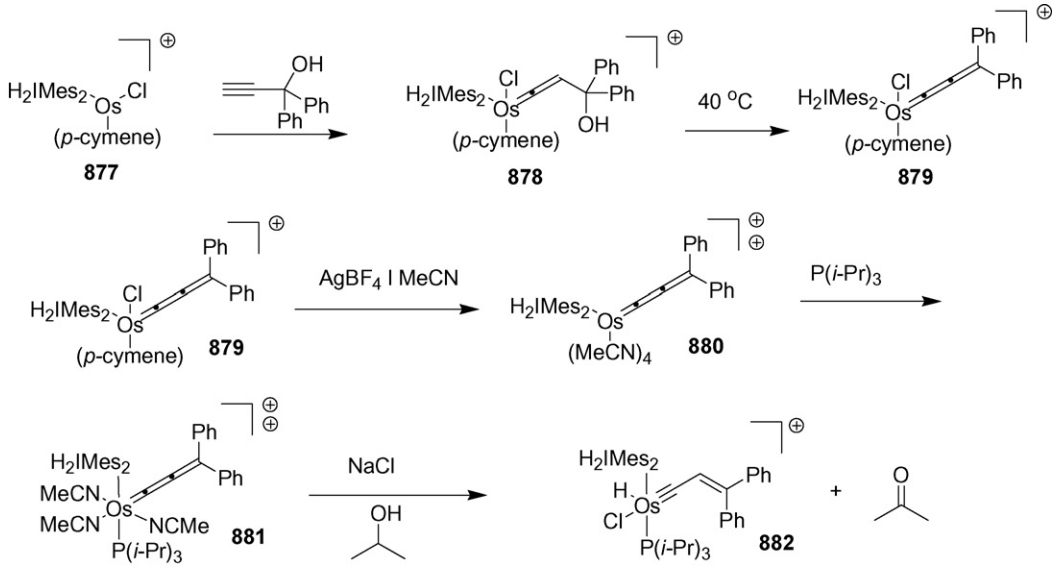
Scheme 97.



Scheme 98.



Scheme 99.



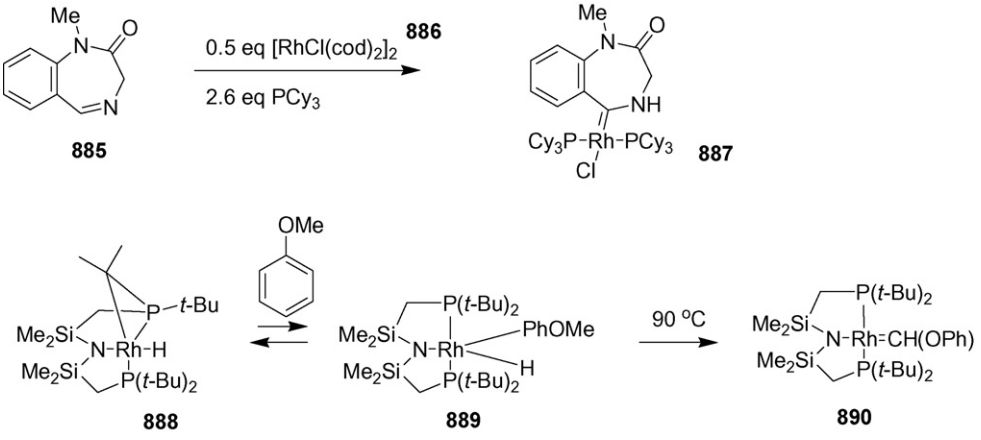
Scheme 100.

complex intermediates (*e.g.* **945**) in the generation of propene through oxidation of dimethylbis(μ-methylene)dirhodium complex **944** (model for Fischer–Tropsch reaction) [1112]; and (6) a DFT study implicating the involvement of hydroxycarbene–rhodium complexes in rhodium-catalyzed hydrocarbonylation of alkenes [1113].

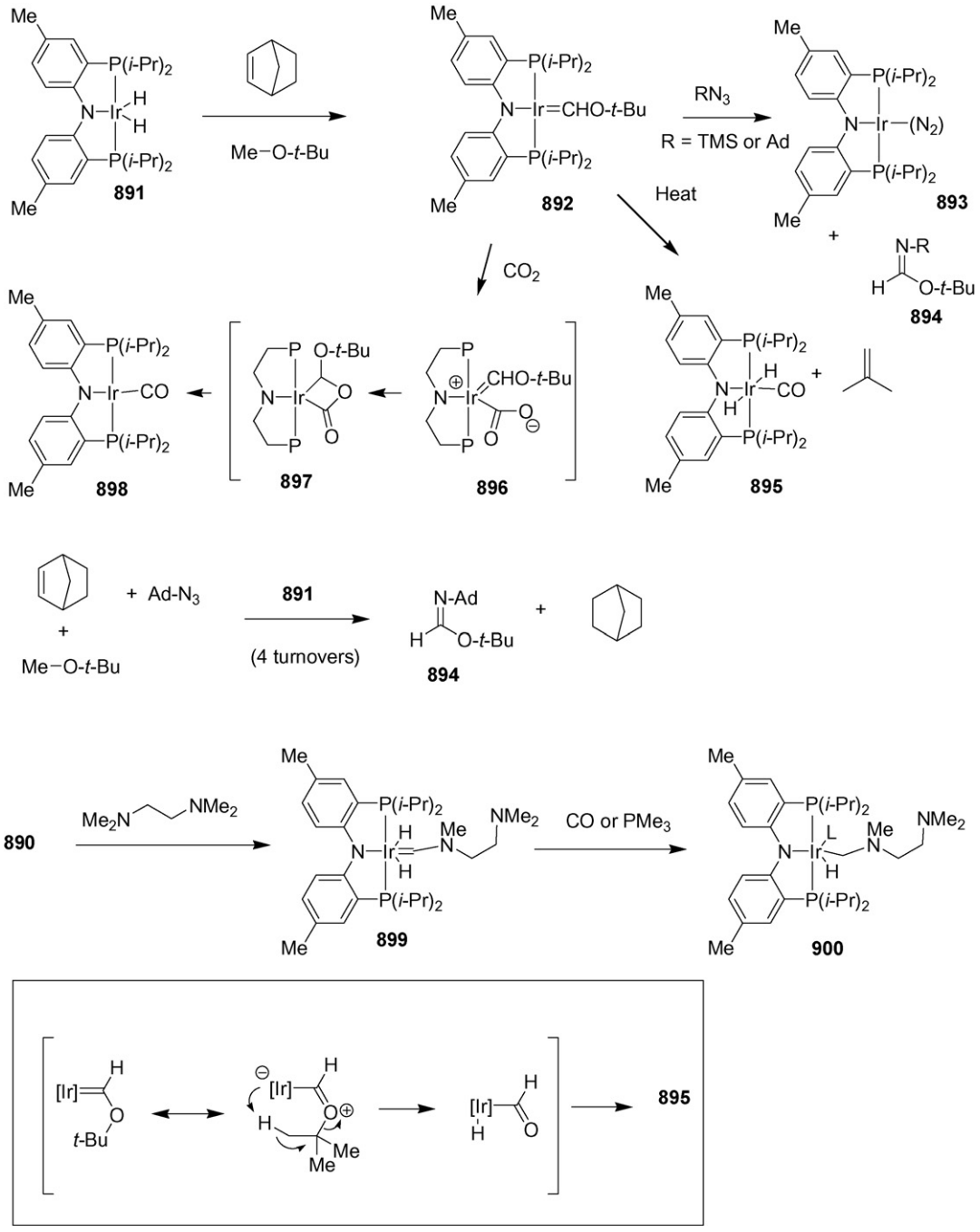
2.3.6.2. Group 9 metalla-aromatics. Iridabenzenes (*e.g.* **948**, Scheme 110) were obtained through methylation of (thiocarbonyl)iridacyclopentadienes (*e.g.* **947**) [1114]. Various electrophilic aromatic substitution reactions were reported for iridabenzene derivative **948**. Substitution occurs para to the SMe group. Synthesis and protonation reactions were reported for iridapyrroles (*e.g.* **953**, **956**) [1115]. Addition of methyllithium to aromatic nitriles (**950**) followed by capture with the iridium complex **951** leads to the chelate complex **952**, which affords the iridapyrrole (**953**) upon protonation. Iridapyrrole **953** (Y=N), which possesses an additional basic site, undergoes protonation at the pyridine nitrogen to afford the dicationic iridapyrroles **954**, while the complex lacking this extra ligation site (**953**, Y=CH) reacts with triflic acid to afford H_2 and iridium triflate complex **955**. The thiophene-pyrrole **956** undergoes ligand isomerization upon protonation to afford the S-coordinated dicationic complex **957**. The structure of iridathiabenzenes were studied by DFT calculations; the π-electrons in these systems were more localized than in benzene ring systems [1116].

2.3.6.3. Group 9 metallacumulenes. Similar synthetic procedures and reactivity patterns were generally observed for Group 9 and Group 8 metallacumulene complexes (Scheme 93).

The conversion of π-alkyne–rhodium complexes (*e.g.* **960**, Scheme 111) to the corresponding rhodium vinylidene complex (**961**) was determined to be a first-order process proceeding through an observable hydrido(alkynyl) complex (**962**) [1117]. This process was also studied computationally. The conversion of alknyl(hydrido)rhodium complexes to vinylidene complexes was studied computationally [1118]. The reaction of (η³-allyl)Rh(P*i*Pr$_3$)$_2$ (**963**) with two equivalents of phenylacetylene to afford propene and the alkynyl vinylidene complex **964** was the focus of these studies. The most reasonable pathway for the hydridoalkyne to vinylidene conversion is formation of the observable π-alkyne complex (**965**), which converts to a C–H bonded complex (**966**) followed by concerted conversion to the vinylidene complex. Formation of the vinylidene complex from the C–H oxidative addition product **967** was excessively high in energy. Rhodium–, cobalt– and ruthenium–vinylidene complexes are likely intermediates in the cyclodimerization of arylacetylenes catalyzed by metal–porphyrin complexes [1119].



Scheme 101.



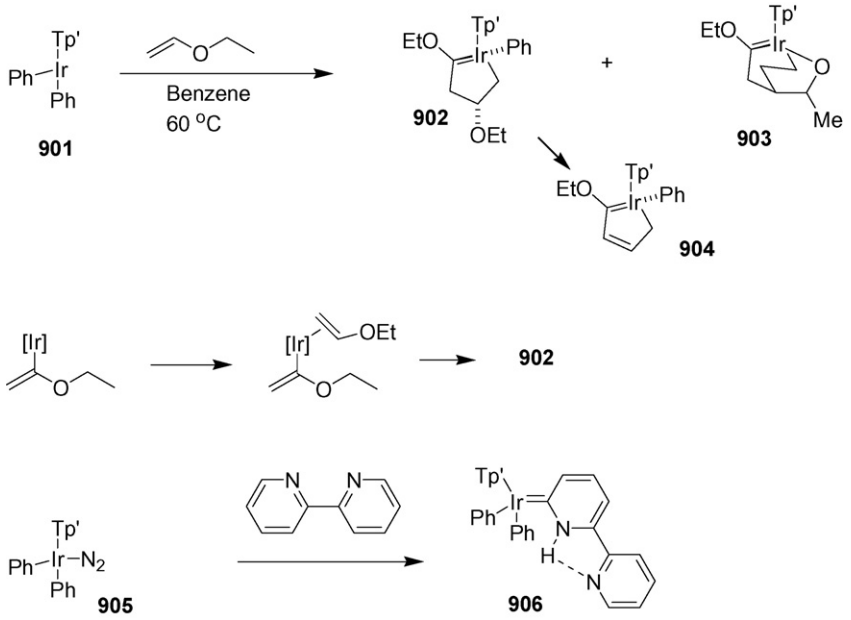
Scheme 102.

The reaction of iridacyclopentadiene derivative **968** (Scheme 112) with various terminal alkynes was reported [1120]. The reaction with 3-butyn-1-ol (**969**) led to the cyclic alkoxycarbene complex **971** via an unobserved vinylidene complex (**970**). Reaction with methyl propiolate led to the iridafuran derivative **973**, formed via vinylidene formation (**972**) followed by chloride migration and carbonyl oxygen complexation. Reaction with the alkynol **974** led to the carbonyl complex **977**. Formation of **977** likely occurs via allenylidene formation (**975**), followed by addition of water to form the acyliridium complex (**976**), followed by deinsertion of CO.

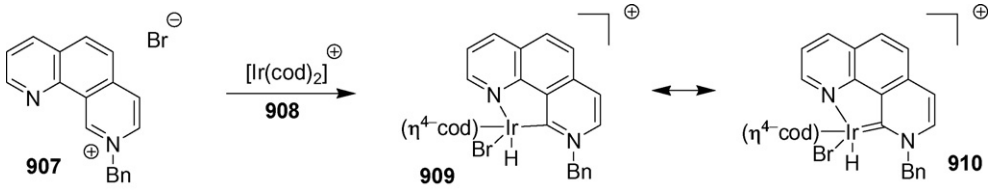
2.3.7. Group 10 metal–carbene complexes

2.3.7.1. Stable carbene complexes. The bonding in NHC–Group 10 metal complexes was studied computationally [1121]. Significant π-bonding interactions were noted for the nickel complexes. Lesser but measurable π-bonding was noted for palladium and platinum complexes. A patent involving the synthesis of carbocyclic palladium–carbene complexes and their use as C–C coupling catalysts was reported [1122]. Palladium–carbene complexes (*e.g.* **981**, **982**, Scheme 113) were prepared from the thermolysis of bis(η^3-cycloheptatrienyl)palladium complexes (*e.g.* **980**) [1123]. Their subsequent use as catalysts of C–N bond formation was also reported. Platinum– or palladium–carbene complexes (*e.g.* **986**) were generated through reaction of metal bis(isonitrile) complexes (*e.g.* **983**) with iminoisoindolinones **984** [1124]. The synthesis of biaryl Bertrand carbene–palladium (*e.g.* **988**) complexes was reported [1125].

The reaction of protonated diacetylplatinum(II) complex **989** (Scheme 114) with various ligands/bases was reported [1126].



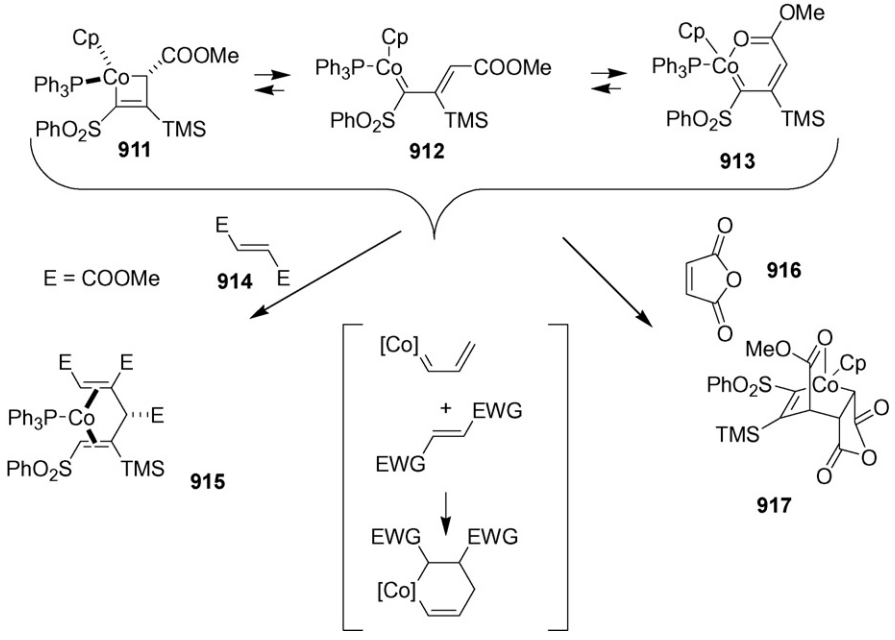
Scheme 103.



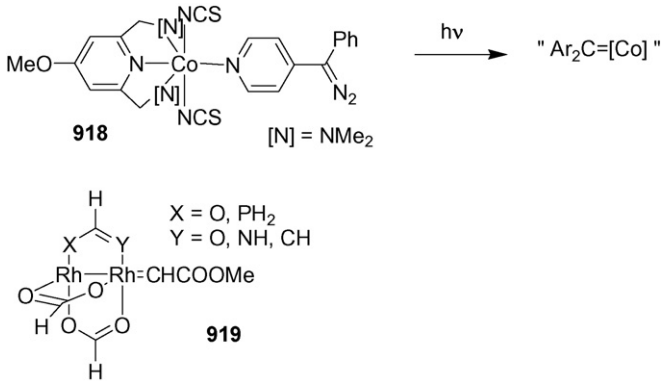
Scheme 104.

Treatment with sodium methoxide and triphenylphosphine led to the mononuclear bis(acetyl) derivative **990**. Treatment of with base in the presence of bipyridyl led to the analogous ligand-substituted complex **991**.

2.3.7.2. Group 10 carbene complexes as transient intermediates. Several processes reported in 2008 proceed through Group 10 carbene complex intermediates (see Scheme 115). Palladium–carbene complexes (*e.g.* **994**) were generated from stable ketenes (*e.g.* **992**)



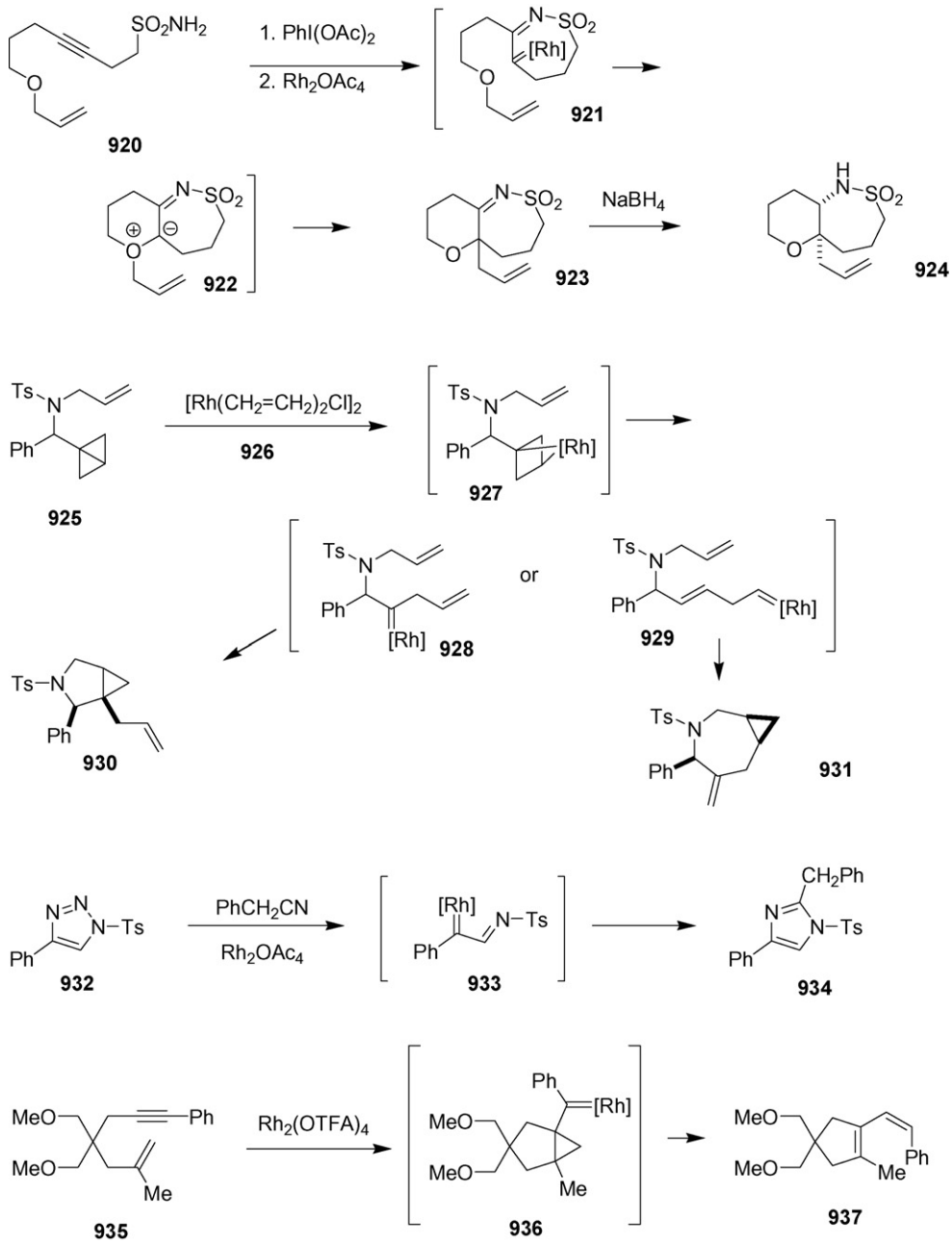
Scheme 105.



918 **919**

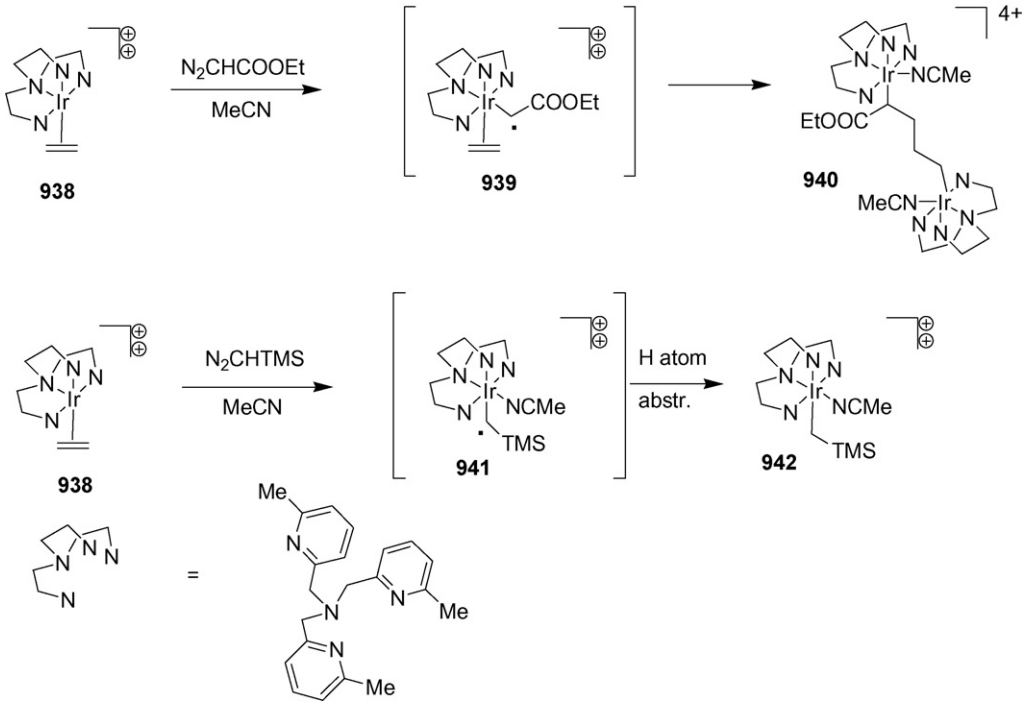
Scheme 106.

[1127]. These carbene complexes react with strained alkenes to afford cyclopropanation products (*e.g.* **995**) and with terminal alkynes to provide C–H insertion products (*e.g.* **996**). A platinum–carbene complex (**999**) was as an intermediate in the ring opening of nortricyclanes (*e.g.* **997**) to provide ring-fused norbornenes (*e.g.* **1000**) [1128]. Palladium–carbene complexes (*e.g.* **1005**) were proposed as intermediates in the cocyclization of aryl bromide-esters (*e.g.* **1001**) and trimethylsilydiazomethane [1129]. Similar carbene complexes (*e.g.* **1008**) were proposed as intermediates in a three-component coupling process involving vinyl iodides (*e.g.* **1006**), trimethylsilyldiazomethane, and nucleophiles [1130]. A π-allylpalladium complex (**1009**) is produced upon carbene insertion, which couples with the nucleophile. A platinum–carbene resonance form was suggested as important in the isomerization of vinylplatinum complexes containing 2-oxonium cation groups [1131]. A palladium–allenylidene complex (*e.g.* **1013**) was suggested



920 **921** **922** **923** **924** **925** **926** **927** **928** **929** **930** **931** **932** **933** **934** **935** **936** **937**

Scheme 107.



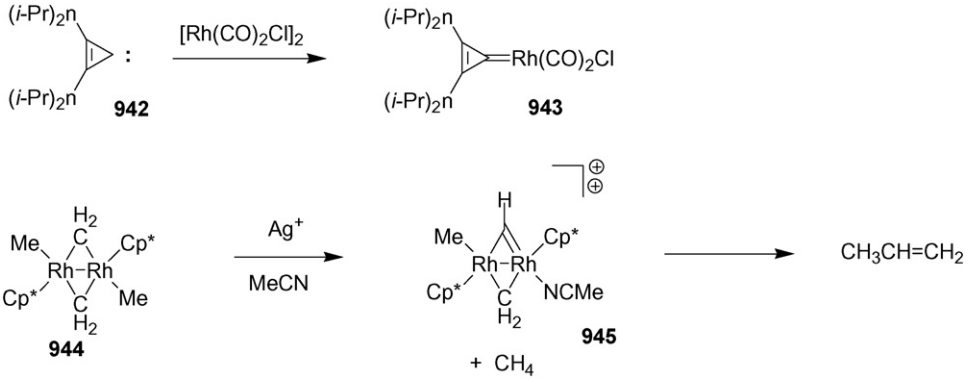
Scheme 108.

as an intermediate in the palladium-catalyzed synthesis of allenylidenecyclopropanes (*e.g.* **1014**) from propargyl esters (*e.g.* **1011**) and norbornadiene [1132]. The contribution of a carbene complex resonance form for bis(porphyrin)–platinum complexes was noted [1133].

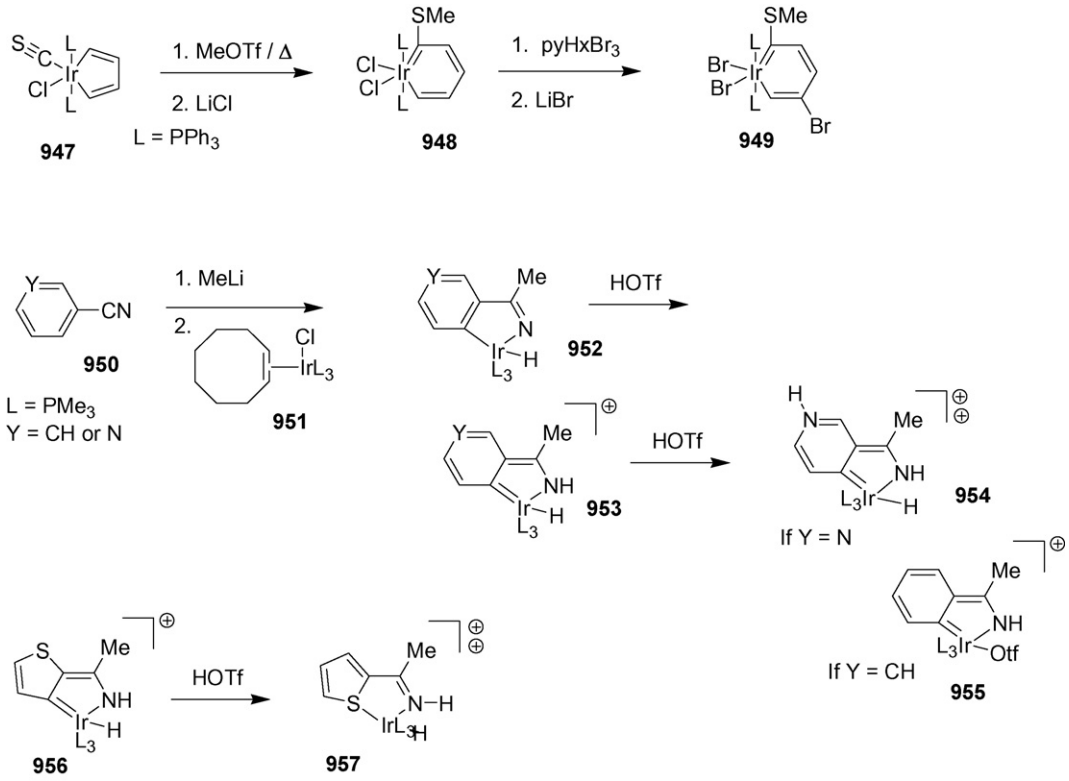
Platinum–carbene complexes were identified as transient intermediates from the reaction of laser ablation-generated platinum atoms with perhalomethane derivatives [1134]. Carbene complexes were identified from argon matrix infrared spectra and compared with calculated (DFT) spectra of geometry-optimized species.

2.3.7.3. Platinum- and gold-catalyzed reactions of alkynes or allenes. Several examples of platinum- and gold-catalyzed reactions of alkynes and allenes have been suggested to involve carbene complex intermediates. One manuscript suggests that these reactions involve metal-stabilized carbocations and not actual carbene complexes [1135].

Several papers report on the development of new reaction processes using cyclopropylcarbene intermediates generated through the reaction of enyne derivatives with platinum or gold complexes; representative examples are depicted in Schemes 116–118. Cyclopropylcarbene–gold complexes (*e.g.* **1019**, Scheme 116) were proposed as intermediates in the gold-catalyzed cycloisomerization of alkyne–cyclohexadienes (*e.g.* **1017**) [1136,1137] and similar annulations of arylalkyne–alkene derivatives [1138]. Gold-catalyzed cycloisomerization of 2-alkynylstyrenes that contain pendant arene chromium complexes were also reported [1139]. The cyclization of propargyl alcohol–allenes (*e.g.* **1021**) to 6-alkylidenebicyclo[3.1.0]hexan-3-ones (*e.g.* **1023**) via cyclopropylcarbene-platinum complexes (*e.g.* **1022**) was also reported [1140]. In this case, the initially formed gold platinum complex **1022** undergoes a 1,2-hydride shift to afford the enol and then the ketone derivative. Platinum- and rhodium-catalyzed enyne cycloisomerizations involving terpenes were also reported [1141]. Enyne cycloisomerization using various gold–NHC complexes that feature N–H bonds as catalysts was reported [1142]. Net intramolecular [6+2]-cycloaddition of alkynes and cycloheptatrienes catalyzed by platinum was proposed to involve cyclopropylcarbene–platinum complex intermediates [1143]. Platinum-catalyzed enyne cycloisomerization was also performed in sequence with allenylidene-mediated reaction processes [1079].

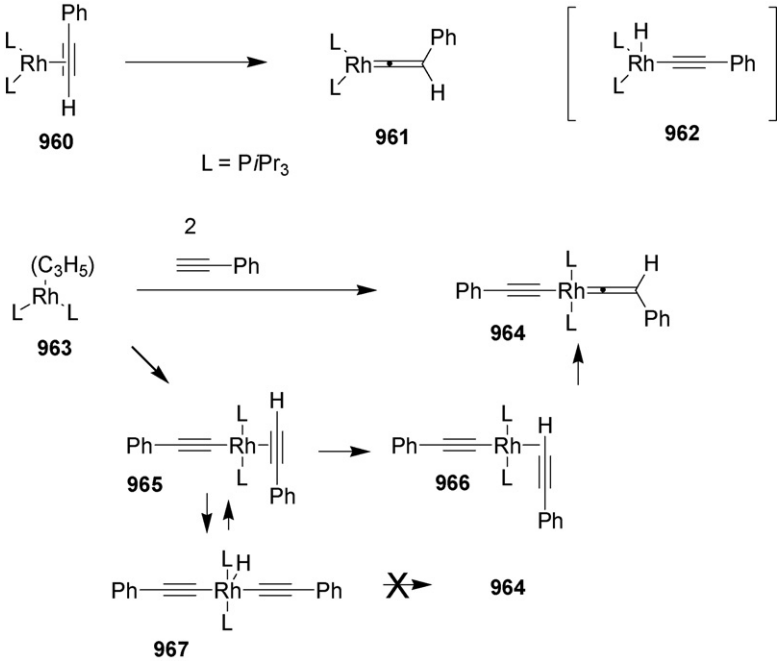


Scheme 109.

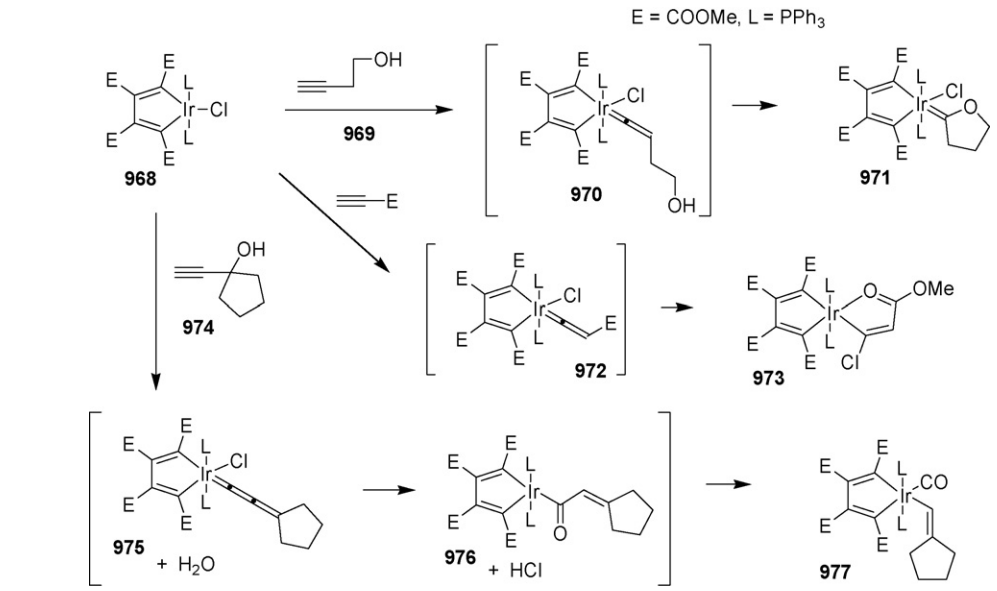


Scheme 110.

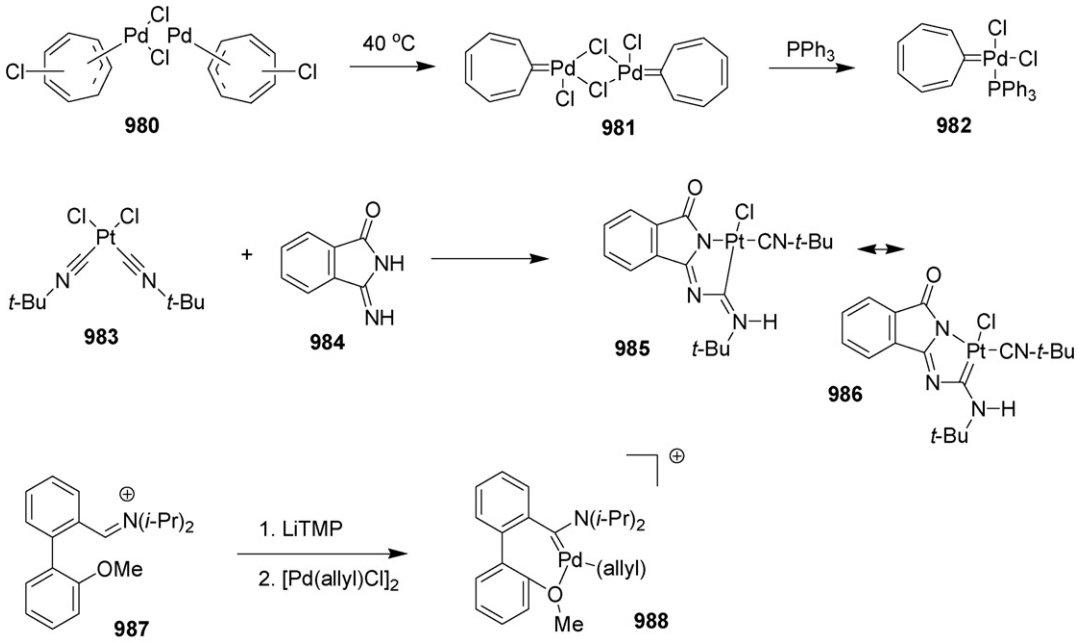
In several cases the cyclopropylcarbene complex intermediates undergo additional skeletal rearrangements (Scheme 117). Ring expansion processes in competition with heterocyclization were observed in oxygenated enyne systems (*e.g.* **1024**) [1144]. In the formation of cyclopropylcarbene (**1026**) derived product **1027**, 1,2-alkyl shift accompanies cyclopropane ring opening to afford the ring expanded product **1027**. Cyclopropylcarbene–gold complexes (*e.g.* **1031**) are important intermediates in the gold-catalyzed cycloaromatization involving furan-ynamides or furan-alkynyl ethers (*e.g.* **1030**) [1145]. A related cyclization–aromatization employing propargyl alcohol–alkenes was also reported [1146]. The later steps of the gold-catalyzed conversion of furan-alkynes to benzene rings were studied by NMR spectroscopy and deuterium labeling [1147,1148]. Several examples where the cyclopropylcarbene complex intermediates were captured through reaction with nucleophiles were reported (Scheme 118). Gold-catalyzed



Scheme 111.



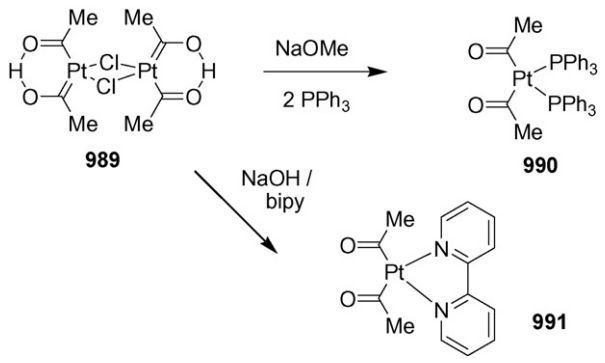
Scheme 112.



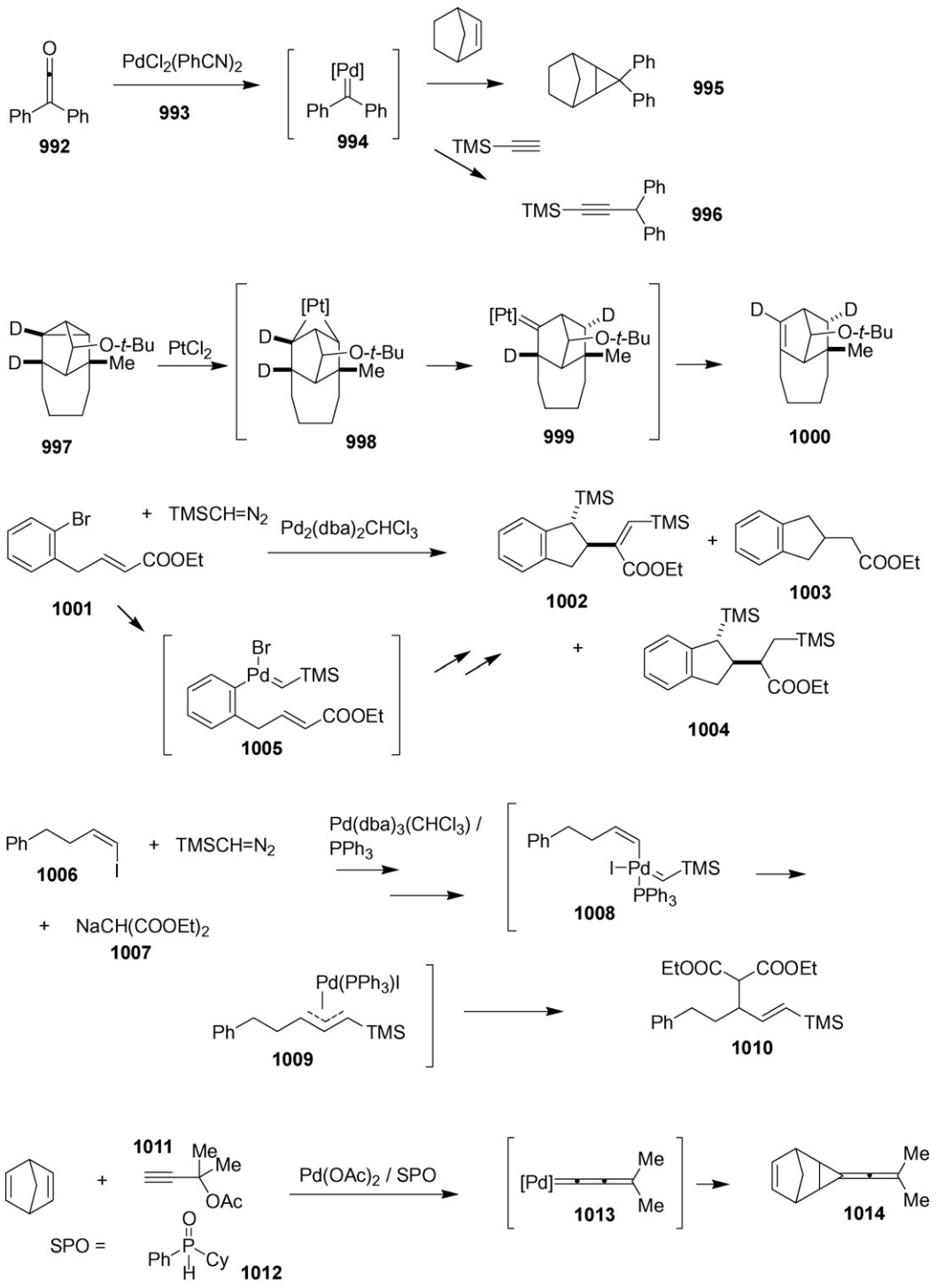
Scheme 113.

cyclization of enynes (*e.g.* **1036**) in the presence of highly electron rich arenes (*e.g.* **1037**) led to cyclization/electrophilic aromatic substitution products (*e.g.* **1040**) [1149]. Trapping of enyne-derived cyclopropylcarbene complex intermediates with methanol and gold-catalyzed intramolecular enyne metathesis reactions were reported [1150]. Trapping with arylboranes was also reported [1151].

Gold– and platinum–carbene complexes were also generated from various heterocyclization processes; representative examples are depicted in Scheme 119. A novel cyclization/cycloaddition cascade reaction process occurred when alkynylcyclopropylketone **1041** was treated with gold complex **1042** in the presence of ethyl vinyl ether [1152]. A mechanism involving nucleophilic addition of the carbonyl oxygen to an alkyne-gold complex to afford the carbene-1,3-dipole **1043**, followed by a 1,3-dipolar cycloaddition and cation rearrangement was proposed. Cyclization of conjugated enyne aldehydes (*e.g.* **1047**) in the presence of



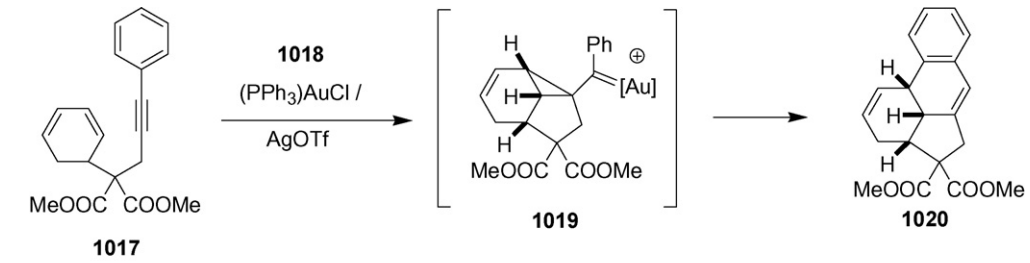
Scheme 114.

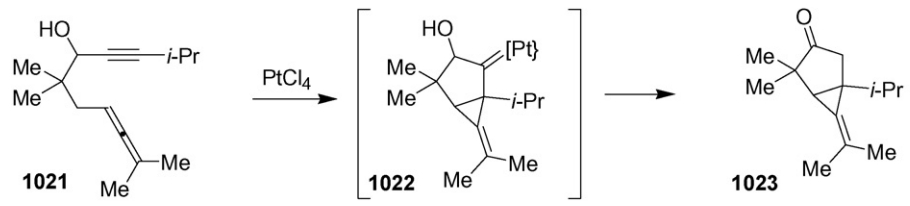


Scheme 115.

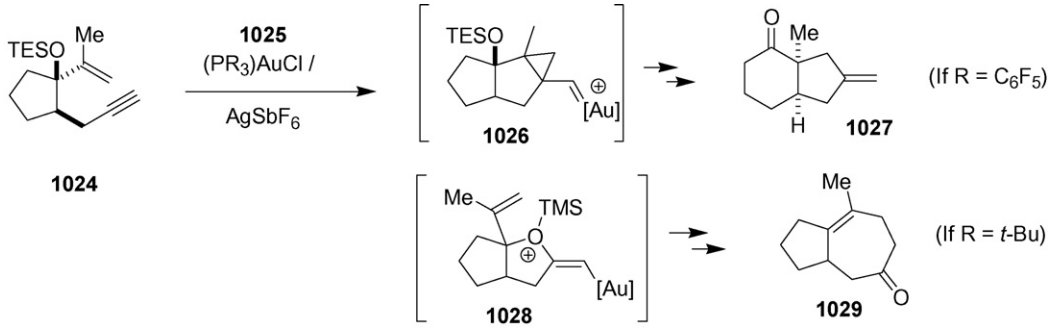
gold(III) chloride followed by capture of the intermediate furylcarbene complex (*e.g.* **1048**) through cyclopropanation led to furylcyclopropane derivatives (*e.g.* **1049**) [1153]. An analogous process where the carbene intermediate was captured through C–H insertion was also reported [1154]. A mechanistically similar process was observed in the platinum-cyclization of enyne ketones (*e.g.* **1050**) [1155]. Similar cascade processes initiated by enyne nitrones were also reported [1156]. Gold-catalyzed cyclization reactions where *o*-alkynylstyrene oxides (*e.g.* **1056**) are converted to indenone derivatives (*e.g.* **1060**) likely involve gold–carbene complex intermediates (*e.g.* **1059**) [1157]. Gold–carbene complexes are likely involved in ring expansion/cyclization reactions of β-lactam-alkynes [1158].

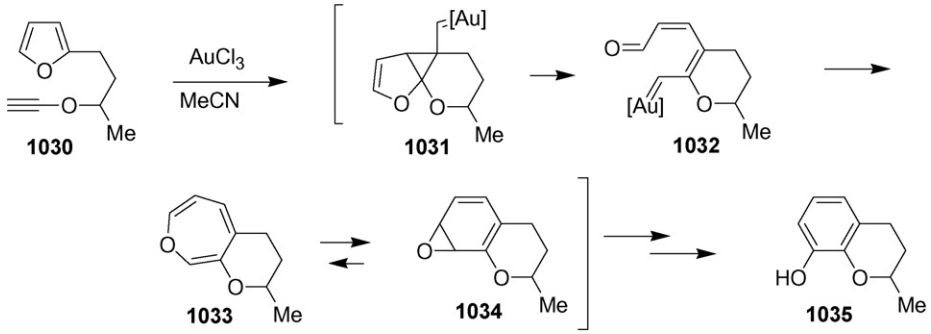
Carbene complexes were also generated from 1,2-shift of propargyl ester derivatives in the presence of platinum and gold complexes (see Scheme 120). The reaction of propargylic acetates (*e.g.* **1061**) and allylic sulfides (*e.g.* **1062**) in the presence of gold catalysts led to 1,5 dienes (*e.g.* **1065**) in a process involving carbene complex formation (**1063**), followed by ylide formation (**1064**) and 2,3-sigmatropic rearrangement [1159]. Gold-catalyzed cycloiso-



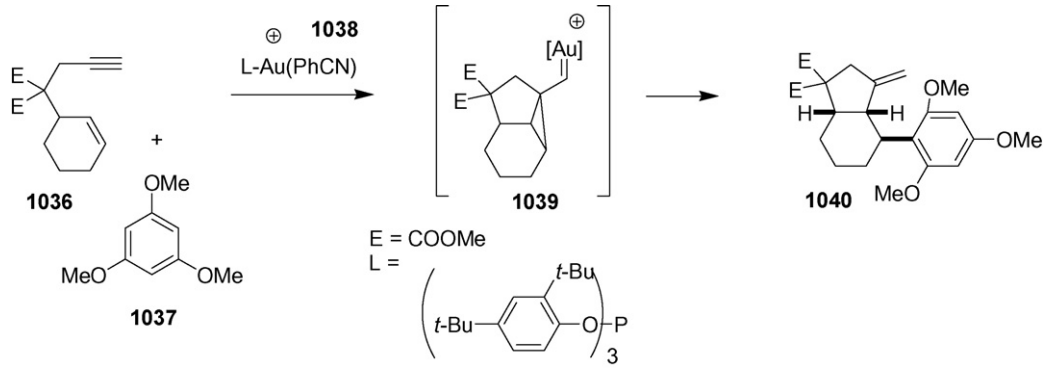


Scheme 116.

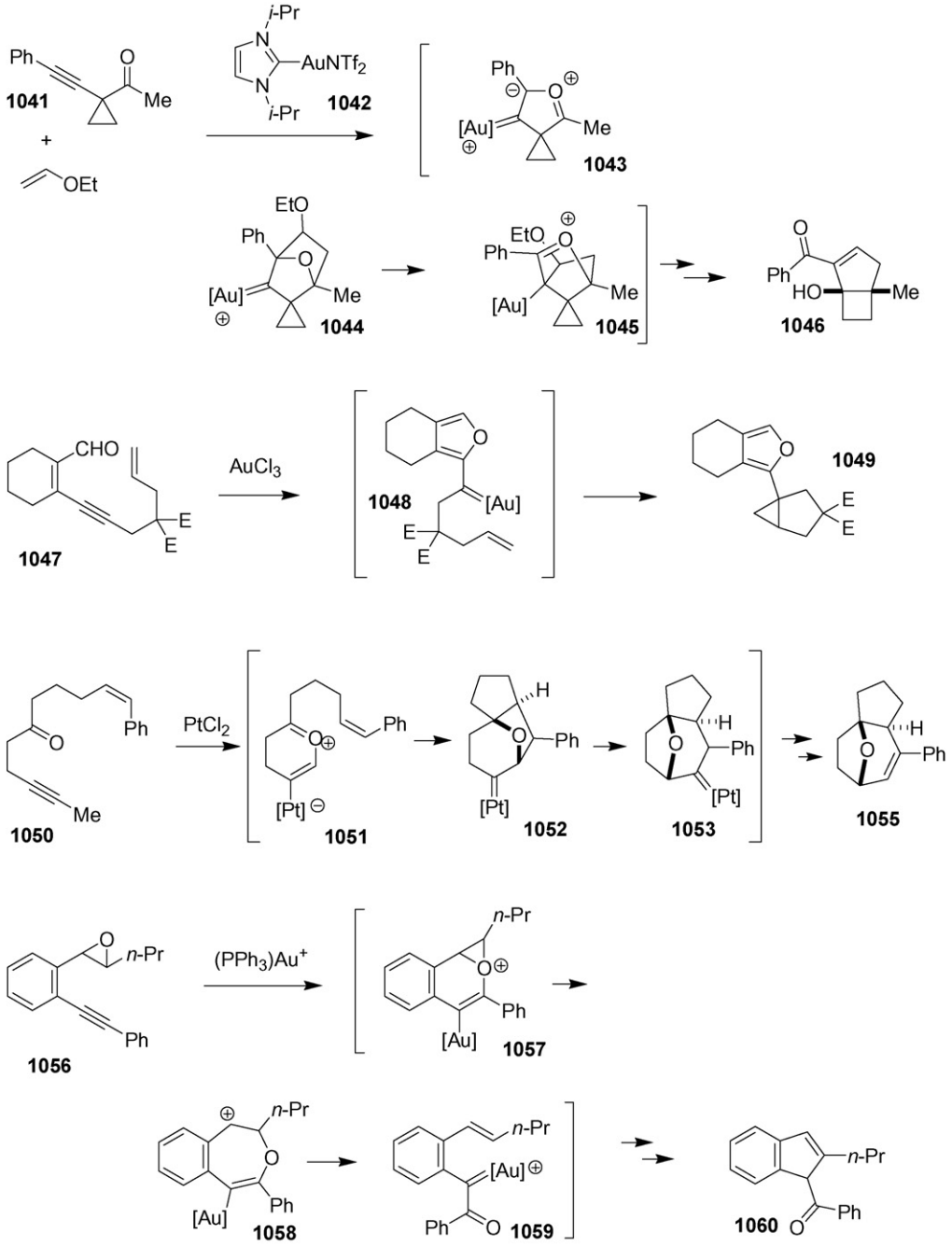




Scheme 117.

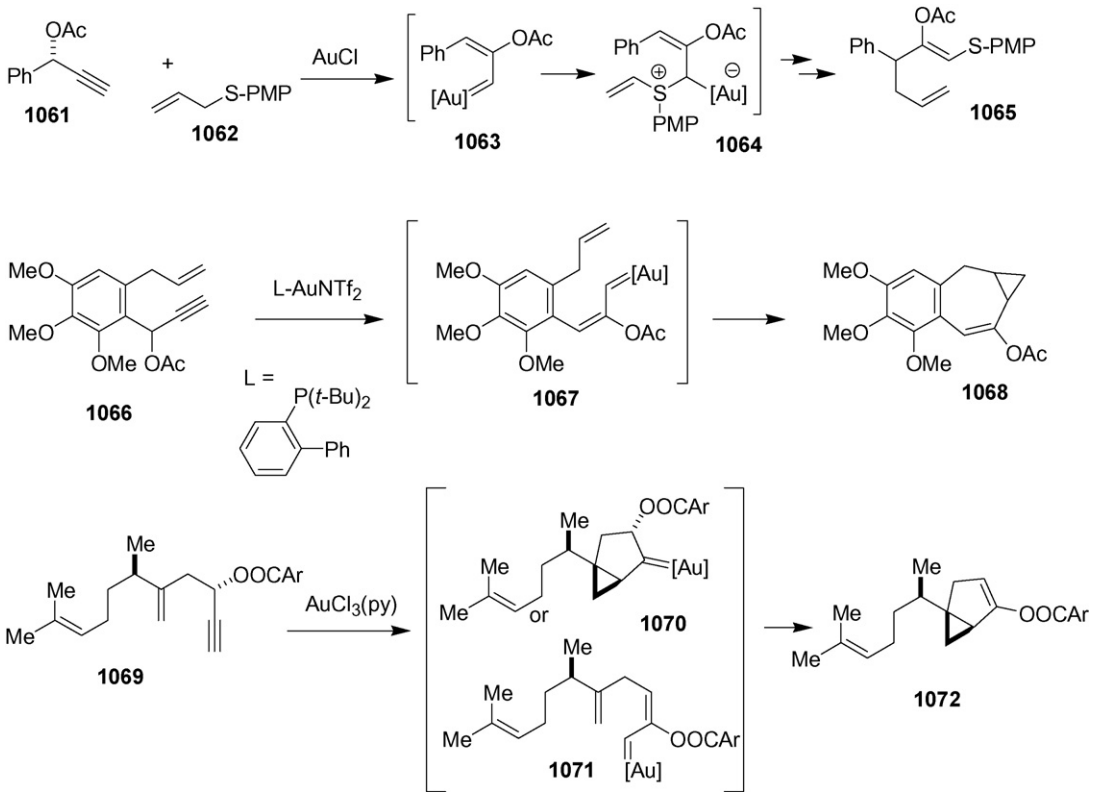


Scheme 118.



Scheme 119.

merization of dienyne **1066** led to the cyclopropane derivative **1068**, which was an intermediate in the total synthesis of allocolchicinoids [1160]. The enantioselective cycloisomerization of enyne propargyl ester **1069** was a key step in the total synthesis of cedrene [1161]. In order to explain the retention of optical configuration, a mechanism where traditional cycloisomerization (as in Scheme 116) occurs to form carbene complex **1070** followed by acetate shift was favored over initial acetate shift to provide intermediate **1071**. A similar rearrangement process was proposed in several other transformations, including: (1) the conversion of propargylic acetates into α-acetoxy-α,β-unsaturated ketones using a gold catalyst in an aqueous environment [1162]; and (2) gold-catalyzed cyclization of allenyl propargyl esters to alkylidenecyclopentenones [1163]. Inter- and intramolecular gold-catalyzed reactions of propargyl acetates were studied computationally [1164]. Additional more elaborate examples based on generation of carbene complexes from propargyl esters and platinum/gold catalysts are depicted in Scheme 121. A novel benzannulation involving the gold-catalyzed coupling of propargyl esters (*e.g.* **1073**) and conjugated enynes (*e.g.* **1074**) was developed [1165]. The reaction proceeds through formation of the cyclopropane-bridged enyne **1075** which undergoes further cyclization to afford aromatic compounds (*e.g.* **1076**, **1077**). The direction of the reaction is highly dependent on the silver source used to ionize the gold catalyst. A tandem carbene generation/allylation was observed in the reaction of propargylic acetates that contain allylic ether groups (*e.g.* **1078**) $PtCl_2$ [1166]. The key mechanistic event in this process is the tandem hydrogen shift allyl transfer from intermediate platinum–carbene complex **1079**. Tandem carbene formation–cyclopropane ring opening was



Scheme 120.

observed in the transformation of cyclopropane-bridged enynes (*e.g.* **1082**) to cyclohexadienes (*e.g.* **1084**) [1167]. A net [4+3]-cycloaddition process was observed in the coupling of propargylic esters (*e.g.* **1085**) with α,β-unsaturated imines (*e.g.* **1086**) [1168]. In this process, nitrogen adds to the carbene carbon of intermediate **1088** to afford ylide **1089**, which then undergoes intramolecular allylation to afford the azepine derivative (**1090**). The reaction of propargyl acetates with cationic gold complexes was evaluated computationally (Scheme 122) [1169]. Emphasis was on the interconversions of alkyne complex (**1091**), the acetate-shifted carbene complex (**1092**), and the allene complex (**1093**). Rearrangement of the gold–alkyne complex to the acetate-shifted carbene complex was a nearly isoergonic process that occurs with a low activation energy. Conversion to the complexed allene was more thermodynamically favorable, however the net conversion via the carbene complex occurred with a lower activation energy.

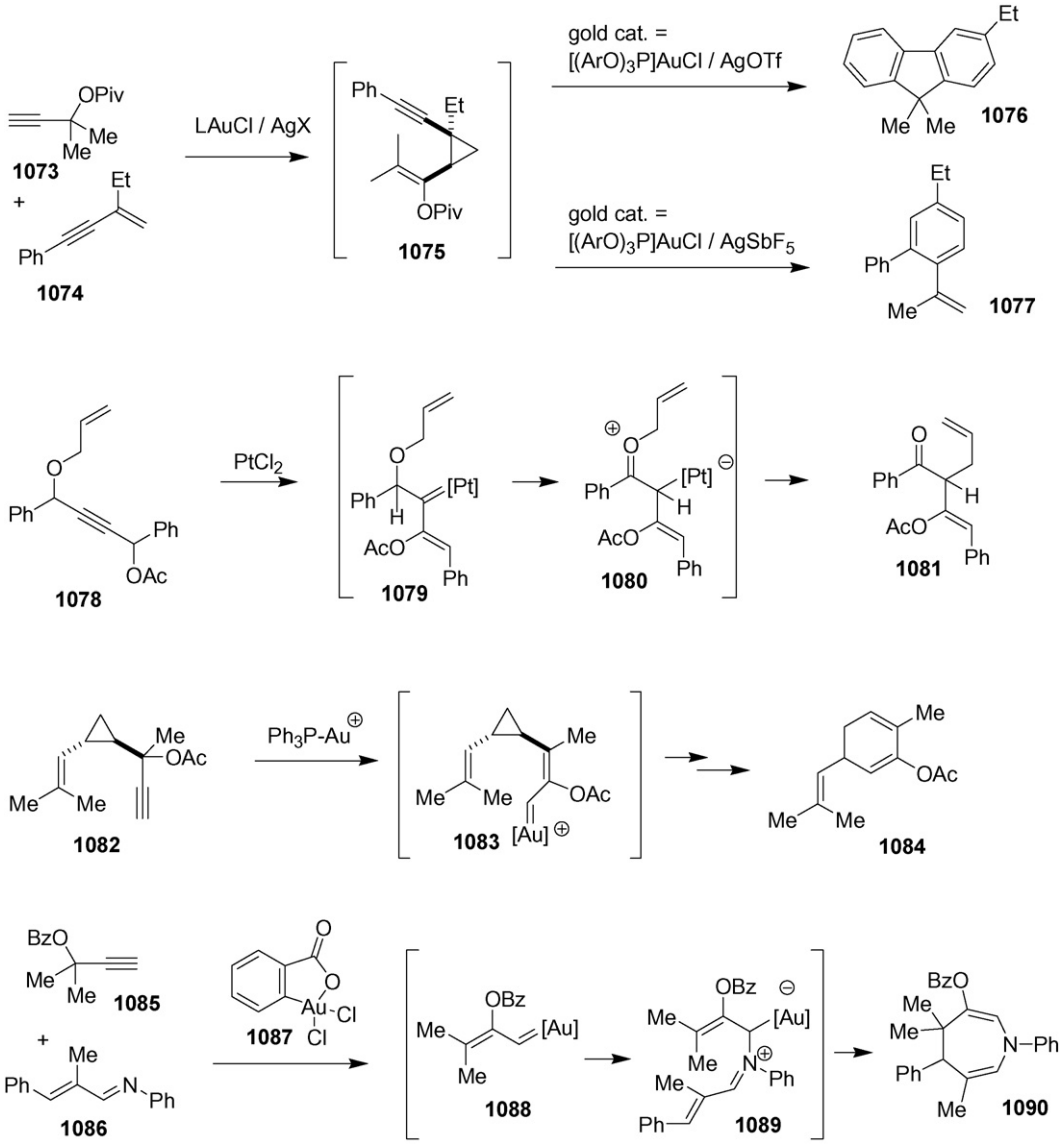
In several cases, gold and platinum complexes initiate oxyallyl cation and Nazarov-like processes that involve gold–carbene complex intermediates (see Scheme 123). The gold-catalyzed intramolecular [4+3]-cycloaddition of allene-furans (*e.g.* **1094**) was proposed to involve gold–carbene complex intermediates (*e.g.* **1096**) generated through cycloaddition of a furan and gold-substituted allyl cation intermediate (**1095**) [1170]. The reaction of vinylallenes (*e.g.* **1098**) with gold complexes led to bicyclic ring systems (*e.g.* **1101**) in a process involving cyclization to generate a cyclopentenylidene-gold complex (**1099**) followed by intramolecular C–H abstraction and allylmetal addition [1171]. The proposed mechanistic pathway was supported by deuterium labeling studies. A Nazarov-like step was proposed for the cyclization of arylalkynes that contain alkylidenecyclopropane groups using a silver-NHC catalyst [1172]. A related process was proposed in gold-catalyzed conversion of 4-phenyl-4-hydroxyallenes (*e.g.* **1102**) to naphthalenes (*e.g.* **1103**) [1173]. Tandem Nazarov-like reaction processes followed by electrophilic cyclization were evaluated computationally and experimentally [1174].

Several other reaction processes were reported from the reaction of alkynes or allenes and platinum or gold complexes that might involve carbene complex intermediates (see Scheme 124). Platinum-catalyzed enyne metathesis (see Scheme 1) likely involves metal–carbene complex intermediates. Several examples of this process were reported in 2008 [1175]. Gold– or platinum–carbene complexes were suggested as potential intermediates in: (1) a tandem 1,2-shift/aromatic substitution reaction of propargylic indoles (*e.g.* **1105**) to provide indenylindoles (*e.g.* **1107**) [1176]; (2) platinum-catalyzed conversion of N-alkynylphenylsuccinimides to annulated indoles [1177]; (3) platinum-catalyzed benzannulation/cyclopentannulation using phenyl-bridged diene-yne alcohols (*e.g.* **1108**) [1178]; (4) gold-catalyzed conversion of 4-phenyl-3-acetoxy-1-butyne derivatives into naphthalenes [1179]; and (5) the gold-catalyzed isomerization of 1-phenylcyclopropenes to indenes [1180].

2.3.8. Group 11 carbene complexes

A copper–carbene complex (**1116**, Scheme 125) was obtained from the reaction of copper complex **1115** and ethyl diazoacetate [1181]. The brown solid obtained from this coupling (R = Me) was characterized spectroscopically. The complex reacts with cyclohexene to afford the cyclopropanation product, and also serves as a catalyst for the cyclopropanation of alkenes using ethyl diazoacetate.

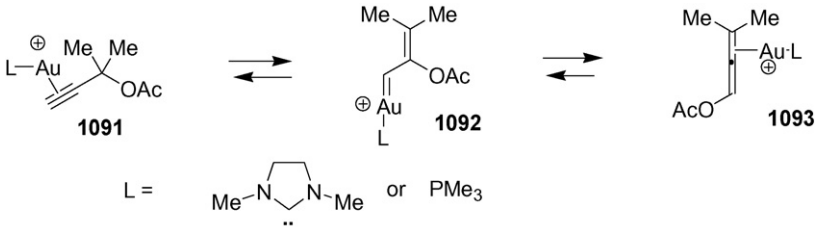
Mononuclear carbene complexes (**1119** (Ar = Ph), Scheme 126) were produced at steady state concentrations from the reaction of copper complexes (*e.g.* **1118**) with diaryl diazomethane derivatives [1182]. Attempted isolation of the carbene complex resulted in the dimeric species **1120**. The dimeric species reacts with alkenes to regenerate the alkene and carbene complex, thus suggesting that this species is not involved in cyclopropanation. The *p*-nitrophenyl



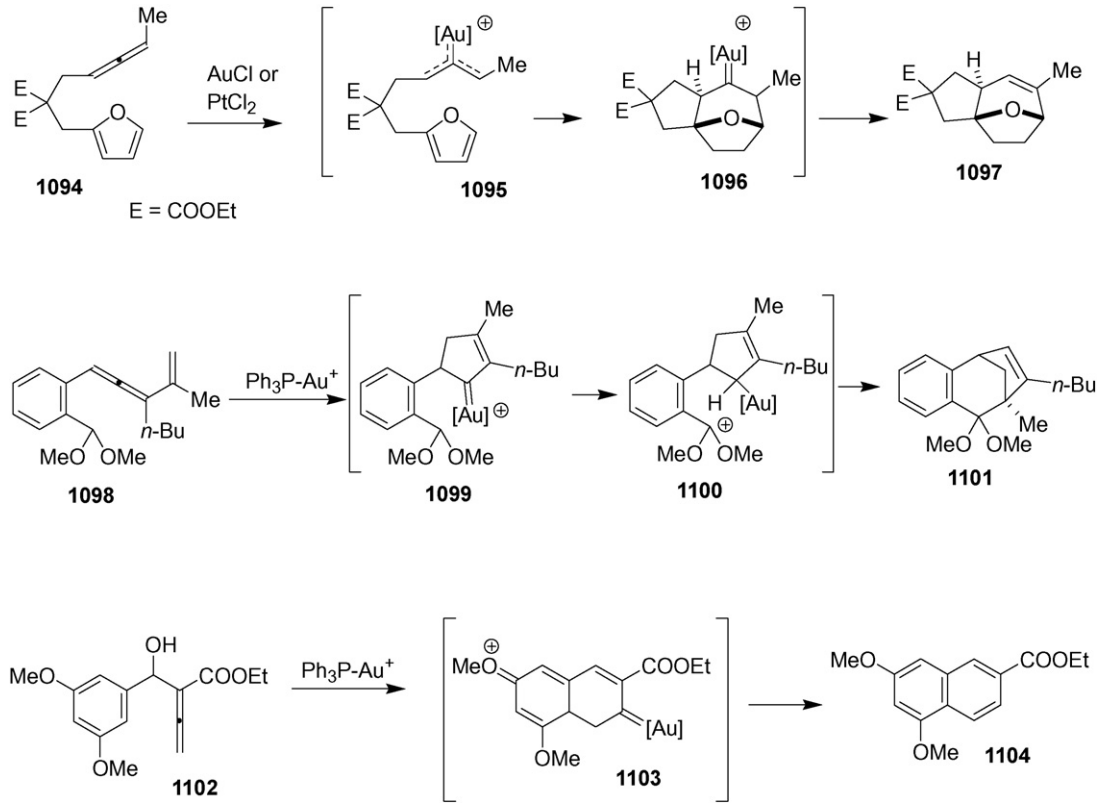
Scheme 121.

analog led to a stable carbene complex that could be isolated and an X-ray structure could be obtained. Reaction of the stable carbene complex with styrene led to the cyclopropanation product **1122**, accompanied by alkene complex **1121** and carbene dimerization product **1123**. An attempt to perform a catalytic cyclopropanation reaction led to a complex reaction resulting in the compounds **1121**–**1124**. The azine (**1124**) and carbene dimer (**1123**) could also be produced from the reaction of the carbene complex **1119** (Ar = *p*-$PhNO_2$) with additional diazo compounds, and accompanied by the carbene-exchanged carbene complex. A copper–carbene complex (**1126**) was also obtained from the reaction of copper complex **1125** with diphenyl diazomethane, accompanied by the copper-azine complex **1127** [1183]. Reaction of the carbene complex with CO led to diphenylketene (**992**), however carbene complex **1126** did not engage in cyclopropanation reactions.

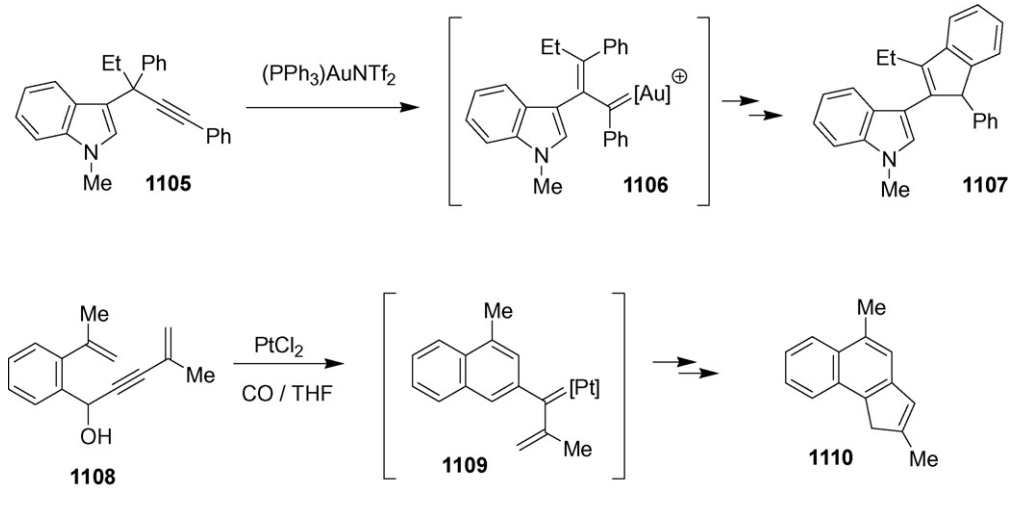
Several papers report on mechanistic aspects of copper–carbene complex intermediates generated from organic diazo compounds (see Scheme 127). Stereoselectivity models were proposed to rationalize the direction of asymmetric induction in enantiose-



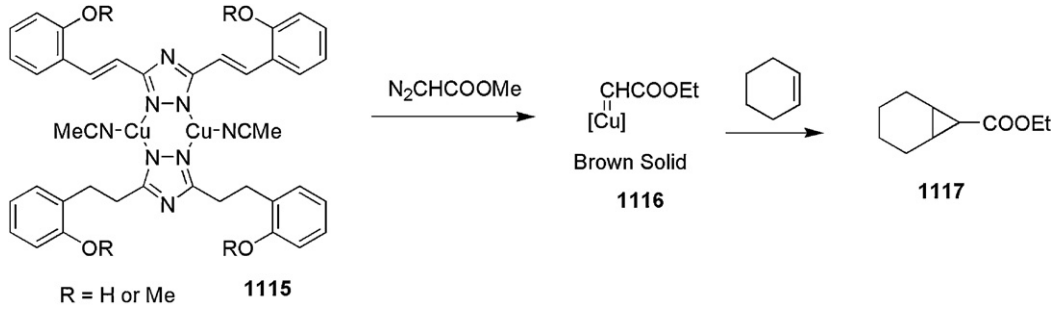
Scheme 122.



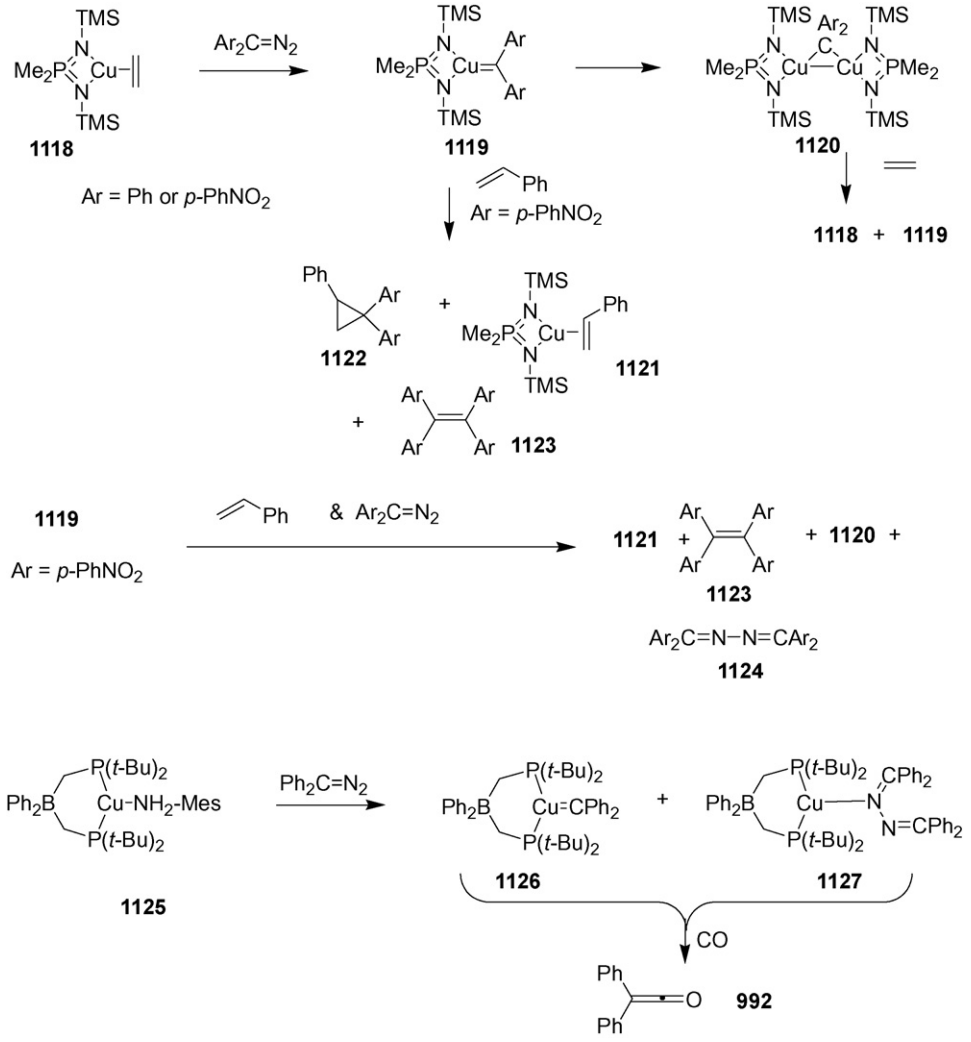
Scheme 123.



Scheme 124.



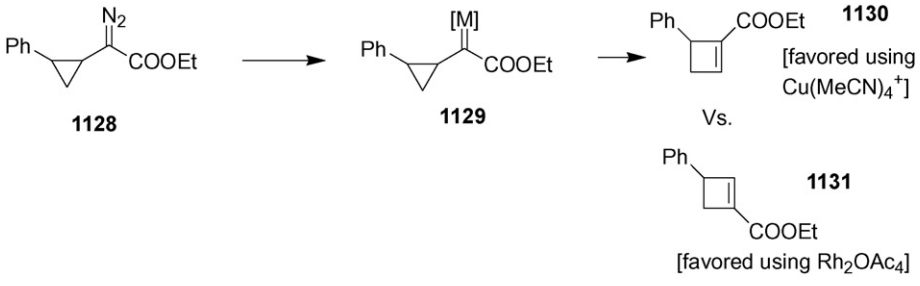
Scheme 125.



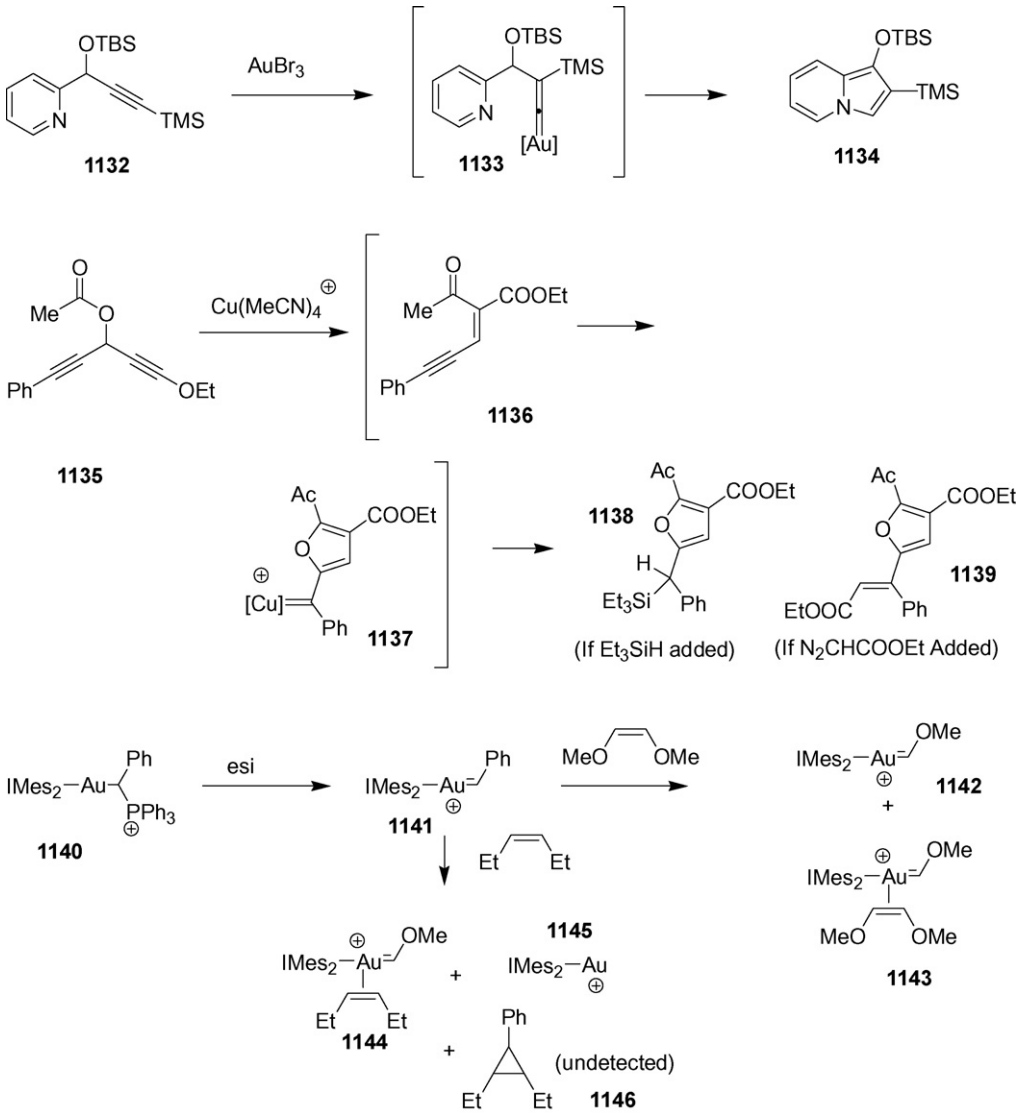
Scheme 126.

lective cyclopropanation catalyzed by chiral oxazolinylquinoline complexes [1184]. The effect of metal (copper vs. rhodium) on the regiochemistry of ring expansion of cyclopropyl-substituted α-diazoesters (*e.g.* **1128**) was examined [1185]. Copper–carbene complexes are likely intermediates in the [3+2]-cycloaddition reaction of aryldiazo compounds and alkynes to afford indenes [1186]. Computational studies of Group 11 metal-induced decompositions of diazo compounds include: (1) a DFT study of copper-catalyzed enantioselective cyclopropanation [1187]; and (2) a DFT study of copper–acac complex-catalyzed C–H insertions using diazoesters [1188].

Additional examples of reaction processes likely involving Group 11 metal–carbene complexes are depicted in Scheme 128. Gold vinylidenes (*e.g.* **1133**) were suggested as intermediates in the isomerization of pyridine-alkynes (*e.g.* **1132**) to indolizines (*e.g.* **1134**) [1189]. The intermediacy of a vinylidene complex was supported by deuterium labeling studies. Copper allenylidene complexes were suggested as intermediates in asymmetric copper-catalyzed propargyl substitution reactions [1190,1191]. A theoretical study of the gold-catalyzed cycloisomerization of 1,5-allenynes revealed the gold–vinylidene complexes are not involved [1192]. Copper–carbene complexes (*e.g.* **1137**) were suggested



Scheme 127.

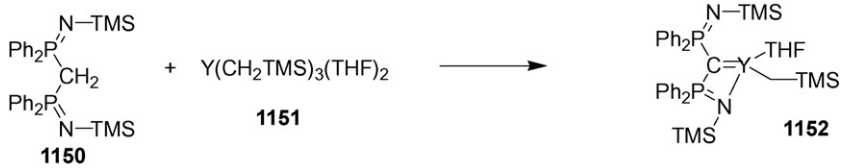


Scheme 128.

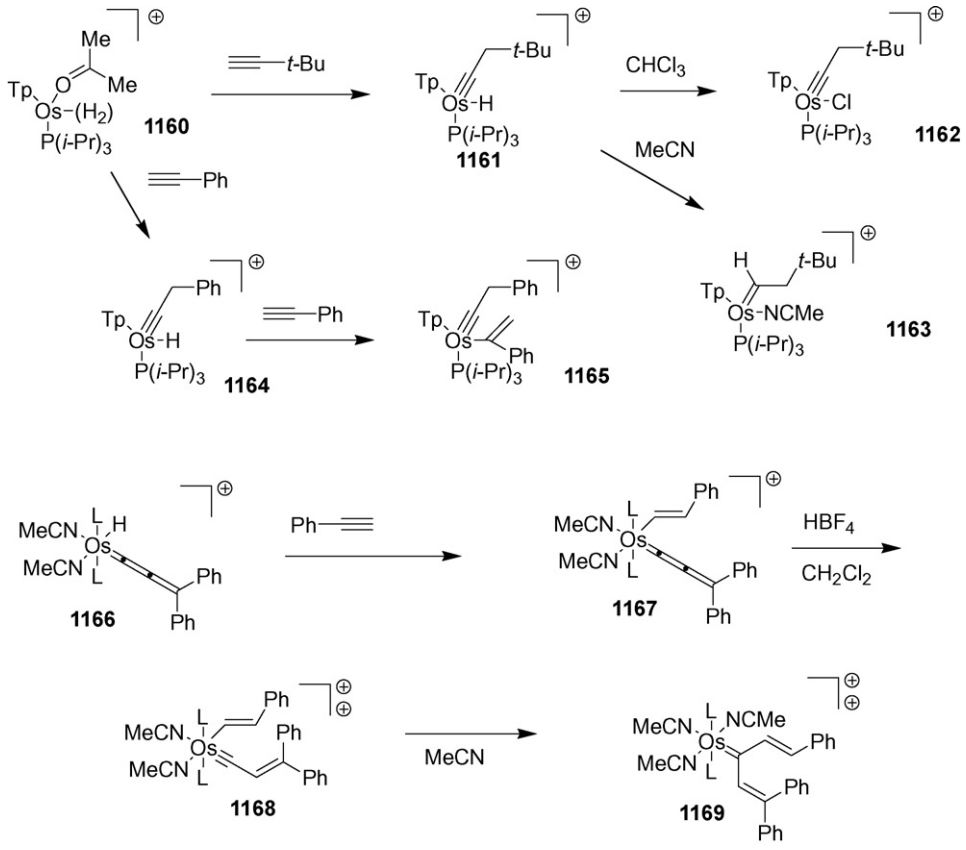
as intermediates in the conversion of doubly propargylic esters (*e.g.* **1135**) into substituted furans (*e.g.* **1138**, **1139**) [1193]. The hypothetical carbene intermediate **1137** could be trapped with hydrosilanes or diazo compounds. Gold–carbene complexes were suggested as likely intermediates in the gold-catalyzed conversion of allenyl ketones to furans [1194]. Gold–carbene complexes were suggested as intermediates in gold-catalyzed cyclopropene ring opening reactions [1195]. Gold–carbene complexes (*e.g.* **1141**–**1144**) were observed in the electrospray ionization mass spectra of gold–NHC complex **1140** [1196]. Various alkene complexes and metathesis products were observed in the presence of alkene additives.

2.3.9. Lanthanide–/actinide–carbene complexes

The attempted formation of pincer carbene complexes of yttrium (*e.g.* **1152**, Scheme 129) was reported [1197]. In this case, the yttrium–carbon bond order was determined to be 0.6. The reaction of thorium atoms generated through laser ablation with various dihalomethane led to carbene complexes [CH_2=ThX_2], which were isolated in an argon matrix [1198]. The analogous reaction with haloform or tetrahalomethane derivatives led to triplet carbyne complexes [HC≡ThX_3] or [XC≡ThX_3]. The compounds were characterized through their infrared spectra and compared with DFT-calculated spectra. Carbene complexes featuring chloride ligands showed higher degrees of agostic inter-



Scheme 129.



Scheme 130.

actions than did carbene complexes featuring chloride ligands. Similar studies were reported for the reaction of methane or halomethanes with scandium atoms, however the degree of multiple bond character of $H_2C{=}ScH(X)$ was calculated to be minimal [1199].

3. Metal–carbyne or metal–alkylidyne complexes

3.1. Review articles

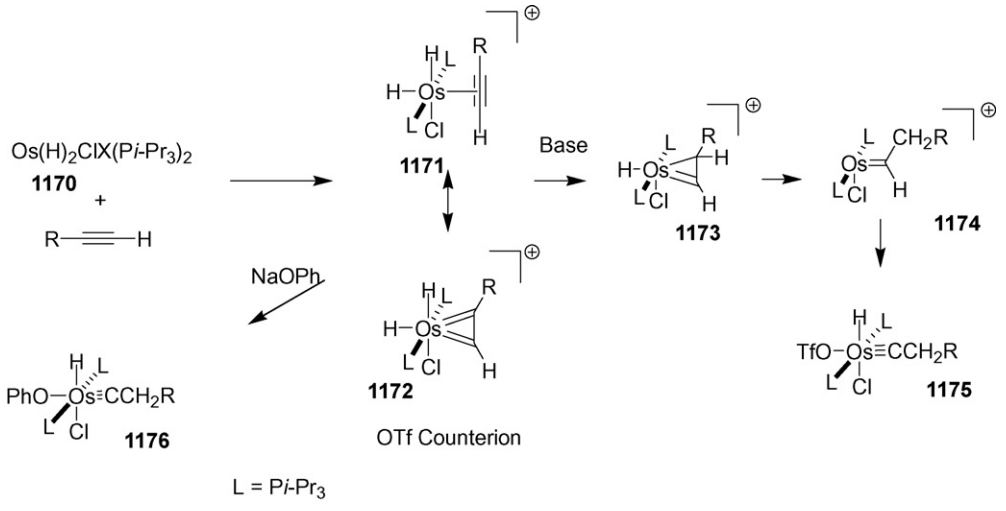
A review article focuses on alkylidyne complexes ligated to poly(pyrazolyl)borate [1200]. Some reviews listed in the carbene complex section also feature discussions on carbyne complexes [159,160].

3.2. Synthesis and/or generation

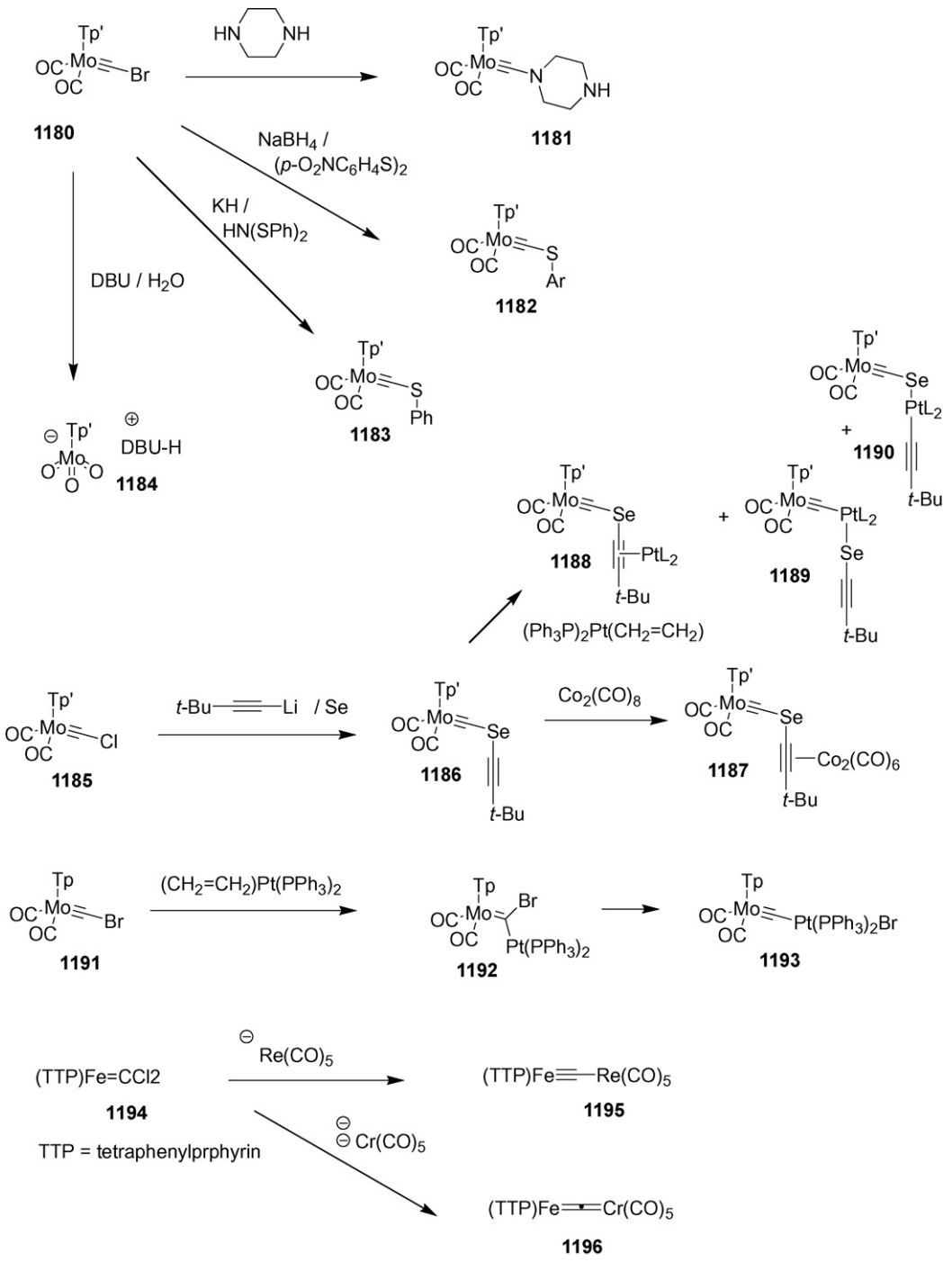
Some papers in the carbene section feature minor segments on carbyne chemistry. These studies include references [241,1015,1080].

The crystal structure for $TMSC{\equiv}Nb(CH_2TMS)_3$ was reported [1201].

Formation and reactivity of osmium carbyne hydride complexes (*e.g.* **1161**, **1164**, Scheme 130) from alkyne and osmium dihydrogen



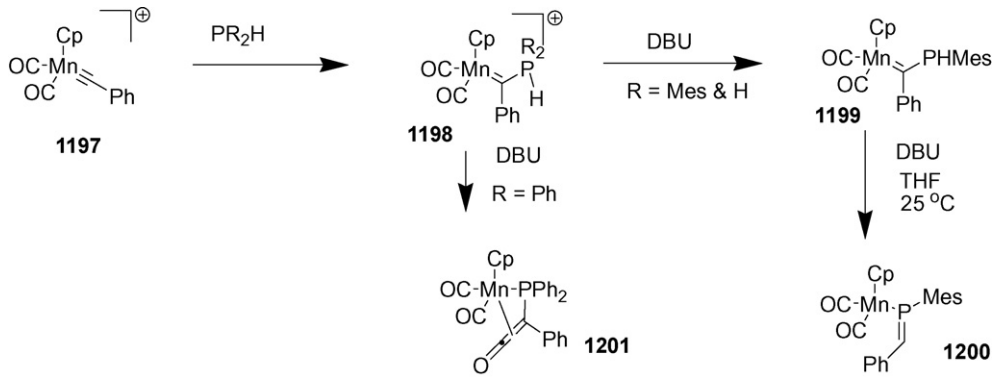
Scheme 131.



Scheme 132.

complex **1160** was reported [1202]. The reaction involves formation of the vinylidene(dihydrogen) complex followed by 1,3-migration of a hydride. Reaction of the carbyne(hydride) **1161** with chloroform leads to the corresponding chloride complex **1162**. Reaction with acetonitrile leads to the hydrogen-migration product, carbene complex **1163**. An oxygen chelated carbene complex analogous to **1163** was produced when the original starting compound **1160** was treated with methyl propiolate. The reaction of complex **1160** with phenylacetylene leads initially to the carbyne complex **1164**. However the alkyne insertion product **1165** was obtained when additional equivalents of the alkyne were employed. An osmium carbyne complex (**1168**) was generated through protonation of vinyl allenylidene complex **1167** [1203]. Carbyne complex **1168** was unstable in acetonitrile and evolved to the divinylcarbene complex **1169**. A mechanism involving alkenyl group migration followed by capture by acetonitrile was proposed and supported by kinetics and DFT calculations.

A mechanistic study of the formation of osmium–carbyne complexes (*e.g.* **1176**, Scheme 131) from osmium hydrides (*e.g.* **1170**) and terminal alkynes was reported [1204]. The alkyne π-complex (**1171/1172**) could be isolated from the low-temperature reaction of complex **1170** (X = OTf) with phenylacetylene or acetylene. Treatment of the alkyne complex with phenoxide or chloride led to a carbyne complex (*e.g.* **1176**). A mechanism involving transformation to the η^2-alkenyl form (**1173**) followed by reductive elimination to form the carbene complex (**1174**), followed by the α-hydride elim-



Scheme 133.

ination to form the carbene complex (**1175**) was proposed. This pathway was supported by deuterium labeling and DFT studies.

The spin states for various molybdenum complexes generated through reaction of laser-ablated molybdenum atoms with methane were studied by DFT calculations [1205]. The reaction leads to the observable carbene complex $H_2C{=}MoH_2$ and the carbyne complex $HC{\equiv}MoH_3$. As the reaction progresses the spin state at molybdenum changes. The initially generated molybdenum atom is most stable as a quintet. An intermediate, CH_3MoH is also a pentet. The carbene complex is a triplet and the carbyne complex is a singlet. The energy of activation in each step is much higher if spin state is conserved during the alkyl to carbene to carbyne transformation.

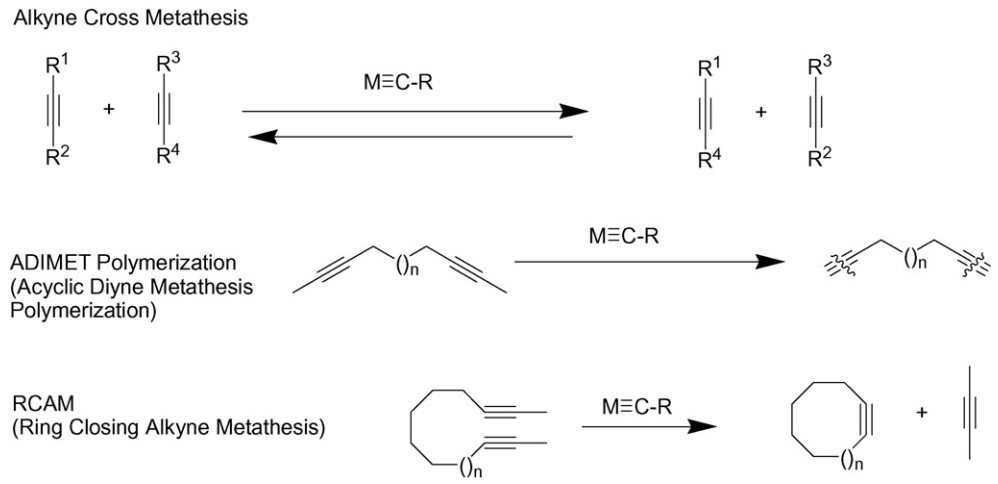
The reaction of laser-ablated osmium atoms with methane and methyl halides was reported [1206]. The reaction leads to observable osmium carbyne complexes. Structures were assigned through comparison of IR spectra with calculated IR spectra. The osmium–carbyne complex $HC{\equiv}OsH_3$ features two kinds of osmium–H bonds. Related $HC{\equiv}MoH_2X$ complexes were obtained from reactions involving methyl halides (X = F, Cl, Br). The reaction of laser-ablated rhenium atoms with methane or methyl halides also led to carbyne complexes, which displayed IR spectra similar to calculated spectra [1207].

3.3. Reactivity

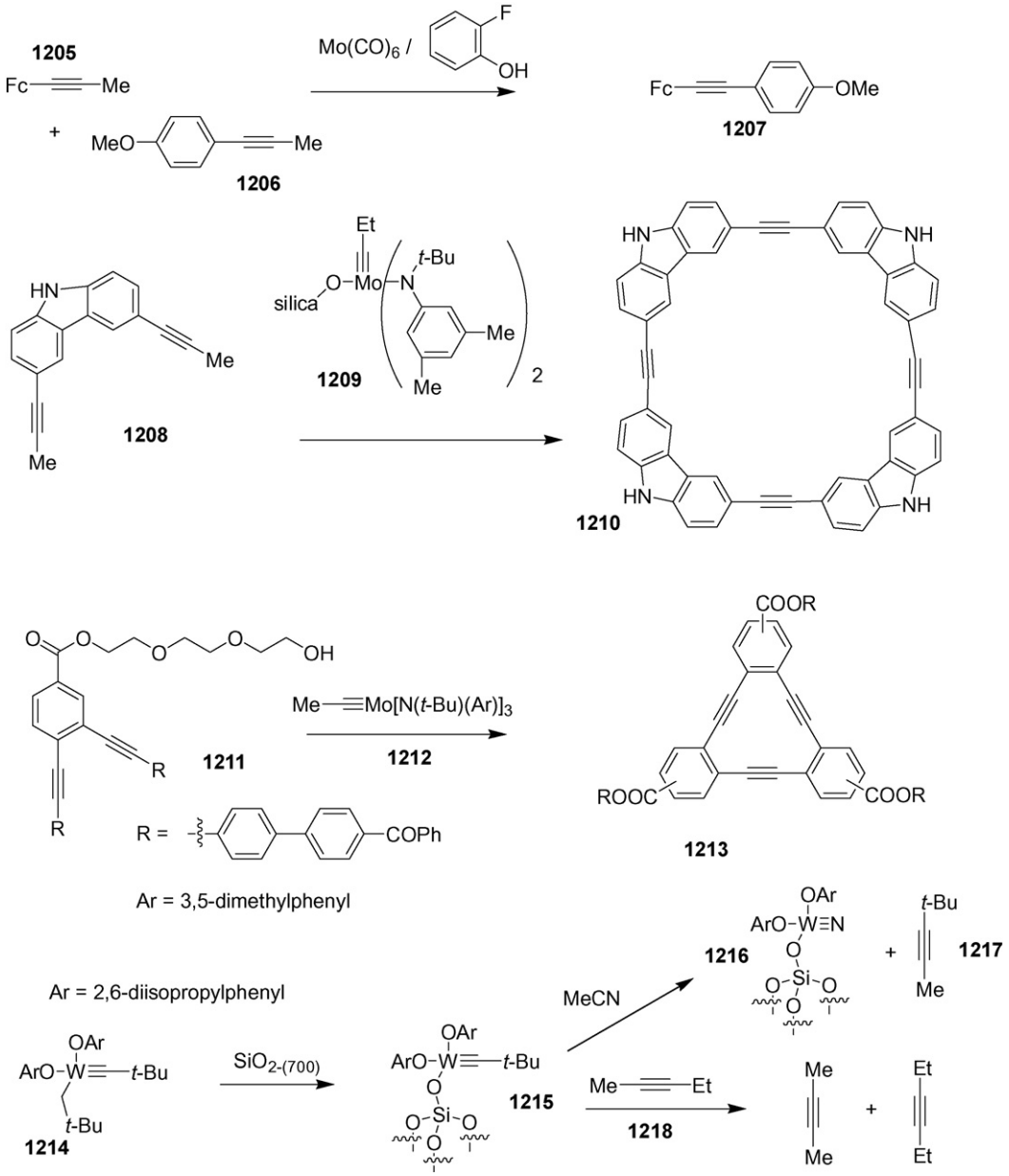
3.3.1. Addition reactions of metal–carbyne complexes

Substitution reactions were reported for bromocarbyne–molybdenum complexes (*e.g.* **1180**, Scheme 132) [1208]. Reaction of bromocarbyne complex **180** with amines led to the corresponding aminocarbyne complexes (*e.g.* **1181**). A variety of different amines, including secondary amines, imidazoles, and pyridines were successful. Reaction of potassium bis(thiophenyl)amide led to the thiocarbyne complex (**1183**) instead of the aminocarbyne complex. A thiocarbyne complex (**1182**) could was also produced through reaction of thiolates with bromocarbyne complex **1180**. Reaction with aqueous DBU led to the trioxo complex **1184**. Related selenoalkynylations of chloromolybdenum carbyne complex **1185** were also reported, resulting in selenocarbyne complexes (*e.g.* **1186**) [1209]. Several alkyne complexation reactions were demonstrated for selenocarbyne complex **1186**. Complexation to cobalt (**1187**) results in significant changes to the Mo–C–Se bond angle. Reaction of selenocarbyne–molybdenum complex **1186** with platinum resulted in the corresponding alkyne complex **1188** and C–Se oxidative addition products **1189** and **1190** [1210]. The reaction of bromocarbyne complex **1191** with $(CH_2{=}CH_2)Pt(PPh_3)_2$ led initially to the addition product, platinacarbene complex **1192**, which then isomerizes to the carbyne complex **1193** [1211]. Other metal-substituted carbyne complexes (*e.g.* **1195**) were successfully prepared from dichlorocarbene–iron complex **1194** and metal carbonyl anions. Reaction of the iron complex with metal pentacarbonyl dianions led to the bridging carbide complexes (*e.g.* **1196**).

Phosphine additions to cationic manganese carbyne complexes (*e.g.* **1197**, Scheme 133) were reported [1212]. Reaction of with secondary phosphines led to the protonated phosphinocarbene complexes (*e.g.* **1198**), which convert to the phospinoketene complexes (*e.g.* **1201**) upon treatment with base. Analogous reaction processes employing primary phosphines led to the neutral phosphinocarbene complexes (*e.g.* **1199**). The phosphinocarbene



Scheme 134.



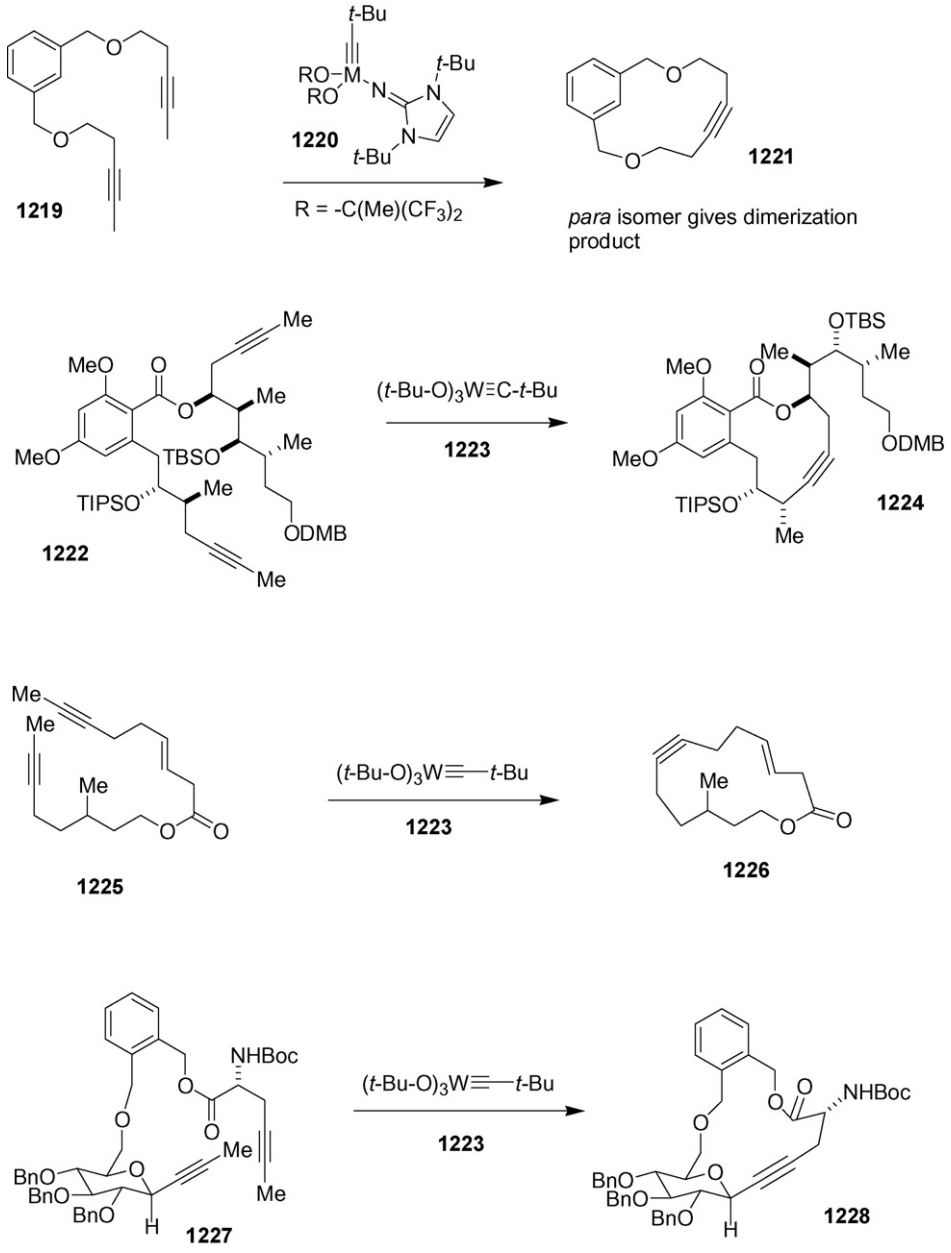
Scheme 135.

complex **1199** eventually evolves into the phosphaalkene complex **1200** at room temperature in THF.

3.3.2. Alkyne metathesis

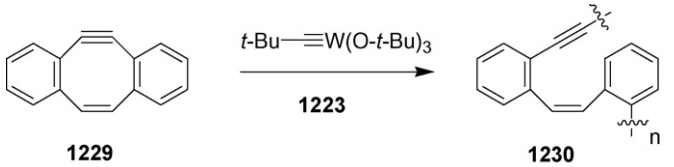
Alkyne metathesis, which involves metal carbyne complexes as intermediates, has been covered comprehensively regardless of whether the initiator is a carbyne complex. General equations describing common modes of alkyne metathesis are presented in Scheme 134.

Several examples of alkyne cross-metathesis, dimerization, and cyclodimerization were reported in 2008, see Scheme 135. Molybdenum hexacarbonyl induced alkyne cross-metathesis was employed in the synthesis of alkynylferrocene derivatives (*e.g.* **1207**) from a simpler alkynylferrocene, **1205** [1213]. Heterogeneous alkyne metathesis catalysts (*e.g.* **1209**) were produced through reaction of a tris (amido) molybdenum carbyne complex with silica [1214]. Cyclic tetramers (*e.g.* **1210**) were formed from bis(alkyne) derivative **1208** using this catalyst system. Cyclic trimers (*e.g.* **1213**) were prepared from the reaction of bis(alkyne) complex **1211** with carbyne complex metathesis catalyst **1212** [1215]. A silica-bound tungsten–carbyne complex (**1215**) was an effective catalyst for alkyne metathesis [1216]. Carbyne complex **1215** also couples stoichiometrically with acetonitrile to form the alkyne **1217** and the nitridotungsten complex (**1216**). Examples of ring-closing alkyne metathesis (RCAM) are depicted in Scheme 136. Competition between RCAM and alkyne metathesis dimerization was observed in the formation of various cyclophane derivatives (*e.g.* **1221**) from bis(alkynes) (*e.g.* **1219**) and carbyne complex **1220** [1217]. Alkyne metathesis of bis(alkyne) **1222** using tungsten carbyne complex **1223** was a key step in the preparation of cruentarin A analogs [1218]. Macrocyclic lactones useful for the synthesis of musk odorants (*e.g.* **1226**) were prepared through tungsten carbyne induce alkyne metathesis [1219]. Macrocycle-bridged carbohydrates (*e.g.* **1228**) were prepared through RCAM [1220]. Ring opening alkyne metathesis polymerization was demonstrated in the reaction of cyclic alkyne derivative **1229** (Scheme 137) with tungsten carbyne complex **1223** [1221]. Reaction of alkyne **1229** with Grubbs catalyst II led to alkyne trimerization products.



Scheme 136.

Nitrile-alkyne metathesis was reported (see Scheme 138) [1222]. The reaction of tungsten nitrides (*e.g.* **1231**) with 3-hexyne led to the tungsten–carbyne complex (**1232**), the metallacyclobutadiene (**1233**), and propionitrile. The nitride complex serves as a catalyst for the co-metathesis of nitriles and alkynes. In the presence of a "sacrificial" alkyne (*e.g.* 3-hexyne) the nitride complexes can also serve as an alkyne metathesis catalyst. A tungsten nitride dimer (**1238**) was prepared through reaction of tungsten carbyne complex **1237** with ^{15}N-labeled acetonitrile [1223]. Gas phase metathesis of a nitride–iron complex with alkynes to form a carbyne complex and a nitrile was demonstrated [1224]. The tungsten nitride catalyzed exchange of the nitrogen atoms of nitriles was evaluated through experimental and theoretical studies [1225]. This mechanistic similarity of this process and alkyne–nitrile metathesis was noted. An additional example of stoichiometric nitrile–alkyne cross-metathesis was previously discussed in Scheme 135 [1216].

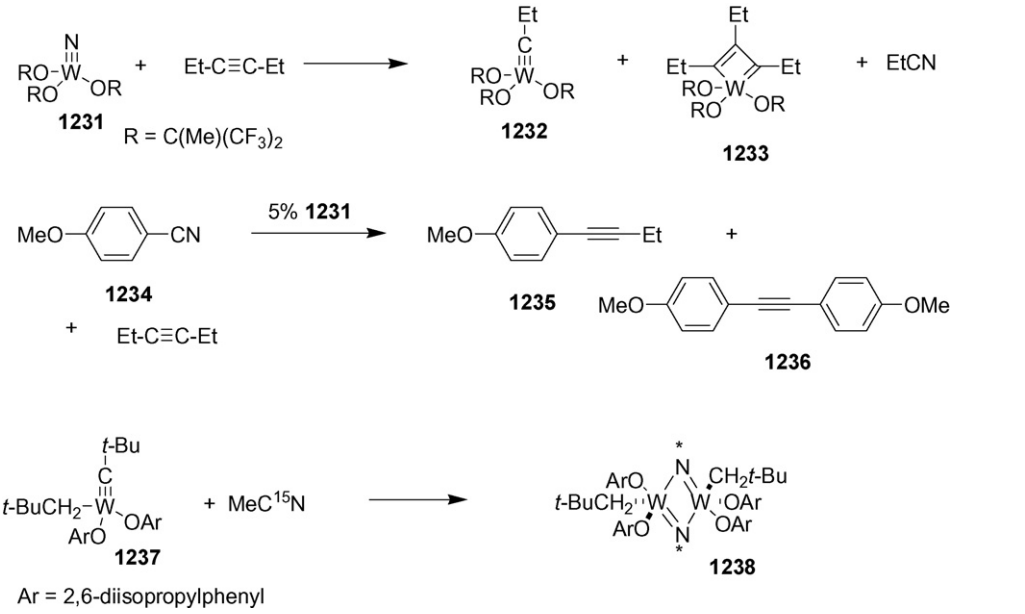


Scheme 137.

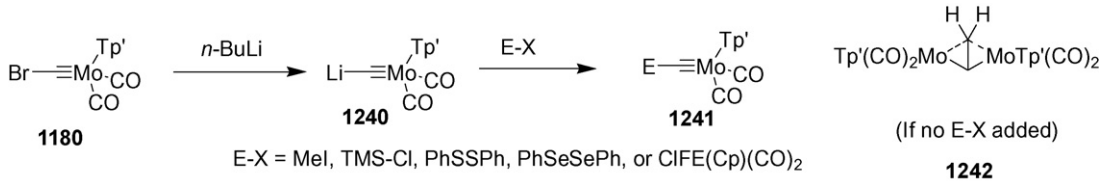
3.3.3. Other processes involving metal–carbyne complexes

The formation and reactivity of lithiocarbyne–molybdenum complexes (*e.g.* **1240**, Scheme 139) was reported [1226]. Reaction of the bromohalide **1240** with *n*-butyllithium affords lithiocarbyne complex **1240**, which then reacts with various electrophiles to afford substituted methylidyne complexes (**1241**). In the absence of an electrophile, a bimetallic vinylidene complex (**1242**) was produced.

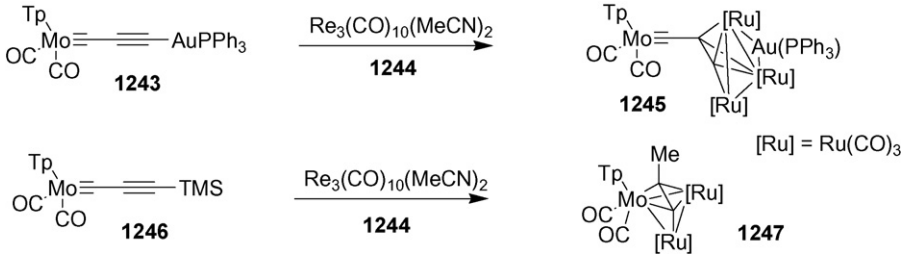
The reaction of alkynylcarbyne–molybdenum complexes (*e.g.* **1243**, **1246**, Scheme 140) with various metal carbonyl clusters was



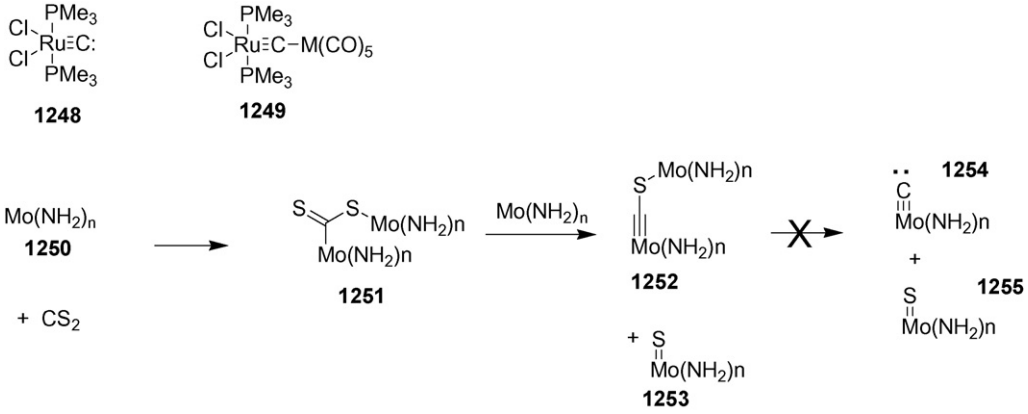
Scheme 138.



Scheme 139.



Scheme 140.



Scheme 141.

reported [1227]. Reaction of the aurylethynylcarbyne complex **1243** with the ruthenium cluster **1244** led to the alkyne complexation product **1245**. A similar reaction employing the silylethynylcarbene complex analog and Group 8 metal clusters led to the carbide complex derivative **1247**. Reaction employing cobalt and nickel clusters led to complexation at the alkyne and retention of the metal carbyne functionality.

The interconversion of ruthenium carbyne complex $(Me)_2(NHt\text{-}Bu)Re{\equiv}CMe(NH_2)$ and $(Me)_2(NHt\text{–}Bu)Re{=}CHMe(=NH)$ [1228] was reported. The process was determined to not be intramolecular. The most energetically accessible pathway involves intervention by a water molecule. Similar studies were reported for hydrogen migrations in analogous chromium–carbyne complexes (*e.g.* $Cl_2Cr{\equiv}CH(CH_2CH_3)$) [1229].

The ability of ruthenium–carbide complexes (*e.g.* **1248**, Scheme 141) to serve as ligands for transition metals (*e.g.* as in complex **1249**) was evaluated computationally [1230]. Ruthenium–carbide complex **1248** was determined to be a very CO-like ligand. The hypothetical formation of molybdenum–carbide complex **1254** through reaction of molybdenum complex **1250** with carbon disulfide was studied computationally [1231]. The conversion to the thiocarbonyl-bridged dimolybdenum complex **1251** was thermodynamically favorable, however further conversion to the carbide and molybdenum sulfide was an endothermic process. Group 6–Group 9 metal–carbide complexes were studied computationally [1232]. The focus was primarily on the metal carbon bond. The carbon atom is best described as unhybridized with the lone pair in a 2s orbital. The C–metal bond strength in these complexes is comparable to that of metal nitrides and metal oxo complexes; for example the bond dissociation energy is 147 kcal/mol in $Cl(PMe_3)_2Ru{\equiv}C$.

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