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Review

The chemistry of the carbon–transition metal double and triple bond: Annual survey covering the year 2006☆

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Contents

Abstract

This is a review of papers published in the year 2006 that focus on the synthesis, reactivity, or properties of compounds containing a carbon–transition metal double or triple bond.

Keywords: Carbene complexes; Alkylidene complexes; Carbyne complexes; Metallacumulenes; Olefin metathesis; Alkyne metathesis

☆ For 2005, see J.W. Herndon, Coord. Chem. Rev. 251 (2007) 1158.

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1. Introduction

This survey is intended to be a comprehensive summary of articles that report on the synthesis, reactivity, or properties of compounds featuring a multiple bond between carbon and a transition metal. Reactions that employ metal–carbene complexes as transient intermediates generated through well-established routes are not covered, unless there is some effort to characterize the carbene complex intermediate. Several reviews in this area appeared in 2006 [1–4]. Although a determined effort has been made to include patents, in general only patents that focus on the metal–carbene or metal–carbyne complex are included. Patents that appear in 2006 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 [5–8]. This area was reviewed several times in 2006 [9–16] and the back donation issue was evaluated experimentally [17,18] and computationally [19], and found to be present to some extent. 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 [20] 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 45, 12, 62, and 47, respectively, which covers the period of December 2005–December 2006 according to some search engines.

Abbreviations (see also the front of issue #1 of the *Journal of Organic Chemistry* [21]).

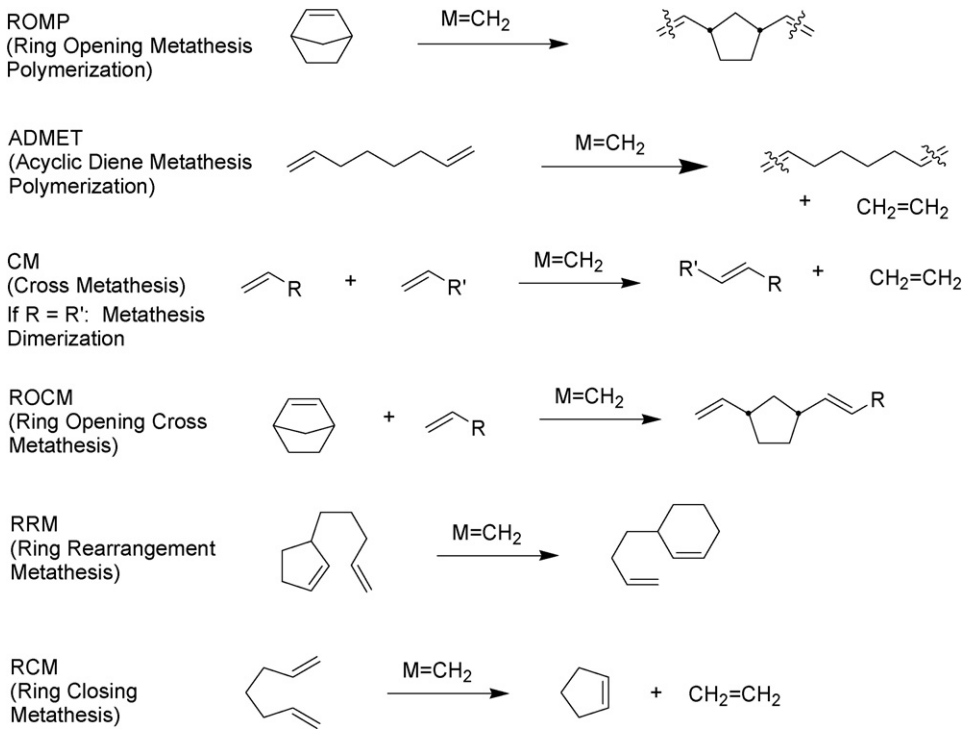
DFT	Density functional theory
NHC	*N*-heterocyclic carbene
Grubbs catalyst I	Structure **1** (Fig. 1)
Grubbs catalyst II	Structure **2** (Fig. 1)
Grubbs catalyst III	Structure **3** (Fig. 1)
Hoveyda–Grubbs catalyst	Structure **4** (Fig. 1)
Schrock catalyst	Structure **5** (Fig. 1)

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

2. Metal–carbene or metal–alkylidene complexes

2.1. Review articles and comments

Several reviews/comments covering aspects of metal–carbene complex chemistry appeared in 2006. Many articles focusing on some aspect of carbene complex-initiated olefin metathesis were published, including the following specific subjects: (1) the early days of olefin metathesis [22]; (2) the



Scheme 1.

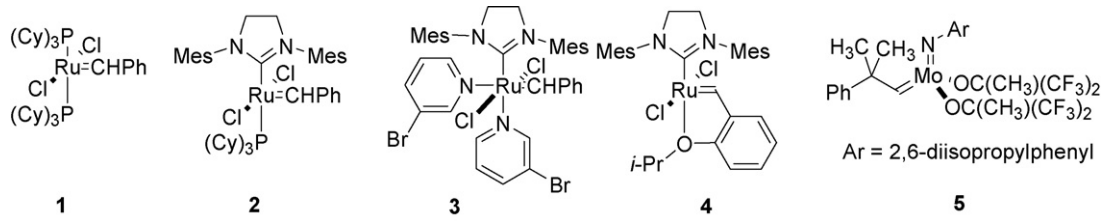


Fig. 1. Structures of alkene metathesis catalysts **1**–**4**.

history of olefin metathesis [23]; (3) general olefin metathesis [24,25]; (4) ruthenium catalysts for alkene metathesis [26]; (5) tunable ruthenium catalysts for olefin metathesis [27]; (6) olefin metathesis catalysts [28–30]; (7) carbon–metal multiple bonds for catalytic metathesis reactions [31]; (8) olefin metathesis for the preparation of molecules and materials [32]; (9) olefin and alkane metathesis [33]; (10) preparation of silicon-containing materials using ADMET polymerization [34]; (11) synthesis of heterocycles via RCM [35]; (12) formation of *N*-heterocycles using RCM [36]; (13) synthesis of aromatic rings via RCM [37]; (14) formation of macrocycles via RCM [38]; (15) formation of macrocyclic crown compounds using RCM [39]; (16) formation of macrocyclic cyclophanes via ring-closing metathesis [40]; (17) RCM using substrates that contain more than two alkene groups [41]; (18) factors influencing ring closure in the RCM reaction [42]; (19) formation of strained ring systems using metathesis reactions [43]; (20) use of heteroatom-substituted alkenes and alkynes in metathesis reactions [44]; (21) use of cross metathesis in organic synthesis [45]; (22) use of cross metathesis for the formation of trisubstituted alkenes [46]; (23) use of cross metathesis for the synthesis of bioactive phosphonates [47]; (24) use of cross metathesis involving alkenes attached to fluorophores [48]; (25) occurrence of metathesis reactions in tandem with other reaction processes [49]; (26) use of metathesis for the formation of nanostructures [50]; (27) surface-bound olefin metathesis catalysts [51]; (28) metathesis polymerization to and from surfaces [52]; (29) olefin metathesis and isomerization [53,54]; (30) enyne metathesis [55]; (31) reactivity and selectivity problems in enyne metathesis [56]; (32) use of ruthenium allenylidene complexes as metathesis catalysts [57]; (33) "green" techniques in olefin metathesis [58]; and (34) metathesis in ionic liquids [59]. Several review articles report on synthesis of various compound classes where carbene complex initiated olefin metathesis is a commonly employed synthetic route. Specific compound classes represented include: (1) azaspirodecane derivatives [60]; (2) guanacastapene A [61]; (3) heliannuol sesquiterpenoids [62]; (4) ingenol derivatives [63]; (5) amino acids [64]; (6) imidazoles [65]; (7) indole alkaloids [66]; (8) tamiflu [67]; (9) oxepine syntheses [68]; (10) polycyclic ethers [69]; (11) macrocidin pesticides [70]; (12) epothilones [71]; (13) azaepothilones [72]; (14) vinyl sulfones [73]; (15) ceramides [74]; (16) ansa metallocenes [75,76]; (17) multivalent ligand receptors [77]; (18) *cyclo*-olefin copolymers [78]; and (19) supported metal catalysts [79]. Additional review articles include some segments on carbene complex-initiated metathesis. Articles in this category focus on the following subjects: (1) fluorous solid phase extraction [80]; (2) domino reactions [81]; (3) metallodendrimers as catalysts for olefin polymerization [82]; (4) olefin polymerization catalysts [83]; (5) polymer synthesis in 2005 [84]; (6) cascade reactions initiated by ruthenium catalysts [85]; (7) ruthenium NHC complexes in catalysis [86], and (8) PEG-supported catalysts [87]. Several reviews on carbene complex chemistry featuring some aspect other that metathesis appeared in 2006. These reviews include the following subjects: (1) titanium and vanadium carbene and carbyne complexes [88,89]; (2) heterocyclization reactions using Fischer carbene complexes [90]; (3) carbene complexes of heme proteins and iron porphyrin models [91]; (4) metallabenzene chemistry [92,93]; (5) metal vinylidenes and allenylidenes in catalysis [94]; (6) pericyclic reactions involving metal vinylidene complexes [95]; (7) catalysis of C–C bond forming reactions of propargylic alcohols by ruthenium complexes [96]; (8) metallacumulenes as potential electronic devices [97]; and (9) platinum and gold-catalyzed enyne cycloisomerization [98];. Although not specifically focusing on metal–carbene complexes, some review articles place some emphasis on this subject. Subjects reviewed in this category include: (1) syntheses of 1-oxadecalins (frequently employing Fischer carbene complexes) [99]; (2) coupling of laser-ablated metal atoms with simple organics and matrix isolation studies [100]; (3) ruthenium catalysts for the Kharasch reaction [101]; (4) radical polymerization catalyzed by ruthenium–NHC complexes [102]; (5) free radical reactions [103]; (6) gold-catalyzed benzannulation [104]; (7) gold catalysis [105]; (8) chemistry of the noble metals [106]; (9) formation of alkene complexes from metal carbonyls [107]; and (10) multiply bonded actinides [108].

2.2. Alkene metathesis

Alkene metathesis was the most common reaction process reported for metal–carbene complexes in 2006, 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 discreet 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 2006; 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 either ROMP

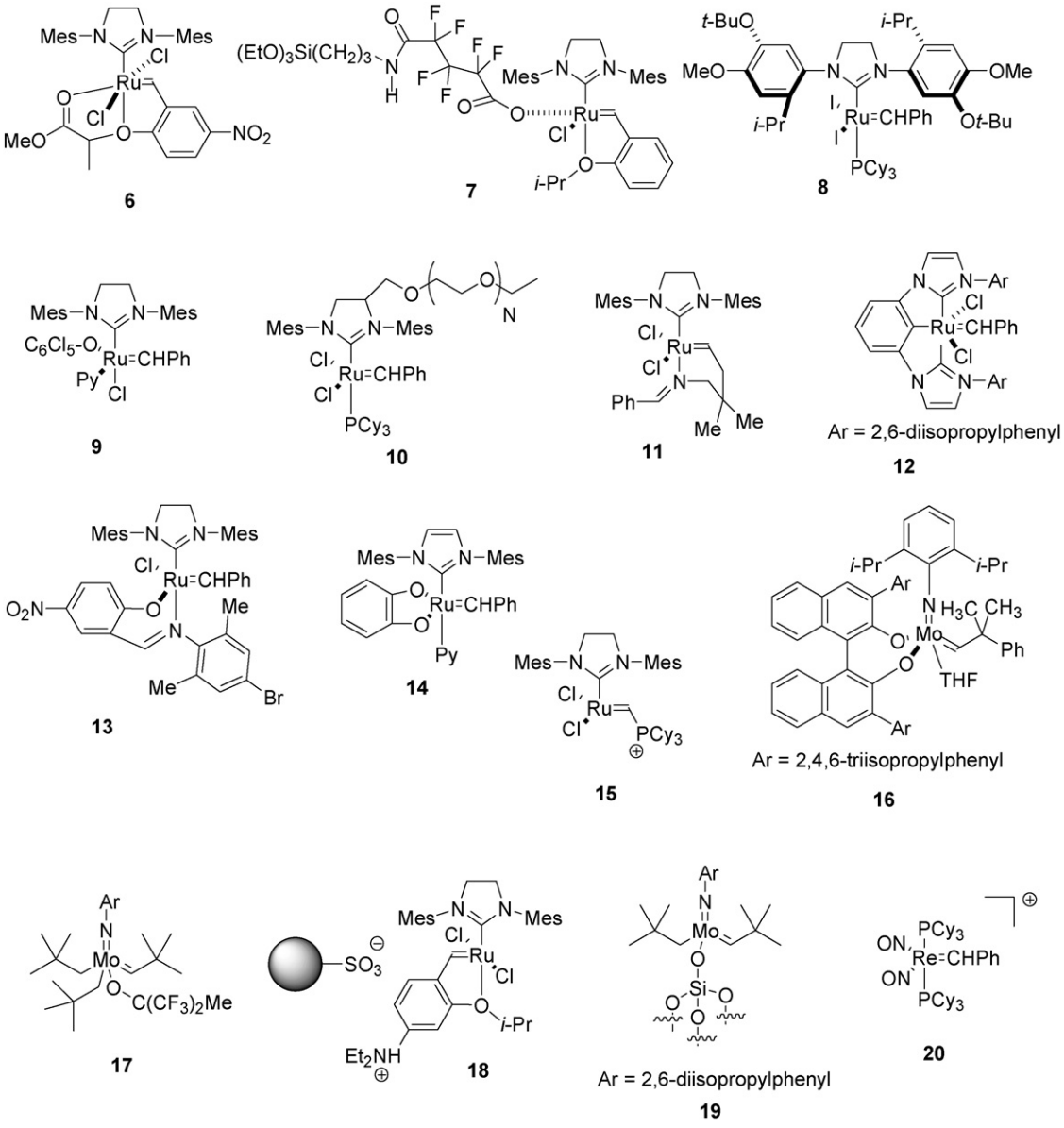
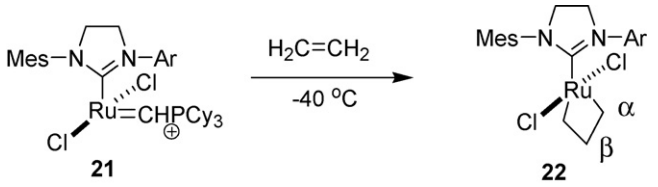


Fig. 2. Representative examples of new catalysts for alkene metathesis.

or RCM processes, including: (1) highly active analogs of Grubbs catalyst II and the Hoveyda–Grubbs catalyst where fluorinated aromatic rings replace the mesityl groups [109]; (2) an octahedral analog of the Hoveyda–Grubbs catalyst (**6**) [110]; (3) analogs of the Hoveyda–Grubbs catalyst featuring sulfonated aromatic rings and a dimeric sulfone-linked analog [111]; (4) analogs of the Hoveyda–Grubbs catalyst featuring nitrated arylcarbene groups [112]; (5) an analog of the Hoveyda–Grubbs catalyst featuring a tetraalkylammonium group on the arylcarbene group [113]; (7) an analog of the Hoveyda–Grubbs catalyst featuring a perfluorinated silyl group on the arylcarbene group and a silica-immobilized analog of this catalyst [114]; (8) analogs of the Hoveyda–Grubbs catalyst featuring unsymmetrical NHC ligands [115]; (9) chiral and chelated analogs of the Hoveyda–Grubbs catalyst featuring mixed anionic ligands (*e.g.* **7**) [116,117], (10) analogs of the Hoveyda–Grubbs catalyst that feature imidazolinium cation groups for metathesis reactions in ionic liquids [118]; (11) a reusable analog of the Hoveyda–Grubbs catalyst [119]; (12) water-soluble analogs of Grubbs catalyst I [120]; (13) analogs of Grubbs catalyst II featuring sterically bulky *N*-aryl groups [121]; (14) analogs of Grubbs catalyst II where one of the mesityl groups has been replaced by various alkyl groups [122]; (15) analogs of Grubbs catalyst II featuring 2,6-dimethylphenyl groups in place of mesityl groups and arylphosphines in place of tricyclohexylphosphine [123–125]; (16) fluorescently labeled analogs of Grubbs catalyst II [126]; (17) chiral analogs of Grubbs catalyst II (*e.g.* **8**) for asymmetric metathesis reactions [127,128]; (18) highly active phenoxy analogs of Grubbs catalyst II [129] and analogs featuring phenoxy and pyridine ligands (*e.g.* **9**) [130]; (19) water-soluble polyethylene glycol-bound analogs of Grubbs catalyst II for aqueous-phase metathesis reactions (*e.g.* **10**) [131]; (20) analogs of Grubbs catalyst II featuring fluorinated phosphine ligands [132]; (21) chelated analogs of Grubbs



Scheme 2.

catalyst II that initiate metathesis reactions only at elevated temperatures (*e.g.* **11**) [133]; (22) analogs of Grubbs catalyst II featuring quinoline–carbene chelates [134]; (23) ruthenium carbene complexes containing pincer–NHC ligands (*e.g.* **12**) [135]; (24) a chelated imine–ruthenium carbene complex (**13**), which becomes an active catalyst upon treatment with trichlorosilane [136,137]; (25) catechol-ligated ruthenium carbene complexes (*e.g.***14**) [138]; (26) a phosphonium alkylidene–ruthenium complex (**15**) [139]; (27) ruthenium acyclic amine complexes [140]; (28) indenylidene analogs of Grubbs catalyst III [141]; (29) chiral analogs of the Schrock catalyst for asymmetric RCM reactions (*e.g.* **16**) [142,143]; (30) a trifluoroacetate-bridged dimeric analog of the Schrock catalyst, which becomes an active catalyst upon breaking up the dimer with quinuclidine [144]; (31) analogs of the Schrock catalyst featuring alkyl ligands (*e.g.* **17**) [145]; (32) immobilized analogs of the Hoveyda–Grubbs catalyst using a variety of support materials [146,147]; (33) an ionically immobilized analog of the Hoveyda–Grubbs catalyst (*e.g.* **18**) [148]; (34) silica-bound analogs of the Hoveyda–Grubbs catalyst [149]; (35) polydimethyl siloxane-occluded Grubbs catalyst II [150]; (36) polymer-bound analogs of Grubbs catalyst I [151]; (37) analogs of the Schrock catalyst bound to a resin through the imido group [152]; (38) a germanium-substituted tungsten alkylidene complex [153]; (39) silica-bound analogs of the Schrock catalyst (*e.g.* **19**) useful for alkene and alkane metathesis and efforts to better understand the reactivity of these systems [154–157]; (40) placement of Grubbs catalyst II into a polydimethylsiloxane slab resulting in a catalytic system selective for nonpolar substrates [158]; and (41) cationic rhenium dinitrosyl complexes (*e.g.* **20**) [159]. Several patents were issued for the synthesis and development of metal–carbene containing olefin metathesis catalysts [160–164].

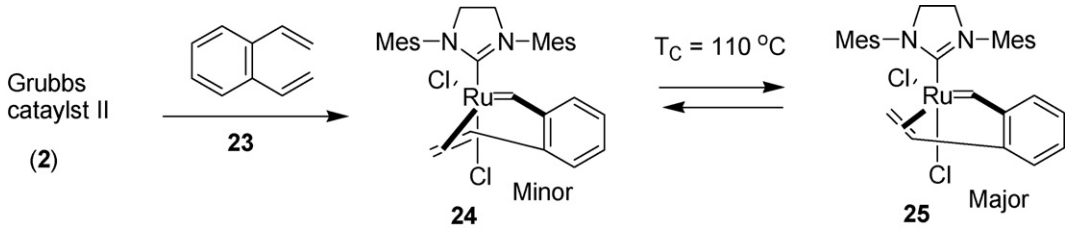
The formation of stable ruthenacyclobutanes (*e.g.* **22**, Scheme 2) from the reaction of carbene complex **21**, which features an unsymmetrical NHC ligand with ethylene or propene at low temperature, was reported [165]. The structure of the complexes is more consistent with bottom face attack of the alkene. A dynamic exchange process interconverting the α and β carbons was revealed through 2D NMR experiments which was indicative of metallacycle retrocycloaddition and reformation. This interconversion is an internal process since there was no exchange observed with external olefin.

Formation of alkene-coordinated ruthenium carbene complexes (**24**, **25**, Scheme 3) was achieved through the reaction of Grubbs catalyst II with *o*-divinylbenzene (**23**) [166]. The reaction affords a mixture of the side-bound alkene complexes **24** and **25**, which coalesce at 110 °C. The minor isomer could be crystallized and the structure was verified through X-ray crystallography. There was no evidence for a bottom-bound alkene (alkene ligand *trans* to the NHC ligand) complex. A computational study of the mechanism of alkene metathesis suggested that the alkene coordinates *trans* to the NHC ligand [167], however solvent effects were determined to play a very important in determining the relative stability of intermediates.

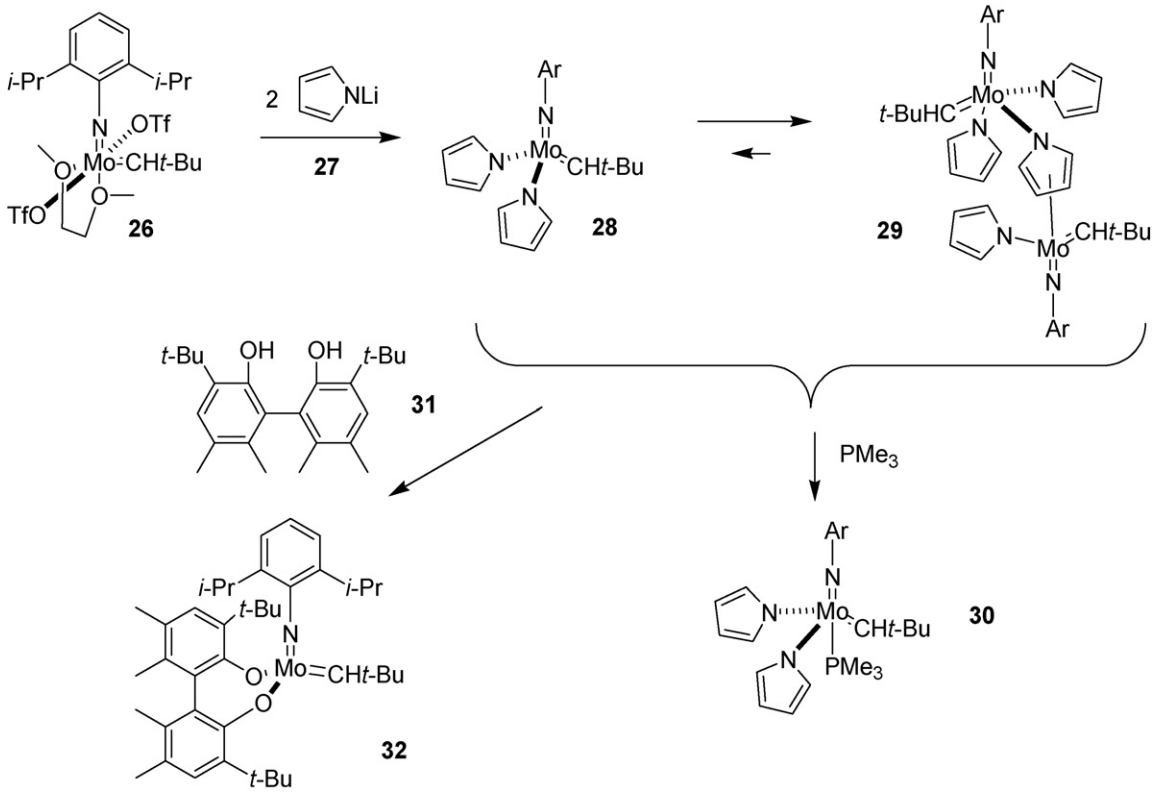
The 14-electron intermediates of ruthenium catalyzed metathesis reaction [$LCl_2Ru{=}CH_2$] and ruthenacyclobutanes were studied computationally (DFT) and a QSAR model was developed for metathesis catalysts [168]. The most effective L groups in metathesis reactions stabilize the high oxidation state metal of the metallacyclobutane intermediate via σ-donation. Steric repulsion from the L group is also beneficial since it drives the metallacyclobutane into a less sterically congested orientation.

A general synthetic route for the preparation of derivatives of the Schrock catalyst was reported (Scheme 4) [169]. Reaction of molybdenum carbene complex **26** with lithiopyrrole led to the bis(pyrrolyl) complex **28**, which is in equilibrium with dimeric complex **29**. Reaction of **28/29** with bis(phenol) derivatives (*e.g.* **31**) leads to compounds that are useful as alkene metathesis catalysts (*e.g.* **32**), including asymmetric catalysts. Cationic derivatives of the Shrock catalyst containing acac ligands (*e.g.* **34**, **38**, Scheme 5) were prepared and their reaction with alkenes examined [170]. These complexes were not effective metathesis catalysts. Reaction of complex **34** with ethylene led to the metallacyclobutene **35**, metathesis product **36**, and cyclopropane. The analog featuring a 2,6-dichlorophenyl group on nitrogen (**39**) was a catalyst for the ROMP of norbornene, however was not effective at RCM of diallyl ether due to coordination of the ether oxygen.

A study of rhenium-catalyzed metathesis was reported (Scheme 6) [171]. The cationic rhenium complex **40** is an effective catalyst for ROMP of norbornene. Treatment of complex **40** with phenyldiazomethane affords a carbene complex **41**, which is inactive for ROMP of norbornene. A novel mechanism for generation of the metathesis-initiating carbene species was proposed. Addition to norbornene affords alkylrhenium complex–phosphonium salt **42**, which then rearranges to the



Scheme 3.

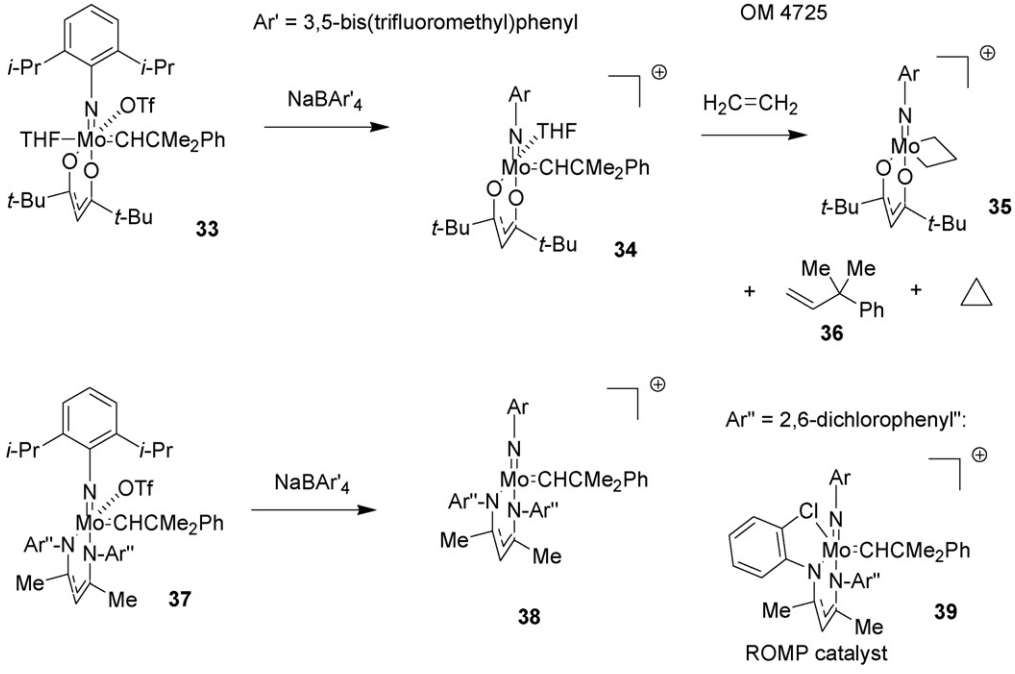


Scheme 4.

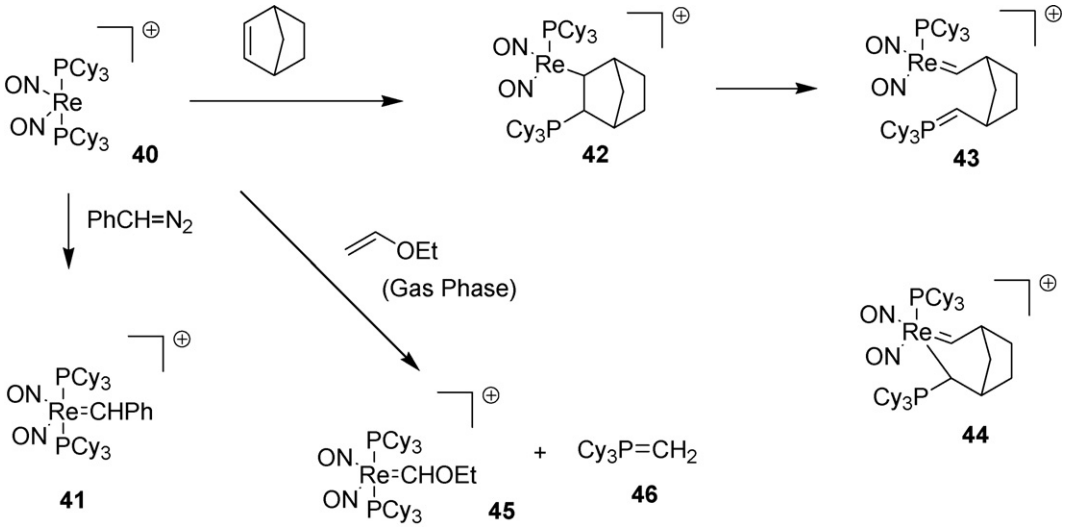
carbene–phosphorane complex **43**, which then cyclizes to afford carbene complex **44**. Gas phase reaction of complex **40** with ethyl vinyl ether led to the alkoxycarbene complex **45** and the phosphorane **46**.

The formation of a metathesis catalyst through reaction of atomic carbon with tungsten(VI) chloride was reported (Scheme 7) [172]. The hypothetical catalyst, **47**, catalyzes the metathesis of 1-octene and afforded the carbonyl olefination product **48** upon treatment of with benzaldehyde. Formation of the catalyst was evaluated computationally, and was found to be a highly exothermic process.

Other general studies of alkene metathesis where carbene complexes were discussed include: (1) metathesis reactions in ionic liquids [173]; (2) exchange of the anionic ligands in



Scheme 5.

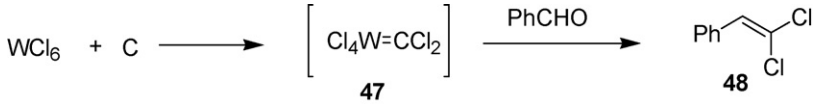


Scheme 6.

the Hoveyda–Grubbs catalysts and potential implications for metathesis reactions [174]; (3) addition of tin and iron halides as additives in ruthenium catalyzed metathesis reactions [175]; (4) dynamic NMR studies for analogs of Grubbs catalyst I [176]; (5) successful performance of metathesis reactions in a continuous flow reactor [177]; (6) monitoring of the progress of a ROMP reaction by Raman Spectroscopy and determining the *cis–trans* ratios within the polymer [178]; (7) XAS studies of ruthenium chloride olefin metathesis catalysts [179]; (8) establishment of standard screening reactions for comparison of alkene metathesis catalysts [180]; (9) use of mesoporous silicates for removal of ruthenium-containing byproducts from metathesis reactions [181]; (10) a computational study of alkene metathesis focusing on the mechanism and the enhanced stability and longevity of catalysts featuring bicyclic phosphine ligands [182]; (11) a computational study of metathesis of 1-octene [183]; (12) a computational study of metathesis reactions catalyzed by phosphorus analogs of ruthenium–NHC complex catalyzed metathesis reactions [184]; (13) a computational study of the cross metathesis of dimethyl maleate and ethylene [185]; (14) the importance of C–C agnostic interactions in metallacyclobutane intermediates of metathesis reactions [186]; (15) a study of the surface-bound molybdenum–carbene complexes generated through reaction of molybdenum carbide surfaces with cyclopentanone [187]; and (16) a DFT study focused on understanding the different behavior of Grubbs catalyst II and analogs where the NHC ligand is aromatic [188]. Patents were issued for: (1) a method to conduct RCM reactions in supercritical CO_2 [189]; and (2) development of a membrane to purify metathesis mixtures [190].

2.2.2. *Polymerization reactions*

Initiation of the ring opening metathesis polymerization (ROMP) (see Scheme 1) reaction using carbene complexes remains a very active area of investigation. The strained alkene norbornene, norbornene derivatives, and copolymerization involving a norbornene derivative and another alkene accounted for a large fraction of all reports of the ROMP reaction in 2006; 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) simple norbornenes [191]; (2) norbornene diesters [192]; (3) norbornenecarboxylic acids [193]; (4) norbornenes linked to polythiophene derivatives (*e.g.* **50**) [194]; (5) norbornenesuccimides (a surface-bound ruthenium catalyst was employed) [195]; (6) dichlorophosphonates (*e.g.* **51**) [196]; (7) norbornenes linked to radical polymerization initiating groups (*e.g.* α-bromo ester **52**) [197,198]; (8) norbornenes linked to fullerenes [199]; (9) norbornenes linked to dendrons [200]; (10) norbornadiene–carbohydrate nitrile oxide adducts (*e.g.* **53**) [201]; (11) benzonorbornenes (*e.g.* **54**) [202]; (12) norbornenes linked to cyclophosphazene rings [203]; (13) norbornenes fused to additional norbornene rings (*e.g.* **55**) [204]; (14) simple norbornenes (initiation via the Schrock catalyst followed by termination with aldehydes connected to bipyridyl ligands) [205]; (15) indole-substituted norbornenes [206]; (16) thiocyanate-substituted norbornenes [207]; (17) bis(norbornenes) connected through a polynuclear aromatic hydrocarbon [208]; (18) bis(norbornenes) connected through a nickel–thiol linkage (*e.g.* **56**) [209]; (19) norbornenes connected to porphyrins [210]; (20) norbornenes connected to fullerenes [211]; (21) norbornenes connected to metal–NHC complexes (*e.g.* **57**) [212]; and (22) norbornenes connected to salen complexes (*e.g.* **59**) [213]. Copolymers prepared through ROMP reported in 2006 include: (1) block copolymers from norbornene-fused succinimides and cyclic acetals derived from 2-butene-1,4-diol [214]; (2) block copolymers from crown ether-



Scheme 7.

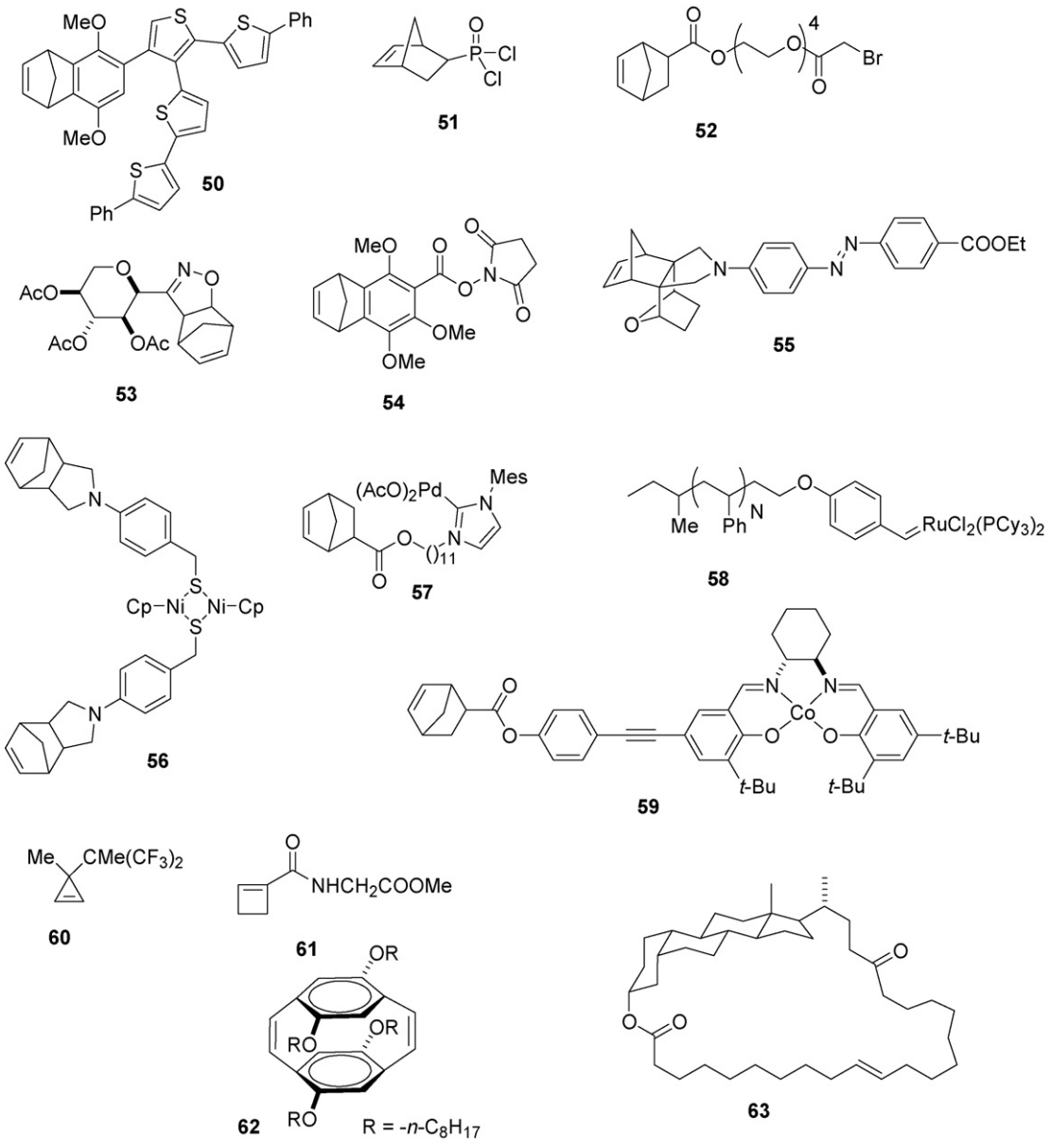


Fig. 3. Representative substrates for the ROMP reaction.

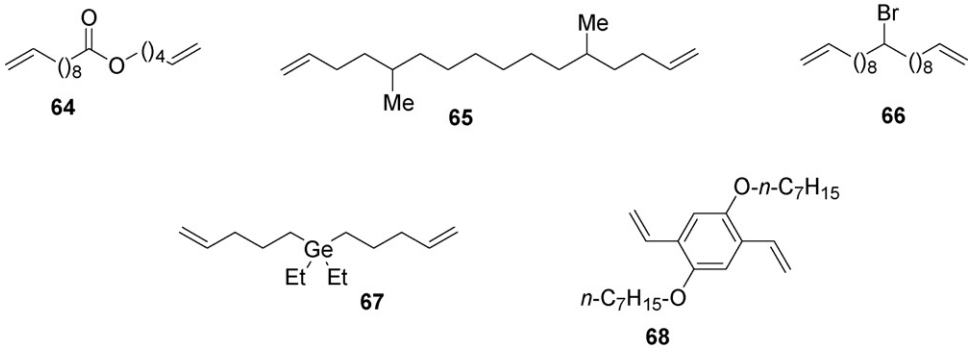
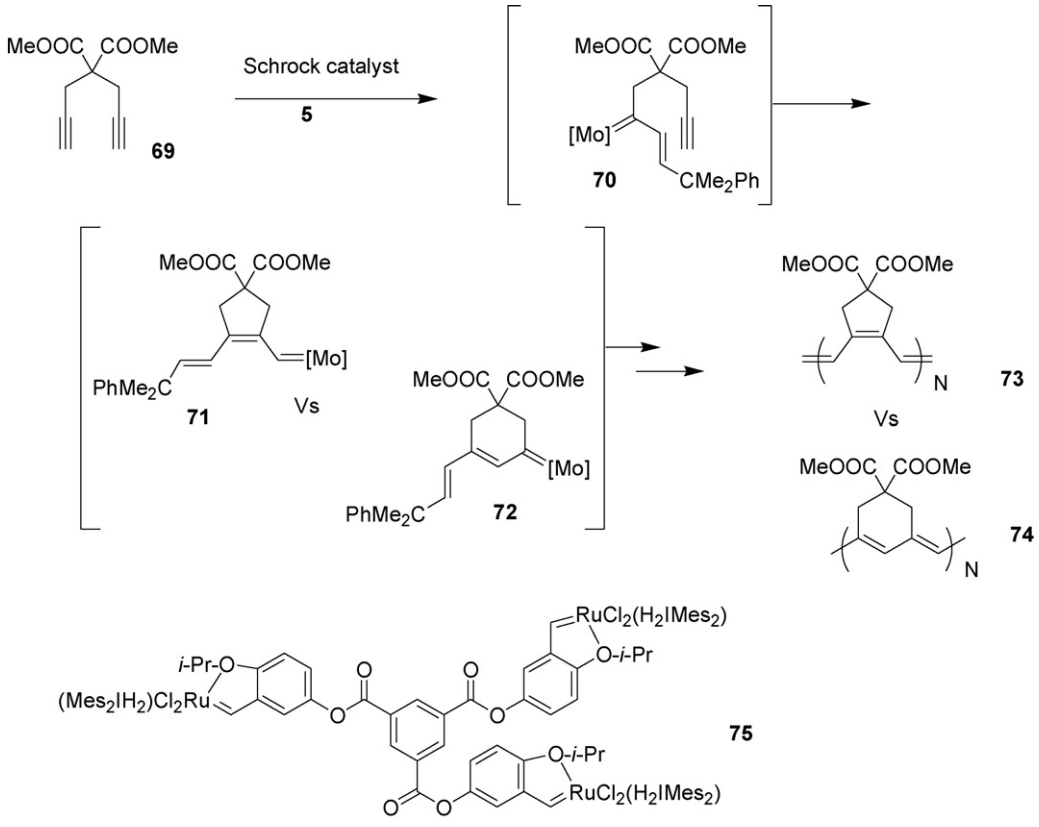


Fig. 4. Representative saubstrates for ADMET polymerization.

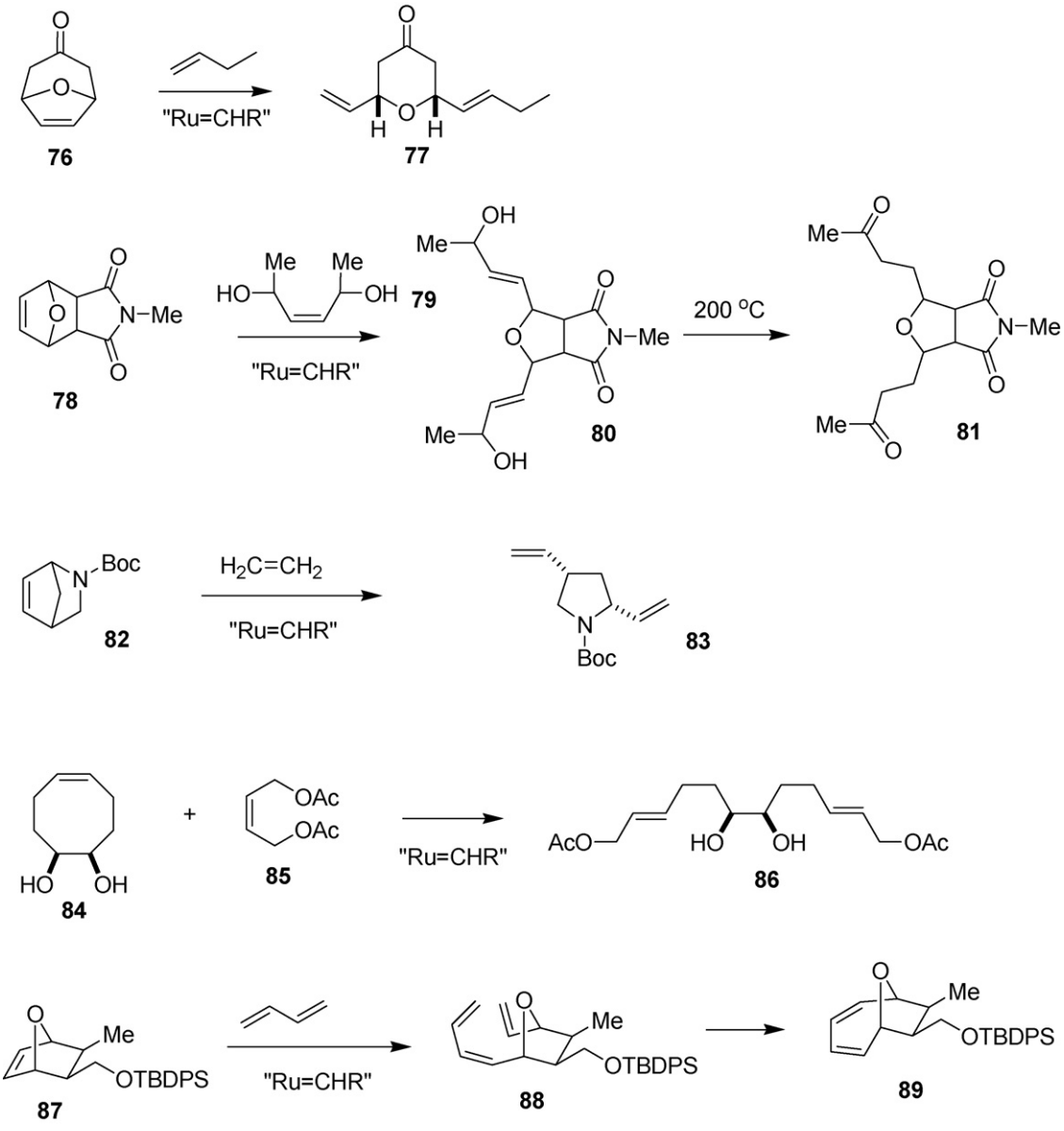


Scheme 8.

linked norbornenes and norbornenes linked to amino groups [215]; (3) copolymers from norbornenes linked to nitrile groups and norbornenes linked to chelating ligands [216]; and (4) copolymers of norbornenes linked to a biphenylcarboxylic acid group [217]. Preparation of a polymeric carbene complex (**58**) through anionic polymerization and stoichiometric metathesis, followed by ROMP with norbornene derivatives was reported [218]. Other ring systems that have been subjected to ROMP reactions include: (1) 3,3-dialkylcyclopropenes (*e.g.* **60**) [219]; (2) cyclobutenecarboxamides (*e.g.* **61**) [220]; (3) cyclobutenes linked to polystyrene chains [221]; (4) *p*-cyclophanes (*e.g.* **62**) [222]; (5) a macrocycle-bridged steroid derivative (**63**) [223]; and (6) carborane-substituted cyclooctenes [224]. The ROMP of norbornene using a bridging germylene–ditungsten complex and formation of the initial carbene complex from norbornene was discussed [225,226]. The ROMP reaction of norbornene was studied computationally, with emphasis on the chain transfer to ethylene, *trans*-1,2-difluoroethylene, and *trans*-1,4-dichloro-2-butene [227]. The rate of chain transfer was strongly correlated with the steric bulk of the alkene substituents. The use of ionic liquids for ROMP reactions was reported [228]. ROMP and ADMET polymerization reactions were reported in limonene, which results in limonene-end capped polymers [229]. Several patents were awarded for various aspects of carbene complex-initiated ROMP reactions [230–235].

Several examples using carbene complexes to initiate acyclic diene metathesis polymerization (ADMET, see Scheme 1) were reported. Monomers subjected to ADMET polymerization are depicted in Fig. 4 and include: (1) α,ω-dienes where the alkenes are linked through an ester group (*e.g.* **64**) [236]; (2) α,ω-dienes where the alkenes are linked though methylated tethers (*e.g.* **65**) [237]; (3) α,ω-dienes where the alkenes are linked through halogenated alkyl chains (*e.g.* **66**) [238]; (4) α,ω-dienes where the alkenes are linked through germanium or tin (*e.g.* **67**) [239]; and (5) various α,ω-dienes linked through anhydride, amide, or imide linkages, and/or ROMP of their RCM products formed at high dilution [240]. An NMR study of Schrock carbene-catalyzed ADMET of *p*-divinylbenzene derivative **68** was reported [241]. Analysis of ADMET polymerization processes using mass spectrometry was also reported [242]. The detrimental effect of allylic methyl groups on ADMET polymerization and cross metathesis was examined in detail by NMR [243]. The alkenes 3-methyl-1-pentene and 3,6,9-trimethylundeca-1,10-diene were employed as model substrates. When the Schrock carbene complex was employed, accumulation of metallacyclobutanes was noted. When either Grubbs catalyst I or II was employed, nonproductive metathesis was the predominant pathway, and the buildup of methylene–ruthenium complexes led to rapid decomposition of metathesis active species.

Polymerization of bis(alkynes) in an ADMET-like process was reported (Scheme 8). The process employing 4,4-bis(alkoxycarbonyl)-1,6-heptadiene derivatives (*e.g.* **69**) results in a polymer containing mostly five-membered rings but some six-membered rings [244]. Synthesis of a related star polymer using tris(carbene) complex **75** as the initiator was also reported [245].



Scheme 9.

2.2.3. Nonpolymer-forming ring opening metathesis reactions

Several examples of RO–CM (see Scheme 1) were reported in 2006. Representative examples are depicted in Scheme 9. Co-metathesis of various [4 + 3]-cycloadducts (*e.g.* **76**) and various gaseous alkenes was reported [246]. A RO–CM–alkene isomerization sequence was observed in the RO–CM of oxanorbornenes (*e.g.* **78**) and alkene–diols (*e.g.* **79**) followed by thermolysis [247]. Thermolysis converts the ruthenium byproducts into an alkene isomerization catalyst. Several examples of simple CM–alkene isomerization were also demonstrated. The RO–CM sequence was reported for 2-azanorbornenes (*e.g.* **82**) and ethylene [248]. The RO–CM of cyclooctenediol **84** and butene–diacetate **86** was a key step in a novel synthetic approach to acetogenins [249]. A ring expansion was observed in the RO–CM of oxanorbornene **87** and butadiene [250]. This process likely occurs through a RO–CM to afford **88** followed by ring-closing metathesis to afford **89**. This reaction was used in the total synthesis of (+)-1893B. The thermodynamics of RO–CM were studied computationally [251].

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 2006. Representative examples are depicted in Fig. 5. Regretfully it is very difficult to rationally organize these reactions. Specific pairs of compounds subjected to cross metathesis include: (1) eugenol and *cis*-2-buten-1,4-diol (an undergraduate laboratory experiment) [252]; (2) hexadienoate esters with allyl bromide [253]; (3) dissimilar styrenes for synthesis of unsymmetrical stilbene derivatives [254]; (4) cross metatheses involving oleate esters [255]; (5) 2-butene with natural triacylglycerides

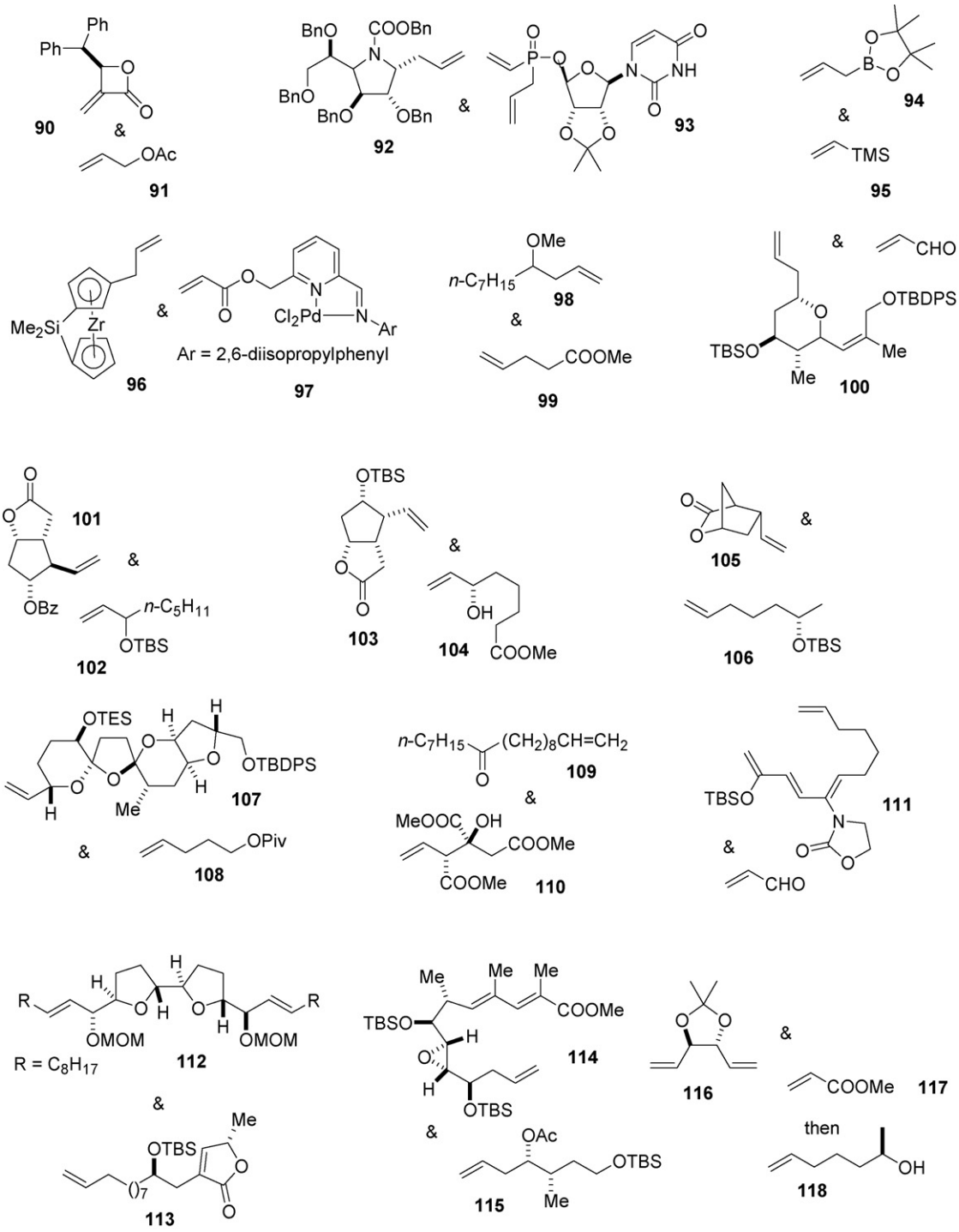


Fig. 5. Represent pairs of alkenes subjected to cross metathesis.

[256]; (6) allylic alcohol derivatives with methyl acrylate [257,258]; (7) allylicly functionalized derivatives [259]; (8) homoallylic alcohols with methyl acrylate [260]; (9) homoallylamine derivatives with monosubstituted alkenes [261,262]; (10) an *exo*-methylene-β-lactone and an allylic acetate (*e.g.* **90** and **91**) [263]; (11) solid phase CM of β-lactam-alkenes and various monosubstituted alkenes [264]; (12) an alkene remotely connected to a β-lactone with 1-nonene [265]; (13) methylenetetrahydrofurans with either acrolein or vinylboranes [266]; (14) alkenes connected to fluorinated imines with methyl acrylate [267]; (15) a vinyltetrahydrofuran derivative with a styrene derivative [268]; (16) *N*-allyl macrocyclic lactams and allyl ester derivative [269]; (17) an alkene nucleoside with an alkene amino acid [270]; (18) a bis(allylic ether) and ethyl acrylate [271]; (19) selective γ,δ-alkene metathesis using conjugated diene–esters and amides [272]; (20) a C-vinyl glycoside with allylic alcohol derivatives [273]; (21) a vinylphosphonate nucleoside (*e.g.* **93**) and a highly oxygenated pyrrolidine derivative (*e.g.* **92**)

[274]; (22) a hydrogen bonding associated alkene–pyrimidone and an alkene–naphthyridine [275,276]; (23) divinyl sulfones with various monosubstituted alkenes [277]; (24) cross metathesis reactions involving porphyrins that contain remote alkene groups [278]; (25) cyclodextrin-linked allylamine derivatives and monosubstituted alkenes containing a perfluoroalkyl group [279]; (26) vinylsilanes and *p*-chlorostyrene [280]; (27) divinylsilanes or divinylsiloxanes with monosubstituted alkenes [281]; (28) allylboron derivatives (*e.g.* **94**) and vinylsilanes (*e.g.* **95**) [282]; (29) allylcarboranes with various mono- and disubstituted alkenes [283]; (30) BH_3-complexed allylphosphines or vinylphosphines with various monosubstituted alkenes (includes several examples of RCM reactions for BH_3-complexed phosphines) [284]; (31) an allylated ansa zirconocene (**96**) with a palladium complexed iminopyridine derivative (**97**) [285]; (32) π-stacked aromatics featuring remote alkene side chains [286]; (33) cross metathesis of alkenes bound to a silicon surface with 1-octadecene [287]; (34) cross metathesis of alkenes bound to a gold surface with alkenes connected to ferrocene groups [288]; (35) selective double cross metathesis of an unsymmetrical diene and two different complex alkenes [289]; and (36) failure to cross metathesize styrene and vinylgermanes using Grubbs catalysts I and II [290]. Several publications appeared in 2006 where cross metathesis was employed in natural products synthesis. Examples of alkenes employed in CM for natural products synthesis include: (1) an allylic alcohol with phenyl vinyl ketone for total synthesis of dispongin A [291]; (2) an allylic alcohol derivative and a complex allylbicyclic compound for zaragozic acid total synthesis [292]; (3) an allylic ether and a terpenoid alkene for vitamin E synthesis [293,294]; (4) a homoallylic ether (**98**) with methyl 4-pentenoate (**99**) for lyngbic acid total synthesis [295]; (5) a homoallylic alcohol derivative and acrolein as an early event in the total synthesis of amphidinolide A [296]; (6) two different complex alkenes for the total synthesis and structural revision of amphidinolide W [297]; (7) a homoallylic alcohol derivative with a 2-vinylbenzoate derivative for apicularen A total synthesis [298]; (8) a homoallylic alcohol derivative and an alkene–tetrahydropyrone derivative for methyl monate C total synthesis [299]; (9) a homoallylic diol and methyl vinyl ketone for *endo* and *exo* brevicomin total synthesis [300]; (10) an allylic amine and an α,β-unsaturated ketone for hyacinthacine A_2 total synthesis [301]; (11) a homoallylamine with an α,β-unsaturated ketone for total synthesis of pinidinol [302]; (12) a triene and a complex allylic ether for losonolide A total synthesis [303]; (13) a complex alkene (**100**) with acrolein for preparation of lasonolide A segments [304]; (14) a bicyclic lactone (**101**) and an allylic alcohol derivative (**102**) for synthesis of prostaglandins [305] and a similar process involving the formation of carbacyclins [306]; (15) two complex alkenes (**103** and **104**) for synthesis of isoprostane derivatives [307]; (16) two structurally complex partners for the total synthesis of pyragonacin [308]; (17) a vinyl oxanorbornene derivative (**105**) and an alkenol derivative (**106**) for total synthesis of brefeldin A [309]; (18) acrylic acid with complex alkenes in two separate events for total synthesis of colletol [310]; (19) an allylic ester and ethyl 4-pentenoate for stemoamide total synthesis (a later step employs a lactone-forming RCM reaction) [311]; (20) undeca-1,10-dien-6-one with excess acrylonitrile (double CM) for histrionicotoxin total synthesis [312]; (21) a complex alkene (*e.g.* **107**) with a simple alkene–alcohol derivative (*e.g.* **108**) for azaspiracid A total synthesis and synthesis of stereoisomers of azaspiracid A [313–317]; (22) a complex alkene with various simple monosubstituted alkenes for the synthesis of seco loganin analogs [318]; (23) a complex symmetrical 1,2-disubstituted alkene and a monosubstituted alkene for sovaphen A total synthesis [319]; (24) a complex alkene with methyl acrylate for total synthesis of bistramide A [320]; (25) a complex alkene–diester (**110**) and a 10-undecenylketone derivative (**109**) for total synthesis of viridofungin [321]; (26) a complex alkene–pyrone system and electron-deficient alkenes for total synthesis of mitorubrinal and mitorubrinic acid [322]; (27) a complex triene (**111**) (reaction occurs at the monosubstituted alkene) and acrolein for total synthesis of galbulimima alkaloid 13 (product spontaneously undergoes intramolecular Diels–Alder reaction) [323]; (28) two complex alkenes for total synthesis of macrolide antibiotic A26771B [324]; (29) a complex C_2-symmetric bis(tetrahydrofuran) derivative (**112**) and another complex alkene (**113**) (1:1 metathesis) for total synthesis of asimicin [325]; (30) two different complex monosubstituted alkenes for synthesis of dictyostatin segments [326]; (31) two different complex alkenes (**114** and **115**) for total synthesis of FD-891 [327]; (32) 1-pentadecene with an allylic alcohol for sphingosine synthesis [328]; and (33) a complex allylic alcohol with various monosubstituted alkenes for synthesis of laulinamide analogs [329]. A one-pot tandem cross metathesis – rhodium-catalyzed carbene formation – carbonyl ylide cycloaddition process was reported [330]. A serial two-step double RCM reaction was reported using C-2 symmetric diene **116** and two different alkenes (**117** and **118**) for cladospolide C total synthesis [331]. A catalytic system that isomerizes alkanes via a tandem alkane metathesis – dehydrogenation – cross metathesis sequence was reported [332]. Several patents were awarded for the preparation of alkenes via cross metathesis [333–341].

Several examples of dimerization via metathesis (see Scheme 1) were reported in 2006. Compounds subjected to carbene complex-catalyzed metathesis dimerization are depicted in Fig. 6, and include: (1) allylic alcohol derivatives (*e.g.* **120**) [342]; (2) diene **121** (forms **122** with excision of cyclopentene) [343]; (3) allylglycines [344]; (4) *N*-acryloylamino acids (*e.g.* **123**) (also includes cross metatheses) [345]; (5) vinylglycine and allylglycine derivatives [346]; (6) styrene [347] or 1-octene [348] in an ionic liquid; (7) C-alkenyl glycosides (*e.g.* **124**) [349]; (8) a nucleoside derivative (**125**) followed by a macrocyclic RCM in a later synthetic event [350]; (9) alkenes connected to aromatic rings by seven CH_2 groups (includes several examples of RCM) [351]; (10) vinylphosphane oxides (*e.g.* **126**) (includes cross metathesis also) [352]; (11) alkene-linked cyclodextrins [353]; (12) *p*-vinylphenylferrocene (**127**) [354]; (13) ferrocene–alkenes [355]; and (14) a bis(ruthenium) complex featuring a bridging carboxylate ligand having remote alkene functionality (**128**) [356]. Metathesis dimerization and RCM were noted for several aromatic compounds where the alkene groups feature a non-*ortho* aromatic arrangement (*e.g.* **129**) [357]. More favorable tethers (*e.g.* as in **130**) undergo the

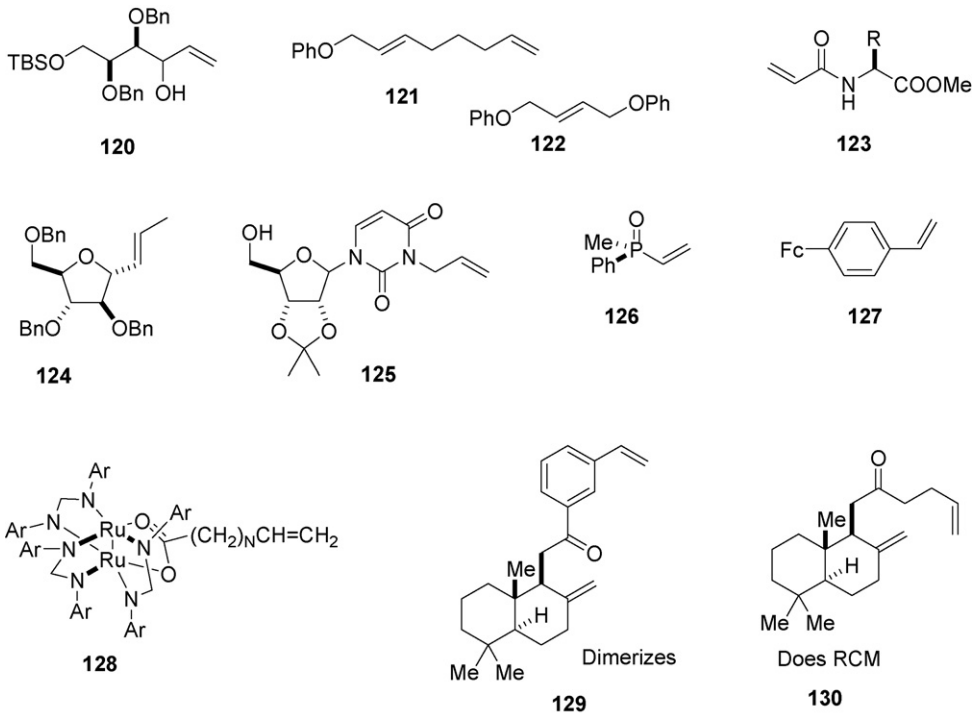


Fig. 6. Representative alkenes subjected to metathesis dimerization.

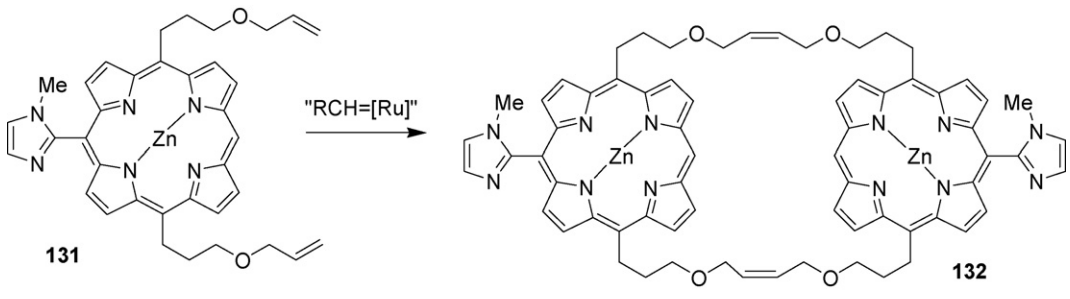
RCM reaction. Metathesis dimerization was used for the cross-linking of 10-undecenoyl-cellulose derivatives [358]. A failed attempt to cross metathesize a self-assembled pentenylguanosine derivative was noted [359].

Additional examples feature cross metathesis in tandem with some other metathesis mode. Examples are depicted in Scheme 10. Tandem dimerization-RCM was observed for bis(alkyne)-linked porphyrin derivatives featuring pendant vinyl groups (*e.g.* **131**) [360,361]. Tandem double CM and RCM were assisted by preorganization through self-assembly of the monomers around a template molecule, and resulted in a cyclic trimer [362].

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 2006, 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) simple five-membered rings [363]; (2) five-membered ring spiroketals [364]; (3) α-aminocyclopentenecarboxylates [365]; (4) cyclopentenes for total synthesis of herbertenediol [366,367], HM-3 total synthesis and structure revision [368], preclavulone A methyl ester [369], lagopodon [370], cuparenone [371], guanacastepene E [372], carbapentofuranoses [373], and ent-bacillariolide 11 [374]; (5) highly oxygenated cyclopentenes for total synthesis of homo-apioneplanocin A (**135**) [375], 6-epipentenomycin B (**136**) [376], carbocyclic nucleosides [377], and cundaramines [378]; (6) α,β-unsaturated cyclopentenones [379]; (7) α,β-unsaturated cyclopentenones for total synthesis of D-noviose [380] and partial synthesis of nitiol [381]; (8) the five-membered ring of the steroid ring system [382]; (9) bicyclo[3.3.0]octadienones (*e.g.* **137**) for merillactone A total synthesis [383]; (10) bicyclo[3.1.0]hexane derivatives for total



Scheme 10.

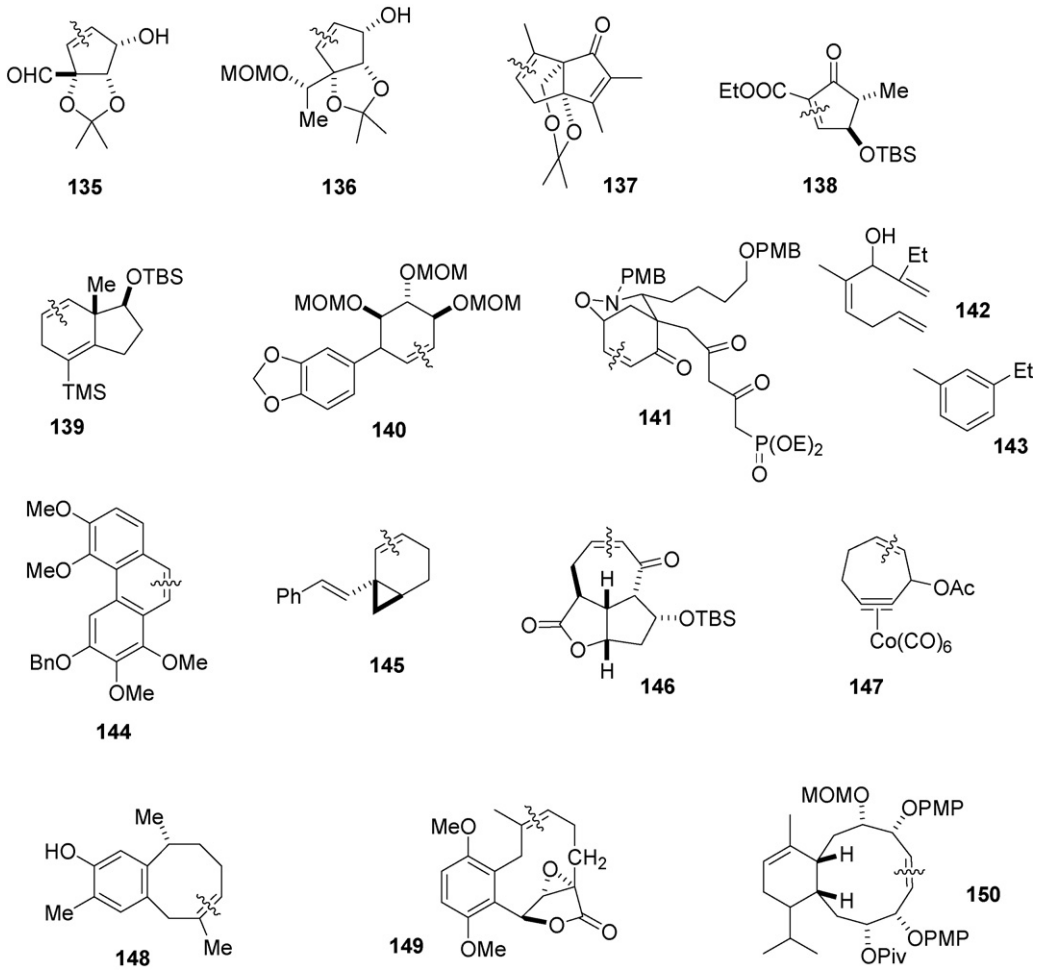
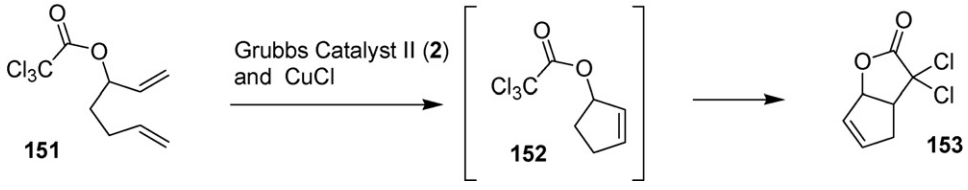


Fig. 7. Representative carbocycles produced through an RCM reaction (bond constructed through RCM indicated).

synthesis of L–N–MCd4T [384]; (11) highly electron deficient cyclopentenone systems (*e.g.* **138**) and heterocyclic analogs [385]; (12) indenes and indenones [386]; (13) a cyclopentene ring in the process of cleaving a chiral auxiliary [387]; (14) five- to eight-membered rings fused to cyclohexadiene rings [388]; (15) five- to eight-membered rings spiro fused to dihydro-3-pyrone rings [389]; (16) a cyclohexene ring for bisabolol total synthesis [390]; (17) cyclohexene rings spiro fused to pyrrolidine rings [391], oxygenated cyclohexenone rings [392], and tetrahydrofurans [393]; (18) a cyclohexene ring spiro fused to a tetrahydropyridone ring for quinic acid total synthesis [394]; (19) a cyclohexene ring spiro fused to a pyrrolidine ring for lepadiformine total synthesis [395]; (20) cyclohexene rings fused to cyclobutanes for merillactone A total synthesis [396]; (21) cyclohexadienes fused to five-membered rings (*e.g.* **139**) [397]; (22) a hexahydronaphthalene ring for total synthesis and structural revision of trihydroxycadinane [398] and for total synthesis of branimycin [399]; (23) cyclohexenol derivatives spiro fused to the bicyclo[2.2.2.]octane ring system [400] and to cyclohexane rings [401]; (24) highly oxygenated cyclohexene rings for total synthesis of 1-*epi*-MK7607 [402], simplified conditurols (**140**) [403], and pancratistatin [404]; (25) diaminocyclohexenes [405]; (26) a densely functionalized cyclohexane ring for total synthesis of GS4104 [406]; (27) an α,β-unsaturated six-membered ring ketone (**141**) for total synthesis of phyllantidine [407]; (28) carbocycle-bridged nucleoside derivatives [408]; (29) six-membered ring fluorinated cyclic amino acid derivatives [409]; (30) fusion of a cyclohexene ring onto a porphyrin ring [410]; (31) benzene rings (*e.g.* **143**) directly through RCM of trienols (*e.g.* **142**) [411]; (32)



Scheme 11.

phenanthrene ring **144** for hasubanonine total synthesis [412]; (33) benzene rings for the construction of helicenes [413]; (34) diastereoselective formation of bicyclo[4.1.0]heptene derivatives (*e.g.* **145**) [414]; (35) various fused bicyclic carbocycles and total synthesis of periplanone C [415]; (36) six-membered rings fused to propellanes [416]; (37) six- to seven-membered ring fluorinated cyclohexenes fused to a uracil ring [417]; (38) six- to eight-membered rings fused to five-membered ring lactones [418]; (39) a cycloheptene ring for total synthesis of cananodine [419]; (40) a seven-membered ring fused tricyclic system (**146**) for preparation of the rameswaralide core [420]; (41) a cycloheptene ring fused to a cyclopentene ring [421]; (42) a cycloheptene ring of a pentacyclic system [422]; (43) cobalt-cycloheptyne complexes (*e.g.* **147**) [423]; (44) seven- to eight-membered ring α-thioketone derivatives [424]; (45) seven- to eleven-membered ring carbocycles and heterocycles that feature chiral sulfonimine groups [425]; (46) eight-membered ring carbocyclic ring systems [426]; (47) eight-membered rings fused to a benzene ring (*e.g.* **148**) for parvifolene total synthesis [427]; (48) eight-membered rings fused to cyclohexane rings [428]; (49) a lactone-bridged ten-membered ring (**149**) for ent-clavilactone total synthesis [429]; and (50) a ten-membered ring (**150**) for total synthesis of eleutherobin [430]. One-pot tandem RCM–Kharasch reactions were observed in the reaction of trichloroacetate ester–diene **151** (Scheme 11) with ruthenium carbene complexes and CuCl [431]. Similar reactions using only ruthenium metathesis catalysts and/or catalyst combinations were also reported [432]. Patents were awarded for carbocycles prepared through RCM [433,434].

Numerous examples of the formation of nitrogen heterocycles using the RCM reaction (Fig. 8) were reported in 2006, including: (1) dihydropyrroles [435]; (2) dihydropyrroles followed by further conversion to pyrroles when the RCM reaction was conducted at 150 °C [436]; (3) pyrrole derivatives (*e.g.* **156**) from diene precursors (*e.g.* **155**) by performing the reaction in the presence of benzoquinone oxidants [437]; (4) dihydropyrroles for total synthesis of hydroxypipecolic acid [438], hygrine [439], broussonetine (**157**) [440], and the attempted total synthesis of uniflorine B [441]; (5) the five-membered ring of the indolizidine skeleton [442]; (6) pyrrolidinium salts (*e.g.* **158**) [443]; (7) fluorinated α,β-unsaturated five-membered ring lactams [444]; (8) five- and six-membered ring cyclic amines [445]; (9) cyclic five- and six-membered ring β-amino acid derivatives [446]; (10) five- through nine-membered ring nitrogen heterocycles [447]; (11) five- to six-membered ring *N*-amino cyclic amines (*e.g.* **159**) [448]; (12) a tetrahydropyridone (**160**) for total synthesis of deoxygulonojirimycin [449], (13) a combinatorial mixture of five- to six-membered ring nitrogen heterocycles formed through either RCM or intramolecular enyne metathesis [450]; (14) tetrahydropyridines for total synthesis of pipecolic acid [451] and sedamine [452]; (15) tetrahydropyridines fused to dihydropyridone rings [453]; (16) tetrahydropyridines fused to

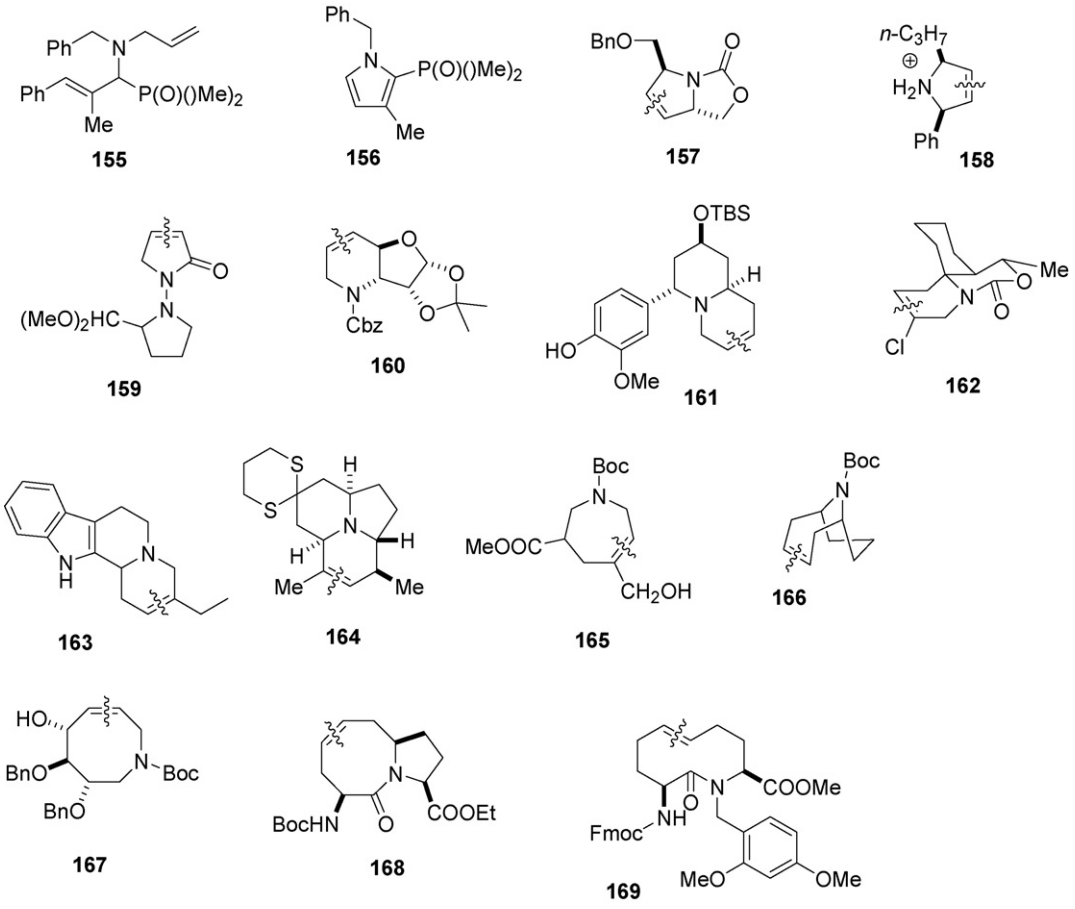


Fig. 8. Representative *N*-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).

a pyrrolidine ring for total synthesis of deoxynupharidine [454]; (17) tetrahydropyridines followed by osmium-catalyzed dihydroxylation in the same reaction pot as the RCM step [455]; (18) a tetrahydropyridine fused to a piperidine ring (**161**) for total synthesis of lasubine I [456]; (19) fluorinated tetrahydropyridine derivatives [457]; (20) tetrahydropyridine–chloroalkenes fused to a bicyclic ring (*e.g.* **162**) for preparation of cylindricine B segments [458]; (21) a failed attempt to form a six-membered ring amine derivative when one of the alkenes is chlorinated [459]; (22) tetrahydropyridines fused to oxazolone rings [460]; (23) α,β-unsaturated six-membered ring lactams [461,462]; (24) a six-membered ring amine fused to a carboline ring (**163**) for mitralactonine 1 total synthesis [463]; (25) a six-membered ring heterocycle for synthetic approaches to swainsonine derivatives [464–467]; (26) two six-membered ring amines for total synthesis of isosparteine [468]; (27) a tricyclic amine (**164**) for total synthesis of indolizidine 223AB [469]; (28) an azabicyclo[2.2.2]octene derivative for catharanthine total synthesis [470]; (29) six- to seven-membered ring cyclic amines [471]; (30) six- to seven-membered ring cyclic amines spiro fused to various *N*-heterocycles [472]; (31) six- to seven-membered ring cyclic amino acid derivatives [473]; (32) six- to seven-membered rings spiro-fused to an oxindole ring [474]; (33) six- to eight-membered ring cyclic enamines fused to benzene rings (the cyclization precursor was prepared through Petassis olefination) [475]; (34) six- to eight-membered ring lactams–enamides (through tandem RCM and alkene isomerization) [476]; (35) six- to nine-membered ring lactams [477]; (36) seven-membered ring cyclic amines (*e.g.* **165**) [478–481]; (37) seven-membered ring amine–ketones [482]; (38) a seven-membered ring fused to a lactam ring for stemoamide total synthesis [483]; (39) seven-membered ring lactams [484]; (40) seven-membered ring α,β-unsaturated lactams [485]; (41) 10-azabicyclo[4.3.1]decanes (*e.g.* **166**) and 9-azabicyclo[4.2.1]nonanes [486,487]; (42) one-pot seven-membered ring formation and double bond oxidation [488]; (43) seven- to eight-membered rings fused to oxazolone rings [489]; (44) eight-membered ring cyclic amine derivatives (*e.g.* **167**) [490]; (45) eight-membered ring lactams [491]; (46) eight-membered ring lactams fused to proline (*e.g.* **168**) [492]; and (47) nine- and ten-membered ring lactams (*e.g.* **169**) [493]. Simultaneous RCM and cleavage of alkene-bound chiral auxiliaries was also reported for the synthesis of various *N*, *O*, and *S* heterocycles and carbocycles [494].

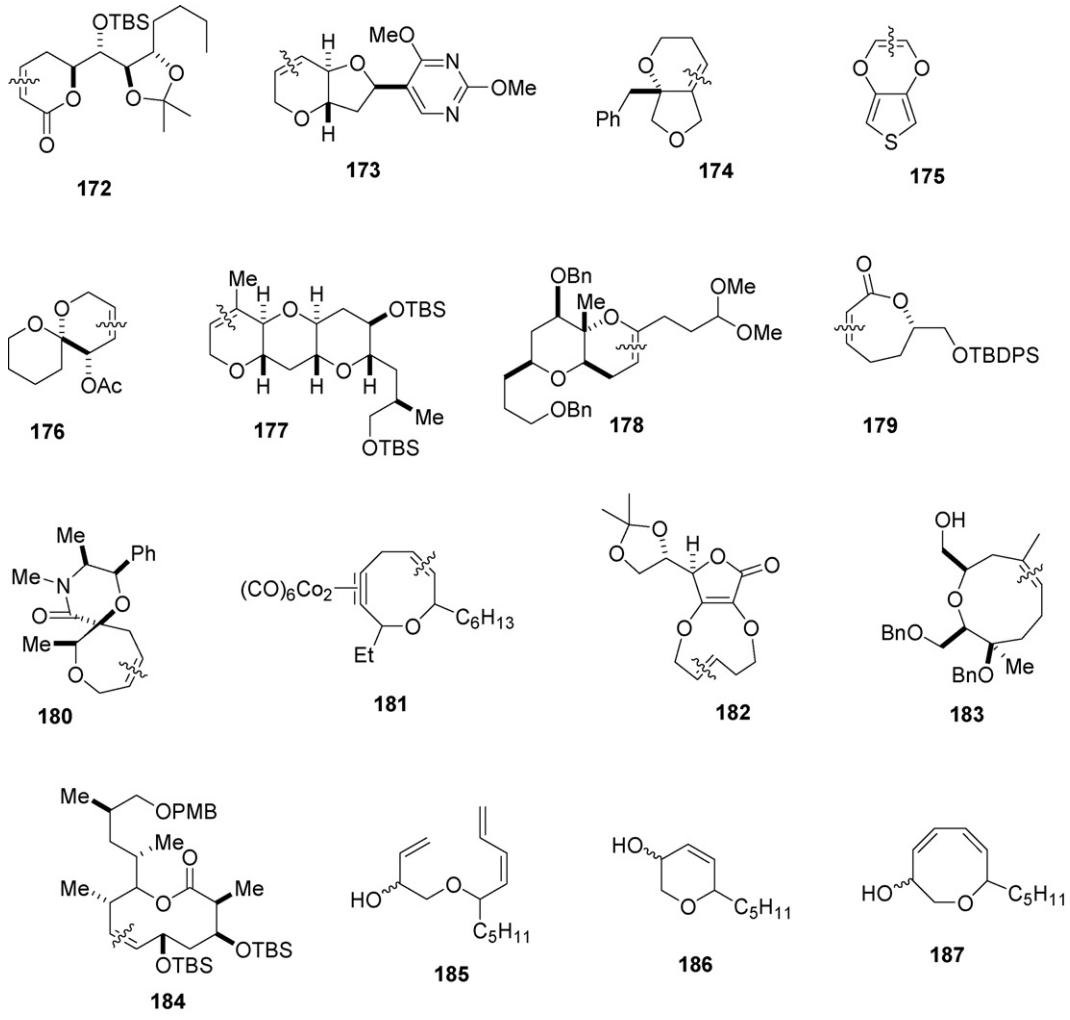
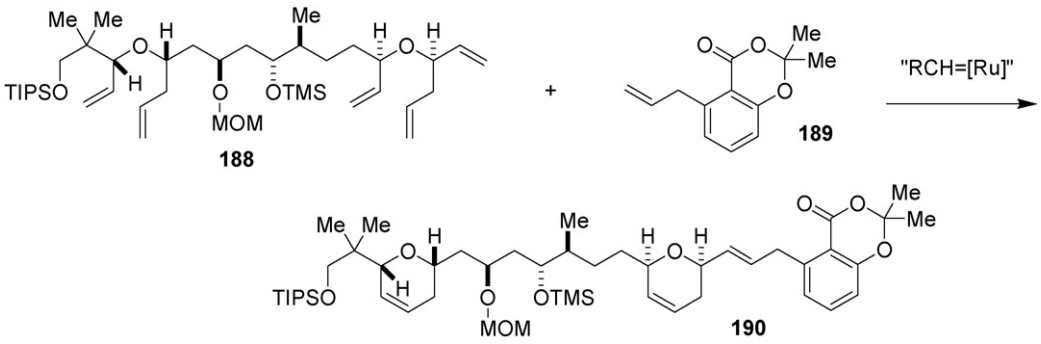


Fig. 9. Representative oxygen-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).



Scheme 12.

Many diverse oxygen heterocycles were synthesized via the RCM reaction in 2006 (Fig. 9), including: (1) five- and six-membered ring ethers for total synthesis of mucocin [495]; (2) five- and six-membered rings from a triene and control of the ring size selectivity through alkene substitution pattern [496]; (3) five- to seven-membered ring oxygen heterocycles [497]; (4) five- to seven-membered ring *O*-heterocycles and carbocycles fused to a benzene ring [498]; (5) six-membered ring allylic ethers [499–501]; (6) a six-membered ring ether for total synthesis of triacetoxygoniotriol [502] and 11-desoxylaulinamide [503]; (7) six-membered ring α,β-unsaturated lactones for total synthesis of boronolide (**172**) [504–506], passifloricin and analogs [507,508], fostriecin [509], fragments of rhizoxin D [510], FR 901464TS (the synthesis also employs a CM reaction in a separate event) [511], obolactone [512], diospongin A and B [513], cryptocarya diacetate [514], apratoxin A [515], migrastatin (the synthesis features two other metathesis events) [516], and goniothalamin epoxides [517]; (8) six-membered ring α,β-unsaturated lactones via solid phase RCM [518]; (9) a six-membered ring β,γ-unsaturated lactone fused to a vitamin D-like structure [519]; (10) a six-membered ring alkenyl ether for DAH and KDO total synthesis [520]; (11) a six-membered ring ether fused to a tetrahydrofuran (**173**) for malayamycin total synthesis [521]; (12) six-membered rings fused to carbohydrate rings [522]; (13) six-membered ring enol ethers formed enantioselectively using a chiral molybdenum carbene complex catalyst [523]; (14) furo[3,4-c]pyrans (*e.g.* **174**) [524]; (15) dioxane rings 3,4-fused to furan rings (*e.g.* **175**) [525]; (16) six-membered ring spiroketals (*e.g.* **176**) [526]; (17) coumarins [527]; (18) tandem six-membered ring ether formation and transfer hydrogenation [528]; (19) the six- to nine-membered ring ethers present in brevitoxin and related compounds (*e.g.* **177**, **178**) [529–536]; (20) seven-membered ring oxygen heterocycles featuring fluorinated side chains [537]; (21) seven-membered ring α,β-unsaturated lactones (*e.g.* **179**) for synthesis of kumausynes [538]; (22) a seven-membered ring ether for methyllycaconitine total synthesis [539]; (23) seven- to eight-membered ring ethers fused to benzene rings [540,541], pyridine rings [542], and carbazole rings [543]; (24) seven- to nine-membered ring cyclic ethers fused to oxalactam rings (*e.g.* **180**) [544]; (25) an eight-membered ring cyclic ether containing a cobalt–alkyne complex unit (**181**) for lauthisan total synthesis [545]; (26) eight- to nine-membered ring bridged vitamin C analogs (*e.g.* **182**) [546]; (27) nine-membered ring ethers for total synthesis of ophirin B and astrogorgin (*e.g.* **183**) [547]; (28) a nine-membered ring *m*-cyclophane–diether via RCM on a self-assembled nucleoside derivative [548]; and (29) ten-membered ring lactones for total synthesis of dictyostatin (**184**) [549] and herbarumin III [550]. The RCM of triene derivatives (*e.g.* **185**) led to predominantly the smaller ring compounds (*e.g.* **186**) and not the larger ring dienes (*e.g.* **187**) [551]. A simultaneous double RCM/CM sequence was employed in the total synthesis of SCH 351448 [552]. This process involves the coupling of penta-ene **188** (Scheme 12) and allylbenzene derivative **189** [552]. Patents were awarded for oxygen heterocycles prepared via RCM reactions [553,554].

Heterocyclic compounds involving elements other than *N* and *O* were also constructed via the RCM reaction (Fig. 10). Examples include: (1) six- and seven-membered ring silacycles and carbocycles (*e.g.* **191**) [555]; (2) cyclic siloxanes (*e.g.* **192**) for total synthesis of streptazolin [556]; (3) a seven-membered ring cyclic siloxane for total synthesis of monocerin [557]; (4) nine-membered ring cyclic siloxanes for preparation of epothilone analogs (*e.g.* **193**) (also features metathesis dimerization processes) [558]; (5) cyclic phosphate esters fused to ribose rings (*e.g.* **194**) [559]; (6) cyclic enol phosphonamidates (*e.g.* **195**) [560]; (7) six-membered ring cyclic phosphates [561]; (8) bicyclic phosphate esters (*e.g.* **196**) (also includes examples of cross metathesis) [562]; (9) seven-membered ring cyclic phosphates [563]; (10) five- to eight-membered ring cyclic sulfonates (*e.g.* **197**) for synthesis of the proposed structure of mycothiazole [564]; (11) cyclic sultams (*e.g.* **198**) [565,566]; (12) ansa nickelocenes (*e.g.* **199**) [567]; and (12) bridged enantiomerically pure ferrocenes (*e.g.* **200**) through kinetic resolution using a chiral molybdenum carbene complex catalyst [568]. A tandem RCM–CM sequence was employed as the key step in the total synthesis of gigantecin (Scheme 13) [569]. Success in this reaction requires the addition of a fourfold excess of the cross metathesis partner **202** since undesired macrocycle-forming RCM reaction involving the asterisked alkene of **201** are predominant when lesser amounts of metathesis partner are employed.

Numerous macrocyclic compounds (rings with ≥11 atoms) were synthesized using the RCM reaction in 2006 (Fig. 11), including: (1) a macrocyclic conjugated diene (**205**) for preparation of a borrelidin fragment [570]; (2) a macrocyclic α,β-unsaturated ketone (**206**) for abyssomicin C total synthesis [571]; (3) a macrocyclic bis(ketone) (**207**) for total

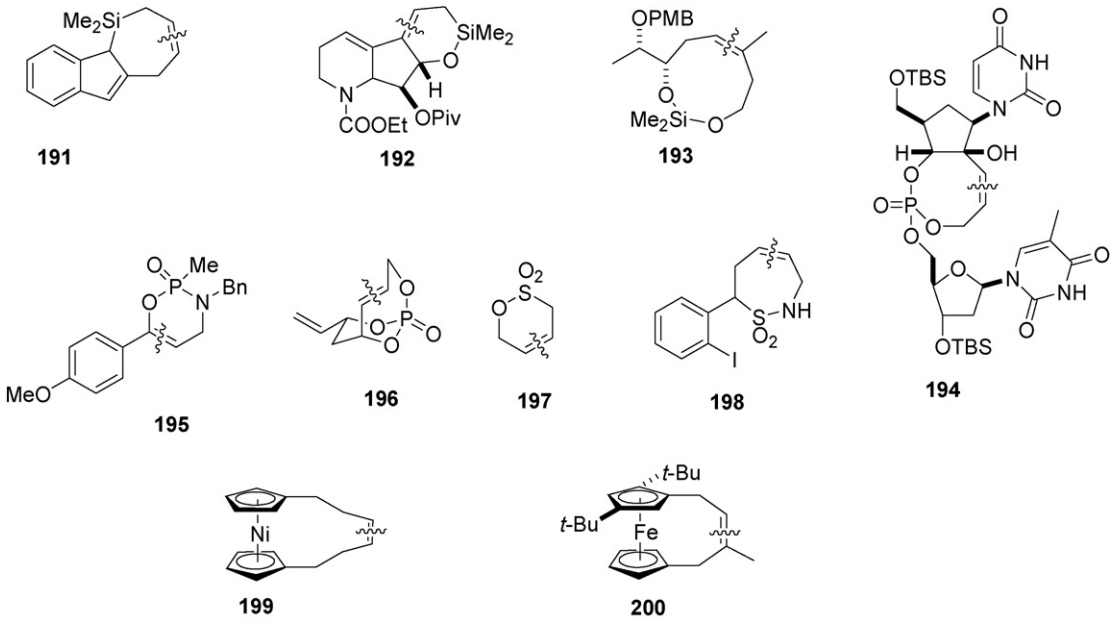
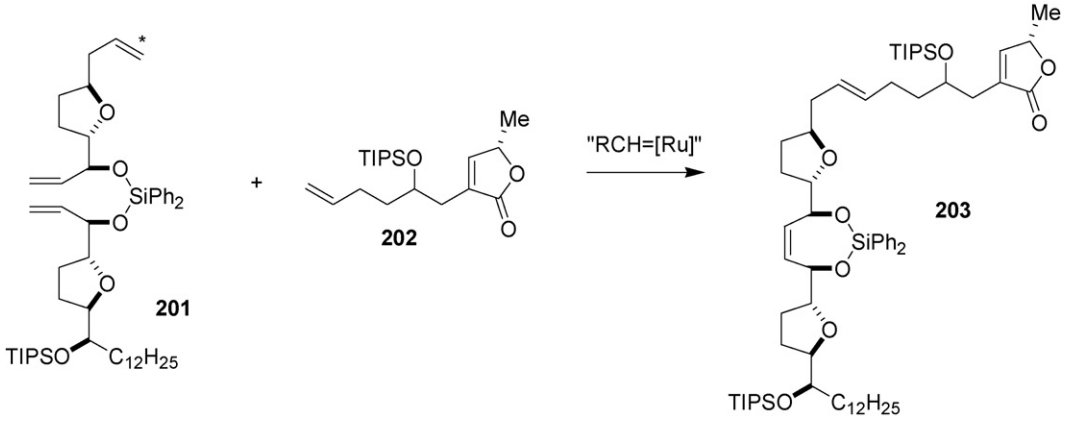


Fig. 10. Representative examples of other heterocycles prepared via the RCM reaction (bond constructed through RCM indicated).

synthesis of 15-acetyl-3-propionyl-17-norcharaciol total synthesis [572]; (4) macrocyclic amines (*e.g.* **208**) for total synthesis of saranin A [573,574]; (5) a macrocyclic ether for kendomycin total synthesis (synthesis also uses a Petassis reaction) [575]; (6) a macrocyclic lactone for preparation of the core of migrastatin [576]; (7) a macrocyclic lactone for mycolactone core synthesis [577], (8) macrocyclic lactones for total synthesis for aigialomycin [578], amphidinolide E (**209**) [579], sporiolide [580], tonantzitlone [581], and sporiolode B [582]; (9) macrocyclic lactones for preparation of epothilone analogs (*e.g.* **210**) [583,584]; (10) a macrocyclic keto-lactone for pochonins total synthesis [585]; (11) a macrocyclic polyene–lactone for total synthesis of lejimalide B [586]; (12) macrocyclic lactone–tetraenes [587]; (13) macrocyclic rings related to bryostatin (*e.g.* **211**) [588]; (13) macrocyclic bis(lactones) bridging a bicyclo[3.3.1]nonane ring system [589]; (14) a macrocyclic lactone diether for phorboxazole A total synthesis [590]; (15) a macrocyclic lactone–lactam for spongidepsin total synthesis [591]; (16) macrocyclic bis(lactone)–lactams for total synthesis of cryptophycin analogs [592]; (17) macrocyclic lactone–lactams [593]; (18) macrocyclic lactams [594]; (19) a macrocyclic bis(lactam) fused to an indole derivative (**212**) [595]; (20) a macrocyclic bis(lactam) (**213**) for BILN 2061 total synthesis [596,597] and mechanistic/model studies of the RCM event [598]; (21) a macrocyclic lactam/*p*-cyclophane (*e.g.* **214**) for preparation of the central core of macrocidins [599]; (22) the macrocyclic lactam/ether of radicicol [600]; (23) macrocycle-bridged bis(urea) derivatives [601]; (24) macrocycle-bridged bis(sulfonamide) derivatives [602]; (25) macrocyclic polyethers or polyamides [603–605]; (26) a macrocycle-bridged pyran derivative (**215**) for dactylolide total synthesis [606]; (27) macrocycle-bridged peptide derivatives [607–610]; (28) macrocyclic siloxanes via RCM or intramolecular enyne metathesis (also includes examples of cross metathesis) [611]; (29) a *m*-cyclophane-lactam [612] and failure to form a *m*-cyclophane lactone [613]; (30) a *m*-cyclophane for floresolide B total synthesis (including an alternative approach involving metathesis dimerization followed



Scheme 13.

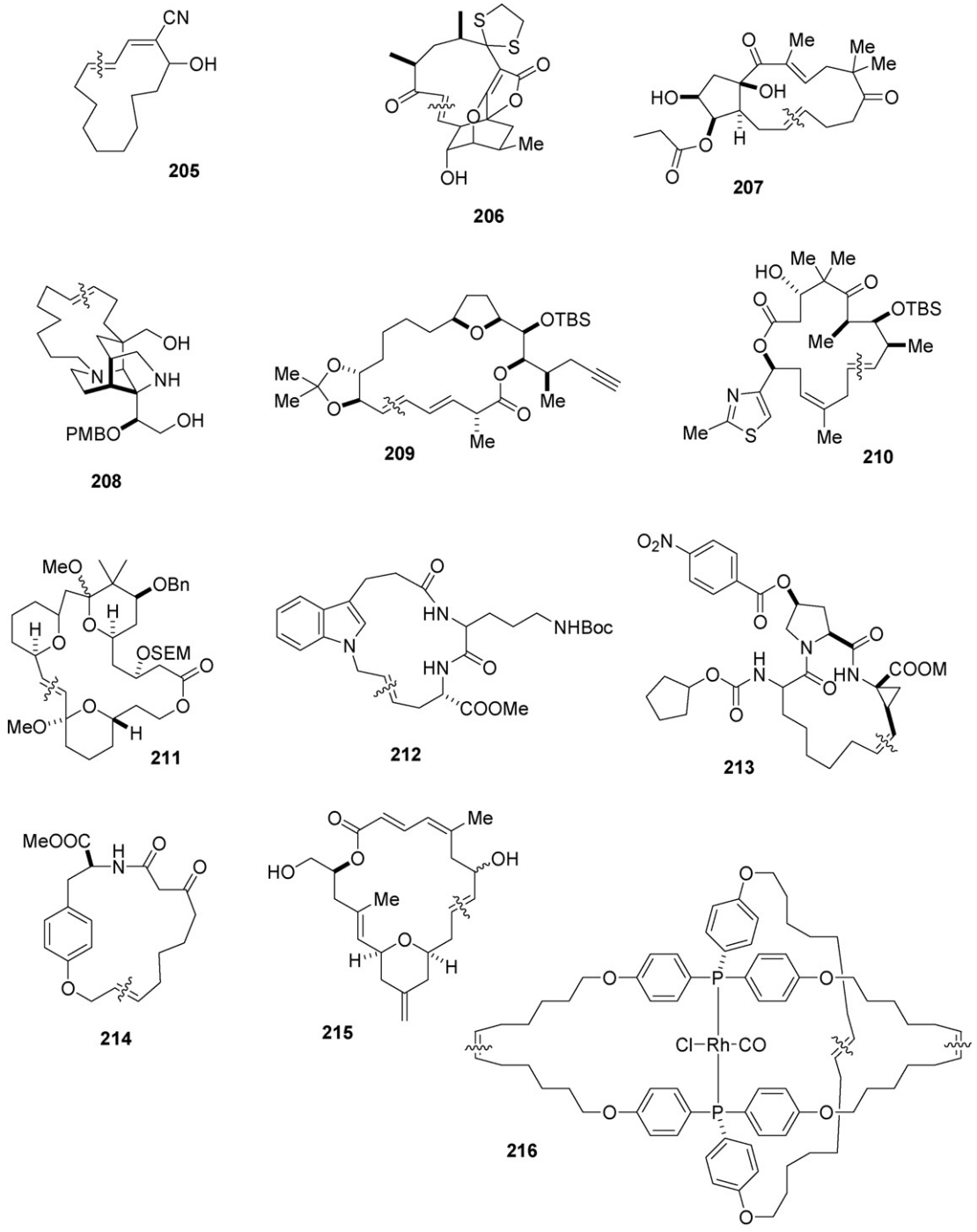
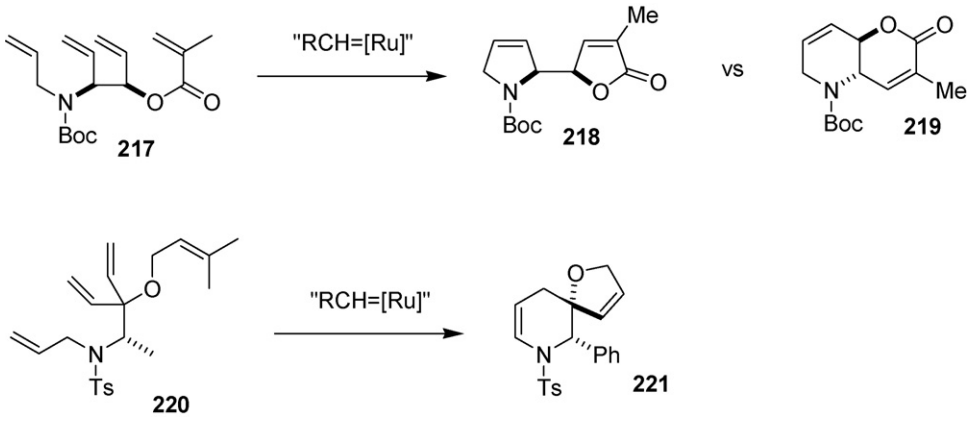


Fig. 11. Representative macrocycles (ring size >10) prepared using the RCM reaction (bond constructed through RCM indicated).

by RCM) [614]; (31) a bis(*p*-cyclophane)-bridged *m*-cyclophane [615]; (32) *p*-cyclophanes in a system where ring closure is aided by π-stacking interactions [616]; (33) *p*-cyclophanes where the rings are further linked through several conjugated alkene and alkyne units [617]; (34) macrocycle-bridged disaccharides [618]; (35) ligand-bridged rhodium bis(phosphine) complexes (*e.g.* **216**) [619,620], palladium bis(phosphine) complexes [621], platinum bis(phosphine) complexes [622], and butadiyne-bridged diplatinum complexes that are also linked through the phosphorus ligands [623]; (36) pyridine analogs of *m*-cyclophanes in systems where the pyridine is complexed to either palladium or platinum [624]; (37) macrocycle-bridged porphyrins [625]; (38) macrocycle-bridged tetraporphyrins [626]; (39) rotaxanes and calixarenes [627]; (40) catenanes [628]; (41) bridged cyclodextrins [629]; and (42) carbon chain-bridged dinucleotides [630]. A patent was awarded for synthesis of macrocyclic compounds via RCM [631].

Several examples using tetraenes to effect selective double RCM reactions were reported in 2006 (see Scheme 14). Reaction of tetraene **217** with the Hoveyda–Grubbs catalyst (**4**) led



Scheme 14.

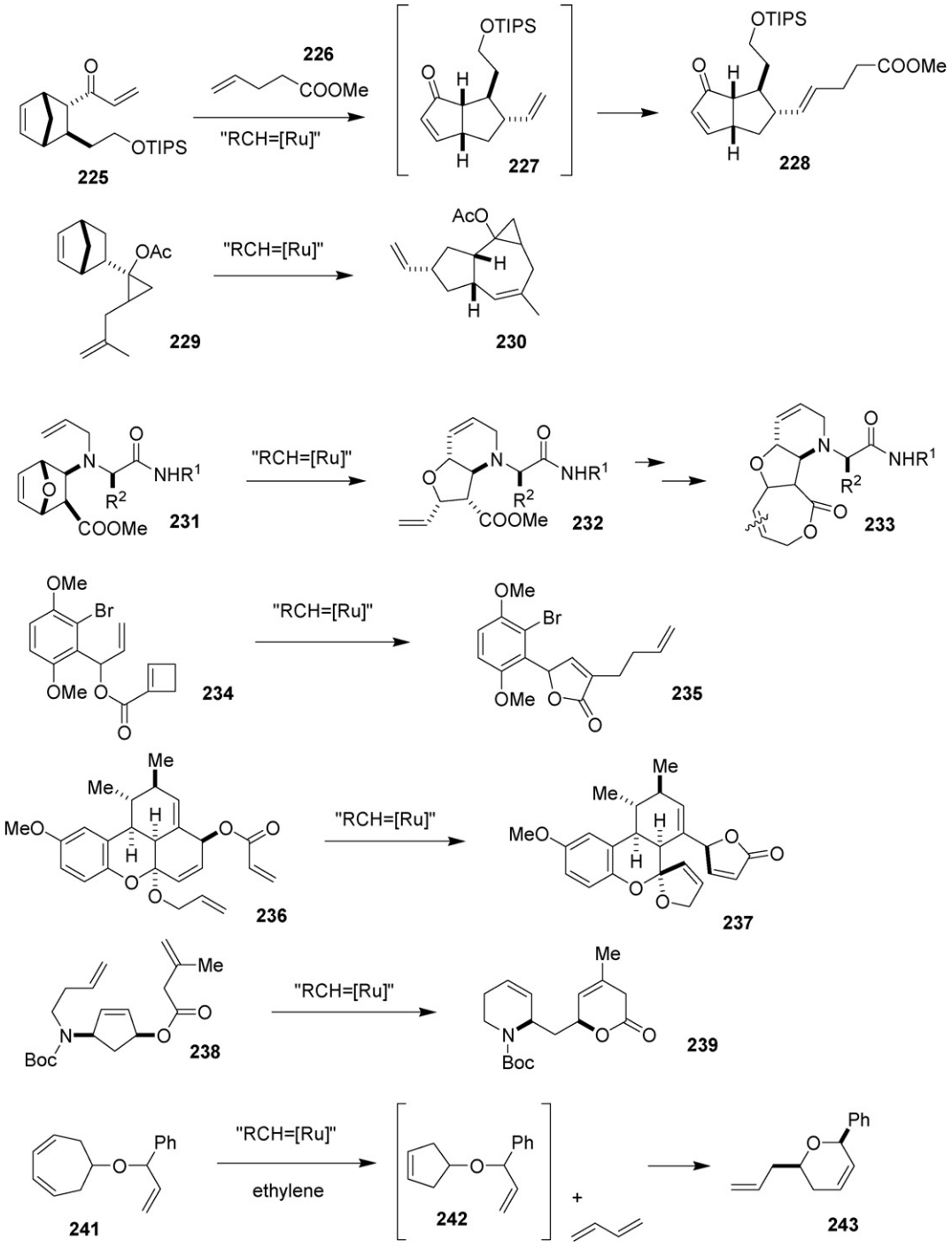
to predominantly the dumbbell shaped molecule **218** and not the fused ring compound **219** [632]. The reaction mixture was accompanied by monocyclic compounds. Compound **218** was an intermediate in the total synthesis of pandamarilactone A. Diastereoselective double RCM was reported in the synthesis of spirocycles (*e.g.* **221**) from acyclic tetraenes featuring diastereotopic vinyl groups (*e.g.* **220**) [633].

Several examples of ring rearrangement metathesis (RRM) were reported in 2006 (see Scheme 15). These examples include: (1) formation of a bicyclo[3.3.0]octenone derivative (**228**) for preparation of the core of geodin A through a tandem RRM–CM sequence [634] and use of a similar reaction sequence for the total synthesis of cylindramine A [635]; (2) formation of cyclopropane-fused seven- and eight-membered rings (*e.g.* **230**) through RRM of cyclopropylnorbornenes (also includes simple RCM processes) [636]; (3) conversion of azanorbornenes to hexahydroindole derivatives [637]; (4) the conversion of oxanorbornene derivative **231** to the fused bicyclic compound **232** (subsequent RCM and intramolecular enyne metatheses were also reported) [638]; (5) conversion of an oxanorbornene to a dihydrofuranopyrone derivative [639]; (6) a related conversion of *N*-allylaminonorbornene derivatives to analogous compounds fused to a cyclopentene ring [640]; (7) conversion of an allylic dihydrofuran to a dihydropyran for total synthesis of centrolobine (a later step of the synthesis employs cross metathesis) [641]; (8) conversion of ester-cyclobutene **234** to the corresponding lactone **235** and unanticipated formation of a naphthalene ring in a failed attempt to form a macrocyclic ring [642]; (9) conversion of triene **236** to diene **237** and ethylene in a tandem RRM–RCM process [643] and a related process for the preparation of **239**, an intermediate in the total synthesis of dumetorine [644]; and (10) a tandem butadiene excision/ring contraction and diastereoselective RRM for the conversion symmetrical cycloheptadiene derivatives (*e.g.* **241**) to allyldihydropyrans (*e.g.* **243**) and butadiene [645].

2.2.6. Alkene metathesis involving alkyne components

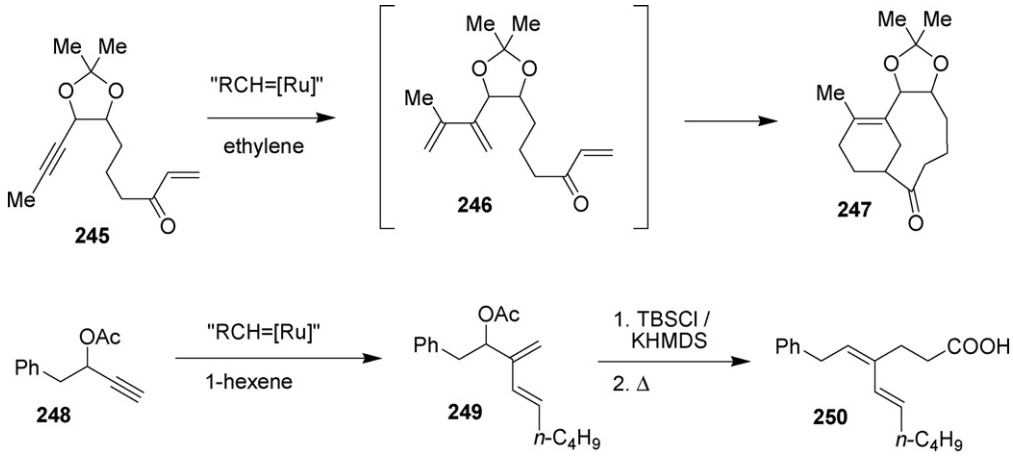
Several examples of the synthesis of conjugated dienes through the intermolecular (enyne CM) or intramolecular (enyne RCM) metathesis of enynes (see Scheme 1) using carbene complexes were reported in 2006. Examples of intermolecular enyne metathesis are depicted in Scheme 16 and include: (1) formation of the taxane ring system (**247**) in a process involving intramolecular enyne metathesis of alkyne **245** with ethylene followed by intramolecular Diels–Alder reaction [646]; (2) enyne metathesis of propargylic esters (*e.g.* **248**) and monosubstituted alkenes (the products then undergo the Claisen rearrangement) [647]; and (3) enyne metathesis of vitamin D alkynones with ethylene [648]. Failure of an allene-yne to undergo enyne RCM using Grubbs catalyst I was reported [649], however the process was successfully catalyzed by $GaCl_3$. Examples of intramolecular enyne metathesis are depicted in Fig. 12 and include: (1) formation of dihydropyrroles for diversity-oriented synthesis [650]; (2) formation of 2-vinyl dihydropyrrole derivatives (*e.g.* **252**) using ynamines (*e.g.* **251**) as starting materials [651]; (3) formation of spirocyclic cyclopentenes through either enyne metathesis or RCM [652,653]; (4) formation of vinylcycloheptene **254** from enyne **253** for micrandilactone synthesis [654]; (5) formation of seven-membered ring ethers fused to pyridine rings [655]; (6) formation of seven-membered ring oxygen heterocycles fused to pyrone rings [656]; (7) formation of bicyclic amine derivative **256** from enyne **255** for anatoxin A total synthesis [657]; (8) a ring opening intramolecular enyne metathesis for the preparation of cyclopentene-fused tetrahydropyrroles (*e.g.* **258**) using norbornene **257** and ethylene [658]; (9) ring opening enyne metathesis for the preparation of ring-fused pyrrolidinones (*e.g.* **260**, **261**) from alkyne–azanorbornenes (*e.g.* **259**) and the effect of alkyne substituents on the efficiency and regiochemistry of the metathesis event [659]; (10) a related ring rearrangement enyne metathesis using simple *N*-propargyl-aminocyclopentenes [660]; and (11) enyne metathesis involving pyridinium salts (*e.g.* **262**, **263**) [661]. Synthetic and mechanistic studies were reported for enyne metathesis reactions [662], and criteria correlating steric effects and catalyst requirements were developed.

Tandem intramolecular enyne metathesis and cross metathesis [*e.g.* conversion of **265** (Scheme 17) and methyl vinyl ketone to **266** in the presence of Grubbs catalyst II] was employed for the total synthesis of 8-*epi*-xanthatin [663]. A similar tandem intramolecular enyne metathesis–CM process was employed in the synthesis of a six-membered ring ether for total synthesis of TMC-69–6H [664]. Tandem intramolecular enyne metathesis–CM was employed for the preparation of seven-membered ring heterocycles fused to benzene rings [665]. Representative examples of tandem enyne metathesis–RCM



Scheme 15.

are depicted in Scheme 18. A moderately efficient tandem enyne metathesis–RCM was observed in the formation of the fused tricyclic compound **269** from dienyne **267** [666]. Tandem enyne metathesis–RCM reactions were also employed for the synthesis of: (1) steroid-like molecules (*e.g.* **271**) [667]; (2) a key intermediate (**273**) in the total synthesis of erythroidine [668]; (3) bicyclo[5.3.0]decadiene systems (*e.g.* **275**) [669]; (4) formation of bicyclo[5.4.0]undecane ring systems [670]; (5) a tetrahydroindole ring system for total synthesis of erythrocarine [671]; and (6) silicon-tethered tandem intramolecular enyne metathesis–RCM sequences as key steps in the total synthesis of acylfulvene and isofulvene [672], and in a synthetic approach to tartrolon B [673]. A tandem intermolecular enyne metathesis and RCM (a net cycloaddition) was observed in the reaction of alkyne amino acids (*e.g.* **276**, Scheme 19) with 1,5-hexadiene (**277**) [674]. The cycloaddition product (**281**) arises from the Z enyne metathesis product **279**. Treatment of the crude reaction mixtures with acrylic acid leads to a cross metathesis product (**282**) from the E enyne metathesis product **280**, resulting in an easy separation. A similar cycloaddition process was reported that used 1,5-cyclooctadiene in place of 1,5-hexadiene [675,676].



Scheme 16.

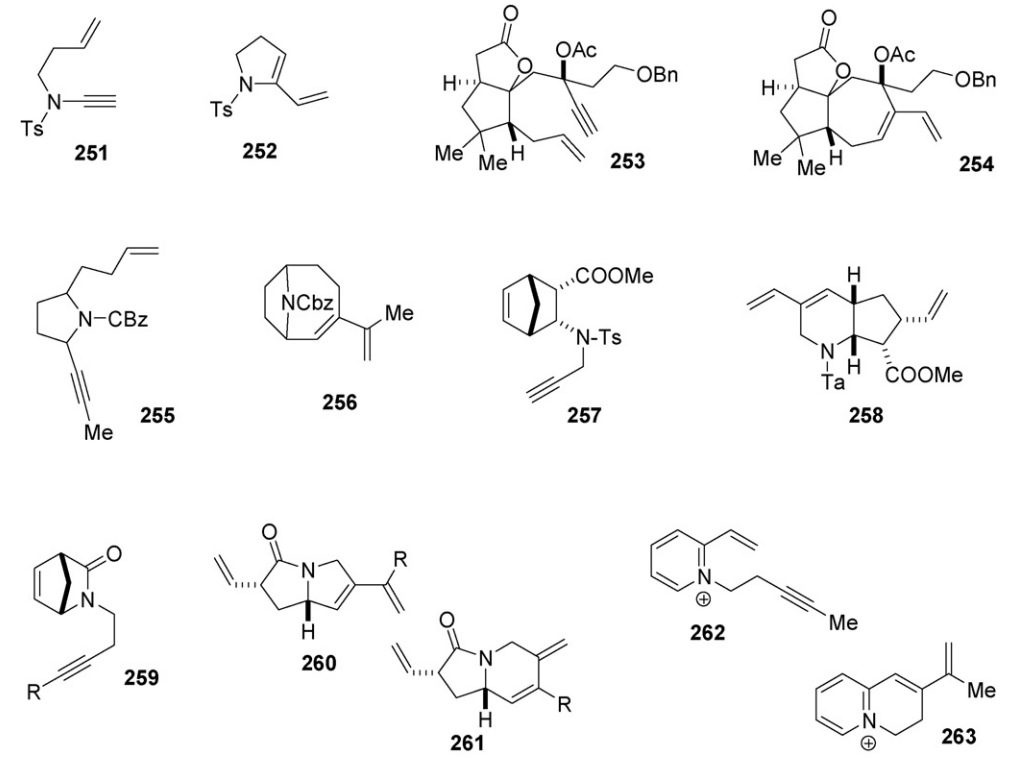
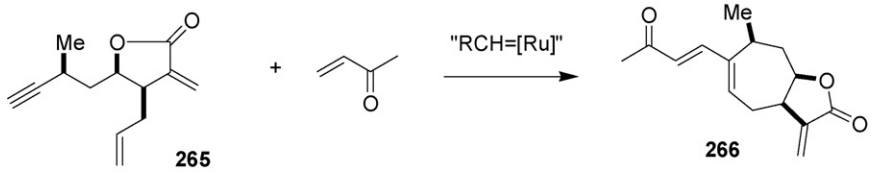


Fig. 12. Representative reactants and products from intramolecular enyne metathesis.

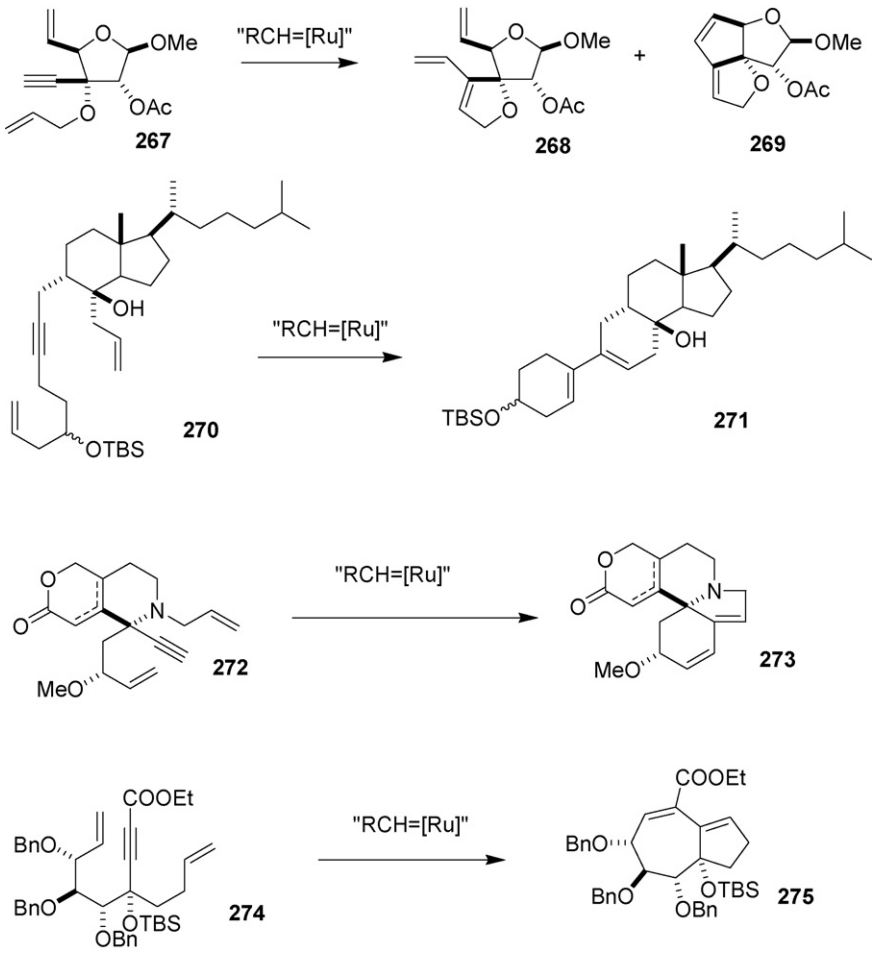
Tandem enyne metathesis–cyclopropanation was observed in the reaction of enynes (*e.g.* **283**, Scheme 20) with Grubbs catalyst I in the presence of ethyl diazoacetate (**284**) [677]. The reaction process destroys the metathesis activity of the catalyst. Addition of a 1,5-diene after the tandem enyne metathesis–cyclopropanation resulted in no RCM product.

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

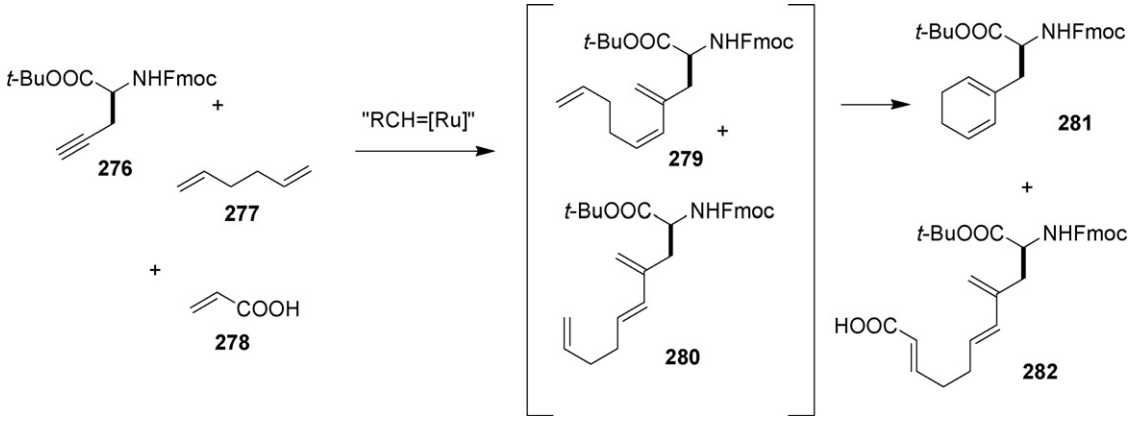
Several publications in 2006 report on processes unrelated to metathesis that are initiated by ruthenium carbene complex catalysts **1**–**4** and structurally related carbene complexes. Use of ruthenium carbene complexes to simultaneously initiate RCM



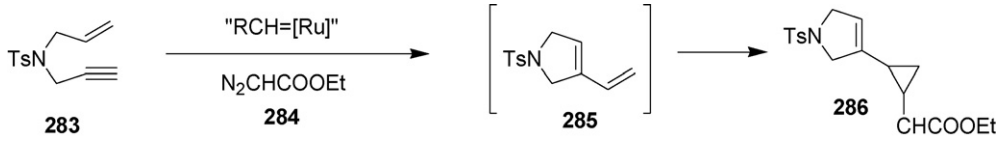
Scheme 17.



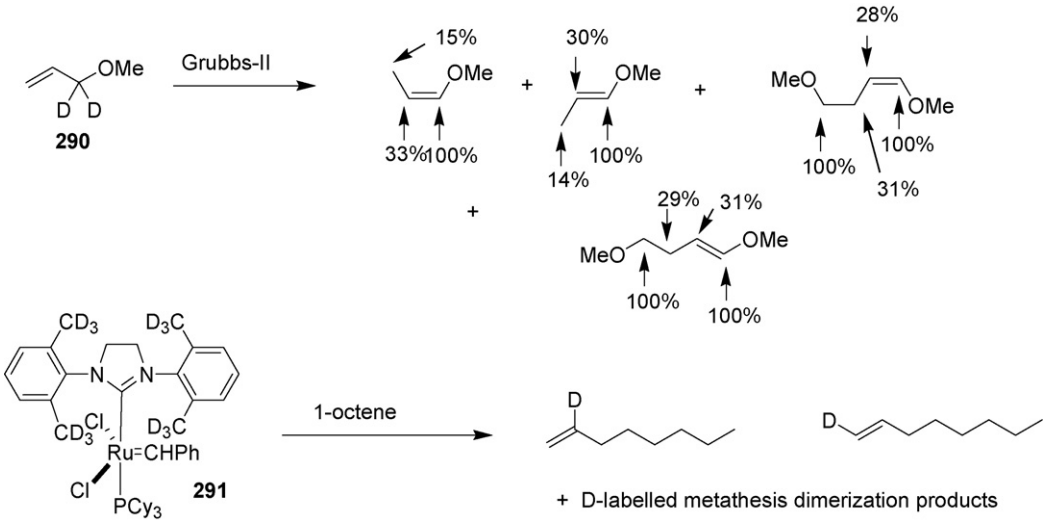
Scheme 18.



Scheme 19.



Scheme 20.



Scheme 21.

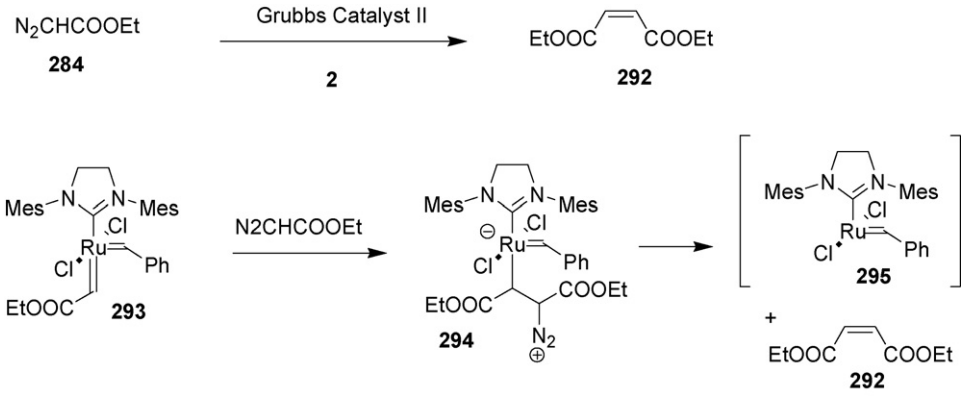
and free radical reactions was discussed in the RCM section (Scheme 11) [431,432].

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. A determined effort to better understand alkene isomerization during metathesis using deuterium labeling was reported using deuterium labeled alkenes (*e.g.* **290**, Scheme 21) [678]. A deuterated metathesis catalyst (**291**) also led to a deuterium labeled alkene product. Alkene isomerization was attributed to formation of metal hydride intermediates through decomposition of the methylidene complex intermediate. Thermally modified Grubbs catalyst II was shown to be an effective alkene isomerization catalyst [679]. Deprotection of *N*-allyl groups through conversion of *N*-allyl amides to the corresponding enamides using Grubbs catalyst I followed by hydrolysis was reported [680]. Other previously presented references also discuss alkene isomerization by metathesis catalysts [247,476].

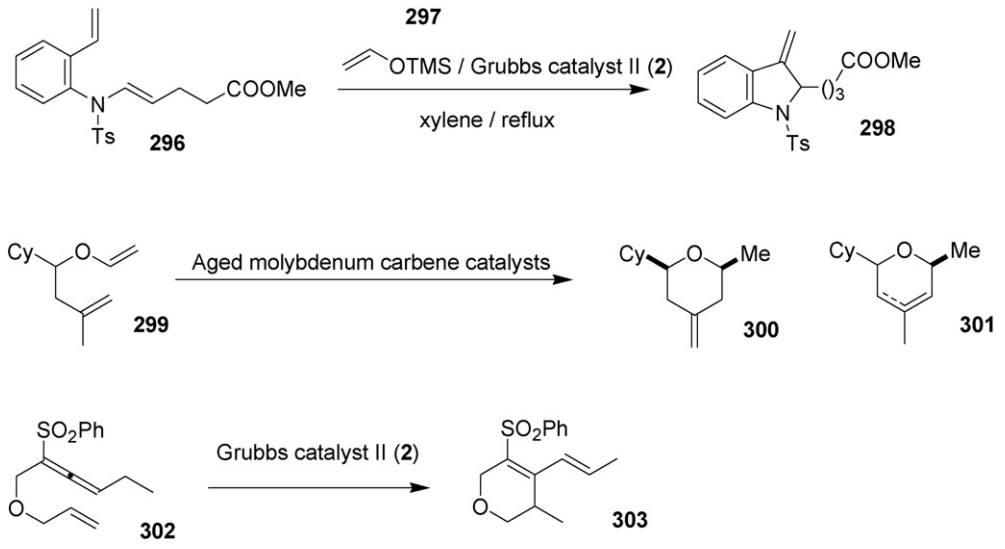
Use of Grubbs-catalysts II for the generation of carbenoids from diazo compounds was reported (Scheme 22) [681]. Reaction of Grubbs catalyst II (**2**) with ethyl diazoacetate (**284**) led to diethyl maleate (**292**). The proposed mechanism involves phosphine dissociation and formation of tris(carbene) complex **293**, followed by addition of ethyl diazoacetate to the acylated carbene carbon to afford intermediate diazonium salt **294**, followed by elimination of unsaturated carbene complex **295** and formation of diethyl maleate. Several examples of the formation of unsymmetrical maleates from dissimilar α-diazo esters were presented. Another example of cyclopropanation (in tandem with enyne metathesis) was presented earlier (Scheme 20) [677].

The use of Grubbs catalyst II and trimethylsilyl vinyl ether (**297**, Scheme 23) to initiate the cycloisomerization of dienes (*e.g.* **296**) was reported [682,683]. It was noted that at higher temperature the silyl ether additive converts Grubbs catalyst II into a species that prefers alkene isomerization and/or cycloisomerization in preference to alkene metathesis. Similar metathesis reactions performed in refluxing dichloromethane lead to alkene isomerization and RCM processes. It was also noted that aged chiral molybdenum carbene complex metathesis catalysts function as diene cycloisomerization catalysts (*e.g.* conversion of **299** to cyclic ethers **300** and **301**) [684]. Trifluoromethanesulfonic acid itself also catalyzed the conversion of **299**–**300/301**. The reaction of allene **302** with Grubbs catalyst II also resulted in the cycloisomerization product **303** and not the RCM product [685].

A DFT study of the conversion of acetoxycarbene–ruthenium complex **305** (Scheme 24) to ruthenium carbide complex **306**



Scheme 22.



Scheme 23.

was reported [686]. Two reasonable mechanistic pathways were uncovered. One mechanism involves coordination of the carbonyl oxygen to ruthenium (*e.g.* formation of **307**) prior to transfer of hydrogen to O. The other involves a concerted transfer of hydrogen to oxygen and triple bond formation through transition state **309**.

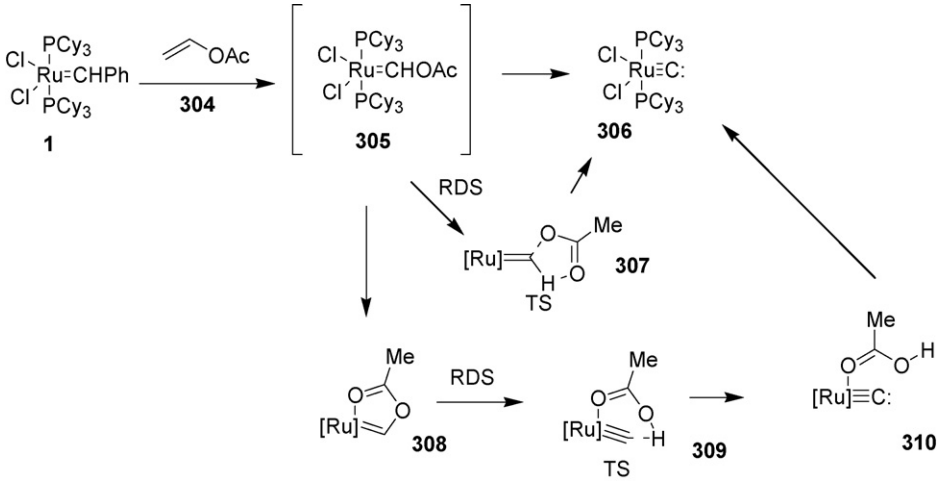
Attempted formation of the Tp complex **314** (Scheme 25) from the reaction of metathesis catalyst **311** (R=H) with KTp resulted in the unexpected complex **313** [687]. The desired Tp complex was formed as part of a complex mixture from the ester complex (**311**, R=OEt). In both cases the reaction mixtures were complex.

2.3. Individual carbene or alkylidene complexes classified according to metal

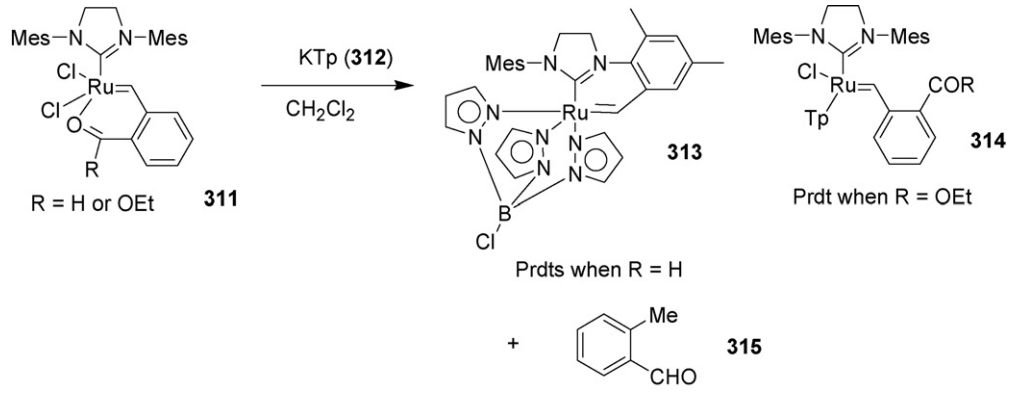
2.3.1. Group 4 metal–carbene complexes

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

Titanium carbene complexes (*e.g.* **321**, Scheme 26) were obtained through oxidation of titanium(III) bis(alkyl) complexes (*e.g.* **320**) [688]. The low temperature oxidation of **320** led to carbene complex **321**, which rearranged to internal metathesis product **322** upon warming to room temperature. The synthesis and reactions of the carbene complex phosphides (*e.g.* **323**) was also reported [689]. In this case the intermediate alkylidene complex undergoes internal hydrogen transfer to afford the phosphinidene complex **324**. Similar studies were reported for PNP-ligated carbene complex **325**, which provided a more stable carbene complex **326**. Reaction with methyl Grignard reagent led to the ligand substitution product **327**. The reaction of titanium carbene complex **328** with pyridine derivatives was reported [690]. The reaction leads to a complex where the pyridine ring has opened (**330**) plus neopentane. The proposed mechanism involves formation of a carbyne complex (**331**), followed by ligation to pyridine to afford complex **332**. Subsequent addition to the C–N π-bond and electrocyclic ring opening affords the metallacycle **334**, which rearranges to the observed product. The proposed mechanism was supported through DFT



Scheme 24.



Scheme 25.

calculations. Another mechanism involving C–H activation was considered but was discarded since DFT calculations showed this intermediate (**335**) to be of very low energy and unlikely to further progress to the observed product.

Zirconium carbene complexes (**338**, Scheme 27) were prepared through treatment of zirconocene dichloride with dianion **336** [691]. A related dimeric carbene complex (**341**) was obtained using $ZrCl_4(THF)_2$. Reaction of either of the carbene complexes with a ketone led to the carbonyl olefination product **339**. DFT studies revealed that the double bond arises through both σ- and π-donation.

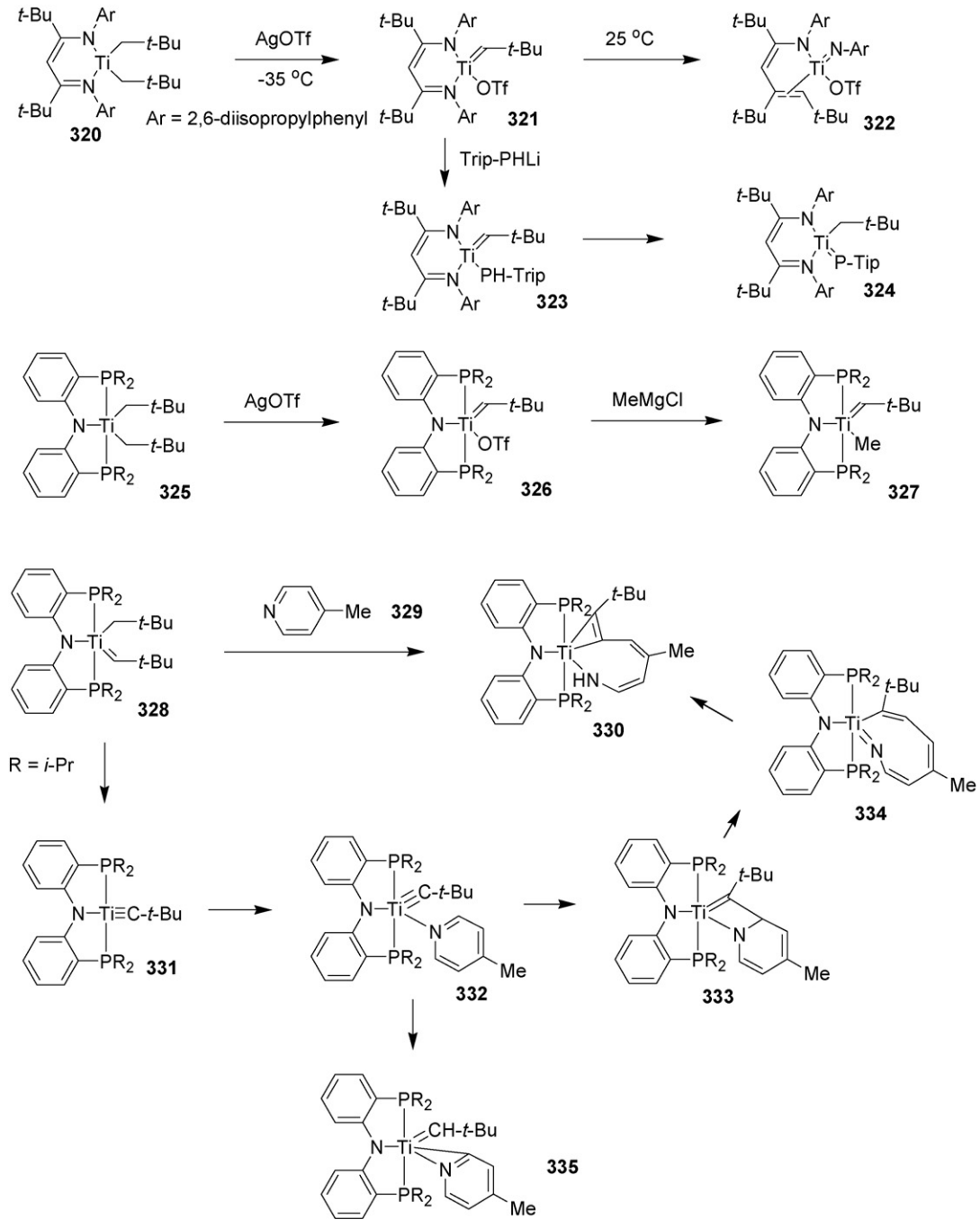
The preparation of silica-bound hafnium–carbene complexes (*e.g.* **343**, **344**, Scheme 28) was reported [692]. Thermolysis of silica-bound tris(neopentyl)hafnium (**342**) led to the carbene complexes **343** and **344**, accompanied by neopentane and isobutylene. A mechanism involving γC–H activation to afford metallacyclobutane **345** followed by retro [2 + 2]-cycloaddition was proposed to account for the formation of **344**. Compound **343** was a catalyst for the splitting of neopentane into isobutylene and methane. Titanium carbene complexes were suggested as intermediates in thermolysis of titanium analogs of **342** [693].

Several examples employing *in situ*-generated titanium–carbene complexes in synthetic organic chemistry were demonstrated in 2006; representative examples are depicted in Scheme 29. Reaction of *gem* dichloroalkenes (*e.g.* **347**) with titanium(II) complex **348** led to *in situ* formation of the vinylidene complex **349**, which subsequently coupled with alkynes to afford dienes (*e.g.* **351**) [694]. A mechanism involving formation of the alkylidenemetallacyclobutene (**350**) followed by protonation was proposed. Deuterium labeling studies support the intermediacy of the alkylidenemetallacyclobutene. Reaction of bis(acyloxymethyl) alkynes led to conjugated vinylallenes (*e.g.* **353**) via an elimination reaction from the allenylidenemetallacyclobutene intermediate (**352**). Titanium carbene complexes (*e.g.* **355**) were instrumental in a solid-phase stereoselective synthesis of piperidine derivatives (*e.g.* **358**) [695]. Chiral bis(thioether) **354** reacts with the titanium(II) species **348** in the presence of a polymer-bound ester (**356**) to afford the polymer-bound carbonyl olefination product **357**, which leads to the piperidine during cleavage from the resin. A similar intramolecular carbonyl olefination was employed for the total synthesis of one of the rings in a polycyclic marine toxin [696].

Formation of titanium carbene complexes (*e.g.* **360**, Scheme 30) through reaction of carbon tetrachloride with titanium tetrachloride and magnesium was reported [697]. Reactions with alkenes to afford cyclopropanation products (*e.g.* **361**) and reactions with carbonyl compounds to provide olefination products (*e.g.* **362**) were both demonstrated for this reagent. Titanium carbene complexes were suggested as intermediates in the cyclopropanation of enamines using dichloromethane, titanium tetrachloride, and magnesium [698]. Various carbonyl olefination reactions were accomplished using *in situ*-generated titanium carbene complexes. Olefination of carbonyl compound **363** as part of an amphidinolide total synthesis was effected using the Petassis reagent (**364**) [699]. The olefination of a carbohydrate lactone (**366**) with Petassis reagent for preparation of clavosilide A was also reported [700]. A similar reaction of the Petassis reagent with a lactone derivative was also reported [701]. Several of the olefin metathesis papers noted previously utilize titanium-based carbonyl olefination as part of a synthetic effort [475,575]. A method to recover titanocene dichloride from the residue of Petassis reactions was reported [702]. The carbohydrate formate ester **368** was converted to the corresponding enol ether (**370**) using the Tebbe reagent (**369**) [703].

Zirconium vinylidene complexes (*e.g.* **377**, Scheme 31) were proposed as intermediates in the formation of cyclopentenones (*e.g.* **378**) from the reaction of zirconacyclopentenes (*e.g.* **371**) with carbenoids followed by alkynyllithium reagents [704]. The key step in this transformation is the metalla-Cope rearrangement of bis(alkynyl)zirconium intermediate **376** to the zirconium vinylidene complex **377**, which affords the observed product upon protonation. Coordination of the second alkynyl group was proposed based on the requirement for two moles of alkynyllithium reagent.

Titanium carbene complexes were suggested as intermediates in the photochemically induced C–H activation of methane by titanium oxide species [705]. The photochemical process affords $H_2C{=}Ti(H)(OH)$, which was identified spectroscopically, and reacts further with methane to afford $(CH_3)_2Ti(H)(OH)$. The complex features an agostic interaction between the C–H bond and titanium. This structural feature was also predicted by DFT calculations. Laser-ablated zirconium or hafnium atoms generated in the presence in methyl halides afforded a carbene complex, $H_2C{=}MHX$, that could be isolated in an argon



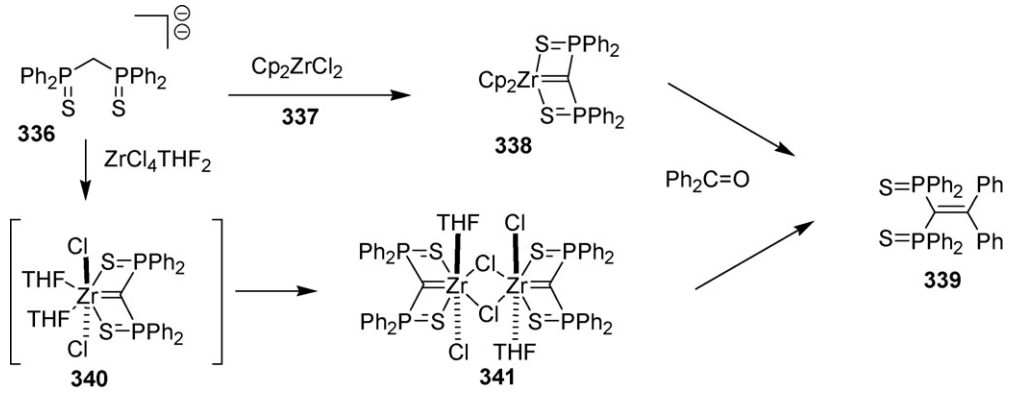
Scheme 26.

matrix and observed by IR spectroscopy [706]. The hypothetical carbene complexes, which feature agnostic C–H interactions, were studied by DFT and the calculated IR spectrum compared with the observed IR spectrum. A related study of laser-ablated titanium atoms and difluoromethane was also reported [707].

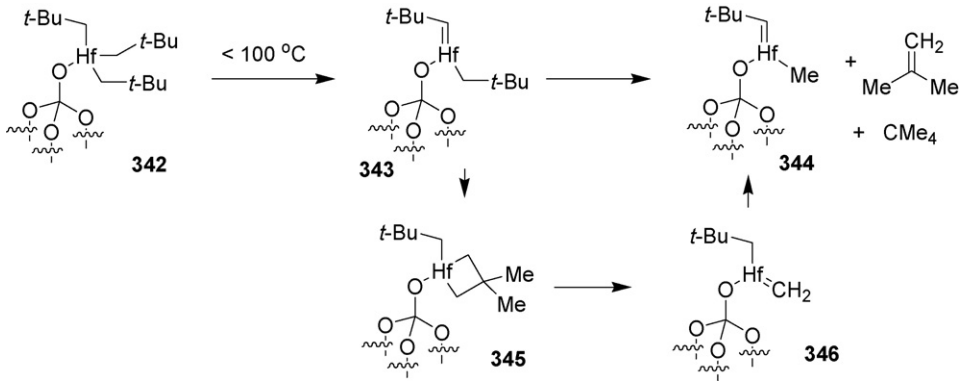
The strength of agostic C–H–metal interactions were evaluated computationally for several titanium and tungsten carbene complexes (Scheme 32) [708]. DFT methods were determined to give the best fit to experimental observations. The strength of an agostic C–H interaction was determined to be less than 10 kcal/mol, and similar in magnitude to a hydrogen bond. Agostic interactions were evaluated for a variety of titanium complexes, including carbene complexes [709]. Bond length and angle distortions were attributed to steric interactions with the titanium center and not to bonding interactions.

2.3.2. Group 5 metal–carbene complexes

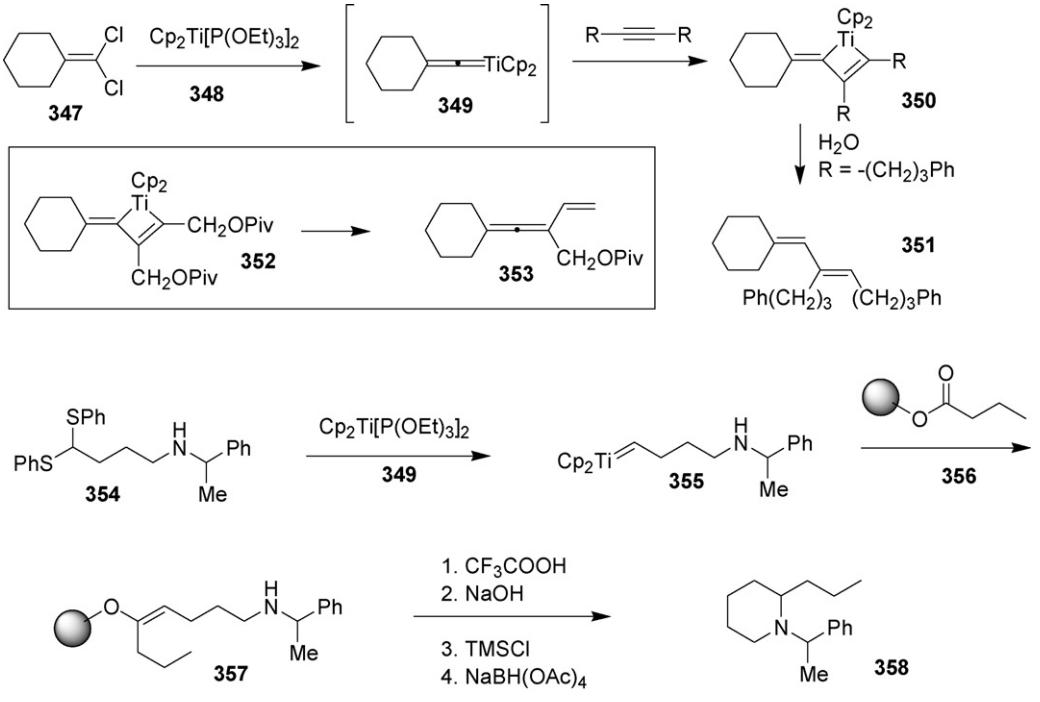
Niobium carbene complexes (*e.g.* **385**, Scheme 33) were prepared through thermolysis of niobium complex **380** [710]. Reaction of the initially formed complex **380** with H_2 establishes an equilibrium between the metal hydrides **381** and **382** and the original starting material. Thermal degradation of the mixture affords either carbene complex **385** or metal–phosphorus double



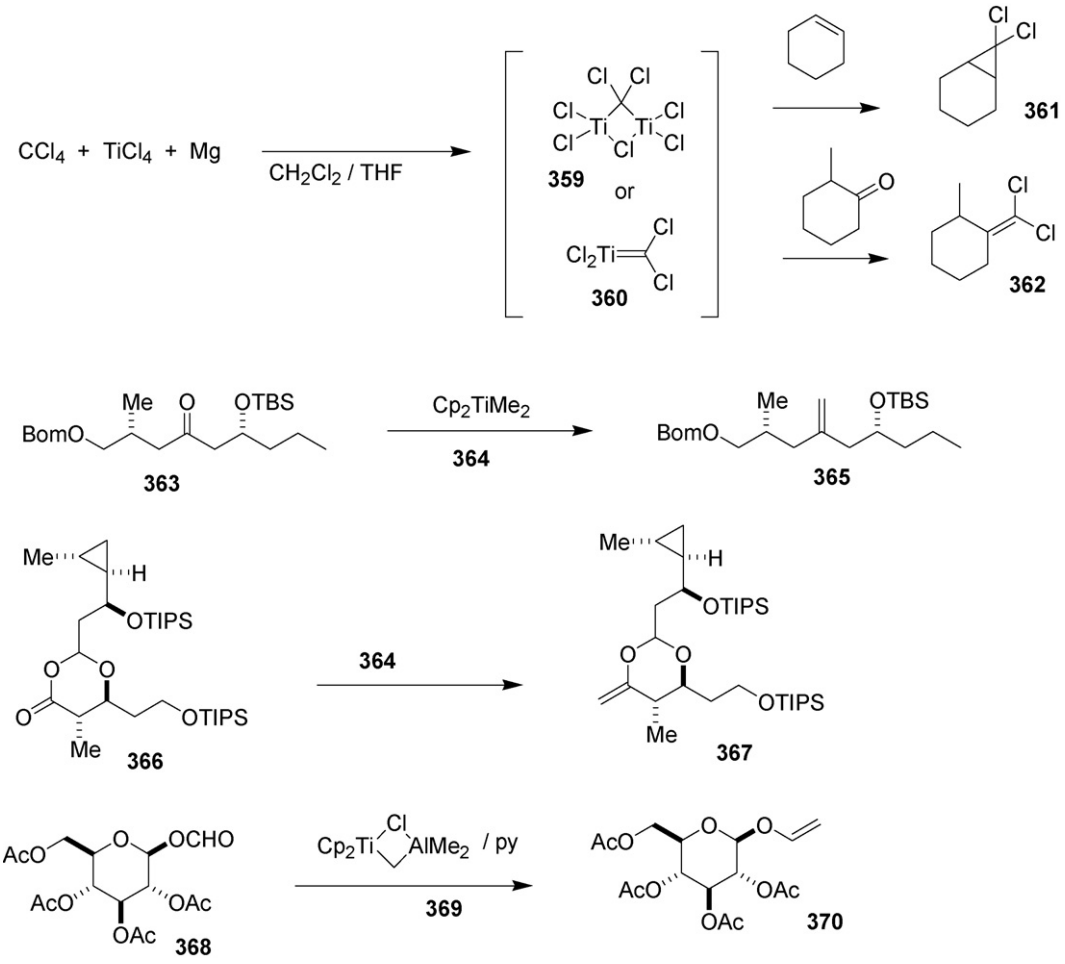
Scheme 27.



Scheme 28.



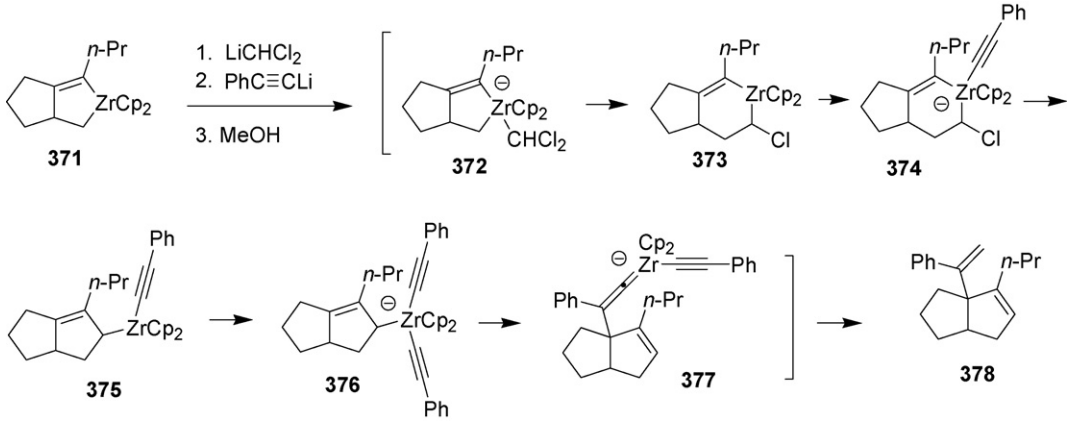
Scheme 29.



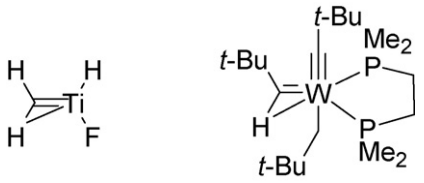
Scheme 30.

bonded species **386** and **387**. Thermolysis of the starting material without hydrogen affords only the carbene complex, which can also be prepared by the reaction of the starting complex with $CH_2{=}PPh_3$. A mechanism involving initial C–H activation or C–P activation was proposed.

Tantalum carbene complexes (*e.g.* **389**, Scheme 34) were prepared through the reaction of tantalum trichloride complex **388** with trimethylsilylmethyllithium [711]. Reaction of the same dichloride complex with neopentyllithium led to the monoalkylation product (**390**), however further reaction with methyllithium led to the neopentylidene complex **391**. Tantalum carbene complexes (*e.g.* **393**) were generated from the reaction of tantalum trichloride complex **392** with ethyllithium [712]. Reaction with higher alkyllithiums (*e.g.* *n*-BuLi) resulted in simple dialkyltantalum chloride complexes.



Scheme 31.



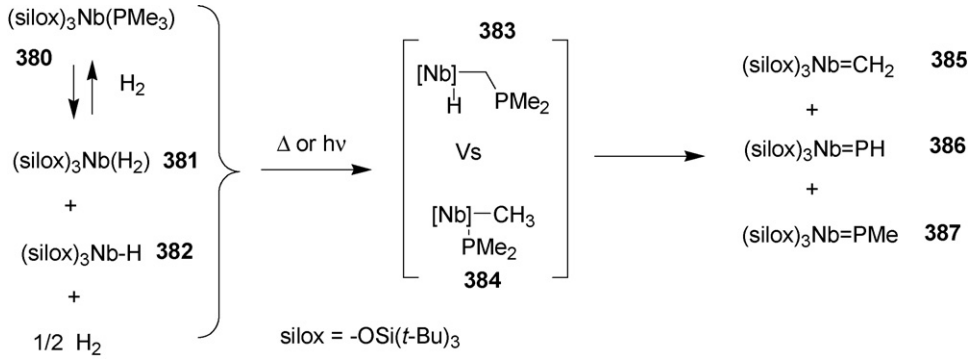
Scheme 32.

Tantalum carbene complex **396** (Scheme 35) was generated from the reaction of carbene **394** with pentabenzyltantalum (**395**) [713]. Carbene complex **396** features an agnostic C–H interaction. Reaction of the dianionic carbene ligand **397** with carbene complex **398** led to the η^2-imine complex **399**. Potential reaction pathways and structural effects were evaluated using DFT calculations.

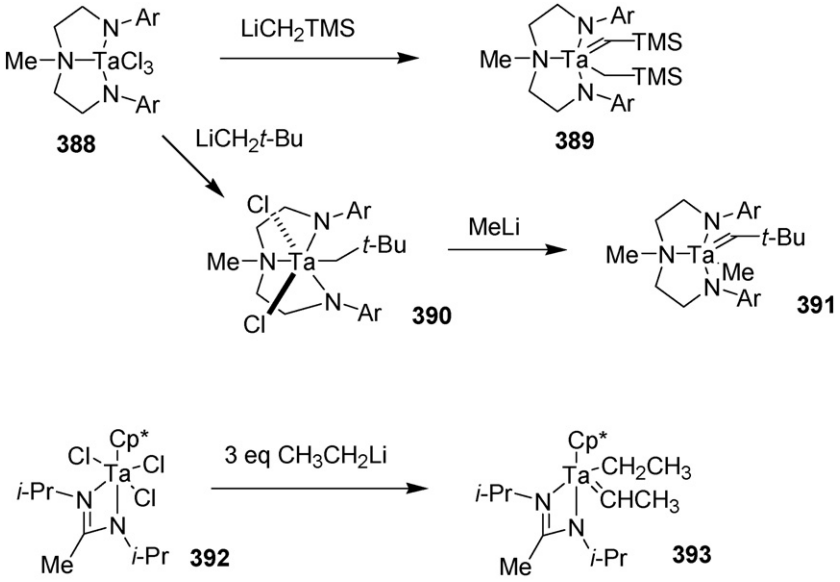
The synthesis and reactivity of tantalum carbene complex **400** (Scheme 36) was reported [714]. The carbene complex reacts with both nucleophilic and electrophilic reagents. Reaction with pyridine-*N*-oxide led to the tantalum oxo complex **401**. Reaction with iodomethane led to the ethyl complex **402**. Insertion products (*e.g.* **403**) were formed from reaction with nitriles or isonitriles. Reaction with carbonyl compounds led to carbonyl olefination products and a titanium–oxo complex (**401**). The sulfide complex **404** was obtained from reaction with tritylthiol, while the sulfur transfer product **405** was obtained from reaction with propylene sulfide.

The reaction of tantalum carbene complex **407** (Scheme 37) with mesoporous silica to afford an immobilized carbene complex (**408**) was reported [715]. Hydrogenation of the silica-bound carbene complex led to a silica-bound tantalum hydride (**409**). Reaction of the hydride with labeled methane led to a species that contains tantalum methylene and methylidyne ligands according to solid state NMR.

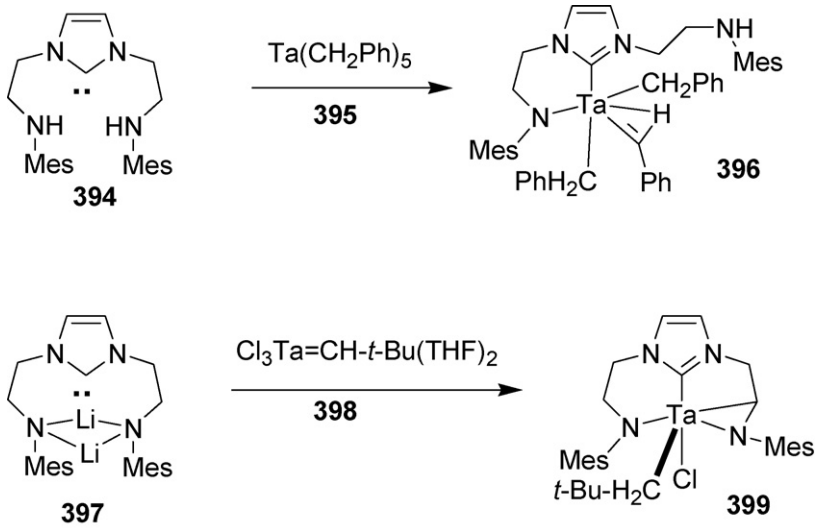
Tantalum carbene complexes were generated through alkylation of tantalum halide complexes followed by thermolysis (Scheme 38) [716]. Reaction of titanium dichloride **412** with alkyllithium reagent **413** led to ditantalum complex **414**, metallacyclopentane derivative **415**, and tantalum methylene complex **416**. Thermolysis of the tris(trimethylsilylmethyl) complex **417** led to the tantalum carbene complex **418**. A related process was reported for the analogous benzyl complex, which leads to carbene complex **420**.



Scheme 33.



Scheme 34.



Scheme 35.

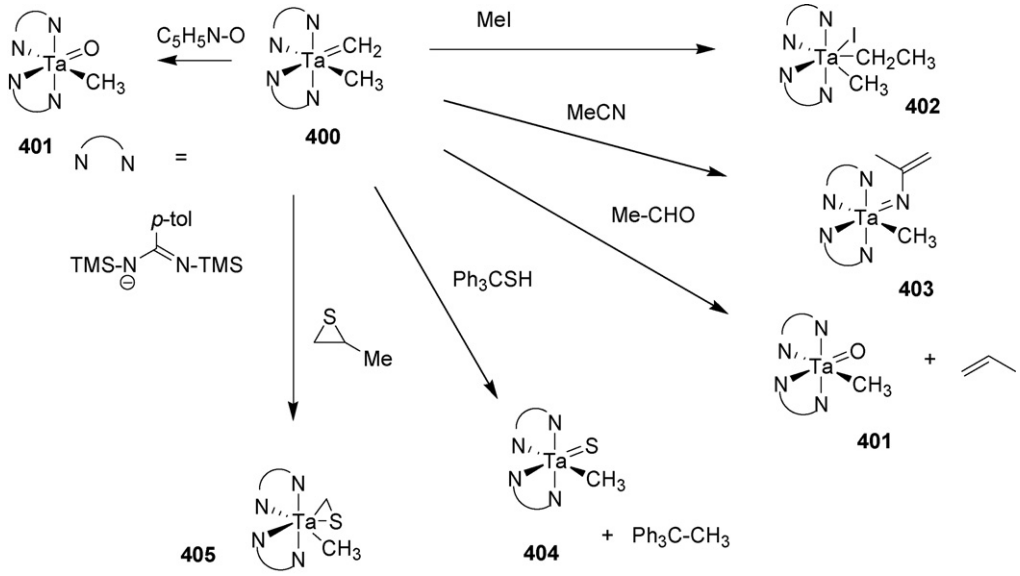
The reaction of tantalum carbene complexes (*e.g.* **421**, Scheme 39) with triarylaluminum reagents was reported [717]. Reaction with one mole of triarylaluminum led to the simple adduct **423**, while reaction with a second mole led to the bridging aluminum species **424**. The initial adduct **423** does not feature an agnostic interaction, however agnostic C–H interactions are present in the boron analog. The bridging species was a catalyst for addition polymerization of acrylate esters and amides.

Carbene and carbyne complexes were observed in the coupling of laser-ablated niobium atoms with fluoromethane [718]. This reaction produced CH_3NbF, $H_2C{=}Nb(H)F$, and $HC{\equiv}Nb(H)_2F$, which were identified by IR spectroscopy and the observed spectra compared with spectra calculated by DFT. A similar study was reported for the interaction of methane or methyl halides with laser-ablated vanadium, niobium, and tantalum, atoms [719,720]. A DFT study of $H_2C{=}NbH_2$ was reported [721].

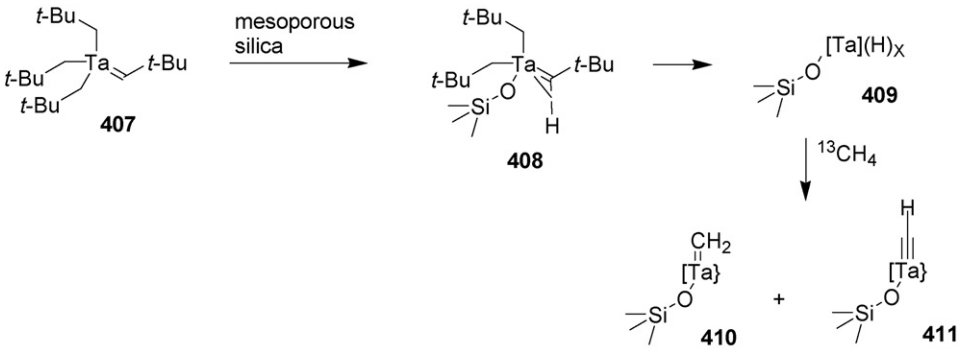
2.3.3. Group 6 metal–carbene complexes (further classified according to structure and reaction type)

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

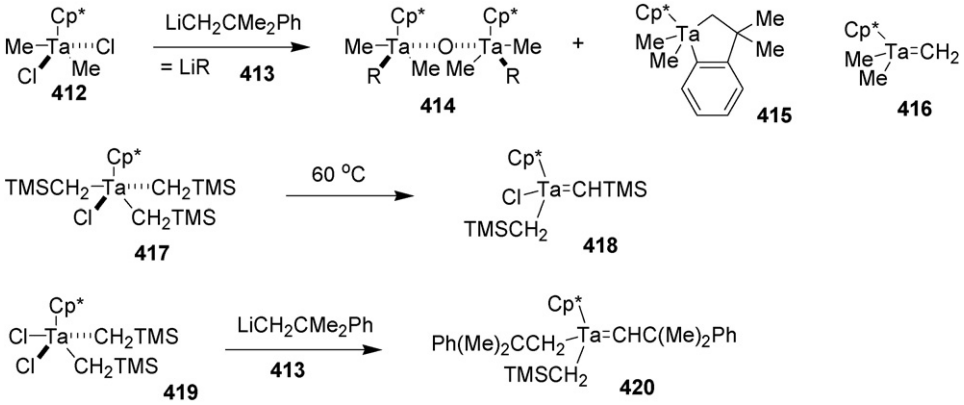
The reaction of tungsten carbene complexes (*e.g.* **430**, Scheme 40) with phenol derivatives was reported [722]. Reaction of carbene complex **430** with pentafluorophenol led to the tris(neopentyl)tungsten complex **431**, which led to an unstable carbene complex (**432**) and eventually a tungsten–tungsten doubly bonded species (**433**) upon thermolysis. A similar dimer was formed upon treatment of tungsten carbene complex **434** with



Scheme 36.



Scheme 37.



Scheme 38.

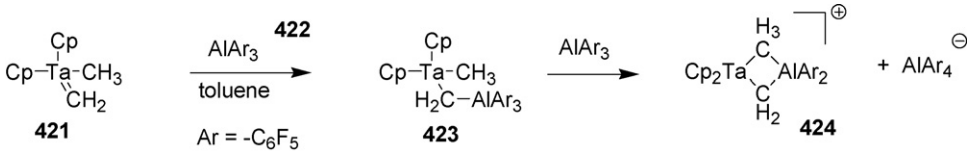
2-pentene. Reaction of bis(imido)–bis(carbene) complex **436** with triflic acid led to imido(carbene)tungsten complex **437**.

Stoichiometric reaction of the Schrock catalyst (**5**) and derivatives with various cyclic triene derivatives (*e.g.* **438**, **442**, Scheme 41) was reported [723,724]. These complexes bear structural similarity to the propagating five- and six-membered ring intermediates of ADMET bis(alkyne) polymerization discussed in Scheme 8. Reaction of **438** with 2 equiv. of a molybdenum carbene complex led to the bis(carbene) complex (**439**), which couples with aldehyde **440** to afford the respective hepta-ene. Similar studies were reported for the analogous six-membered ring triene derivative **439**.

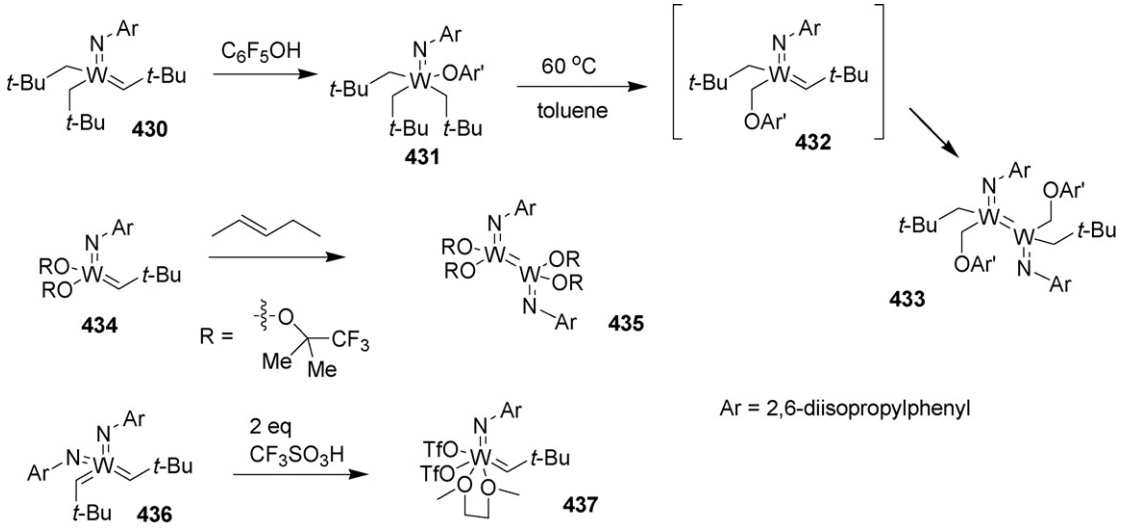
The reaction of tris(sulfide)-bridged dimolybdenum isocyanide complexes (*e.g.* **444**, Scheme 42) with base to afford Cp-bridged aminocarbene complex **445** was reported [725]. Reaction of related complexes (*e.g.* **446**) with propargylic alcohol derivatives to afford bridging allenylidene complexes (*e.g.* **448**) was also reported [726]. Reaction of complex **446** with diphenylpropargyl alcohol (**447**) initially leads to dimetallacyclobutene **449**, which afforded the chelated alkyne complex **450** upon protonation. Reaction with acid affords the bridging allenylidene complex **448**.

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, however other methods were also reported in 2006. The reaction of molybdenum bromopropyl complexes (*e.g.* **454**, Scheme 43) with nucleophiles led to unstable but isolatable molybdenum cyclic carbene complexes (*e.g.* **455**) [727]. Similar reactions with tungsten analogs afforded only the S_N2 products.

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



Scheme 39.



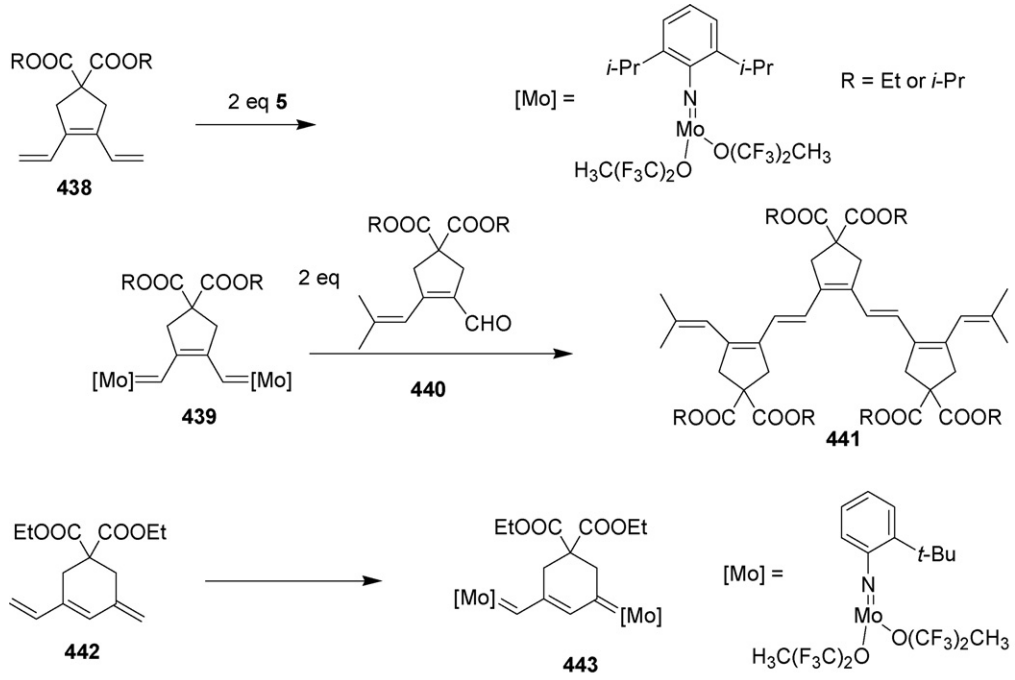
Scheme 40.

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).

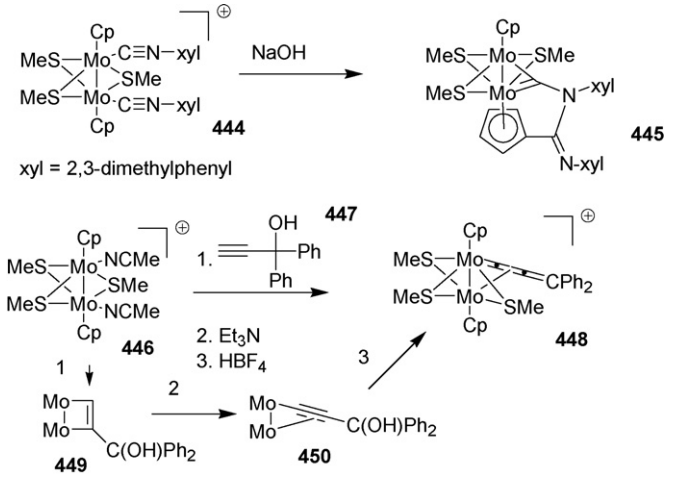
Cyclopropanation using alkynylcarbene–chromium complexes (*e.g.* **456**, Scheme 44) was reported [728]. The reaction using electron-deficient alkenes was facile, however unactivated alkenes required use of a large excess. The formation of pyrroles (*e.g.* **461**) through the reaction of aminocarbene complexes (*e.g.* **457**) and α,β-unsaturated aldehydes was reported [729]. A mechanism involving cyclopropanation to afford a donor–acceptor activated cyclopropane (**459**), followed by ring opening and aldehyde–enamine condensation was proposed.

Synthesis of chelated ferrocenylcarbene complexes (*e.g.* **454**, Scheme 45) was reported [730]. Reaction of metal carbonyls with ferrocenyllithium followed by an alkylating agent let to the alkoxycarbene complexes (*e.g.* **452**) which reacted with allylic amines to afford the aminocarbene complexes (*e.g.* **453**) as predominantly the Z rotamers. Photolysis led to the internally coordinated complexes (**454**). The chelate complexes were studied by cyclic voltammetry.

The coupling of iminocarbene complexes with alkenes and alkynes was reported (Scheme 46) [731]. In all cases, cyclopentannulation products that feature no incorporation of CO ligands



Scheme 41.



Scheme 42.

Scheme 43.

were obtained. The role of the imino group is to suppress CO insertion, thus the primary photoreaction is simply CO loss and not ketene formation.

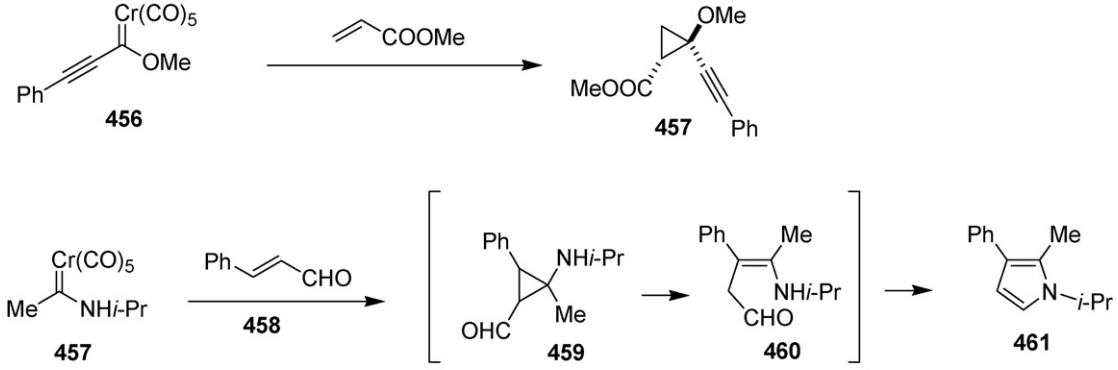
2.3.3.4. Reaction of Group 6 metal–carbene complexes with alkynes–benzannulation. Many examples of benzannulation using α,β-unsaturated chromium–carbene complexes (Scheme 47) and alkynes (commonly known as the Dötz reaction) were reported in 2006. Examples include: (1) synthesis of carbene complex **468** and subsequent Dötz reaction to afford benzene ring **469** for total synthesis of aflatoxins [732]; (2) related reaction of carbene complex **468** with *gem* diester-substituted alkynes (*e.g.* **470**), resulting in net benzannulation–lactonization (forming **471**), accompanied by five-membered ring annulation products [733]; (3) synthesis of naphthopyrans (*e.g.* **473**) via coupling pyran complex **472** with alkynes; reaction with a triyne resulted in a secondary cyclization process to afford a tricyclic compound (**475**) [734]; and (4) direct synthesis of a quinone-fused carbohydrate (**478**) using a β-fluoro-α,β-unsaturated carbene complex (**476**) [735].

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 2006.

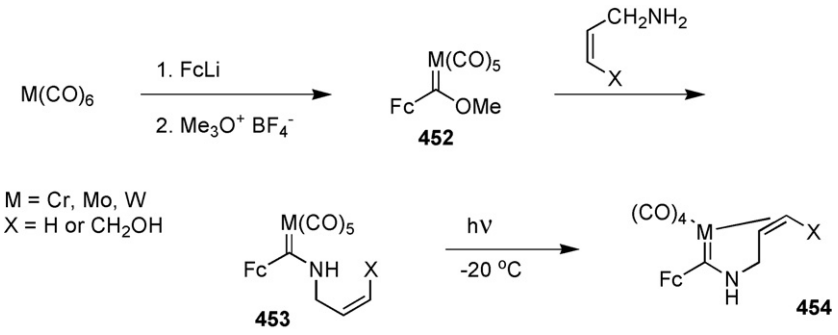
The coupling of 4-oxygenated-2,6-dimethylphenylcarbene–chromium complexes (*e.g.* **480**, Scheme 48) and various alkynes was examined [736]. This coupling reaction in benzene affords predominantly the five-membered ring annulation product (*e.g.* **481**) accompanied by varying amounts of the six-membered ring products (*e.g.* **482**) derived from CO insertion. Electronic effects in the carbene complex and alkyne, as well as ring size effects for intramolecular reactions all played an important effect on the course of the reaction.

The coupling of a γ,δ-unsaturated Fischer carbene complex (**484**, Scheme 49) with electron rich 2-alkynylbenzaldehyde **483** was used as the key step in the total synthesis of the anticancer agent antofine (**487**) [737]. Coupling of the carbene and alkyne initially generates an isobenzofuran intermediate (**485**), which undergoes intramolecular Diels–Alder reaction and dehydration to afford the observed dihydrophenanthrene derivative **486**. A mechanistically related process employing β-cyanocarbene complexes (*e.g.* **489**) was also reported [738]. In this case the intermediate isobenzofuran (**492**) undergoes a Diels–Alder reaction with the nitrile to afford the isoquinoline (**494**) after deoxygenation of the Diels–Alder adduct (**493**). A major by-product from the amide reaction is diene–nitrile **491**, which can result from decomposition of the initially formed carbene complex **490**.

The coupling of γ,δ-unsaturated Fischer carbene complexes (*e.g.* **484**, Scheme 50) with alkynylheterocycle carboxaldehydes (**495**) was reported [739]. The course of the reaction was very dependent on the electron-donating ability of the heterocyclic ring – strong donors led to products derived from the furan intermediate (**495**), while more weakly electron donating heterocyclic rings led to products derived from the pyrone intermediate (**496**). A related process using enyne epoxides (*e.g.* **503**) in place of enyne-aldehydes led to a mixture of oxepins (*e.g.*



Scheme 44.



Scheme 45.

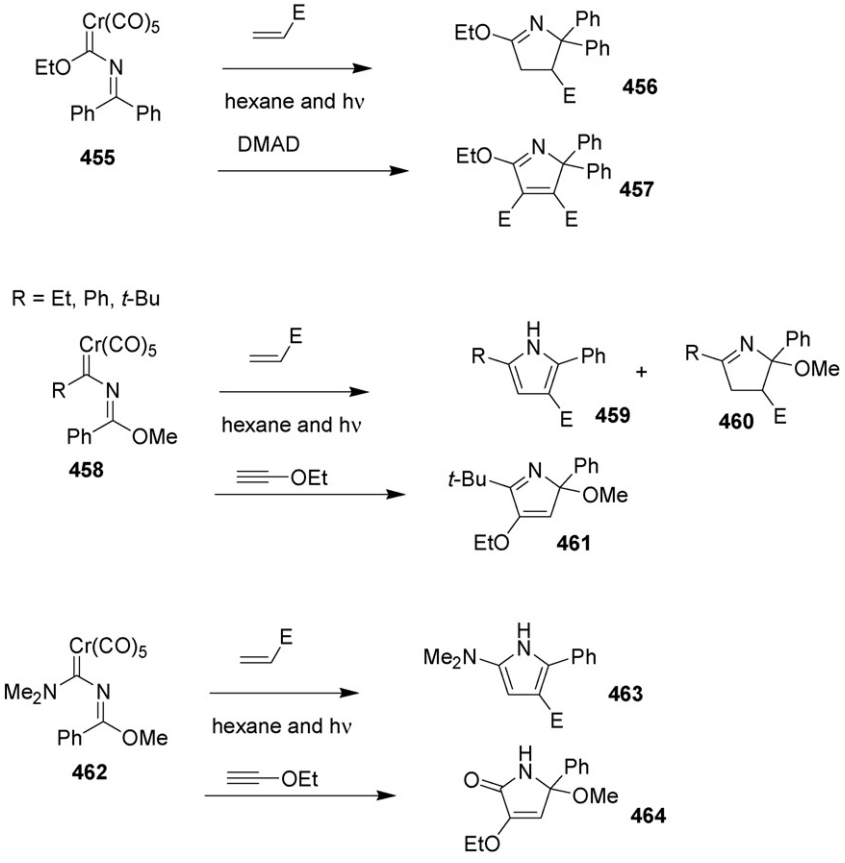
510), derived from ketene **508,** and oxygen transfer products (*e.g.* **507**), derived directly from the vinylcarbene complex intermediate (**505**) [740]. The direction of the reaction was highly dependent on the identity of the R group.

The coupling of 2-alkynylphenylcarbene–chromium complexes (*e.g.* **512**, Scheme 51) with alkyne–aldehydes linked through heterocyclic ring systems (*e.g.,* **511**) was reported [741]. A mechanism that involves formation of a heterocycle-fused pyrone derivative (**513**), followed by intramolecular Diels–Alder reaction and elimination of CO_2 was proposed.

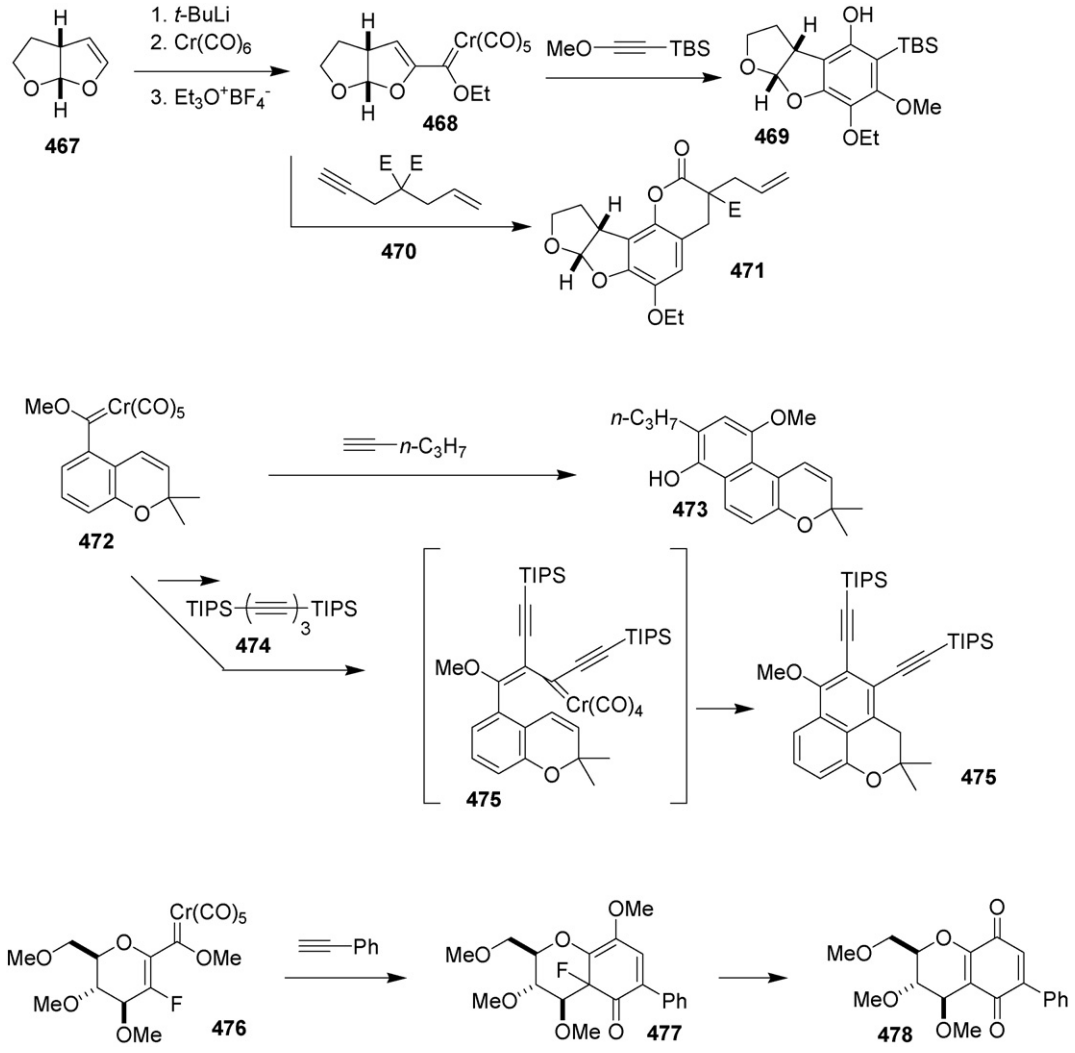
The coupling of silylated alkynes featuring sterically bulky silyl groups (*e.g.* **517**, Scheme 52) with Fischer arylcarbene complexes (*e.g.* **516**) was reported [742]. The reaction initially afforded the stable silylketene complexes (**518**), which subsequently react with sulfur ylides to afford cyclopentenones (**519**).

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 2006 where a carbene complex activates a π-bond for nucleophilic addition or cycloaddition reactions (*i.e.* the carbene complex is a surrogate for an "activated ester").

Coupling of copper enolates (*e.g.* **522**, Scheme 53) with α,β-unsaturated carbene complexes (*e.g.* **523**) was reported [743]. Coupling followed by reaction with acid chlorides resulted in the net "umpolung" product **524** via reaction of the anionic intermediate (**523**) with the acid chloride. The reaction proceeded



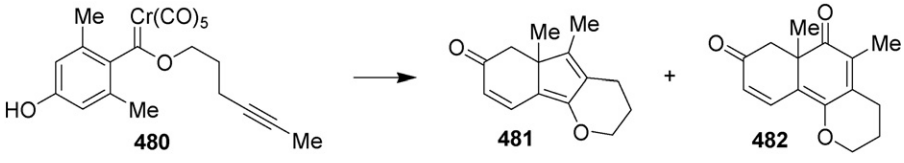
Scheme 46.



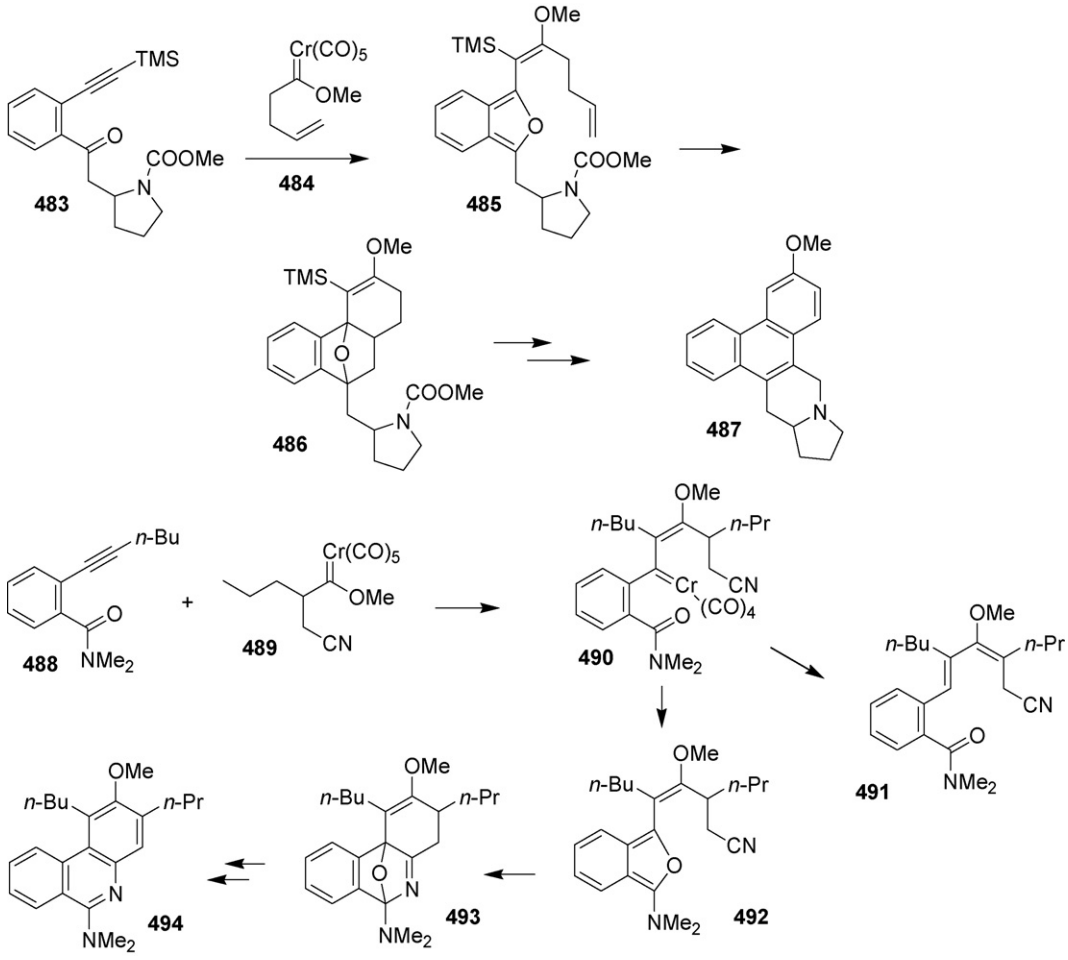
Scheme 47.

with a high degree of diastereoselectivity and *E*/*Z* selectivity. A mechanistically related process involving the nucleophilic addition of ketene acetals (*e.g.*, **526**) to α,β-unsaturated carbene complexes (*e.g.* **525**) was also reported [744]. In this case, the initially formed Michael addition product **527** undergoes silyl transfer and alkyne formation to afford alkyne ketone **528**. This alkyne then undergoes the Dötz reaction with a second equivalent of carbene complex followed by lactonization to afford the observed complex.

The reaction of pyrazolones (*e.g.* **532**, Scheme 54) with alkynyl Fischer carbene complexes (*e.g.* **531**) was reported [745]. A variety of net cycloaddition products (*e.g.* **533**–**536**) were produced in this coupling. A mechanism involving competitive N and O Michael addition followed by addition to the carbene complex was proposed. The corresponding organic carbonyl compounds were formed upon oxidation of the carbene complexes. The mechanism for the Michael addition of pyrazoles (*e.g.* **537**) to alkynylcarbene complexes was studied by kinetics and DFT calculations [746]. The most reasonable mechanism is depicted in Scheme 54, where the initially formed Michael adduct **538** rotates to afford **539**, which then undergoes proton transfer to afford the observed product. The individual rotamers could be observed by low temperature NMR. The Michael addition step was rate determining. An NMR study of the Michael addition of 3,5-di-*t*-butylpyrazole to alkynylcarbene complexes was reported [747]. Similar reactions were reported for cyclic imino-esters (*e.g.* **542**) and alkynylcarbene complexes, which lead to net dimerization/cycloaddition products (*e.g.*



Scheme 48.



Scheme 49.

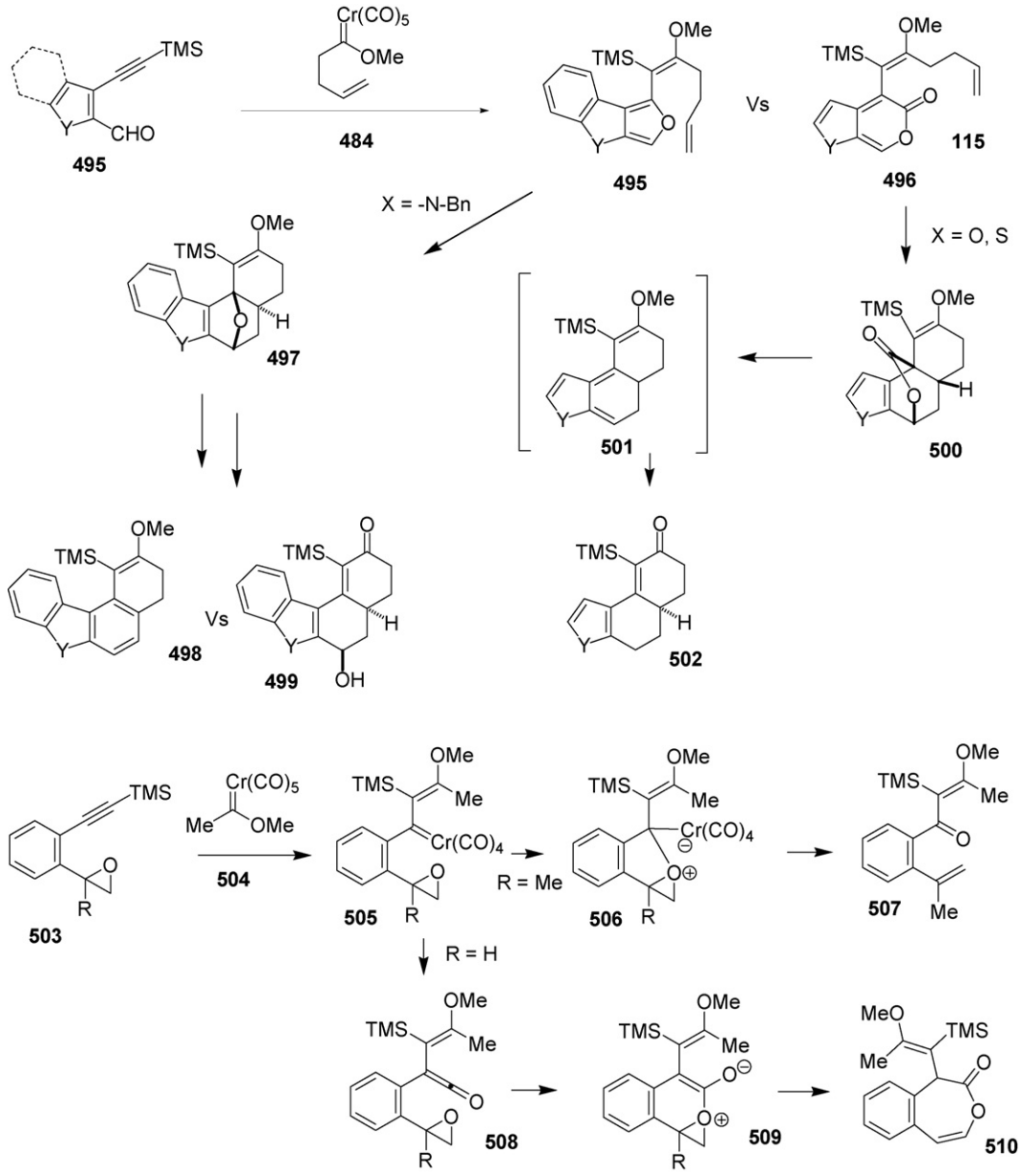
544–**546**) [748]. The initially formed Michael addition product **543** undergoes [2 + 2]-cycloaddition to afford bis(carbene) complex **544**, which undergoes ring opening to **545** upon exposure to silica gel. Thermolysis of the dienylcarbene complex **545** led to the cyclopentenone **546** after silica gel purification.

Diels–Alder reactions were reported for alkynylcarbene complexes (*e.g.* **531/541**, Scheme 55) and fulvene derivatives (*e.g.* **547**) [749]. The Diels–Alder adducts (**548**) are produced at room temperature, however heating transforms the Diels–Alder adducts into either of the compounds **549** or **550**. The distribution of the products was highly dependent on the metal. The cyclopentadienes (**550**) were part of the reaction products only when chromium was employed, while tungsten carbene complexes led exclusively to bicycle[3.2.1]octadienone systems (**549**). Nonconcerted cycloaddition reactions were observed using alkylideneimidazole derivatives (*e.g.* **551**) [750]. A mechanism involving addition of the imidazole nitrogen to the alkyne followed by cyclization of the zwitterions (**552**) was proposed. If an enynylcarbene complex was employed, a secondary cyclization event occurred, resulting in cyclopentadiene derivatives (*e.g.* **554**).

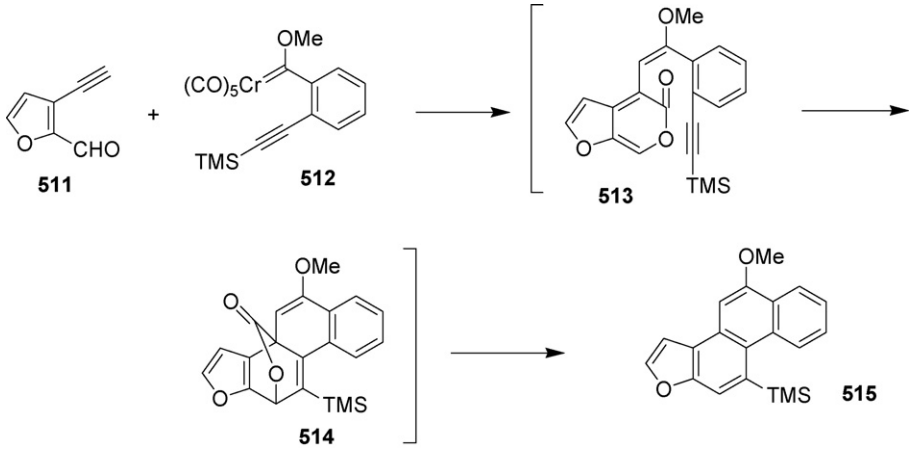
A theoretical study of 1,3-dipolar cycloaddition reactions of alkynyl Fischer carbene complexes was reported [751]. The reaction with alkynyl Fischer carbene complexes proceeds at a much greater rate and with much higher regioselectivity than the analogous reaction with alkyne–esters. The origin of these effects is that the transition state for the reaction involving carbene complexes was more asynchronous than that for esters.

2.3.3.7. Physical organic chemistry of Group 6 Fischer carbene complexes. The rate constants for the reaction of hydroxide ion with Group 6 carbene complexes of general structure **555** (Scheme 56) was determined [752]. The kinetic reactivity follows the order Cr < Mo < W for the thio complexes (X = S) and the order Cr > Mo > W for the alkoxycarbene complexes (X = O). The reactivity order for the thiocarbene complexes was attributed to the relative electronegativities of the metal atoms. The reactivity order for the alkoxycarbene complexes was attributed to the relative resonance stabilization of the carbene complex functionality. The rate constants for nucleophilic attack were generally higher for alkoxycarbene complexes. The kinetic parameters for the addition of hydroxide ion to alkoxycarbene complexes were reported [753]. The rate could be correlated with steric bulk of the alkoxide group. The following relative rates were observed: *O*-*n*-Pr > *O*-neopentyl > *O*-*i*-Pr > *O*-menthyl.

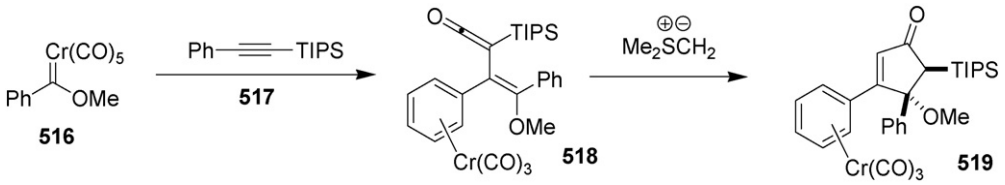
2.3.3.8. Synthesis and reactivity of Group 6 metal–vinylidene complexes, and reactions that involve vinylidene–metal com-



Scheme 50.



Scheme 51.

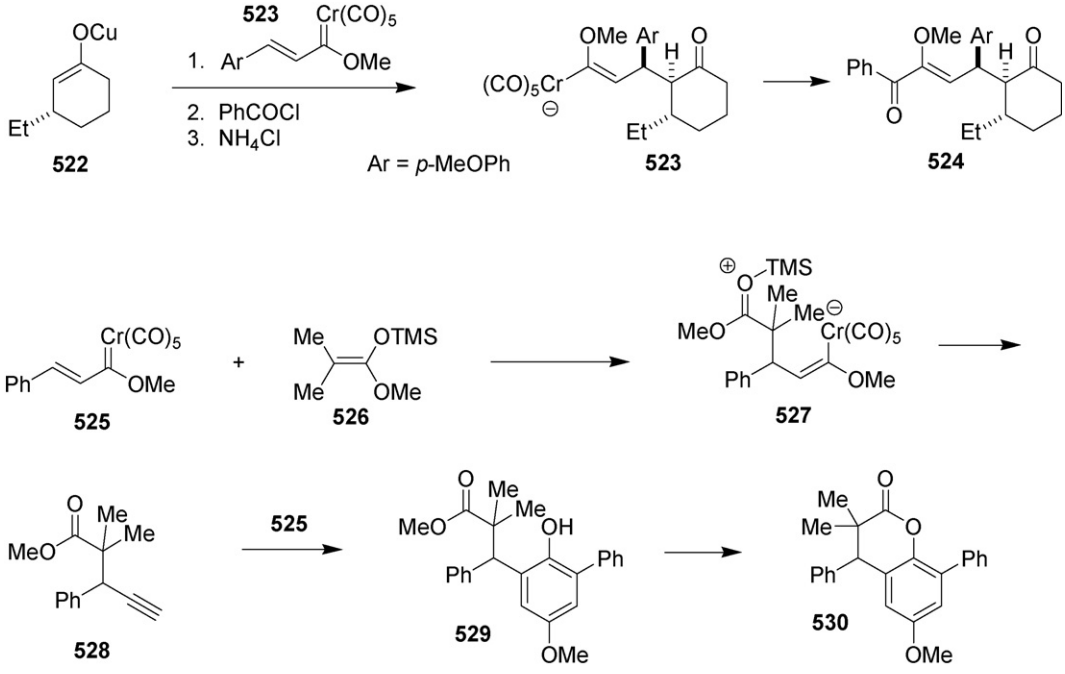


Scheme 52.

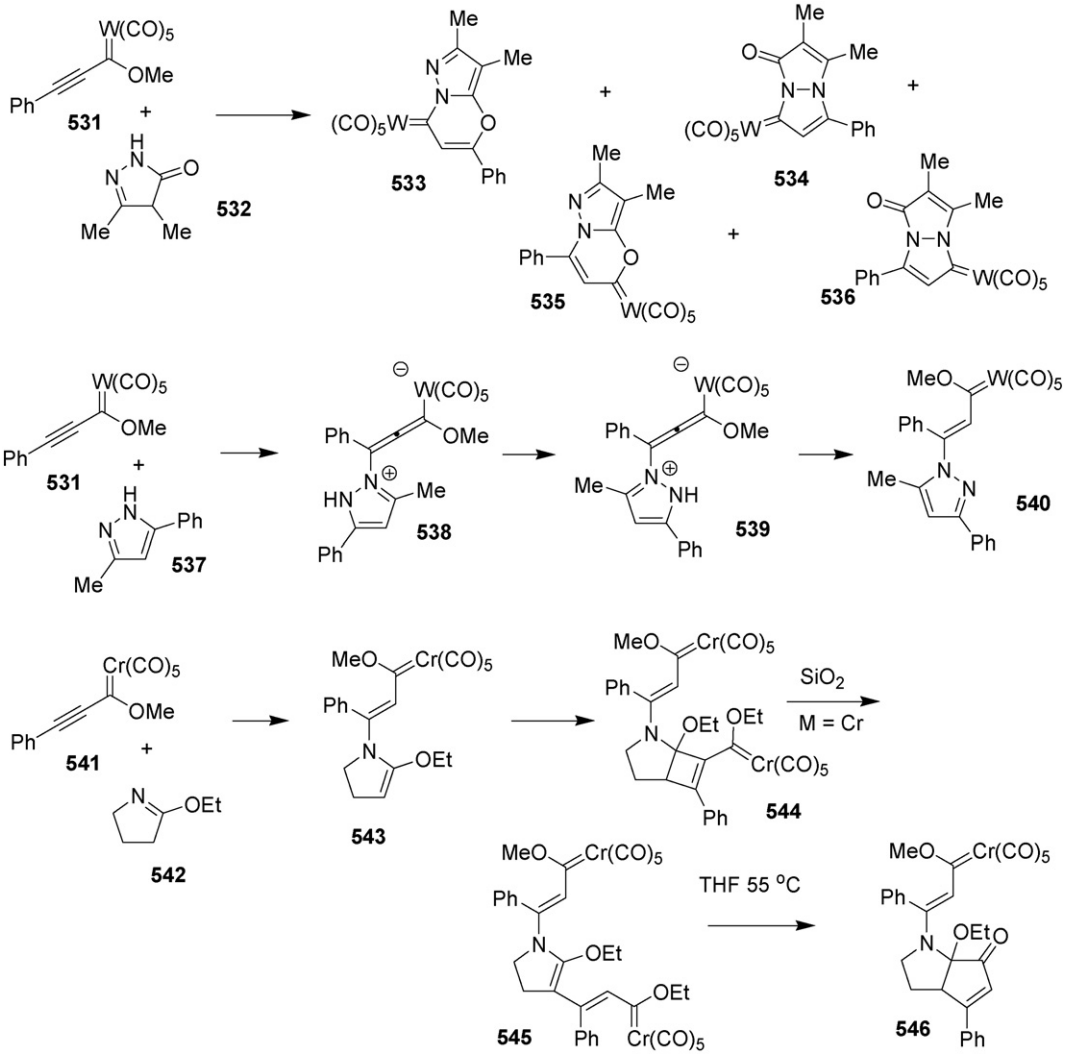
plexes as intermediates; also includes other process that involve the formation of a carbene complex from an alkyne and a noncarbene metal complex. The synthesis and reactivity of heterocycle-substituted allenylidene–chromium and tungsten complexes (*e.g.* **563–567**, Scheme 57) was reported [754]. These complexes were prepared from terminal pyridylalkynes (*e.g.* **560**) in a reaction sequence involving deprotonation and treatment with a $M(CO)_5$ source to generate anionic complex **562**, followed *N*-alkylation to afford the allenylidene complex (**563**). The complexes were compatible with the Sonogashira coupling and alkyne coupling. Highly polarized heterobimetallic systems were prepared (*e.g.* **566, 567**) and their electrochemical and nonlinear optical properties were evaluated. The reaction of chromium allenylidene complex **568** with tungsten pentacarbonyl sources was reported [755]. The reaction of chromium complex **568** with $(CO)_5W(THF)$ led to the transmetallated *N*-complex **570**. The mechanism was evaluated computationally. The key mechanistic event in the carbene transmetallation process is formation of the dimetallacyclopropane intermediate **569**. Formation of cyclic carbene complexes (*e.g.* **573**) through addition of bis(nucleophilic) compounds (*e.g.* **572**) to chromium γ-aminoallenylidene (*e.g.* **571**) or γ-alkoxyallenylidene complexes was also reported [756]. The reaction process involves addition to the γ-position followed by amine/alcohol elimination and cyclization. Formation of γ-alkoxyallenylidene–chromium complexes from alkynyl ester anions and subsequent nucleophilic substitution reactions at the γ-position were also reported [757].

Several examples of the synthesis and reactivity of tungsten vinylidene complexes featuring nitrosyl ligands (*e.g.* **578**, **580**, Scheme 58) were reported in 2006. The complexes were prepared through addition of alkynyllithium reagents to dicarbonyl complex **577** followed by treatment with electrophiles [758]. Reaction with diazo compounds led to the η^2-allene complexes (**579**). Deprotonation of the complexes led to anionic alkynyl complexes, whose one-electron oxidations were probed [759]. Reaction of vinylidene complexes (**580**) with various arsenic compounds was reported [760]. Reaction of the *t*-Bu complex **580** with arsenic compound **581** led to the three-membered ring arsenacyclic species (**582**) and tetrakis(dimethylamino)ethylene (**583**). Reaction of the phenyl complex **580** with the iron-arsenic species **584** led to the aminocarbene complex **585**. A mechanism involving electrophilic attack on the vinylidene complex to afford the intermediate **586**, followed by cyclization to the cyclopropane derivative **587**, followed by ring opening to the carbene–carbocation **588**, followed by addition of the amine and β-C–H insertion of the carbene was proposed.

The reaction of enyne–dienes (*e.g.* **590**, Scheme 59) with tungsten pentacarbonyl sources was reported [761]. This cyclization reaction leads to the hydroazulene system (*e.g.* **591**).



Scheme 53.



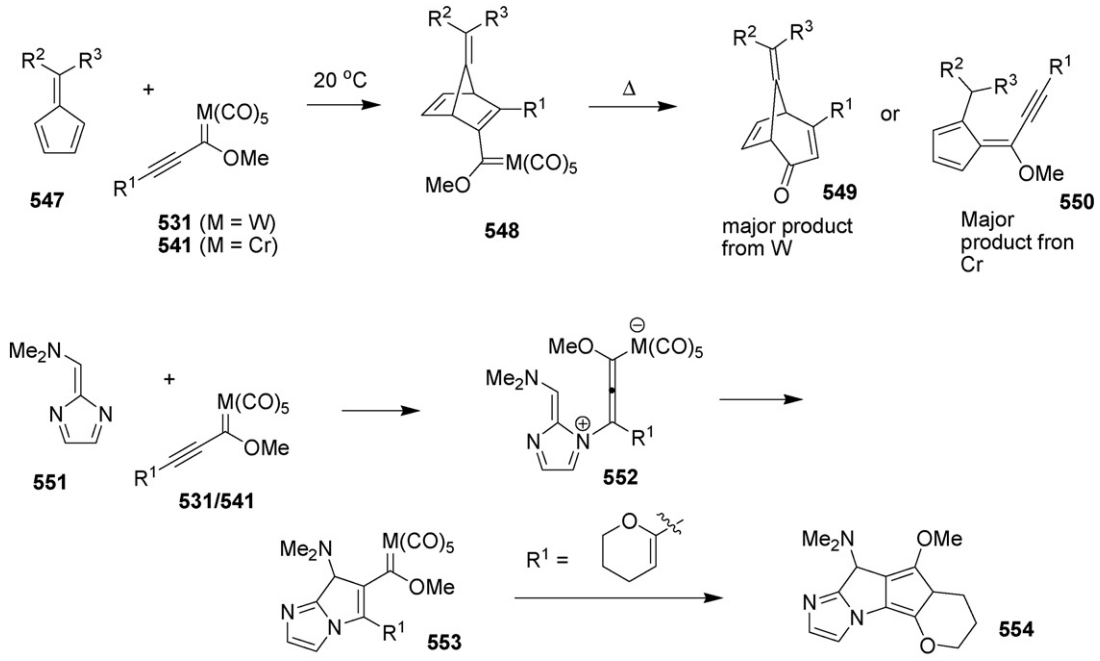
Scheme 54.

The proposed mechanism involves complexation to the alkyne followed by nucleophilic addition of the enol ether to afford the zwitterionic structure (**593**), which cyclizes through intramolecular coupling of the alkenyltungsten complex and oxonium ion moieties to afford the divinylcyclopropylcarbene complex (**594**), which undergoes Cope rearrangement and conversion of the unstabilized carbene unit to an alkene. Tungsten carbene complexes (*e.g.* **597**–**599**) were suggested as intermediates in the net [5 + 2]-cycloaddition of *o*-enynyl aniline imines (*e.g.* **595**) and enol ethers (*e.g.* **596**) in the presence of tungsten pentacarbonyl sources [762]. In this process, the initial reaction of the alkyne with the catalyst affords the 1,3-dipolar species **597**, which then undergoes stepwise cycloaddition followed by demetallation to afford the observed product **600**.

Tungsten(0) vinylidene complexes (*e.g.* **601**, **605**, Scheme 60) were suggested as intermediates in several processes involving the treatment of terminal alkynes with tungsten pentacarbonyl sources. Cyclization of C-propargylated β-lactams (*e.g.* **600**) was proposed to occur through tungsten vinylidene complexes (**601**) [763]. Conversion of the Fischer carbene complex **602** to the alkene complex **603** was studied computationally. The cyclization of C-propargyl-β-lactams using tungsten pentacarbonyl sources according to the proposed mechanism was supported by DFT calculations [764]. The alkylative cyclization of *o*-alkynylbenzoyl ketone derivatives (*e.g.* **604**) was proposed to occur through tungsten vinylidene (**605**) and Fischer carbene tungsten complexes (**606**) [765]. In this reaction, vinylidene formation and cyclization leads to benzopyrilidinium complex **606**, which reacts with various nucleophiles (*e.g.* Grignard reagents) to afford the anionic complex (**607**), which reacts with iodine and triethylamine to afford the ester derivative **608**.

The formation of tribranched polyacetylene derivatives (*e.g.* **613**, Scheme 61) was reported [766]. In this reaction, the tris(vinylidene) complex (**610**) is formed initially from the tris(alkyne) (**609**), which then undergoes reaction with phenylacetylene, followed by capping of the living polymer with an organometallic aldehyde (*e.g.* **612**).

2.3.3.9. Reactions involving carbanions derived from deprotonation of Group 6 metal–carbene complexes. The coupling of carbene complex-derived anions with pyrone-aldehydes (and



Scheme 55.

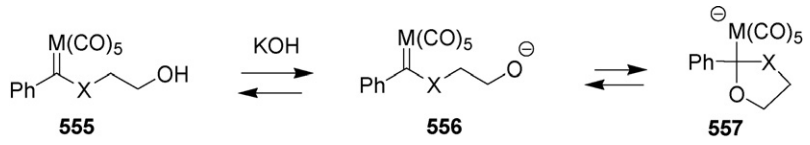
sulfur analogs) (*e.g.* **615**, Scheme 62) was reported [767]. The reaction leads to the extensively polarized conjugated carbene complexes (**617**), which were evaluated for their nonlinear optical properties. The synthesis of alkenylgold complexes (*e.g.* **621**) through reaction of carbene complex stabilized anions with $(Ph_3P)AuCl$ (**620**) was reported [768]. Aminocarbene complexes and thiocarbene complexes were both reported.

2.3.3.10. Reactions involving the addition of nucleophiles to the carbene carbon. The coupling of silylcarbene complexes (*e.g.* **625**, Scheme 63) with alkynyl anions was reported [769]. The reaction initially afforded the anionic complex **626**. The reaction with simple aldehydes afforded either dienes (**627**) or furans (**628**), depending upon the presence or absence of triethylaluminum. The proposed mechanism involves attack at the γ-carbon to afford intermediate zwitterions **630**, which undergoes a hydride shift to afford the ketone (**631**), which affords the product (**627**) after 1,5-silicon shift. Nucleophilic addition of oxygen to afford the dihydrofuran (**633**) followed by methanol elimination leads to the furan (**628**) in the presence of triethylaluminum. Reaction with α,β-unsaturated ketones led to cyclopentenols (*e.g.* **629**) in a mechanism involving Michael addition at the γ-position followed by cyclization.

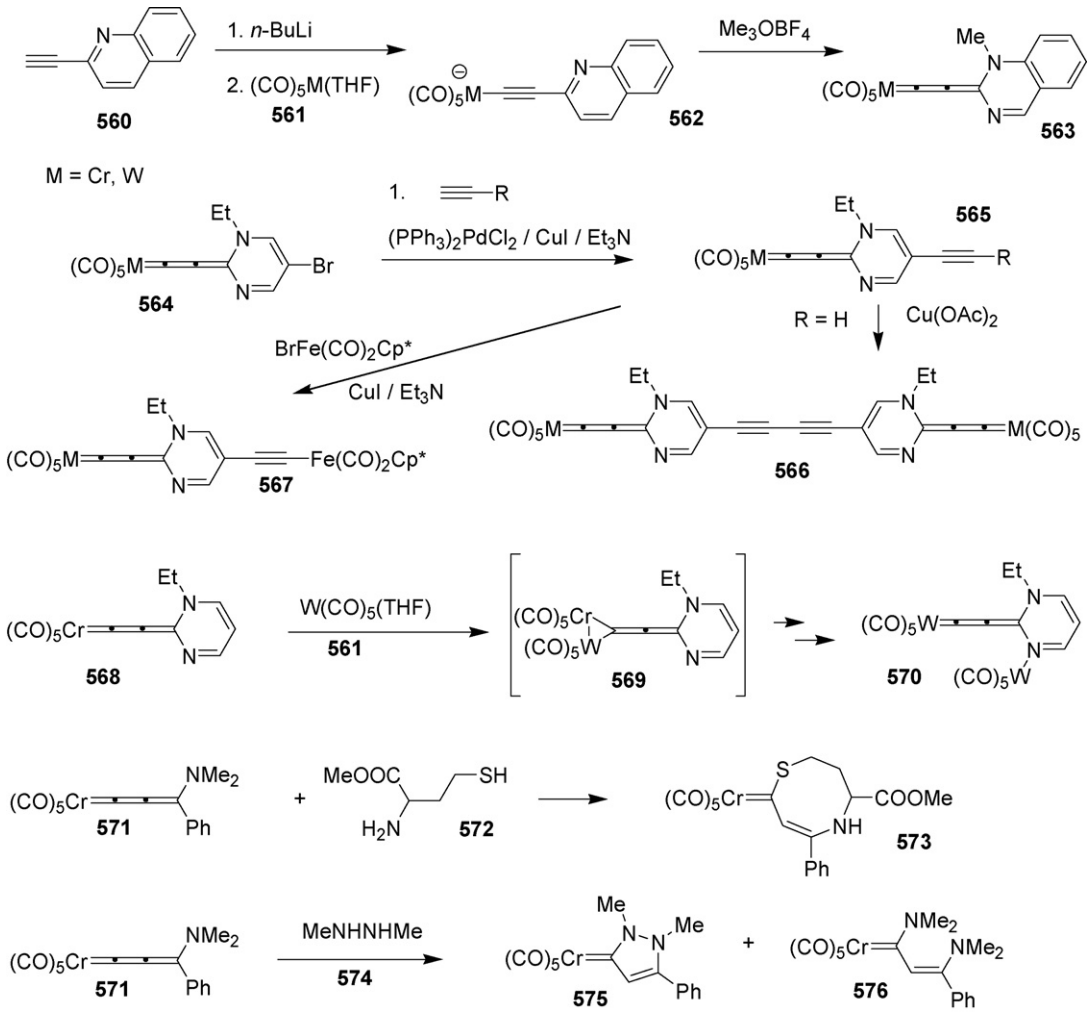
A net alkenylation reaction was observed when α,β-unsaturated carbene complexes (*e.g.* **635**, Scheme 64) were treated with ketone enolates (*e.g.* **636**) followed by iodine [770]. The reaction led to dienoate esters (*e.g.* **639**) and was predominantly E selective. A mechanism involving addition of the enolate to the carbene carbon, followed by reaction with iodine to form the β-iodoketone (**638**), followed by elimination was proposed. A complex ring-forming process involving the sequential addition of ester enolates (*e.g.* **640**) and allylmagnesium bromide to carbene complexes was also reported [771]. The mechanism for formation of the five-membered ring (**641**) involves initial formation of a tetrahedral intermediate (**643**), followed by addition of the allyl group to the resulting ester followed by alkene insertion. Addition of a second mole of Grignard to the ketone occurs prior to the proton quench. In the case of an acetate enolate, CO insertion accompanies the alkene insertion step to afford cyclohexanedione derivative **646**, which eventually leads to methylenecyclohexane derivative **642**.

Reaction of carbene complexes with ketene acetals (*e.g.* **648**, Scheme 65) under CO pressure was reported [772]. The reaction affords both a lactone (*e.g.* **649**) and an alkyne (*e.g.* **650**). The lactone arises through nucleophilic addition to the carbene carbon followed by formation of the unstabilized carbene complex (**653**), which then undergoes intramolecular C–H activation, reductive elimination, and *ortho* ester hydrolysis. Formation of the alkyne occurs through Michael addition followed by conversion of the resulting anionic complex (**655**) to the vinylidene complex (**656**) followed by conversion to the alkyne and *ortho* ester hydrolysis.

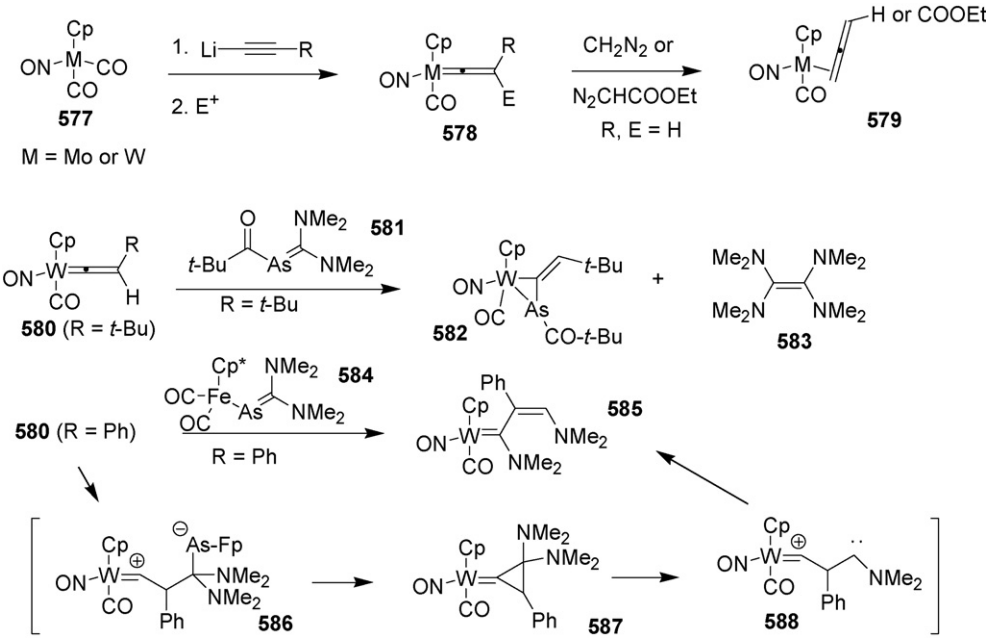
A novel allylation reaction was reported involving the reaction of allylic alkoxides and styrylcarbene complexes where the



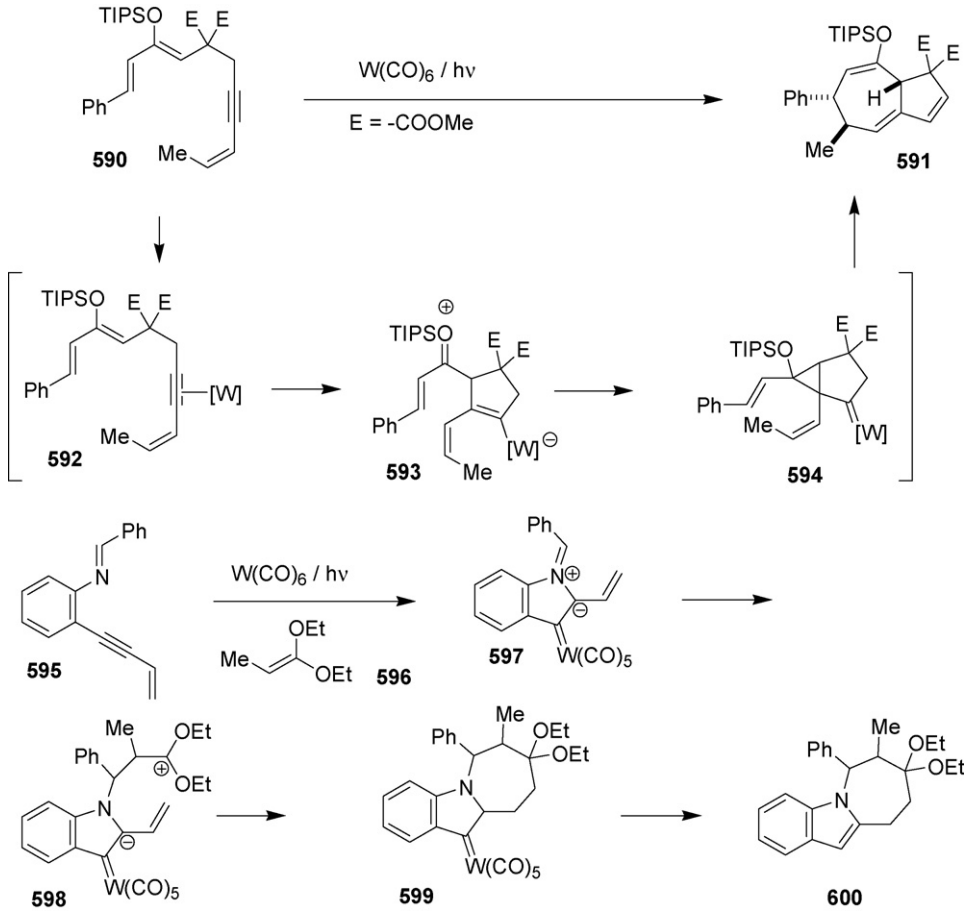
Scheme 56.



Scheme 57.



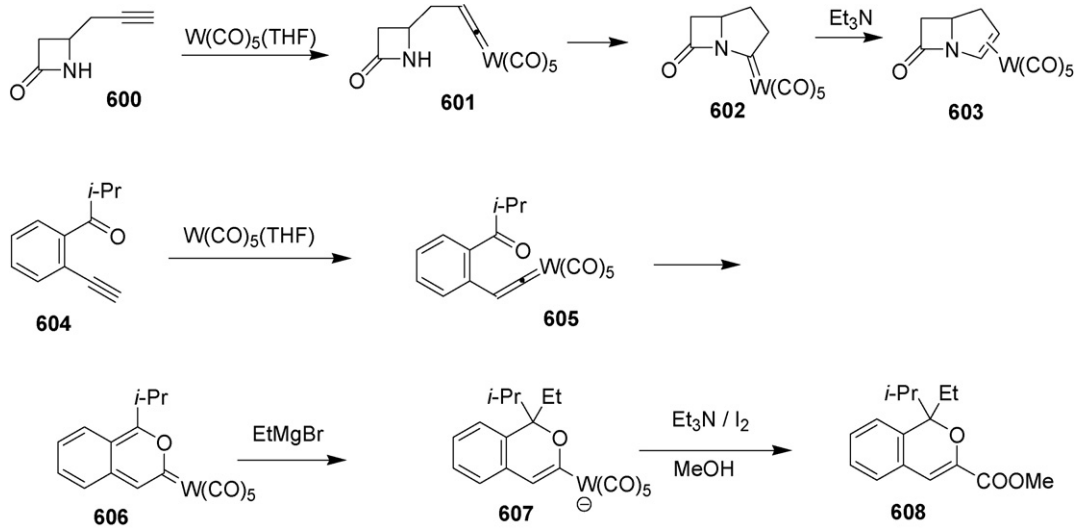
Scheme 58.



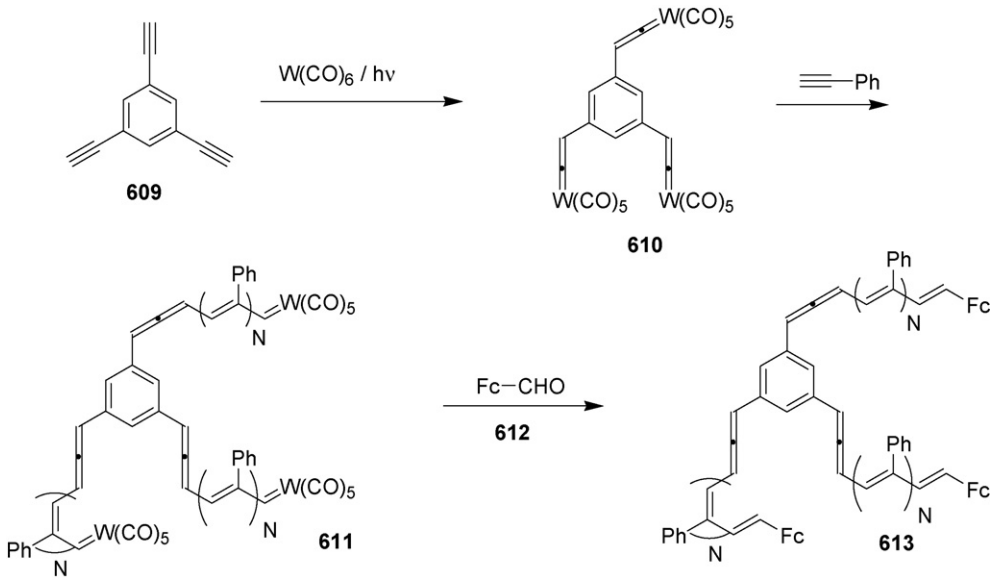
Scheme 59.

arene ring is complexed to the chromium tricarbonyl unit (*e.g.* **657**, Scheme 66) [773]. A mechanism involving addition of the alkoxide to the carbene carbon, followed by 1,3-chromium shift resulting in allyl vinyl ether (**660**), followed by Claisen rearrangement and demetallation was proposed. Chromium carbenes led to the reduced derivatives (**662**, X = H), while tungsten carbenes led to the alcohol derivatives (**662**, R = OH). High de's controlled by the chiral chromium arene functionality were observed.

2.3.3.11. Reactions that involve transfer of a Fischer carbene ligand to another metal. The coupling of alkenylcarbene



Scheme 60.

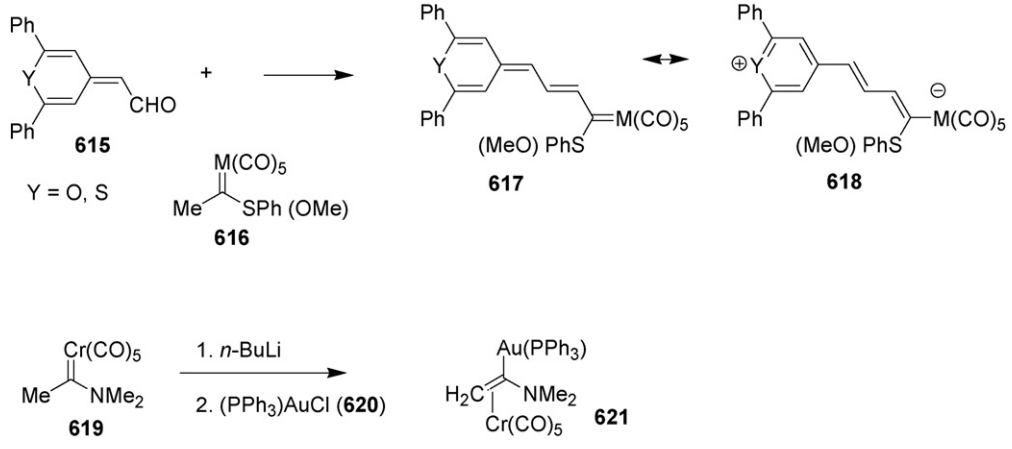


Scheme 61.

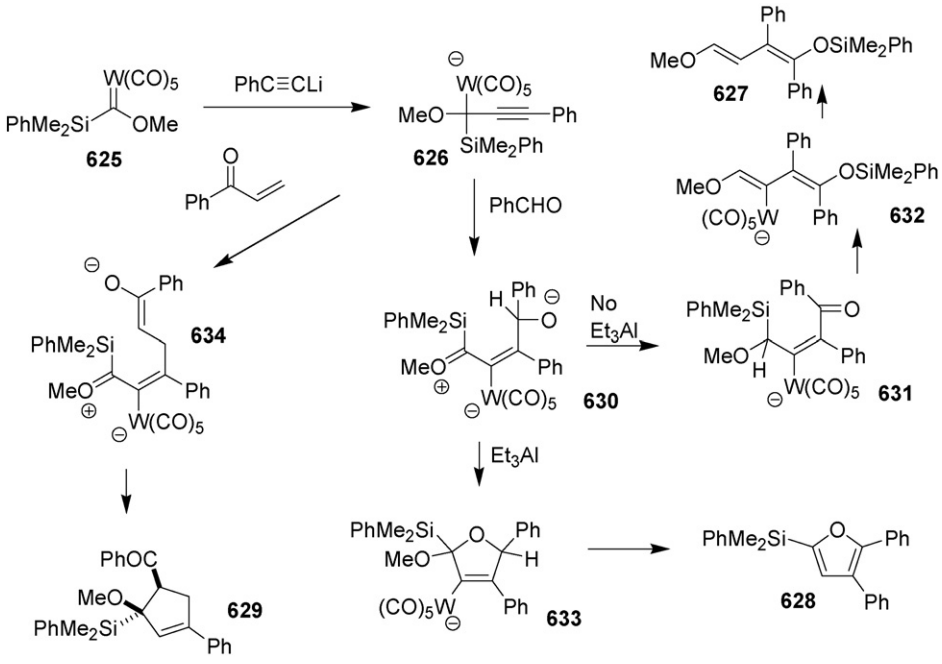
complexes (*e.g.* **525**, Scheme 67) with allenes (*e.g.* **665**) in the presence of a rhodium catalyst (**666**) was reported [774]. The reaction process results in the formation of alkylidenecyclopentene derivatives (*e.g.* **667**). A mechanism involving transmetallation to form a rhodium carbene complex (**668**), followed by metalla Diels–Alder reaction, followed by reductive elimination was proposed. Isolation of a rhodium carbene complex (**671**) from the coupling of chromium complex **525** and rhodium–NHC complex **670** was reported [775]. Thermal decomposition of carbene complex **671** leads to dimeric rhodium complex **673** and the carbene dimerization product **674** The same rhodium complex also catalyzes cycloaddition reactions between α,β-unsaturated carbene complexes and allenes.

The coupling of two alkynes (or a dialkyne) (*e.g.* **677**, Scheme 68) with a Fischer carbene complex (*e.g.* **504**) in the presence of a nickel catalyst was reported [776]. The reaction affords cyclopentadiene derivatives (*e.g.* **679**). A mechanism involving transfer of the carbene ligand to form the nickel carbene complex (**680**), followed by formation of a nickelacyclopentadiene (**681**), followed by alkyl migration and reductive elimination was proposed. Nickel carbene complexes were also proposed in the formation of cyclopentanone (*e.g.* **683**) through nickel catalyzed cycloaddition reaction of carbene complexes (*e.g.* **525**) with methylenecyclopropanes (*e.g.* **682**) [777]. An alternative mechanism involving nickel-catalyzed cyclopropane ring opening was also considered. The preparation of palladium carbene complexes (*e.g.* **686**) through transmetallation from Fischer carbene tungsten complexes (*e.g.* **684**) was reported [778]. The aminocarbene complexes (**686**) transform to the corresponding iminium salts (**687/688**) upon heating to 50 °C.

2.3.3.12. Other reactions of Group 6 metal–carbene complexes. Photoinduced reactions of phosphine-chelated aminocarbene complexes (*e.g.* **690**, Scheme 69) were reported [779]. The reaction affords either the imine-coordinated compound **691** or the carbene excision product **692**, depending on the substitution at nitrogen. Various mechanistic pathways were evaluated com-



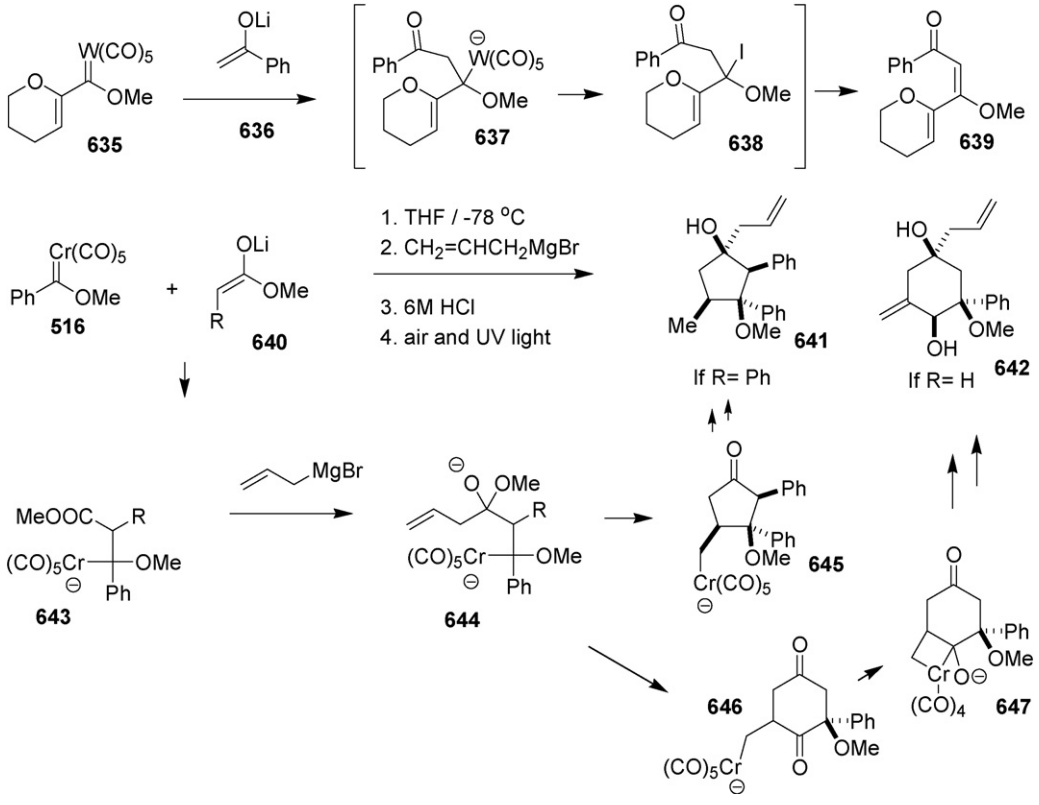
Scheme 62.



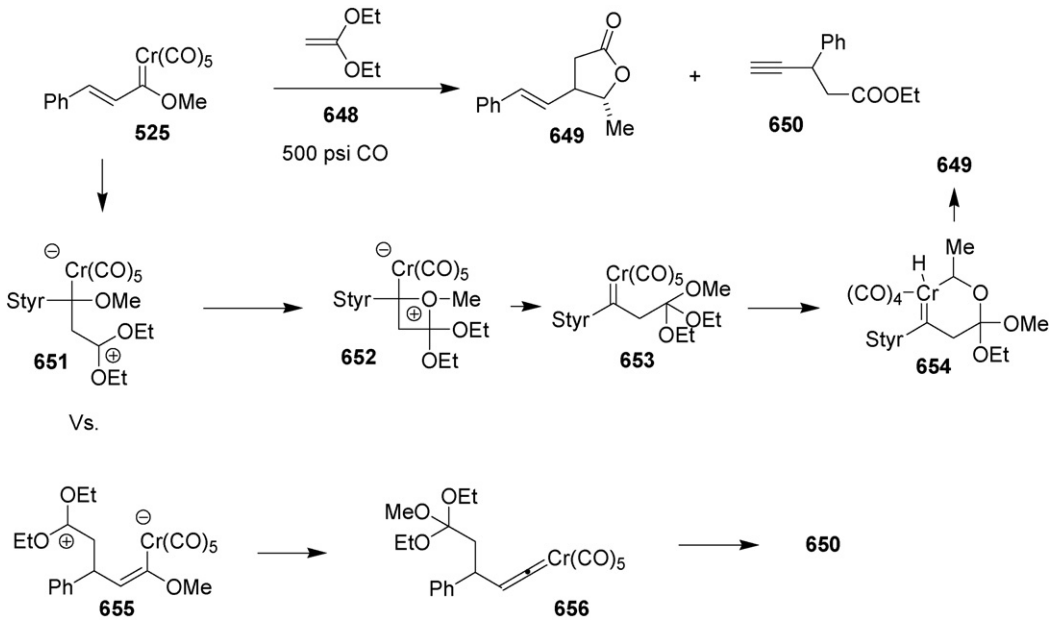
Scheme 63.

putationally. A free radical mechanism was determined to be the most energetically reasonable reaction pathway. The imine complexes arise through rearrangement of the initially generated diradical **693** to the free carbene **694** followed by β-N–H insertion. The carbene excision product arises through cleavage of the C–N bond to form diradical **695** followed by reduction of the diradical to form intermediate **696** which then loses the carbene ligand to afford **692**. Numerous other carbene complexes that do not engage in photochemical ketene generation were also examined.

The coupling of molybdenum dialkyl complexes (*e.g.* **697**, Scheme 70) with cycloalkenes was reported [780]. The reaction



Scheme 64.

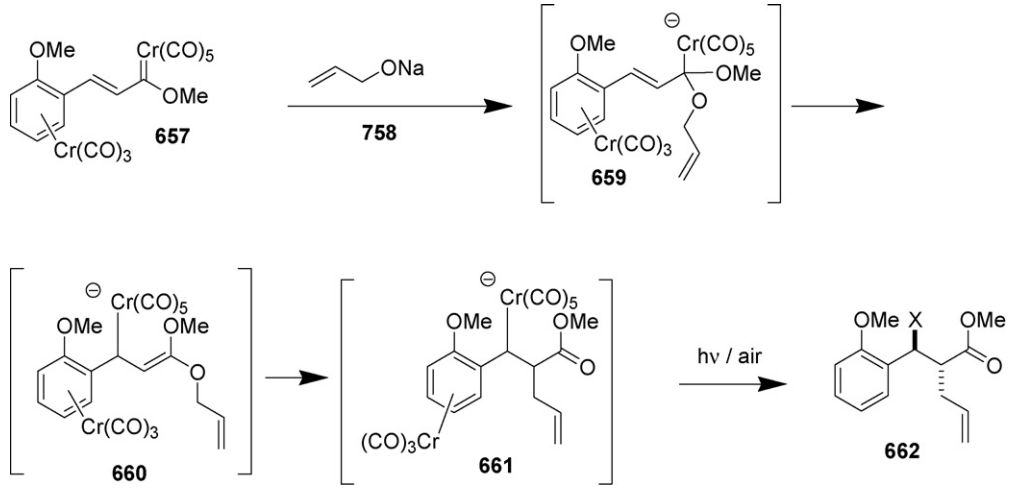


Scheme 65.

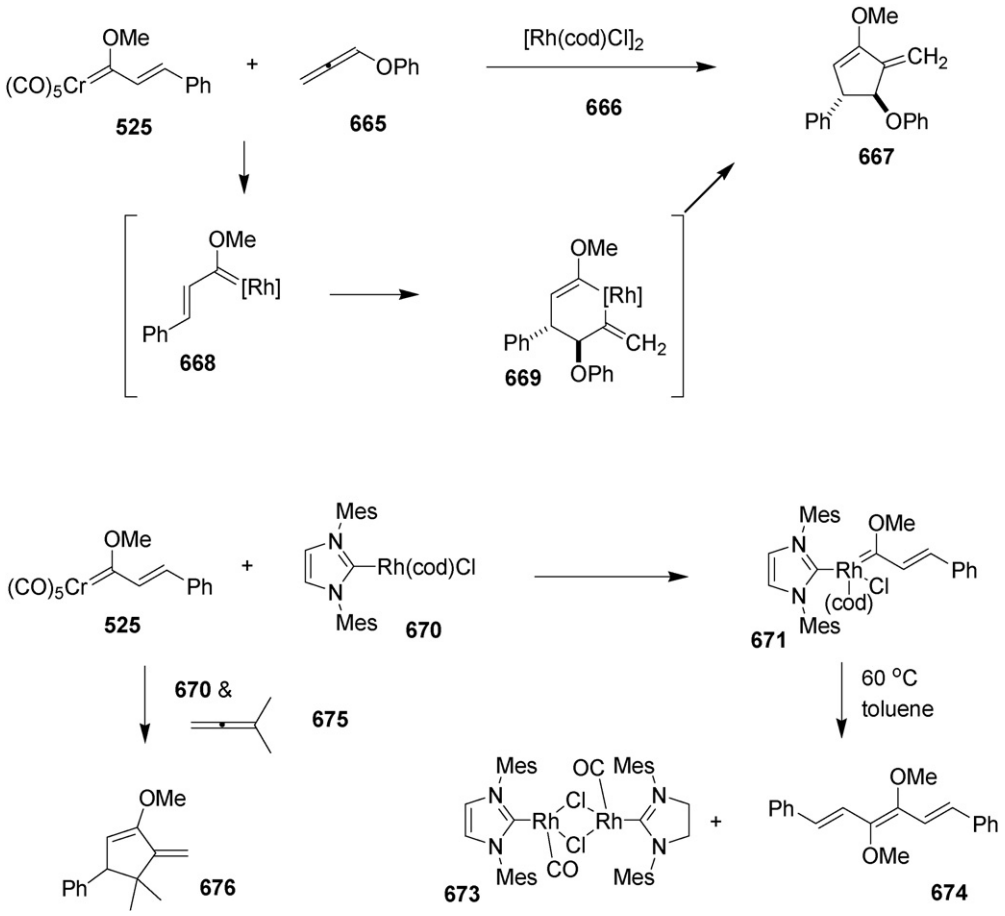
course was highly dependent on ring size, however all processes occur through α-hydride elimination followed by reductive elimination to afford carbene complex intermediate **698**. Reaction with cyclopentene led to the metallacyclobutane **706** in a pathway involving [2 + 2]-cycloaddition to carbene complex **698**. Reaction with cyclohexene led to a mixture of *cis*- and *trans*-metallacyclobutanes (**703/704**) plus the internally coordinated π-allyl complex **702**. Formation of the *trans*-metallacyclobutane was proposed to occur through a reversible β-hydride elimination process (via intermediate **705**). Reaction with cyclooctene led primarily to the *trans*-metallacyclobutane (**699**), which transformed to the internally coordinated π-allyl complex (**700**) upon heating. Further heating a higher temperature in the presence of pyridine afforded η^2-diene complex **701**.

Reaction of analogous tungsten complexes (*e.g.* **707**, Scheme 71) with unsaturated organic compounds was also reported [781]. Reaction with monosubstituted benzene derivatives led to the aryl C–H activation products **709**. A mechanism involving α-hydride elimination and reductive elimination to afford carbene complex **708**, followed by C–H activation and hydride migration was proposed to account for the formation of **709**. Predominantly the *ortho* isomer was formed as a result of the C–H activation. Thermolysis in the presence of 3-hexyne led to the double insertion product **710**. Formation of **710** was proposed to occur through insertion of two alkyne units into carbene complex intermediate **708**.

Other experimental studies of Group 6 metal–carbene complexes are depicted in Scheme 72 and include: (1) the reaction of carbene complexes (*e.g.* **516**) with 1,2-dithiole-3-thione derivatives (*e.g.*, **711**) to afford S–S insertion products (*e.g.* **712**) [782]; (2) the reaction of tungsten–calixarene complex **713** with excess diphenylacetylene to afford alkenylcarbene complex **714** [783]; (3) conversion of alkoxycarbene complexes (RC[=Cr(CO)$_5$]OMe) to the analogous sulfide (RC(=S)OMe)



Scheme 66.



Scheme 67.

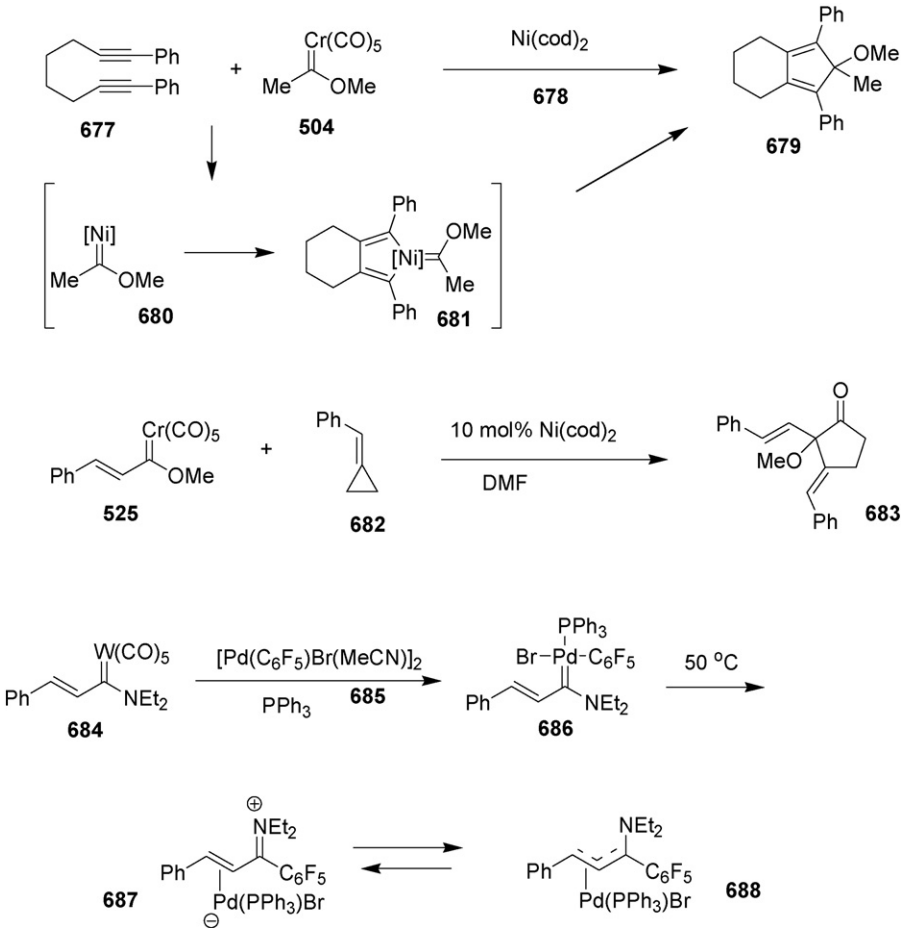
or selenide (RC(=Se)OMe) using S(e)=C=O [784]; (4) oxidation of carbene complexes to the corresponding organic carbonyl compounds using pyridine *N*-oxide (in many cases this reagent was selective for oxidizing one carbene complex group of a bis(carbene) complex) [785]; (5) preparation of tungsten carbene complexes attached to a polyethylene glycol system and subsequent thermolysis in the presence of gold(III) salts to afford gold nanoparticles [786]; (6) acquisition of the crystal structure of MeC(S–*n*-Pr)=Cr(CO)$_5$ [787]; (7) a study of barriers to rotation in internally coordinated carbene complexes of general structures **715** and **716** [788]; (8) discussion of carbene resonance contribution to tungsten–thioacetylene complexes [789]; (9) the guided beam mass spectrometer reaction of molybdenum cations with methane and DFT studies of the possible molybdenum–carbon bonded species [790]; and (10) formation of carbene and carbyne complexes through the coupling of laser-ablated molybdenum and tungsten atoms with methyl halides and comparison of their IR spectra with spectra calculated by DFT [791].

2.3.4. Group 7 metal–carbene complexes

Alkynylcarbene–rhenium complexes (*e.g.* **721**–**723**, Scheme 73) were prepared using the Fischer synthesis applied to decacarbonyldirhenium (**720**) [792]. Both aminocarbene and alkoxycarbene complexes were reported. Reaction of the alkoxycarbene complex **721** with ammonia led to the Michael addition product (**722**). Fluoride-induced desilylation reactions were successfully applied to the carbene complexes.

The reaction of rhenium carbene complex **724** (Scheme 73) with acetonitrile to afford nitrile insertion products was reported [793]. The reaction with acetonitrile afforded the benzonitrile complex **728** at low temperature, which cyclized to the chelating complex **729** at room temperature. Formation of complex **728** was proposed to occur through nitrosyl insertion to afford complex **726**, followed by rearrangement to the NO-ligated complex **727**, followed by a hetero-ene reaction with exogenous acetonitrile. Oxidation of related carbene complexes (*e.g.* **730**) at the nitrosyl ligands was also reported [794] (Scheme 74).

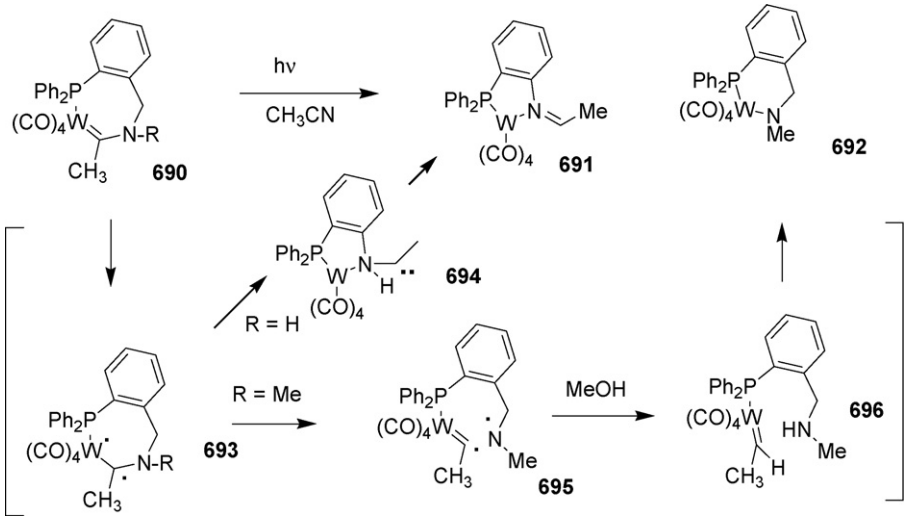
A DFT study comparing rhenium carbene–carbyne complexes (*e.g.* **734**, Scheme 75) and isoelectronic tungsten carbene-imido complexes (**733**) was reported [795]. Both complexes feature agostic interactions with the carbene C–H bond. However the imido is a weaker donor ligand and the agnostic interaction is stronger in the tungsten carbene complexes. The superior performance of tungsten and molybdenum complexes as metathesis catalysts relative to rhenium analogs was attributed to more favorable ligand properties of the imido ligand. A related study of silica-bound rhenium carbene–carbyne complexes was also reported [796].



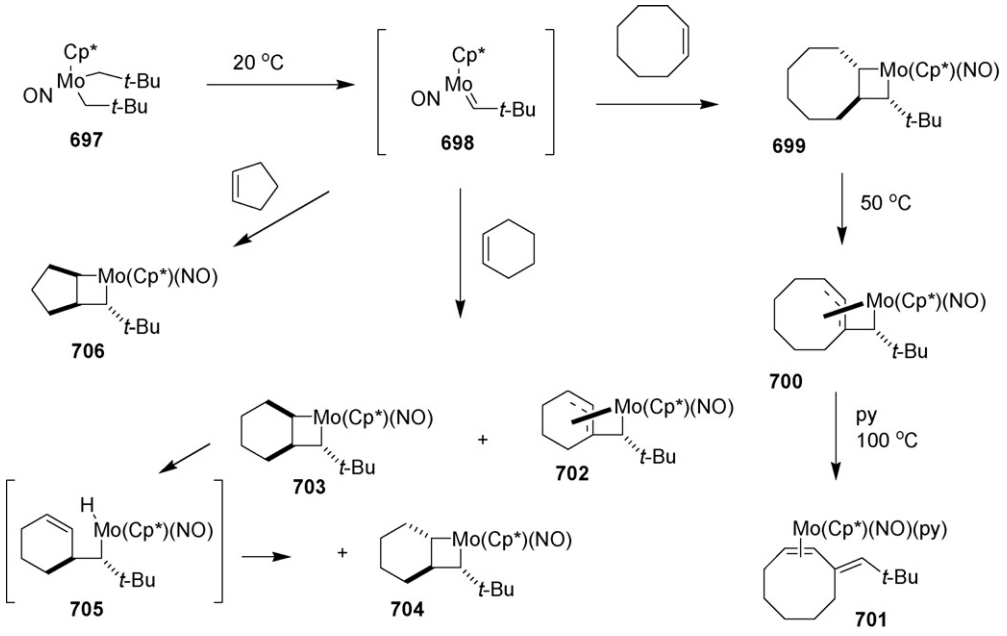
Scheme 68.

Cationic manganese carbene complexes (*e.g.* **736**, Scheme 76) were generated *in situ* through the alkylation of methoxymethylmanganese complex **735** with methyl triflate in the presence of styrene [797]. The product of this reaction is phenylcyclopropane (**738**) and manganese triflate **737**; these products presumably arise from generation of the carbene complex **736** followed by cyclopropanation.

Manganese carbene complexes (*e.g.* **740**, **744**, Scheme 77) were suggested as intermediates in the reaction of manganese–chromium complex **739** with organolithiums followed by methyl triflate, or with diazo compounds [798]. The organolithium reagents add to both the pyridine ring and to the manganese carbonyl ligand. Reaction with the stable carbene led to the simple carbene complex **743**.



Scheme 69.



Scheme 70.

Rhenium carbene complex **752** (Scheme 78) was proposed as an intermediate in the rhenium-catalyzed ring expansive coupling of β-keto-esters (*e.g.* **746**) and alkynes [799]. The key step in the expansion process is the conversion of metallacyclopentene **750** to the hydroxycarbene complex **752**.

The kinetic and thermodynamic acidity of neutral and cationic rhenium carbene complexes (*e.g.* **753**, Scheme 79) was reported [800]. The pKa of complex **753** was determined to be 2.64, and considerably less acidic that carbene complexes where the metal is at the two-position of a dihydrothiophene ring.

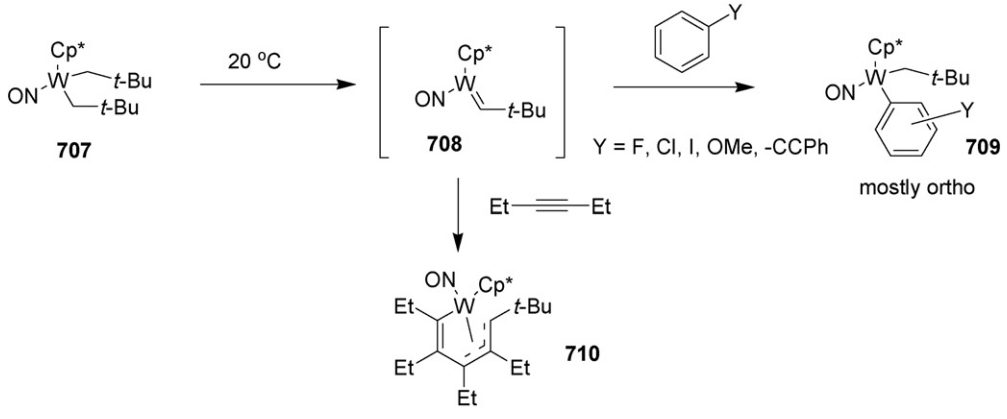
Several examples of Group 7 metallacumulene complexes were reported in 2006 (see Scheme 80). See Scheme 96 for the general reactivity profile of metal vinylidene complexes and metal–allenylidene complexes. The reaction of rhenium allenylidene complexes (*e.g.* **755**) with various nitrogen heterocycles (*e.g.* **756**, **759**) was reported [801]. The reaction leads to new heterocyclic rings (*e.g.* **757**, **760**), which then lead to simple propargyl substitution products (*e.g.* **758**, **761**) upon treatment with base. A mechanism involving the reaction of the heterocycle at the γ-position to generate the alkynyl complex (**758**, **761**), followed by proton transfer and formation of the vinylidene and a second nucleophilic addition was proposed. Reaction of the alkynylrhenium complexes with triflic acid regenerated the starting allenylidene complexes.

2.3.5. Group 8 metal–carbene complexes

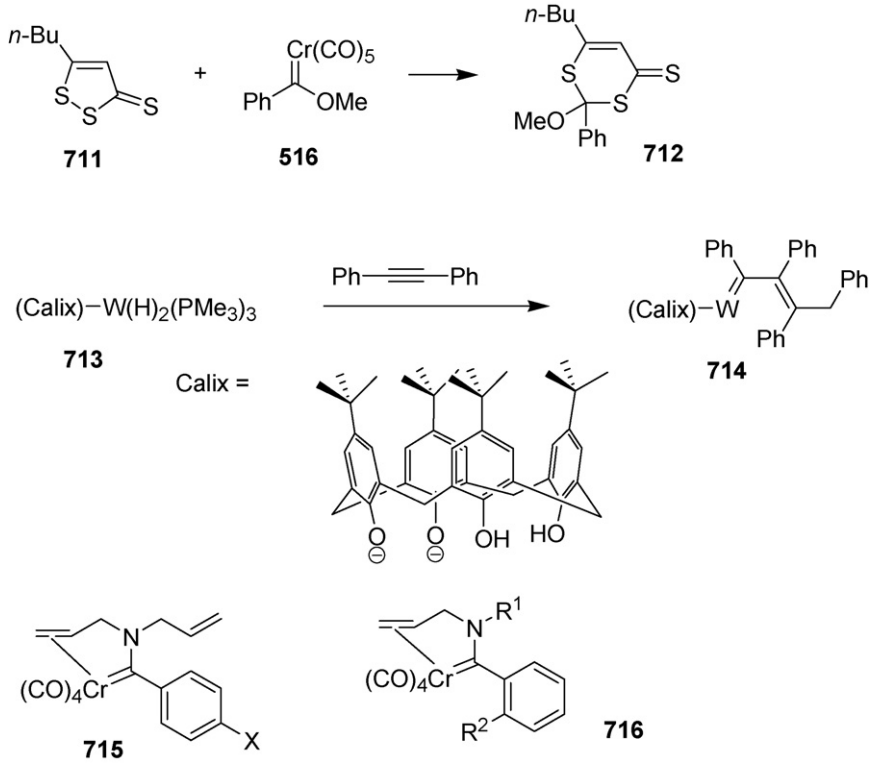
2.3.5.1. Cationic metal–carbene complexes that are notmetalla cumulenes.

Osmium carbene complexes (*e.g.* **766**, Scheme 81) were suggested as intermediates in the conversion of cyclopropenylosmium complex **765** to the hydroxycarbene–osmium complex **768** [802]. Formation of the hydroxycarbene complex was reversible. The optimal formation of cyclopropylosmium complex **767** involves treating the hydroxycarbene complex **768** with aqueous sodium hydroxide.

The generation and reactions of cationic bridging carbyne–diiron complexes (*e.g.* **770**, **774**, Scheme 82) were reported [803]. Treatment of bridging carbene complex **769** with acid at low temperature led to the bridging carbyne



Scheme 71.

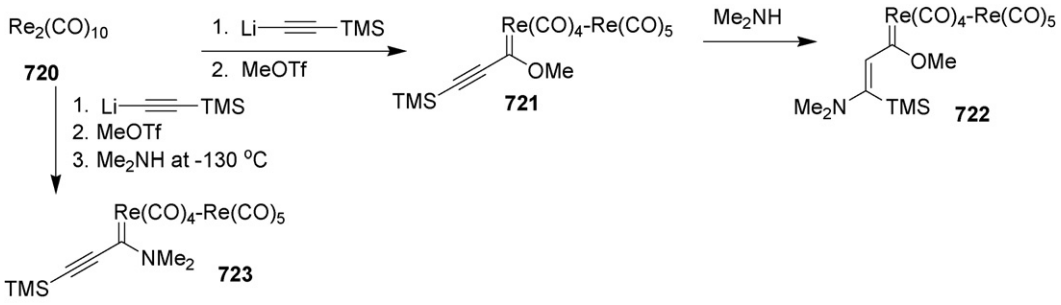


Scheme 72.

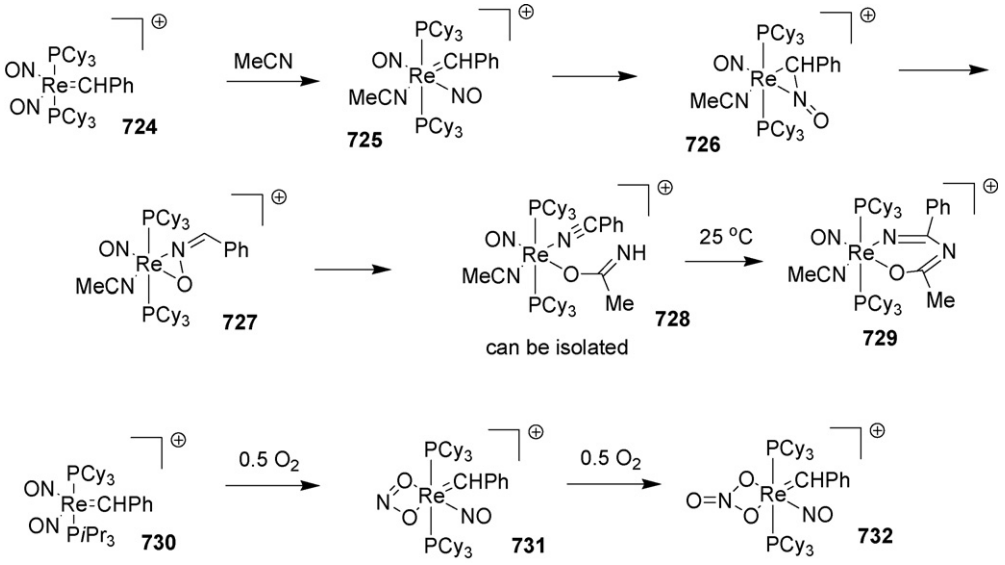
complex **770**. Treatment of the carbyne complex with nucleophiles led to neutral bridging carbene complexes (*e.g.* **771**, **772**). Similar reaction processes were reported for analogous fulvene-bridged complexes (*e.g.* **774**) [804].

Additional studies of cationic Group 8 carbene complexes include: (1) cyclopropanation of alkenes using propargyl esters and $CpRu(PPh_3)_2Cl$, which may or may not proceed through carbene complex intermediates [805]; (2) a mechanistic proposal that ruthenium carbene complexes are involved in the coupling with skeletal rearrangement of benzoxanorbornenes and alkyne–esters [806]; and (3) a mechanistic proposal that diphenylvinylcarbene–osmium complexes are involved in ring opening reactions of η^2-diphenylcyclopropene–osmium complexes [807].

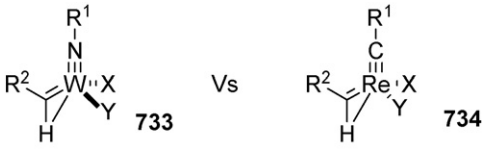
2.3.5.2. Bis(carbene)ruthenium complexes from coupling of two alkynes and a ruthenium complex. Cationic tris(carbene) ruthenium complexes (*e.g.* **777**, Scheme 83) were generated from coupling of ruthenium NHC complex **775** with alkynes or bis(alkynes) (*e.g.* **776**) [808]. The reaction leads to π-allyl(carbene) complexes (*e.g.* **778**) in a process involving formation of the cyclic bis(carbene) complex (**777**) followed by addition of the NHC ligand to the one of the carbene complex groups. The bis(carbene) complex intermediates were observable by low temperature NMR. Formation of an isolatable cationic ruthenium carbene complex (**780**) through reaction of ruthenium complex **775** with a bis(internal alkyne) (**779**) was reported [809]. Reaction with wet chloroform leads to alkene-coordinated carbene complex **781**. Related reaction processes were reported for bis(complex) intermediates that feature an aminophosphine ligand (*e.g.* **783**) [810]. The reaction of complex **782** with bis(alkynes) at room temperature led to the chelate complex **786**. Heating the product at 90 °C leads to the isomeric complex **787**, which leads to the Fischer carbene complex **788** upon refluxing in acetonitrile. The proposed mechanism was supported by DFT calculations.



Scheme 73.



Scheme 74.



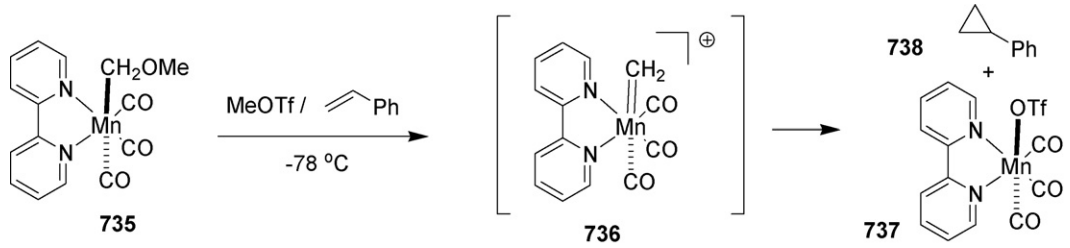
Scheme 75.

The coupling of bis(alkyne) **789** (Scheme 84) and alkynylborane derivative **790** in the presence of ruthenium complex **791** led to the alkyne trimerization product **792** [811]. The regioselectivity in unsymmetrical systems was attributed to steric interactions in the metallacyclobutene (**794**) obtained from the bis(carbene) complex (**793**). A related coupling of bis(iodoalkyne) derivatives with alkynes to afford 1,4-diiodobenzene derivatives was reported [812]. Several related alkyne trimerization reactions were also reported [813,814]. Related coupling of bis(alkynes) with an alkene to form cyclohexadiene derivatives was also reported [815]. Formation of pyridines (*e.g.* **796**) through coupling of bis(alkyne)s (*e.g.* **789**) and nitriles (*e.g.* **795**) in the presence of ruthenium complex **791** was also reported [816]. The greatest success was realized with nitriles that contain a secondary ligation site due to the proposed involvement of a dimeric complex as the catalytically active species. Coupling of bis(alkynes) or two alkynes with a nitrile to form pyridine derivatives was evaluated computationally [817].

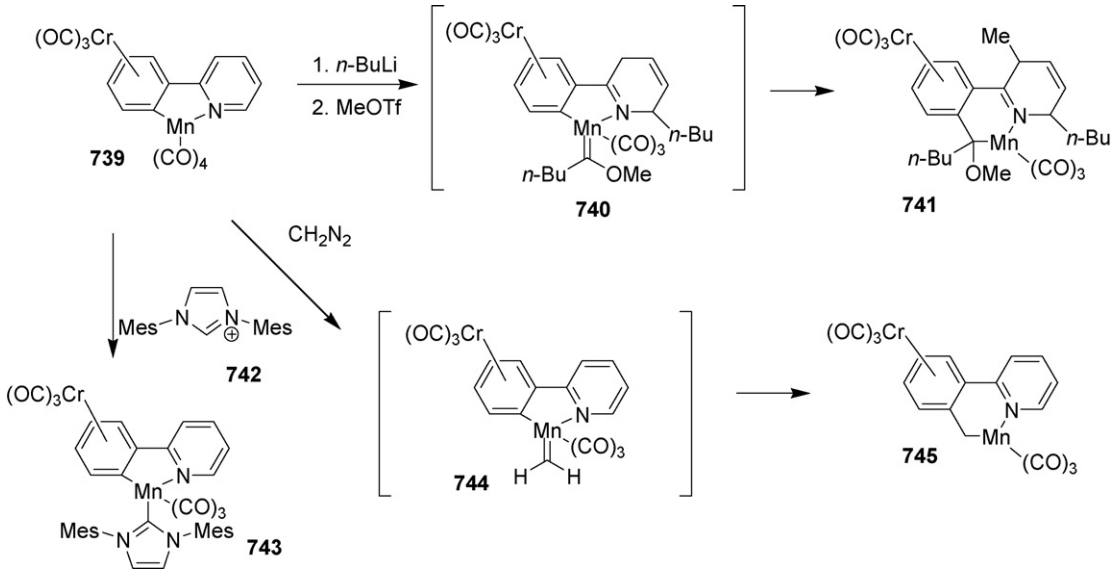
Several reaction processes invoke cyclic ruthenium bis(carbene) complexes as reactive intermediates (Scheme 85). Several examples of the hydrative cyclization of bis(alkynes) (*e.g.* **800**) using cationic ruthenium complex **801** were reported [818]. Reaction of bis(alkyne) derivative **800** with cationic complex **801** leads to cationic bis(carbene) complex **802**, which reacts with water to afford the cyclized product **803**. A ruthenium bis(carbene) complex (**805**) was proposed as an intermediate in the formation of complex ruthenacycle **806**, obtained from the coupling of carboranylruthenium complex **804** and three terminal alkyne units [819].

2.3.5.3. Neutral nonheteroatom-substituted metal–carbene complexes that are not cumulenes. 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.

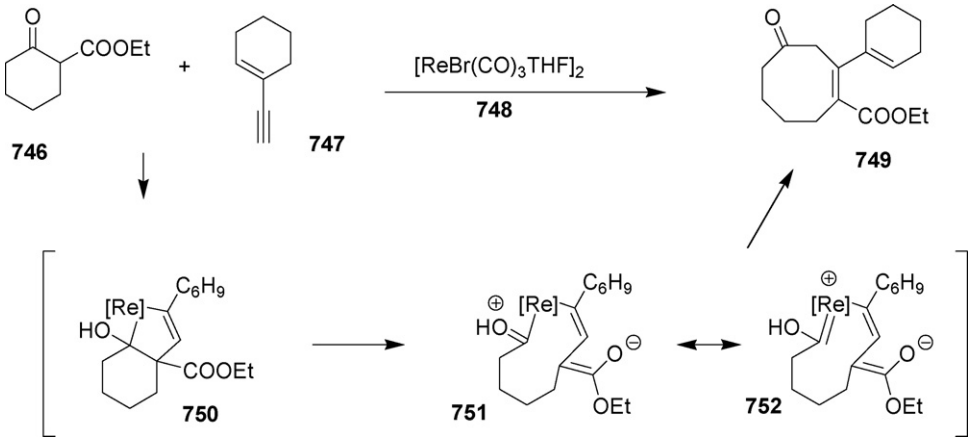
Stoichiometric reaction of Grubbs catalysts I and II with conjugated polyenes (*e.g.* **808**, Scheme 86) was reported [820]. In the stoichiometric reaction with conjugated polyenes, it was noted that Grubbs catalyst I was more reactive than Grubbs catalyst II, which reflects the faster initiation rate of Grubbs catalyst I. The E/Z isomerization process was accelerated by tricyclohexylphosphine, which was attributed to a reversible nucleophilic addition to the polyene ligand. Formation of



Scheme 76.



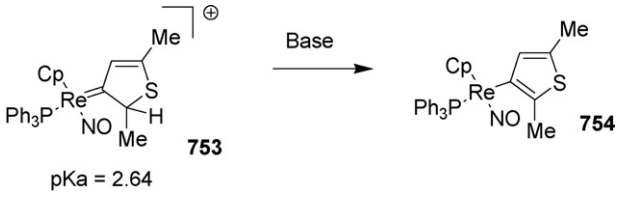
Scheme 77.



Scheme 78.

bis(carbene) complexes from hexatriene and polyacetylene was successful, however the bis(carbene) complex could not be prepared from butadiene. The electrochemical properties of the polyene–monocarbene and bis(carbene) complexes was also examined.

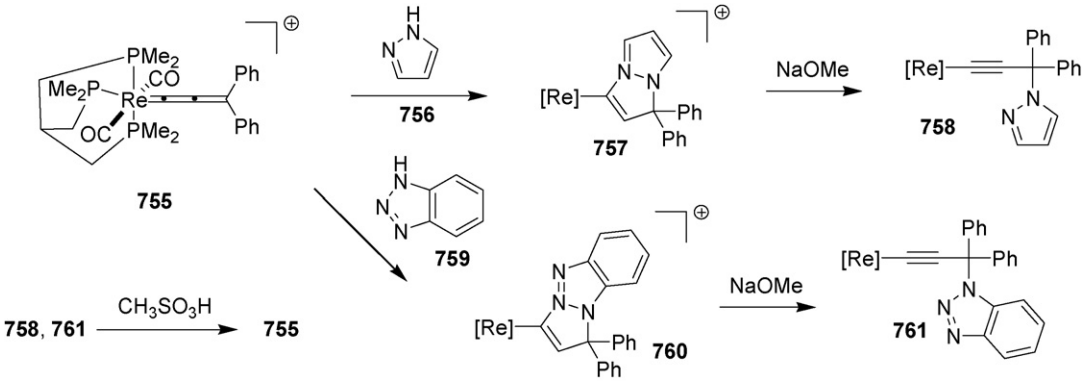
Pincer-ligated ruthenium–carbene complexes (*e.g.* **813**, Scheme 87) were reported [821]. Reaction of the bis(phosphine) complex **811** with ruthenium complex **812** and triethylamine led directly to the hydridoruthenium carbene complex **813**. A variety of reaction processes were reported for the carbene complex. Reaction with a variety of ligand additives led to hydrogen migration products (*e.g.* **814**). Reaction with benzophenone led to the reduction product, alcohol **815**. Reaction with phenylacetylene led to the alkyne insertion product (**816**), which upon further heating led to alkyne dimerization products **817** and **818**.



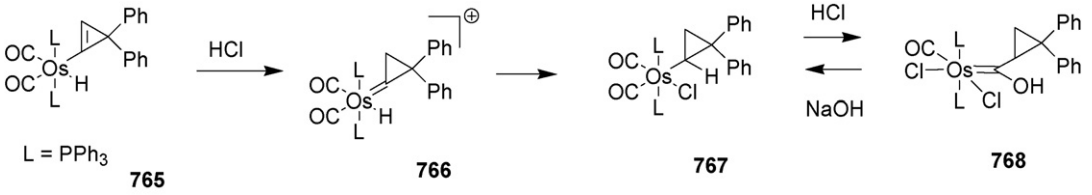
Scheme 79.

Ruthenium carbene complexes (*e.g.* **822**, Scheme 88) were suggested as intermediates in the reaction of propargylic esters (*e.g.* **819**) with methoxyfurans to stereoselectively afford triene–esters (*e.g.* **824**) [822]. Formation of carbene complex **822**, followed by cyclopropanation, followed by ring opening was proposed. Related ruthenium carbene complexes (*e.g.* **828**, **829**) were proposed as intermediates in the isomerization of bis(propargyl esters) (*e.g.* **825**) to conjugated dienynes (*e.g.* **826**) [823]. Formation of an alkynylcarbene complex (**828**) through rearrangement of the complexed propargyl ester followed by 1,3-shift of the alkynylcarbene led to carbene complex **829**, which leads to the observed product **826** after 1,2-shift of the acetate and demetallation.

Ruthenium carbene complexes were proposed as intermediates in the hydrative cycloisomerization of enynes (Scheme 89)



Scheme 80.

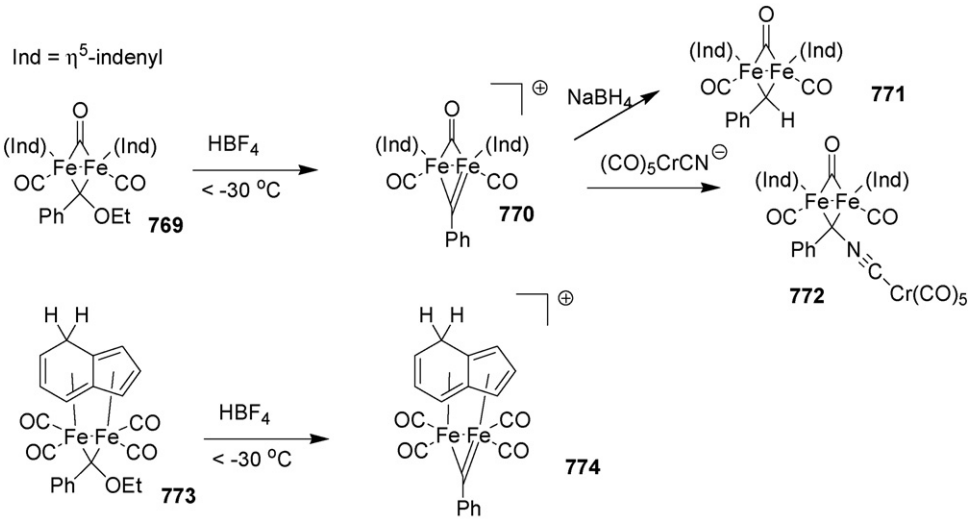


Scheme 81.

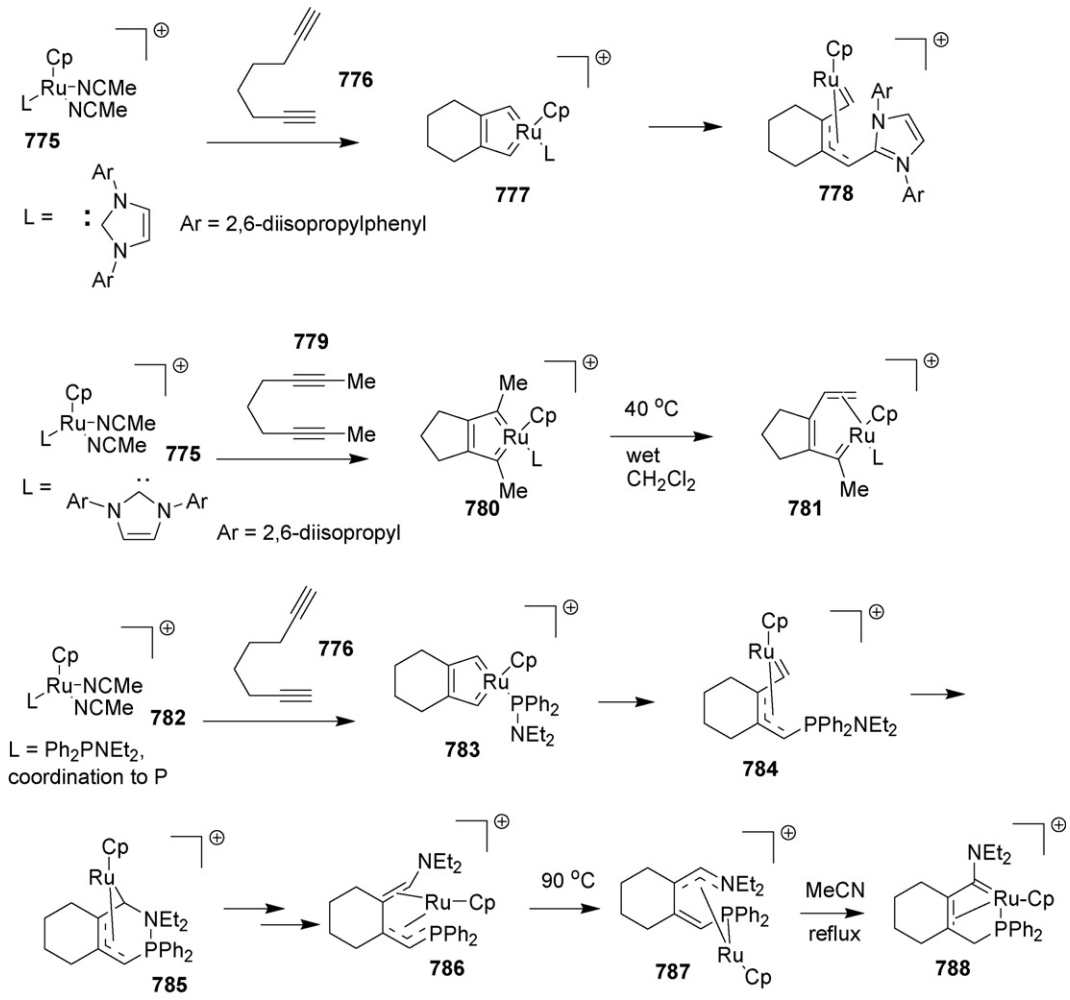
[824]. The mechanism for formation of the three product types **832**–**834** involves addition of either of the carbenylcarbene complex intermediates (**835**, **838**) to the alkene group to form either of the isomeric cyclopropylcarbene complexes (**836**, **839**), which then undergo cyclopropane ring opening processes to afford the observed products. The mechanistic hypotheses were supported by deuterium and C-13 labeling studies.

The formation and reactivity of ruthenabenzene derivatives (*e.g.* **844**, Scheme 90) was reported [825]. Ruthenabenzenes were formed in the coupling of diethynylmethanol (**841**) with ruthenium chlorides (**842** or **843**) in the presence of triphenylphosphine. Various ligand substitution reactions were reported for ruthenabenzene complex **844**.

Osmabenzofurans (*e.g.* **849**, Scheme 91) were obtained from the reaction of osmium–alkyne complexes (*e.g.* **847**) with excess methyl propiolate (**848**) [826]. A mechanism involving formation of a metallacyclopentadiene, followed by reaction with another mole of alkyne to afford the vinylidene complex (**850**), followed by alkyl migration to afford osmacyclohexadiene complex **851**, followed by coordination of the carbonyl oxygen was proposed to account for formation of the metallabenzofuran system. Various reaction processes were reported for the metallabenzofurans. Reaction with HCl in ethanol led to transesterification products. Bromination occurred selectively in the osmafuran ring to afford **852**. Reaction with trifluoroacetic acid led to the cationic protonation product **853**.



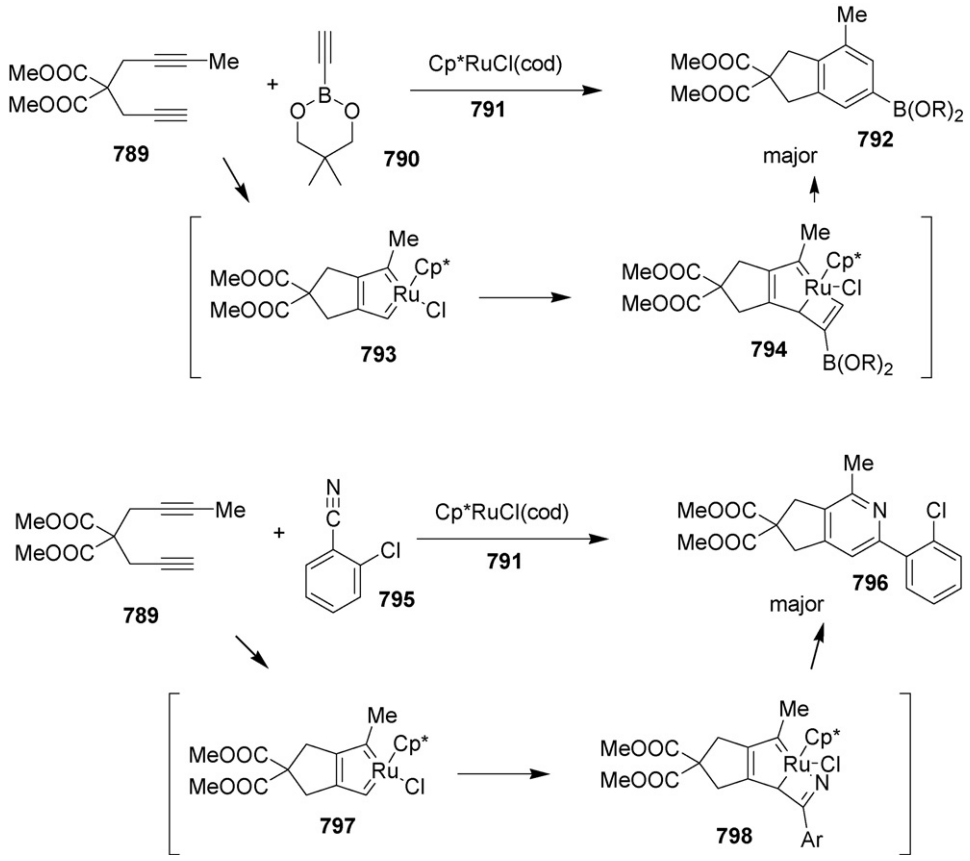
Scheme 82.



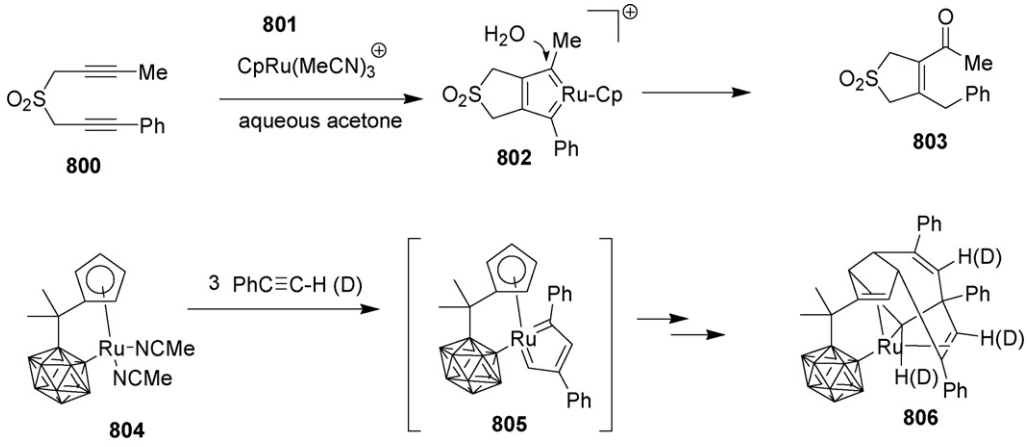
Scheme 83.

Other studies of carbene complexes in this category are depicted in Scheme 92, and include: (1) formation of spectroscopically observable bridging iron carbyne complex (**855**) from the low temperature reaction of diiron complex **854** with phenyllithium followed by triethyloxonium cation [827]; (2) a proposal that ruthenium carbene complexes (**862**) are intermediates in the reaction of enyne–ketone **857** and ethylene with ruthenium complex **858** to generate cyclopropane derivative **859** [828]; (3) a crystal structure of a bis(carbene)-bridged ruthenium porphyrin–dimer [829]; (4) reaction of osmium complex **863** with 2-vinylpyridine to afford the C–H activation product **864**, for which the carbene complex form **865** is an important resonance contributor [830]; (5) use of various NHC ruthenium complexes as catalysts for metathesis and cyclopropanation [831]; (6) use of iron porphyrin complexes to catalyze cyclopropanation, including observation of the carbene complex by electrospray MS [832]; (7) use of ruthenium porphyrin complexes as catalysts for cyclopropanation and discussion of structural effects of the intermediate carbene complex and how they relate to stereoselectivity [833]; (8) formation of ruthenium diaminocarbene complexes as minor products from the reaction of ruthenium carbonyl clusters with thiourea complexes [834]; and (9) a theoretical study of the reaction of $O_3Os{=}CH_2$ (**866**) with ethylene and identification of the *O*,*C*- and *O*,*O*-[3 + 2]-cycloaddition routes as energetically reasonable pathways [835].

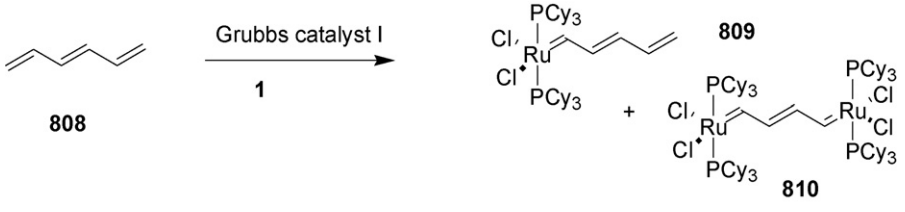
2.3.5.4. Heteroatom-substituted Group 8—metal–carbene complexes. The reaction of cation bridging aminocarbyne diiron complexes (*e.g.* **870**, Scheme 93) with alkynes led to bridging aminocarbene diiron complexes (*e.g.* **871**) through alkyne insertion [836]. Bridging aminocarbene–iron complexes (*e.g.* **872**) were prepared through oxidation of binuclear iron complex **871** [837]. Reaction with trimethylamine–*N*-oxide led exclusively to the ketone–carbene complex **872**. A similar reaction was observed using sulfur or selenium, which led to the alkene-coordinated complexes **873** after treatment with sodium borohydride. The mononuclear cyclic carbene complex **874** was a minor product from the reaction of complex **871** with sulfur. Nucleophilic addition of cyanide and hydride to related vinyliminium complexes (*e.g.* **875**) was also reported [838]. Addition of nucleophiles to the γ-position of related Fischer alkynylcarbene complexes containing the bridging aminocarbyne–diiron framework (*e.g.* **877**) was also reported [839].



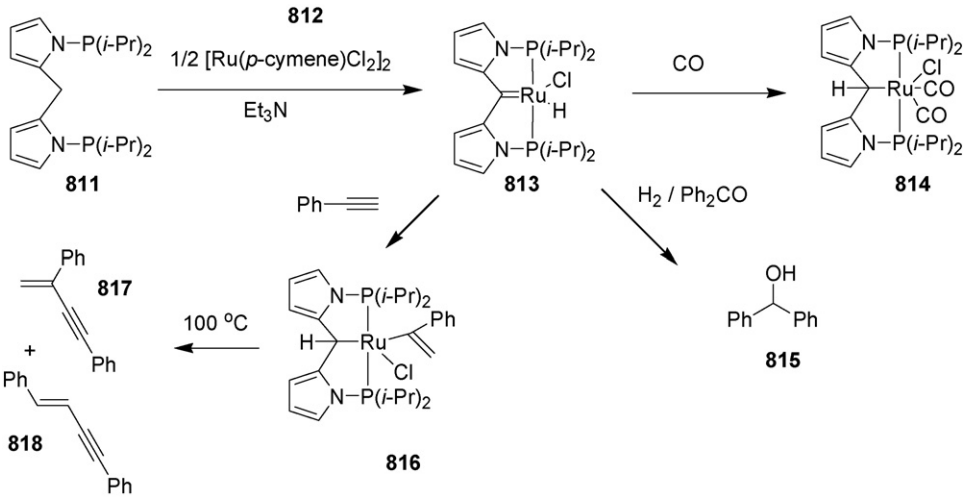
Scheme 84.



Scheme 85.

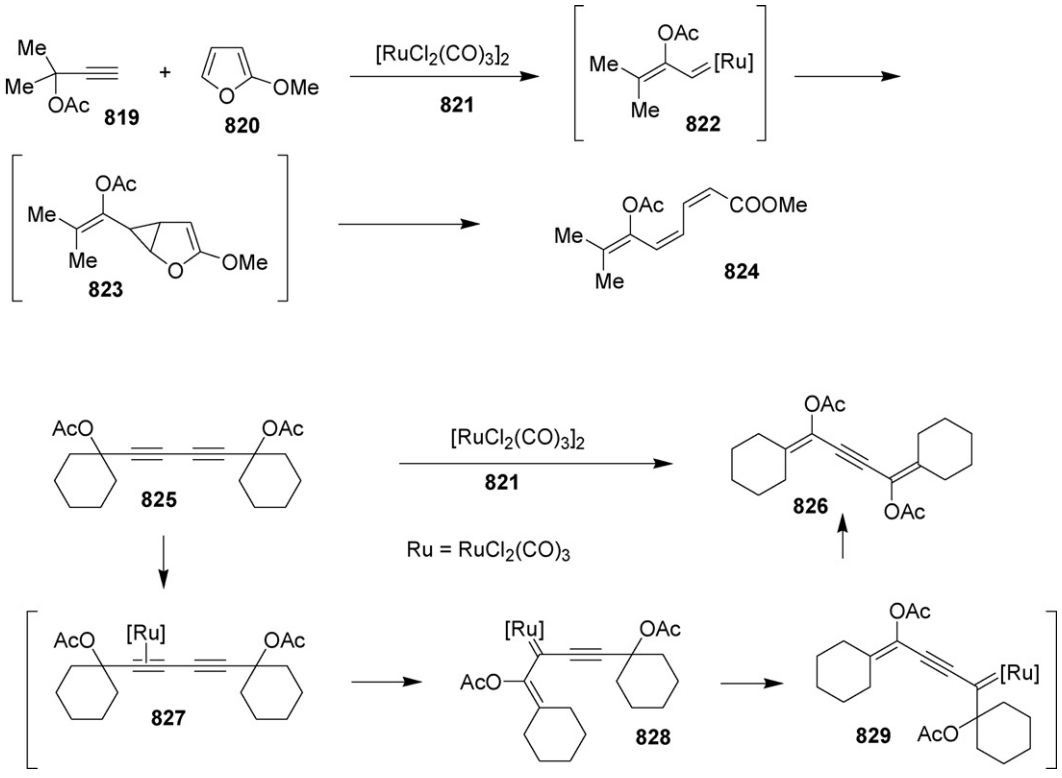


Scheme 86.

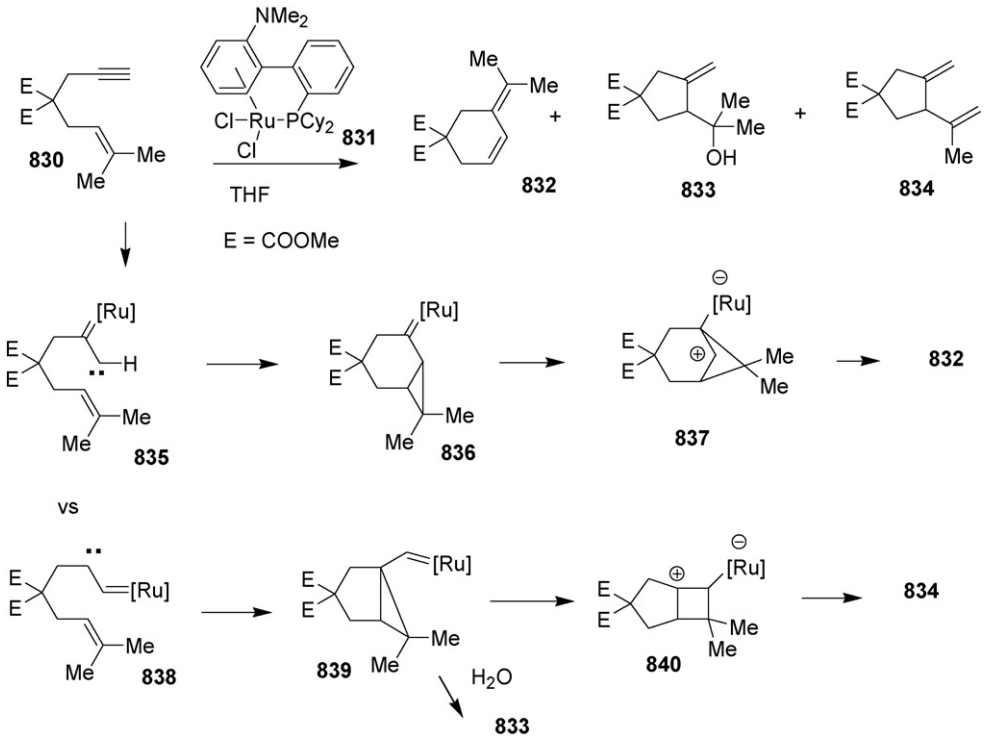


Scheme 87.

Several papers report on the synthesis of carbene complexes through C–H activation processes (Scheme 94). The synthesis of ruthenium– or osmium–carbene complexes (*e.g.* **882/883**) through net C–H activation of quinoline derivatives was reported [840]. This process has been denoted as capturing the minor carbene tautomeric form of quinoline. The five-coordinate complex **883** was obtained in the ruthenium system, which converted to the six-coordinate complex **882-Ru** upon treatment with hydrogen. A mixture of carbene complexes (*e.g.* **886/887**) and alkene complexes (*e.g.* **885**) were obtained in the reaction of bis(aminophosphine) derivatives (*e.g.* **884**) with ruthenium complex **812** [841]. The system was at equilibrium upon mild heating. The complexes were also studied by DFT calculations. The alkene complexes were the major equilibrium species in the open-chain system depicted in Scheme 94. However carbene complexes were the major species for cyclic analogs. Ruthenium thiocarbene complexes (**891**) were produced as a result of C–H activation [842]. When complex **889** was produced from the reaction of complex **888** and two moles of mesitylthiol, the complex **889** and the carbene complex **890** were formed as an equilibrium mixture. Extended reaction time led to irreversible formation of the thiocarbene complex **891** in low yield. Ruthenium aminocarbene complex **893** was formed in a C–H activation process via thermolysis of complex **892** [843].

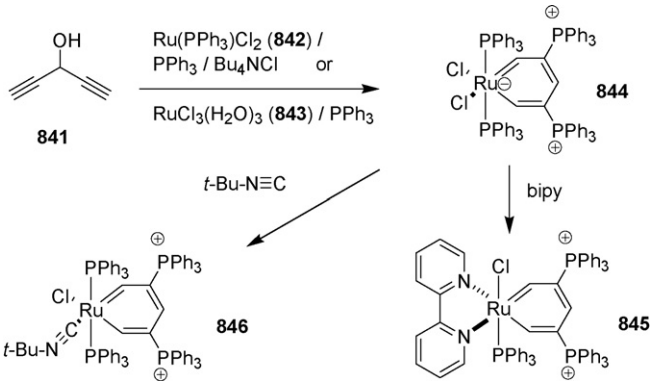


Scheme 88.



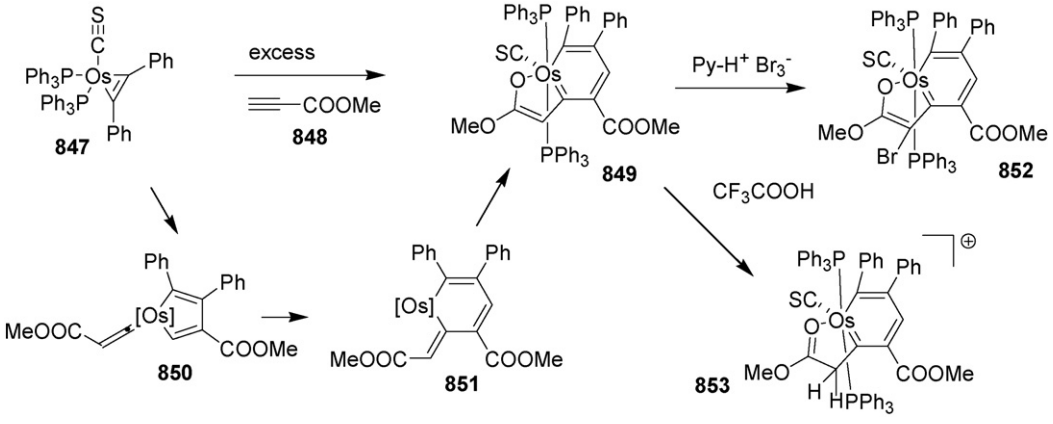
Scheme 89.

Attempted synthesis of iron alkenylcarbene complexes (*e.g.* **895**, Scheme 95) using the Fischer synthesis led directly to the η^4-vinylketene complexes (*e.g.* **896**) [844]. The ketene complexes undergo cycloaddition reactions with alkynes to produce phenols.



Scheme 90.

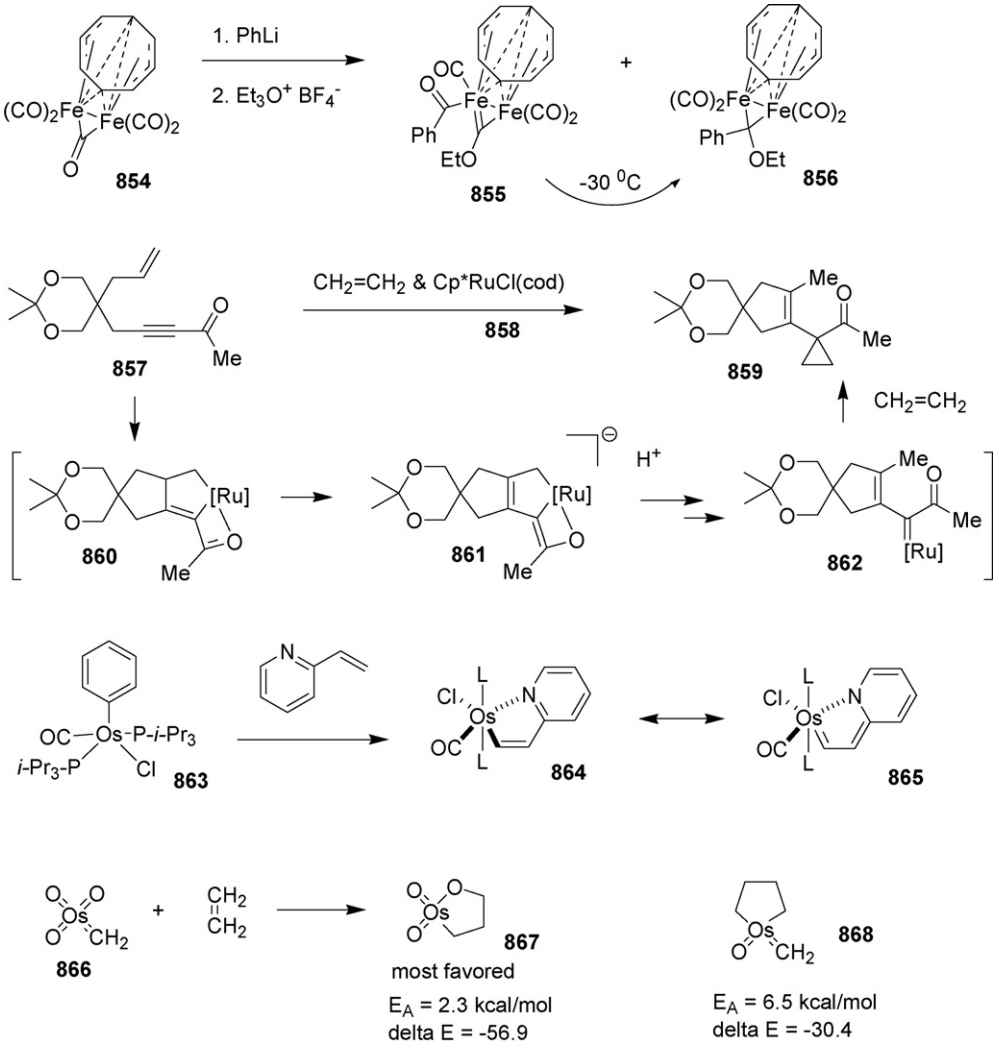
2.3.5.5. Group 8 metallacumulene complexes. Many examples of the formation of metal vinylidene complexes (**897**, Scheme 96) via coupling of coordinatively unsaturated Group 8 metal complexes with terminal or silylated alkynes were reported in 2006. Representative examples are depicted in Fig. 13. Common reaction pathways for these complexes include reaction with nucleophiles to form vinylmetal species (**900**), reaction with alcohols (or amines) to form Fischer carbene complexes (**901**) or water to form metal acyls (**899**), and deprotonation at the β-position to form alkynylmetal complexes



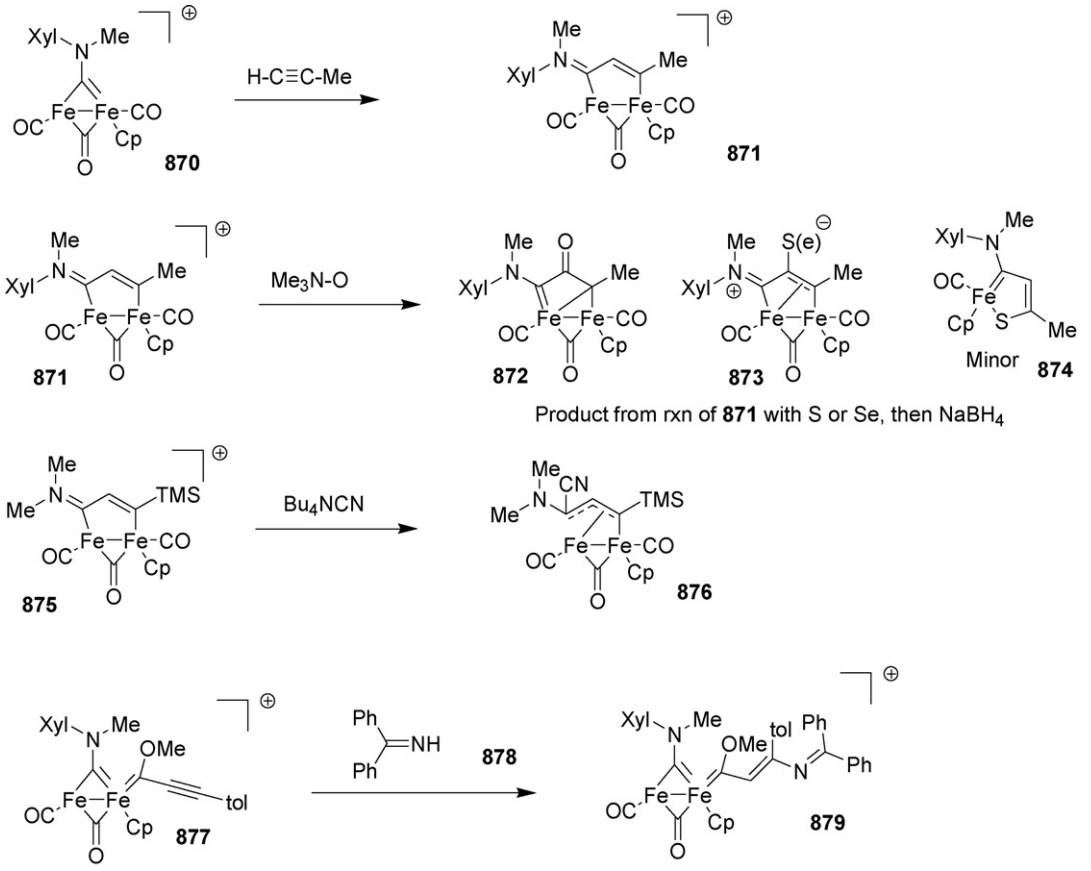
Scheme 91.

(**898**). Other common synthetic routes to metal vinylidenes included addition of electrophiles to metal acetylide complexes (*e.g.* the reverse of the reaction synthesizing **898**), and treatment of acylmetal complexes with dehydrating agents (*i.e.* the reverse of the reaction synthesizing **899**). Metal-higher cumulene complexes (**903**, **908**) 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 (**907**). Common reaction pathways for these complexes include reaction with nucleophiles at the γ-position, resulting in alkynylmetal complexes (**905**), or attack at the γ-position, resulting in allenylmetal complexes (**906**). Reaction with alcohols or amines can lead to α,β-unsaturated Fischer carbene complexes (**904**). Representative examples of this class of compounds are depicted in Fig. 13.

Specific reports which highlight the reaction pathways of Scheme 96 are depicted in Fig. 13 and include the following examples of vinylidene complexes: (1) synthesis and electrochemical studies of iron bis(vinylidenes) (*e.g.* **910**) and the alkynes obtained upon deprotonation [845], mixed iron/ruthenium analogs [846], and bis(ruthenium) analogs [847]; (2) formation of ruthenium vinylidene complexes (*e.g.* **911**) followed by deprotonation to afford the analogous alkynylruthenium species [848]; (3) formation of ruthenium vinylidene complexes from $RuCl_2(Ph_2PC{\equiv}CPh)_4$ and terminal alkynes [849]; (4) formation of an arylvinylidene-linked bimetallic system (*e.g.* **912**) [850]; and (5) cationic ruthenium vinylidene complexes that feature fullerene ligands [851]. Several processes likely involve metal vinylidene intermediates synthesized by these pathways, including: (1) ruthenium-catalyzed conversion of alkynyl *N*-phenyl imines (*e.g.* **913**) to quinolines (*e.g.* **915**) [852]; and (2) ruthenium-catalyzed synthesis of aldehydes via anti-Markovnikov hydration of terminal alkynes [853,854]. Examples that highlight higher cumulenes are depicted in Fig. 13 and include: (1) formation and spectral studies of ruthenium allenylidene complexes featuring pyridine groups (*e.g.* **916**) and subsequent complexation of the pyridine nitrogens [855]; (2) formation of either vinylidene– or allenylidene–ruthenium complexes (*e.g.* **917**) through reaction of a ruthenium chloride complex with a terminal alkyne or propargyl alcohol



Scheme 92.



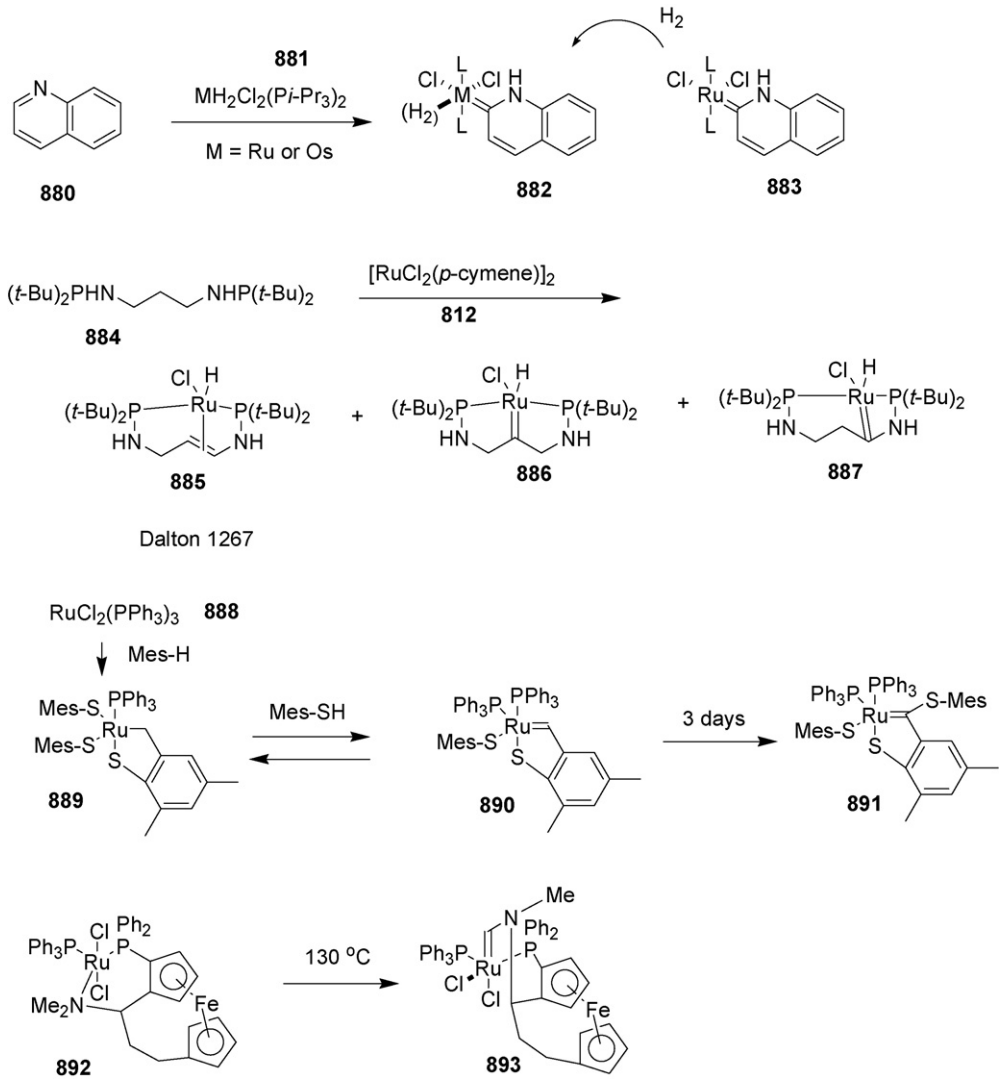
Scheme 93.

[856]; (3) formation of ruthenium vinylidene, cyclic carbene, and allenylidene (*e.g.* **918)** complexes from the reaction of simple alkynes, 4-pentyn-1-ol, or propargylic alcohols with a ruthenium heteroscorpionate complex [857]; (4) formation of cationic alkenylcarbene complex (*e.g.* **920**) from complex **919**, diphenylpropargyl alcohol, and methanol [858], and formation of related vinylidene complexes (*e.g.* **921**) and aminocarbene complexes (*e.g.* **922**) [859]; and (5) a cumulene-like structure obtained through electrochemical oxidation of all-carbon linked bimetallics [860]. Several processes reported in 2006 invoke metal-higher cumulene complexes as intermediates, including: (1) ruthenium-catalyzed rearrangement of propargyl alcohols to α,β-unsaturated aldehydes [861] and tandem rearrangement-aldol condensation [862].

The formation of carbomethoxymethylruthenium–vinylidenes (*e.g.* **924**, Scheme 97) and their subsequent conversion to cyclopropenylruthenium complexes (*e.g.* **925**) by deprotonation followed by cyclization was reported [863]. The kinetic product of the cyclization is the cyclopropenyl system, however extended treatment with base led to the furanylruthenium complex (**926**). A similar process was observed for the extended system **928**, however only the cyclopropene (**933**) was observed in this case. Reaction of the cyclopropenylruthenium complex with TMS–N_3 led to the triazole derivative **932**. A mechanism involving double bond isomerization followed by addition of azide to the vinylidene intermediate, followed by isomerization to the alkenylcarbene complex (**931**), followed by cyclization and demetallation was proposed.

The generation and capture of ruthenium vinylidene complexes that feature a carboranyl-bridged Cp ligand (*e.g.* **935**, Scheme 98) was reported. The reaction of carboranyl-bridged ruthenium–Cp complex **934** with bis(trimethylsilyl)acetylene led to bis(vinylidene)ruthenium complex **935** [864]. Reaction with trimethylsilylacetylene led to alkynylvinylidene complex **936**. A similar process was reported in the coupling of related complex **937** with phenylacetylene [865]. In this case either the chelated aminocarbene complex **941** or the η^4-diene complex **942** was obtained. The product ratio was dependent on the electron-donating ability of the aryl group. More electron-donating aryl groups favored formation of the aminocarbene complex. The key step in formation of the carbene complexes is a [2 + 2]-cycloaddition of the exogenous alkyne to vinylidene intermediate **938**. The diene complex arises through conversion of the initially formed vinylidene complex **938** to the aminoalkenyl complex (**943)** followed by alkyne insertion.

The reaction of osmium vinylidene complexes (*e.g.* **944**, Scheme 99) with Grignard reagents was reported [866]. Reaction of osmium complex **944** with methylmagnesium bromide led to the osmaindene derivative **949** at room temperature, which afforded the allyl complex **950** at higher temperature. A mechanism involving ligand exchange followed by methyl migration and C–H activation was proposed to account for the osmaindene.

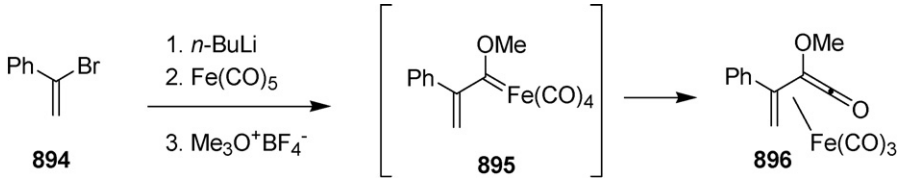


Scheme 94.

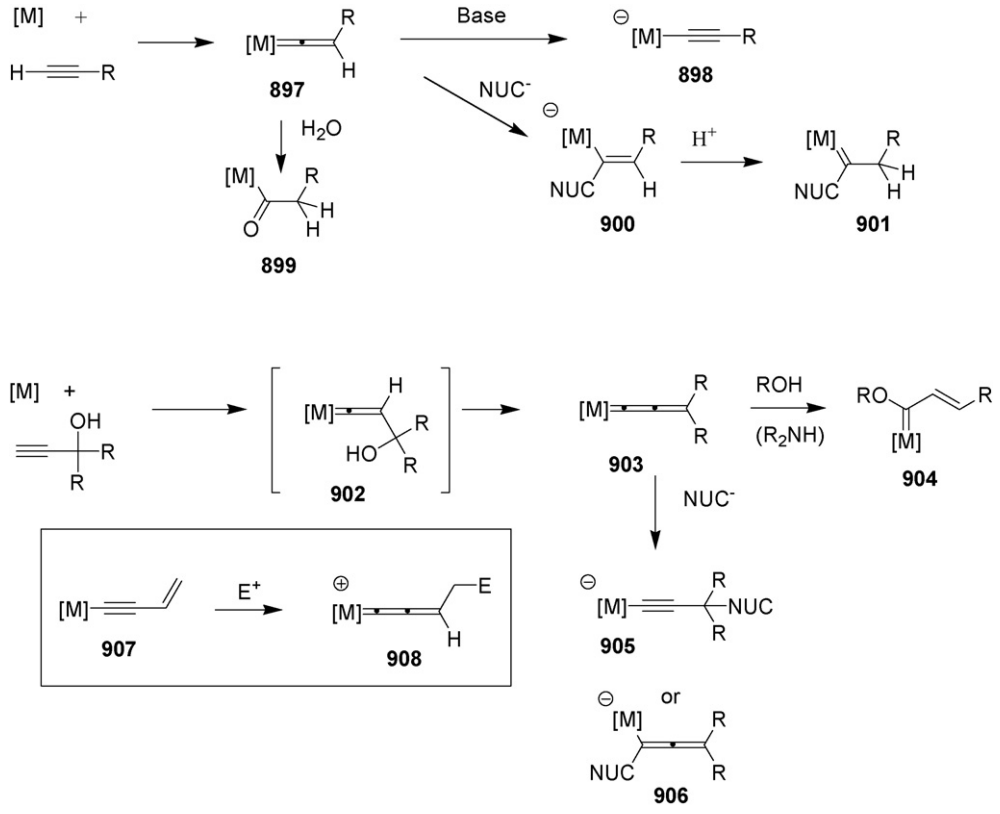
The formation of ruthenium carbyne complexes (*e.g.* **954**, Scheme 100) from ruthenium vinylidene complexes (*e.g.* **953**) was reported [867]. The vinylidene complex **951** was transformed to the octahedral complex **953** by treatment with tridentate ligand **952**. Protonation afforded the spectroscopically observable cationic carbyne complex **954**. The vinylidene ligand could be displaced by treatment with CO or acetonitrile.

The synthesis and reactivity of osmium vinylidene complexes featuring pincer ligands (*e.g.* **957**, Scheme 101) was reported [868]. The simple vinylidene complex was formed from coupling of osmium halide derivative **956** with terminal alkynes. Additional reaction time led to the bimetallic complex **958**. Formation of the Cp ligands was proposed to occur through a mechanism involving [2 + 2]-cycloaddition, alkyne insertion, and reductive elimination to afford the fulvene complex (**961**), which dimerizes through electrophilic attack of the carbocation-like resonance form (**962**) on the nucleophilic alkene of the fulvene complex.

Several cyclization reactions involving terminal alkynes invoke vinylidene intermediates; examples are depicted in Scheme 102. Ruthenium–vinylidene complexes (*e.g.* **964**) have been proposed as intermediates in the cyclization reaction of 5-hexynal derivatives (*e.g.* **963**) [869]. A mechanism involv-



Scheme 95.



Scheme 96.

ing formation of a vinylidene, followed by hydration and an aldol-like process, followed by decarbonylation was proposed. Naphthalenols (*e.g.* **975**, **977**) were prepared through reaction of alkyne–epoxides (*e.g.* **968**) with ruthenium complex **969** [870]. A mechanism involving formation of a cationic vinylidene complex (**970**) followed by a series of pericyclic rearrangements was proposed. The mechanism was supported by C-13 and deuterium labeling studies. Ruthenium vinylidene complexes (*e.g.* **980**) were suggested as intermediates in the cyclization of dienynes (*e.g.* **978**) [871]. In this reaction, after formation of the vinylidene intermediate (**980**), cyclization followed by a series of alkyl shifts occurs to afford the products. The complex mechanistic process was supported by deuterium labeling studies.

Cationic ruthenium allenylidene complexes (*e.g.* **988**, Scheme 103) were prepared through reaction of ruthenium complex **986** with propargylic alcohols [872]. Addition of a second proton to allenylidene complex **988** resulted in dicationic carbyne complex **989**. Coupling of the carbyne complex with acetone led to the substituted vinylidene complex **991**. A mechanism involving proton transfer to acetone followed by coupling of the resulting allenylidene complex (**990**) and enol was proposed.

Reaction of ruthenium allenylidene complexes that contain an allylphosphine ligand (*e.g.* **994**, Scheme 104) with ynamine **995** was reported [873]. The reaction affords the alkenylallenylidene complex (**997**) in a process involving stepwise [2 + 2]-cycloaddition followed by ring opening. Subsequent reaction of the aminoallenylidene complex **997** with lithium triethylborohydride led to the cyclobutenylidene–ruthenium complex **1000** in a process involving nucleophilic addition of hydride to the γ-position to afford alkynyl complex **998**. Treatment with silica led to the allenylidene complex (**999**), which spontaneously cyclized. Reaction of complex **997** with sodium borohydride led to the alkynylruthenium complex **1001**.

The synthesis and reactivity of osmium allenylidene complexes (*e.g.* **1003**, Scheme 105) was reported [874]. Reaction of the alkenylcarbyne–hydride complex **1002** with base led to the allenylidene complex **1003**. Reaction with various ligands led to the allenyl complexes (*e.g.* **1004**). An alkyne insertion product (*e.g.* **1005**) was obtained upon treatment with terminal alkynes. Further reaction with acetonitrile led to azametallacycle **1006**. The proposed mechanism for formation of **1006** involves nucleophilic addition to the nitrile carbon, followed by a Nazarov-type cyclization of the resulting bis(akenyl)carbene complex (**1007**). A similar interconversion of vinylcarbyne and allenylidene complexes was reported for ruthenium analogs (*e.g.* **1009**, **1010**) [875]. The dicationic carbyne complex **1010** was generated through low temperature protonation of the cationic allenylidene complex **1009**. Upon warming to −20 °C, conversion to the indenylidene complex **1011** occurred. Complex **1011** was effective as a ROMP catalyst.

Ruthenium allenylidene complexes (*e.g.* **1013**, Scheme 106) were proposed as intermediates in the propargylation of furan, indole, and azulene rings using propargylic alcohols (*e.g.*, **987**) [876]. Formation of the allenylidene followed by electrophilic

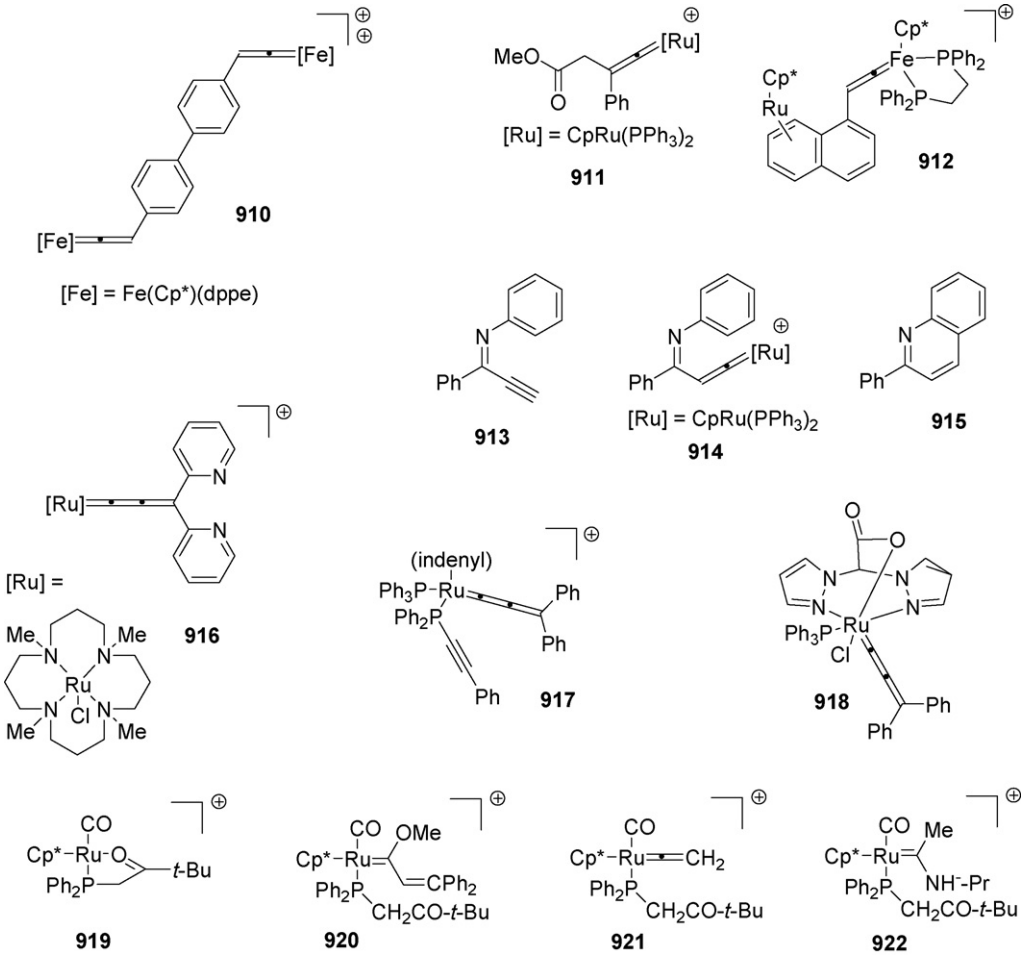
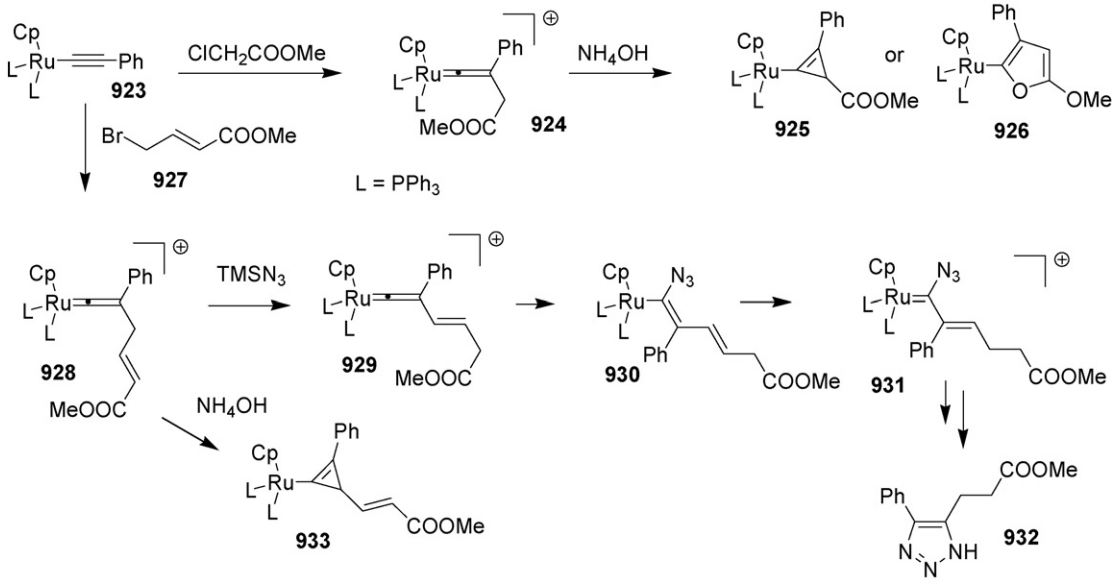
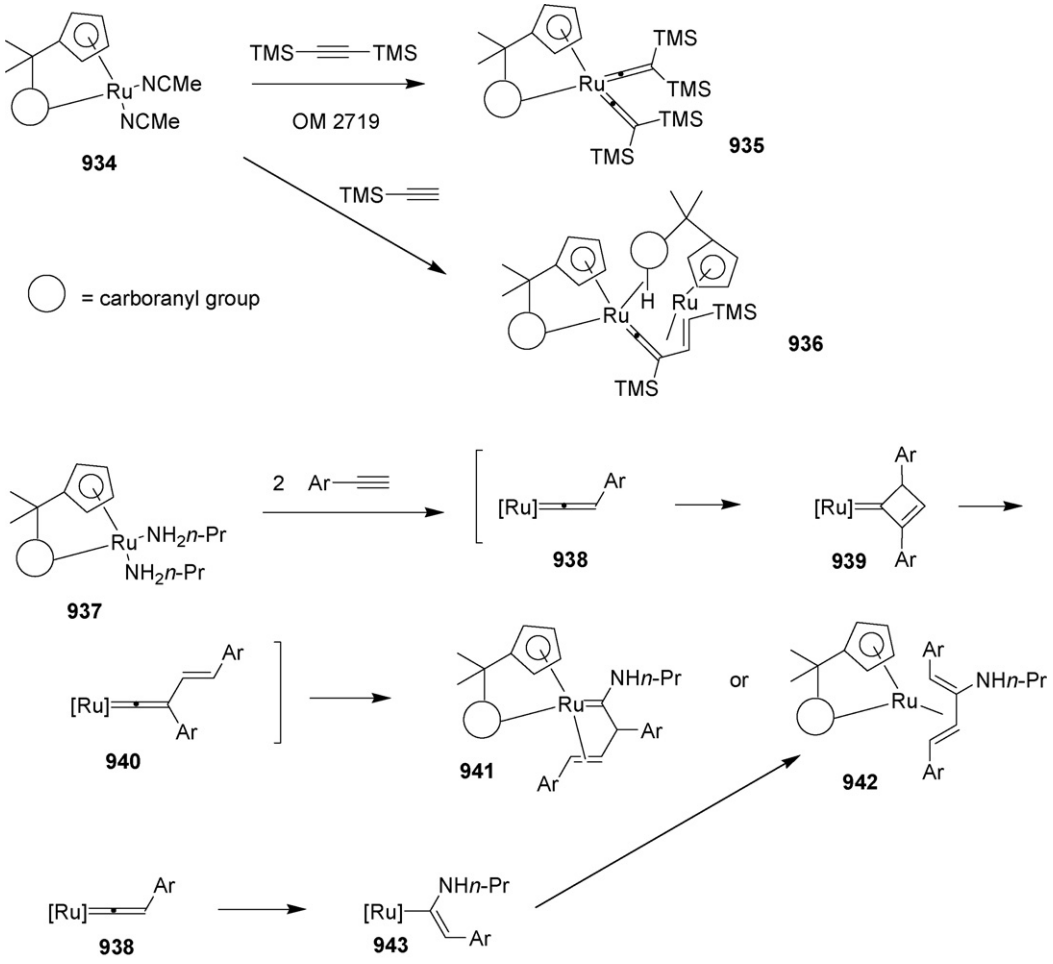


Fig. 13. Representative Group 8 metallacumulene complexes, precusors, and reaction products reported in 2006.

attack on the electron-rich aromatic system was proposed. Ruthenium allenylidene complexes were also proposed as intermediates in the stereoselective formation of 1,5-diynes (*e.g.* **1017**) from propargylic alcohol derivatives in the presence of bis(ruthenium) complex **1012** and boron hydride **1016** [877]. A mechanism that involves allenylidene complex formation, followed by hydroboration of the distal alkene to afford vinylidene **1018**, followed by C–B bond cleavage to afford a radical and



Scheme 97.



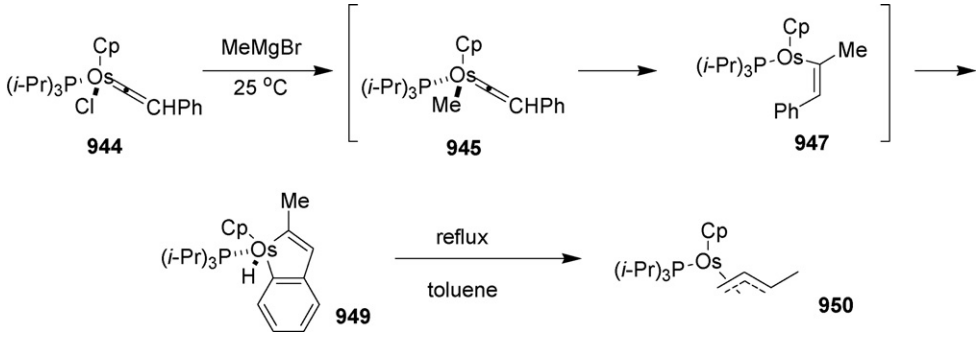
Scheme 98.

then proton loss to afford an alkynyl radical **1020**, which then dimerizes to **1017**, was proposed. The yield of the process was lower in the presence of free radical scavengers.

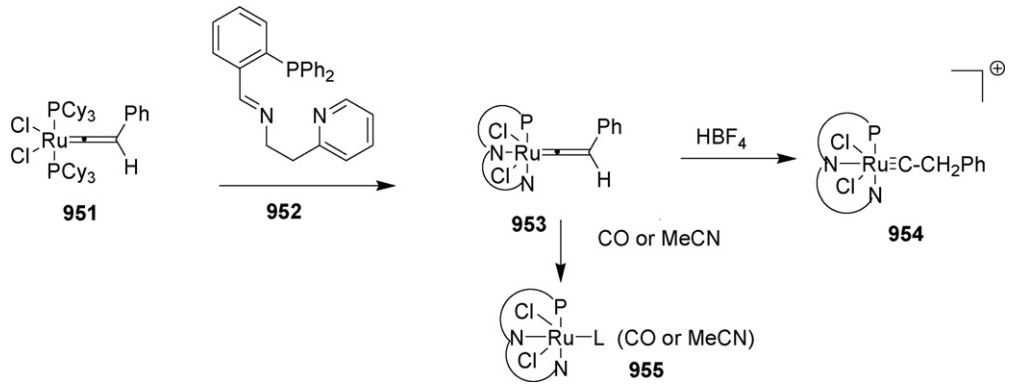
The formation and dimerization of *in situ*-generated butatrienylidene complexes (*e.g.* **1022**, Scheme 107) was reported [878]. The butatrienylidene complex was generated through oxidation of the butadiynyl complex **1021**. Subsequent [2 + 2]-cycloaddition between the butadiynyl complex and the butatrienylidene complex afforded the dimeric complex **1024**. A bimetallic complex featuring a carbon-rich bridge (**1026**) was also obtained through coupling of butadiynyl complex **1021** with allenylidene complex **1023**. A mechanism involving formation of the butatrienylidene complex **1022** and enynyl complex **1025** through proton transfer followed by addition was proposed.

2.3.6. Group 9 metal–carbene complexes

2.3.6.1. Simple carbene complexes. Rhodium carbene complexes (*e.g.* **1031**, Scheme 108) were generated through thermolysis of methylrhodium complex **1030** [879]. Extended thermolysis of the carbene complex leads to the rhodium hydride **1032** and ethylene. Iridium carbene complexes (*e.g.* **1035**) were



Scheme 99.



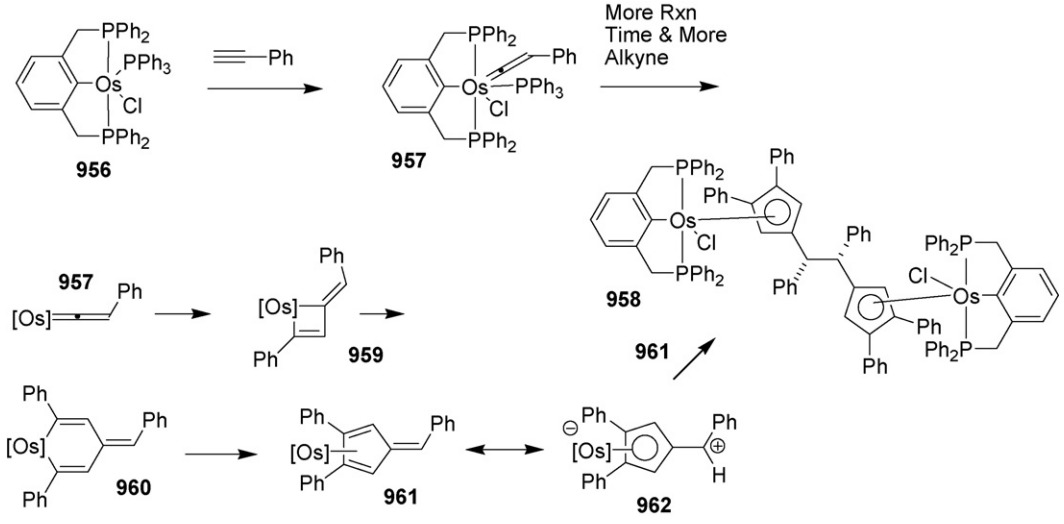
Scheme 100.

formed in the coupling of iridium complex **1034** with pyridine derivatives in an overall C–H activation process [880]. Treatment of the iridium pentadentate ligand complex **1036** with dichloromethane led to chlorocarbene–iridium complex **1037** in a process involving C–H oxidative addition, reductive elimination to convert L′ into L″, and α-elimination of chloride [881]. Reaction of carbene complex **1037** with pyrazole derivative **1038** led to pyrazolylcarbene complex **1039**. Difluorocarbene–iridium complexes (*e.g.* **1042**) were produced through reduction of difluoroalkyliridium complexes (*e.g.* **1040**) [882]. Reduction of the metal followed by α-elimination of fluoride was proposed.

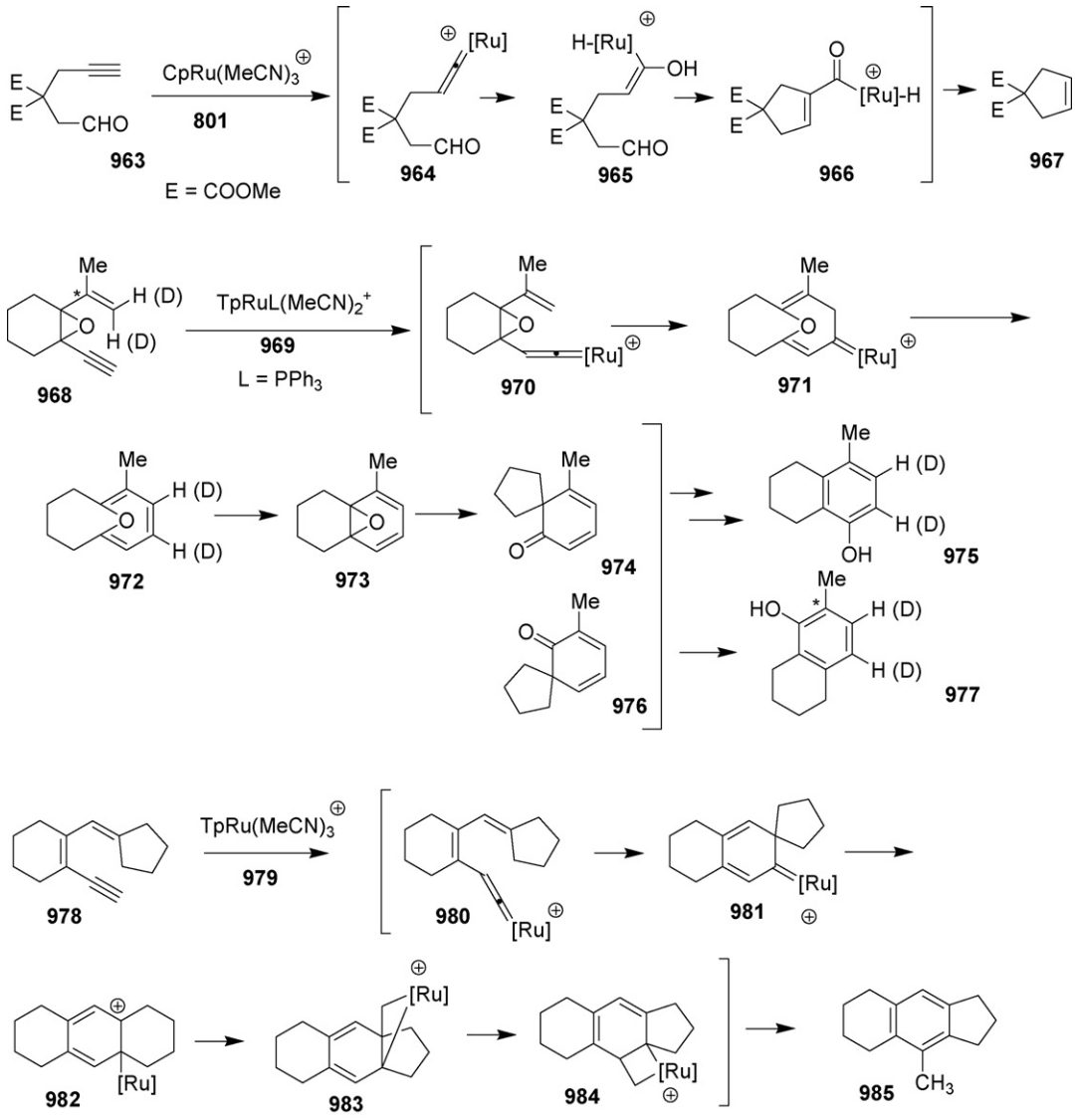
Iridabenzenes (*e.g.* **1045**, **1051**, Scheme 109) or iridacycloheptadienes (**1044**) were obtained upon treatment of iridacyclopentadienes (*e.g.* **1043**, **1050**) with propene [883]. Iridacycloheptadiene **1044** was obtained from lower temperature coupling of iridacyclopentadiene **1044** and propene and iridabenzene–hydride (**1045**) at higher temperature. Reaction of iridabenzene **1045** with acetonitrile at 20 °C led to the iridacyclohexadiene derivative **1046**. Formation of the iridabenzenes involves formation of the alkene complex (**1047**), followed by isomerization to the ethylcarbene complex (**1048**), followed by an alkyl shift. A methyl-iridabenzene (**1051**) was formed in the coupling of lesser substituted iridacyclopentadiene **1050** with propene. This product was proposed to arise from the regioisomeric carbene complex **1053**. An alternative approach to iridabenzenes was also reported. Reaction of diene–iridium complex **1055** with bis(trimethylsilyl)acetylene led to the vinylidene complex **1056**, which afforded the iridabenzene derivative **1057** upon protonation [884]. A mechanism involving formation of the cationic carbyne complex (**1058**) followed by alkyl migration and hydrogen elimination was proposed.

Iridafurans (*e.g.* **1063**, Scheme 110) were obtained from the reaction of iridaindene **1060** with methyl propiolate followed by acid [885]. Initial coupling of the iridaindene with the alkyne led to the alkenyl(alkynyl) complex **1061** which could be isolated in crude form and characterized spectroscopically. Treatment with acid led to the iridafuran in a mechanism involving vinylidene formation, followed by alkyl migration and halide and oxygen coordination.

Iridium carbene complexes (*e.g.* **1067**, **1068**, Scheme 111) were proposed as intermediates in iridium-induced methyl transfer reactions of 2,6-dimethylanisole (**1064**) [886]. The reaction initially forms the iridium carbene complex **1067** through C–H activation. This carbene complex can be detected spectroscop-



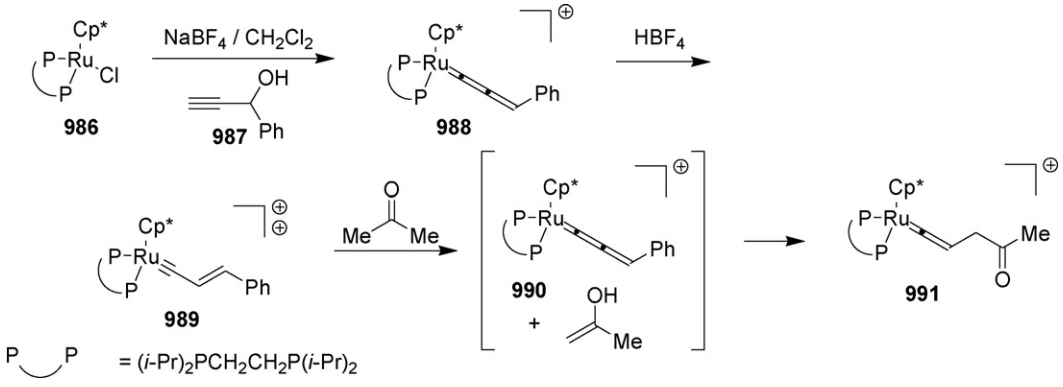
Scheme 101.



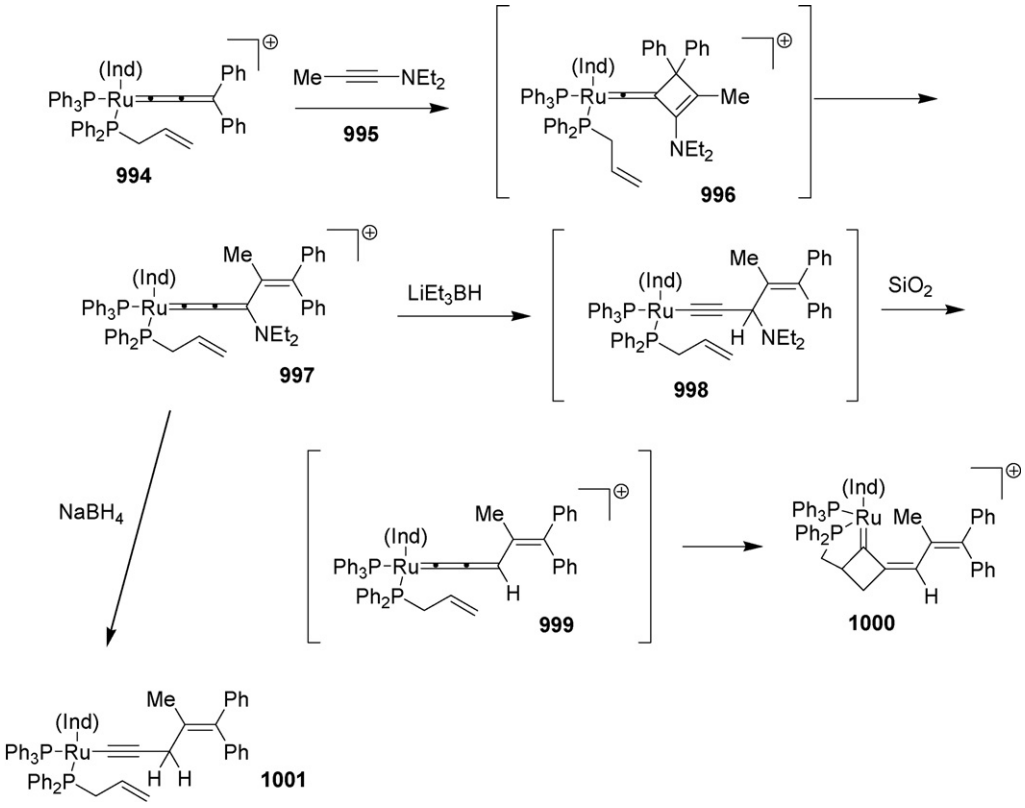
Scheme 102.

ically. Additional C–H activation of the methyl group leads to carbene complex–hydride complex **1068**, which converts to **1066** through metallacycle **1069**. This mechanism accounts for the scrambling of carbons noted during a labeling study.

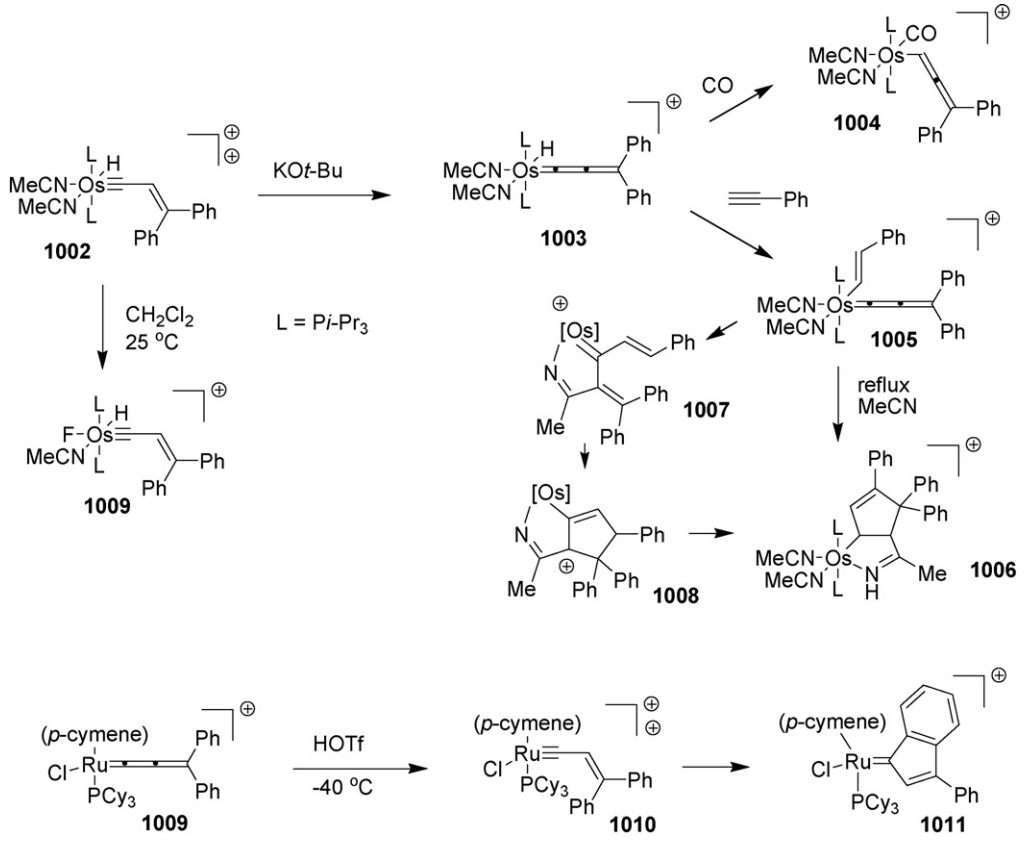
Other studies of Group 9 metal–carbene complexes (excluding metallacumulenes) include: (1) studies of the origins of enantio- and diastereoselectivity in rhodium-catalyzed cyclopropanation [887]; (2) development of models to explain



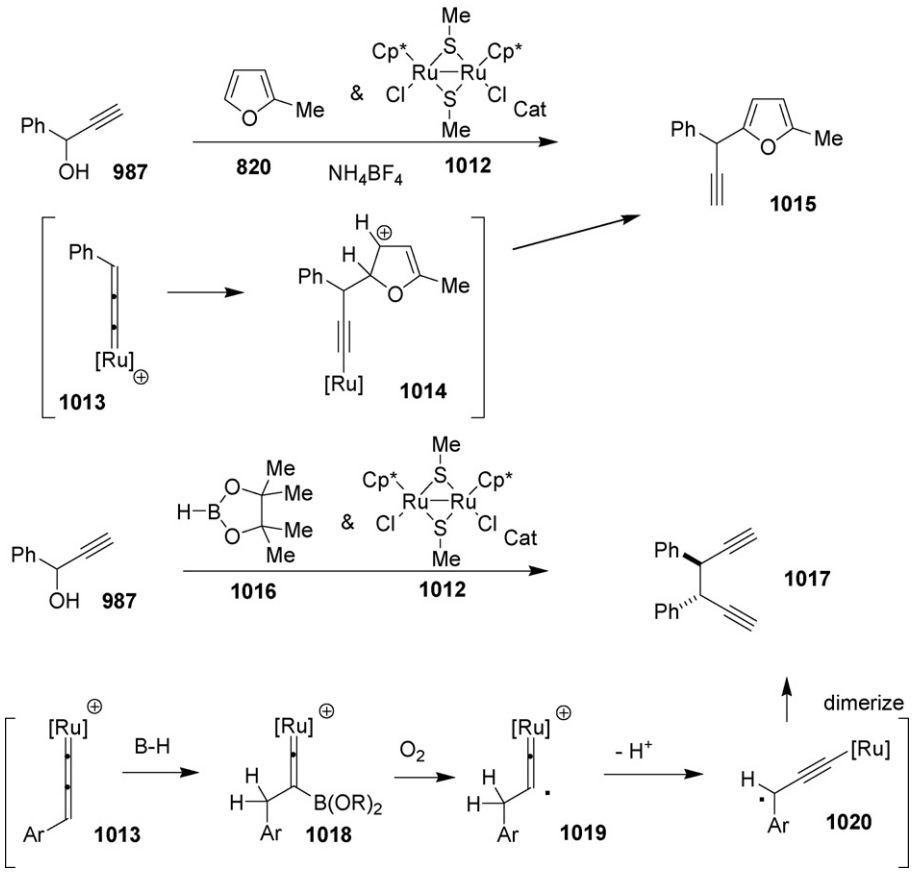
Scheme 103.



Scheme 104.

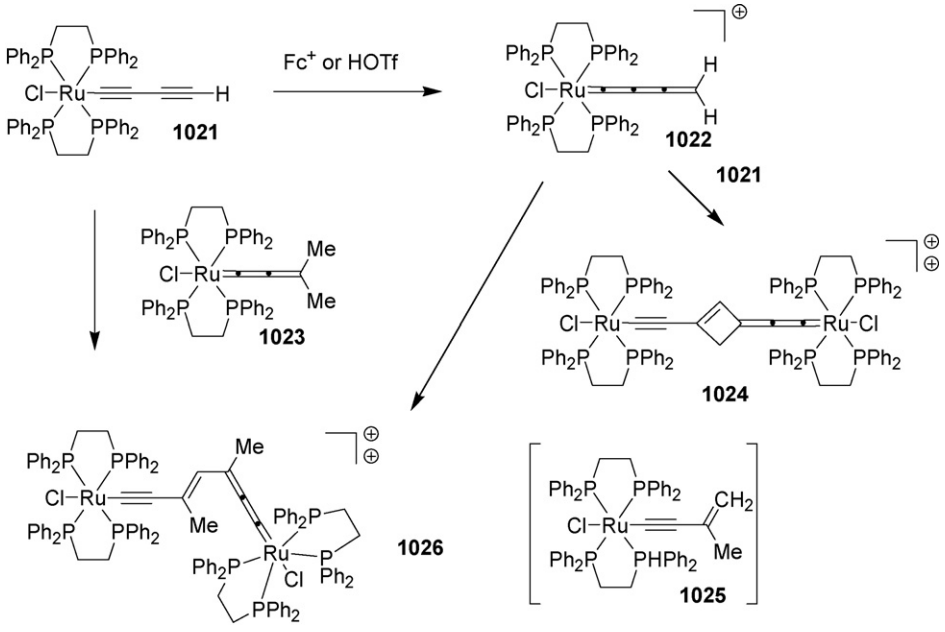


Scheme 105.



Scheme 106.

enantioselectivity in rhodium-catalyzed intramolecular cyclopropanation [888]; (3) discussion of the mechanism and development of models to explain the observed stereoselectivity in cobalt–salen complex catalyzed asymmetric cyclopropanation [889]; (4) formation of rhodium NHC complexes fused to quinone rings (*e.g.* **1070/1071**, Scheme 112), which were shown to have significant back-bonding interactions due to the presence of the electron-withdrawing quinone groups [890]; (5) discus-



Scheme 107.

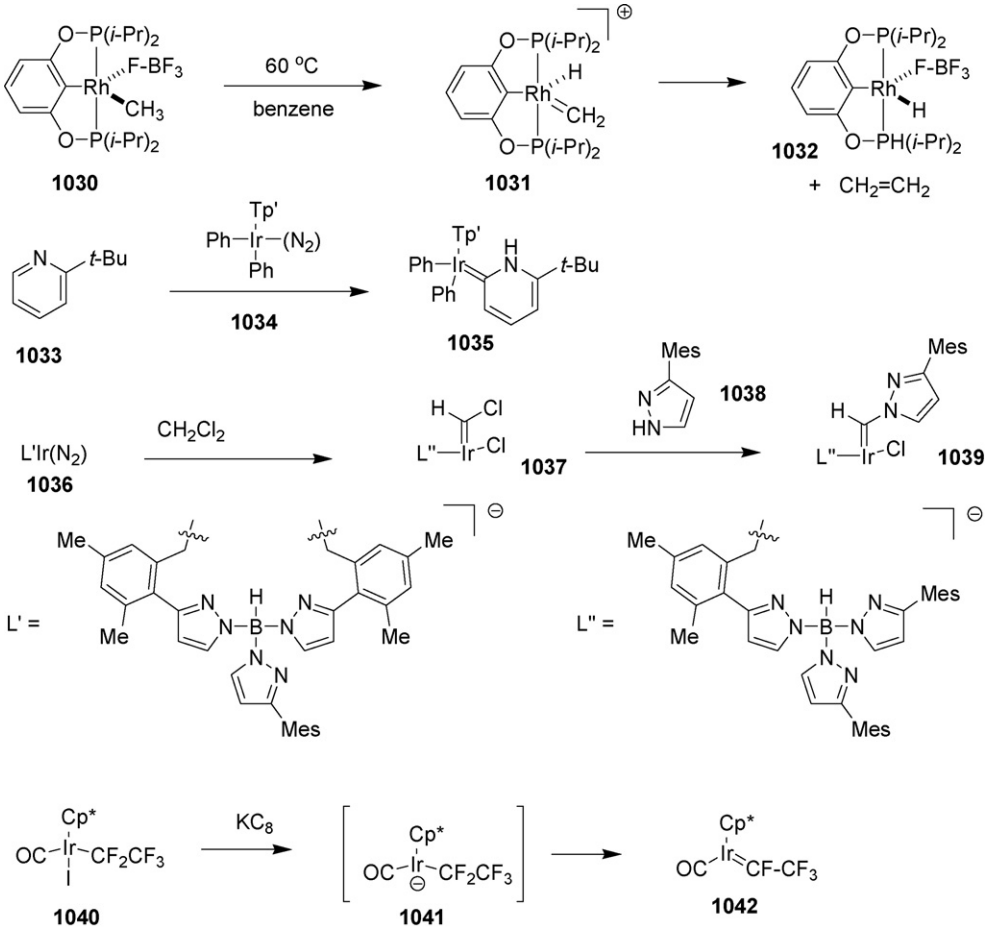
sion of carbene resonance contribution to Lewis acid–acylcobalt complex adducts [891]; and (6) discussion of iridafuran contribution to β-irada-α,β-unsaturated ester complexes [892].

2.3.6.2. Metallacumulene complexes. Similar synthetic procedures and reactivity patterns were generally observed for Groups 9 and 8 metallacumulene complexes (Scheme 96). Rhodium vinylidene complex **1074** (Scheme 113) was generated from rhodium complex **1072**, 1-hexyne, and phosphine **1073** [893]. No (alkynyl)(hydrido)rhodium complex was observed. The π-alkyne complex **1076** was generated at low temperature from the coupling rhodium complex **1075** and 1-hexyne, and it was noted that conversion to the vinylidene complex **1074** occurred without any alkyne crossover. The formation of iridium vinylidene complexes through reaction of iridium dinitrogen complex **1077** with terminal or silylated alkynes and dialkynes was also reported [894].

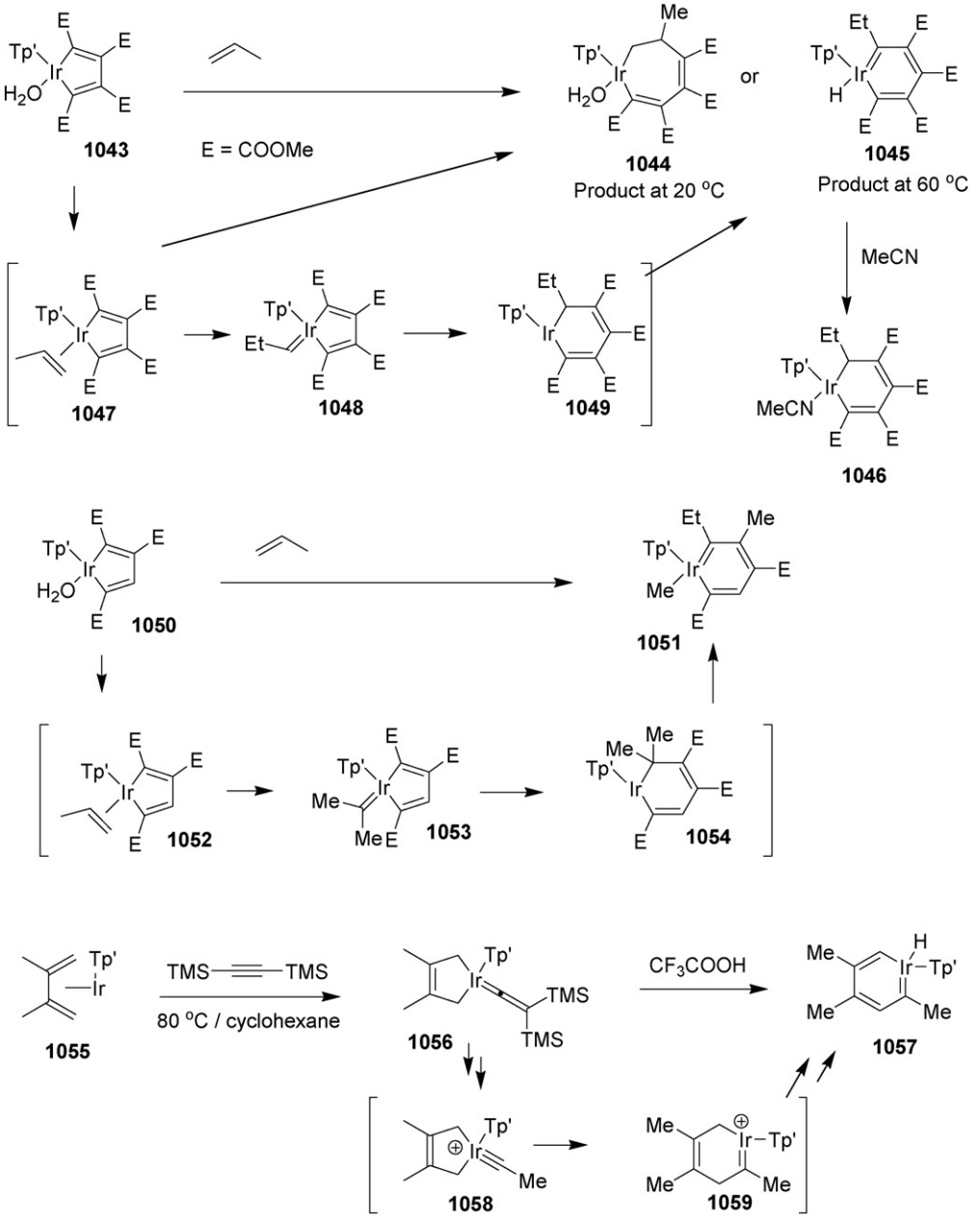
Rhodium vinylidene complexes were proposed as intermediates in several reaction processes involving terminal alkynes. Representative examples are depicted in Scheme 114. Rhodium vinylidene complexes (*e.g.* **1082**) were suggested as intermediates in cycloisomerization of *N*-propargyl enamines (*e.g.* **1080**) to tetrahydropyridine derivatives (*e.g.* **1084**) [895]. They key step in this transformation is nucleophilic addition of the enamine to the vinylidene carbon. A cycloisomerization using iodo enynes (*e.g.* **1085**) was reported, also involving vinylidene complexes (*e.g.* **1088**) [896]. The key event in this transformation is the nucleophilic displacement of iodide by the alkynylrhodium complex (**1087**) to afford the vinylidene complex. The eventual product **1090** arises after [2 + 2]-cycloaddition and ring opening. Cycloisomerization concomitant with arylation was observed in the reaction of enynes (*e.g.* **1091**) with rhodium complexes in the presence of boronic acids [897]. The key events in the process are formation of a vinylidene–aryl complex (**1093**), followed by alkyl migration and intramolecular alkene insertion. Rhodium vinylidene complexes were proposed as intermediates in a novel cycloaddition of alkyne–esters (*e.g.* **1096**) and allylamine [898]. The initial step in this complex process is formation of the chelating aminocarbene complex **1099** via the vinylidene complex, followed by deprotonation, C–H oxidative addition, alkene insertion and reductive elimination to afford iminoacyl complex **1101**, followed by alkyne insertion. Rhodium vinylidene complexes were suggested as possible intermediates in rhodium catalyzed polymerization and oligomerization reactions [899].

2.3.7. Group 10: metal–carbene complexes

Reaction of an NHC ligand (*e.g.* **1110**, Scheme 115) with nickel derivative **1111** led to the NHC-expanded nickel car-



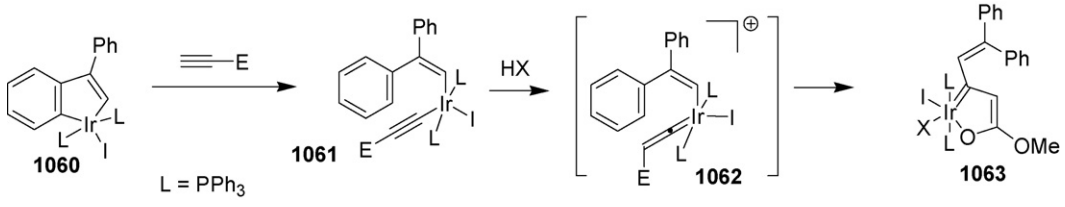
Scheme 108.



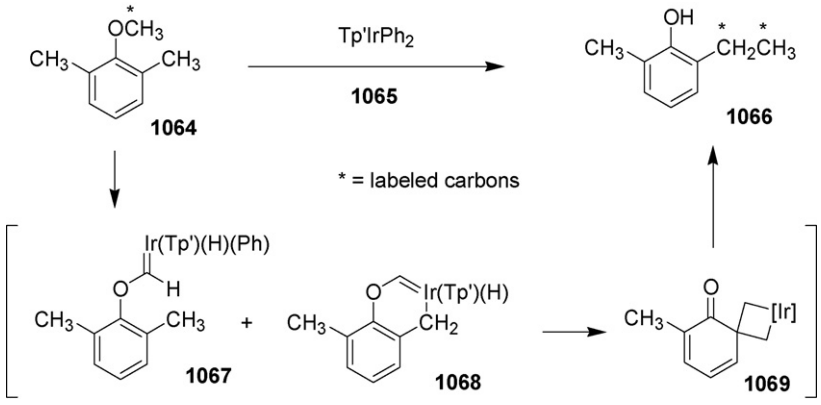
Scheme 109.

bene complex **1112** [900]. The analogous mesitylnickel complex (**1113**) afforded the simple chelating NHC complex (**1114**) under similar conditions. Nickel carbene complexes (*e.g.* **1117**) were studied experimentally and computationally [901]. A variety of chloropyridinium salts (*e.g.* **1115**) react with $Ni(PPh_3)_4$ to afford carbene complexes (**1117**). Substantial double bond character exists in the C–Ni bond.

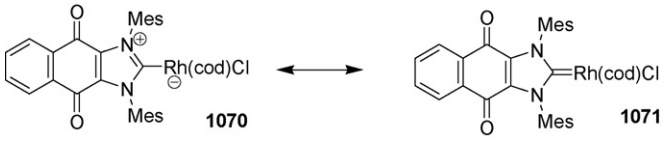
Palladium carbene complexes (*e.g.* **1119**, **1120**, Scheme 116) were prepared from the thermolysis of bis(η^3-cycloheptatrienyl)palladium complexes (*e.g.* **1118**) [902].



Scheme 110.



Scheme 111.

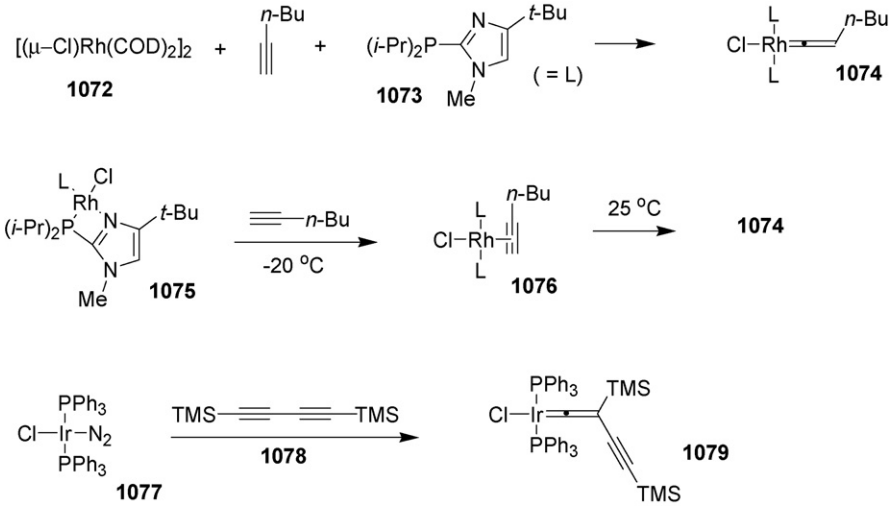


Scheme 112.

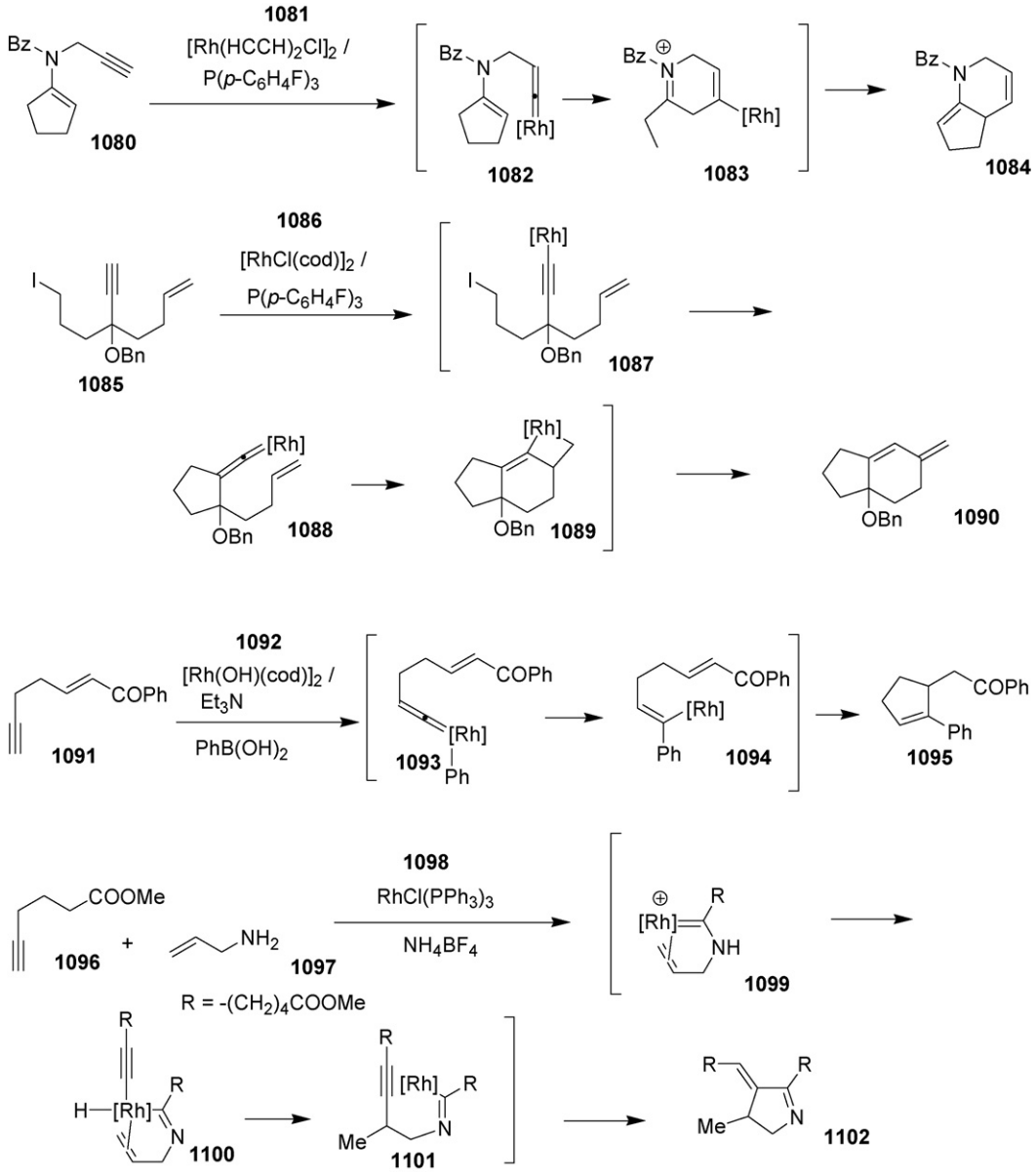
Initially a dimeric complex (**1119**) was obtained, which affords a monomeric complex (**1120**) upon treatment with phosphine ligands. Palladium carbene complexes (*e.g.* **1123**) were formed in the coupling of amine–phosphine derivative **1121** with allyl palladium chloride dimer (**1122**) [903]. Rhodium carbene complexes could also be formed through direct reaction with amine–phosphine **1121**.

Platinum aminocarbene complexes (*e.g.* **1128**, Scheme 117) were obtained through *S*-alkylation of thioacylplatinum complex **1127** with potassium ethyl dithiobicarbonate [904]. The preparation of Fischer carbene–platinum complexes (*e.g.* **1130**–**1133**) through reaction of alkynes and alcohols with platinum salts was reported [905]. The reaction with platinum salt **1129** and methanol/bis(trimethylsilyl)acetylene led to bis(carbene complex) **1130** which afforded the dimeric hydroxycarbene complex (**1133**) upon reaction with water. Reaction with isopropyl alcohol and HBF_4 led to the analogous isopropoxycarbene complex (**1131**). Reaction with higher alkynes was also reported.

Formation of the hydroazulene ring system (*e.g.* **1135**, Scheme 118) through the nickel-catalyzed coupling of diene–ynes (*e.g.* **1134**) with trimethylsilyldiazomethane was reported [906]. A mechanism involving carbene complex formation (**1136**), followed by alkyne insertion and intramolecular cyclopropanation was proposed. Cope rearrangement of the resulting divinylcyclopropane (**1138**) affords the observed product (**1135**). The intermediacy of carbenes was supported by the isolation of alkynylcyclopropanes from coupling of the diazo compound with enynes or with fumarate esters. Palladium carbene complexes were suggested as likely intermediates in the dimerization of conjugated enynes to form styrene derivatives [907]. Platinum carbene complexes (*e.g.* **1142**) were suggested as intermediates in platinum catalyzed conversion of alkylidenecyclopropanes (*e.g.* **1139**) to cyclobutenes (*e.g.* **1143**) [908]. Platinum vinylidene complexes (*e.g.* **1145**) were suggested as intermediates in the formation of indenes (*e.g.* **1146**) from *o*-alkylphenylacetylene derivatives (*e.g.* **1144**) [909]. The key mechanistic event is a net benzylic C–H insertion of the platinum vinylidene complex. The pro-



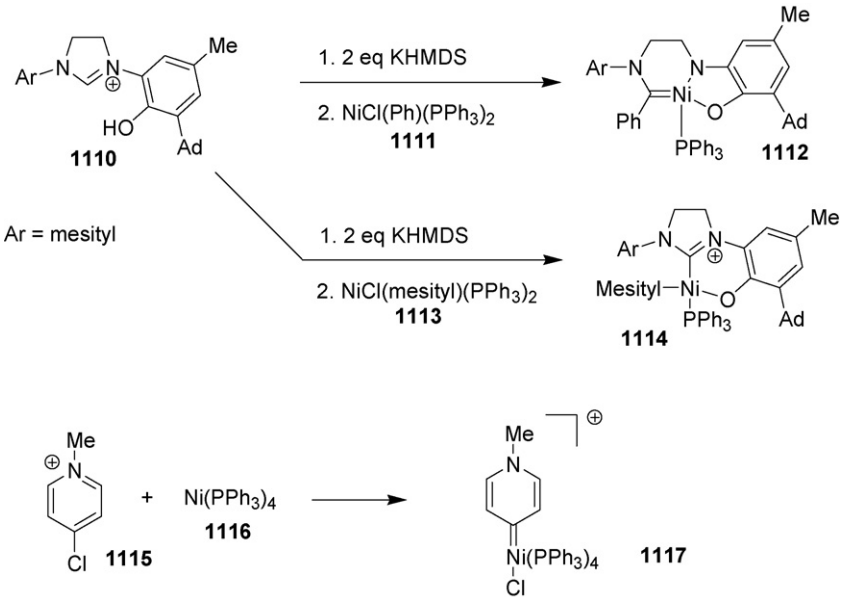
Scheme 113.



Scheme 114.

posed mechanism was supported through deuterium labeling studies.

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 Scheme 119. Gold-catalyzed enyne cycloisomerization of dienyne **1150** led to the bis(cyclopropane) derivative (**1152**) via cyclopropylcarbene complex intermediate **1151** [910]. Related cyclizations of dienynes resulting in tetracyclic compounds were also reported [911]. Cyclopropylcarbene–gold complexes were proposed as intermediates in the cycloisomerization of enynes to either cyclized vinylcyclopropanes or enyne metathesis products [912]. Cyclopropylcarbene–gold complexes (*e.g.* **1154**) were proposed as intermediates in the formation of hydroazulenes (*e.g.* **1156**) from enyne–ketones (*e.g.* **1153**) [913]. The successful capture of cyclopropylcarbene complexes (*e.g.* **1159**) through cyclopropanation of norbornene [914] and through cyclopropanation of hexadiene derivatives were reported [915]. Capture of the gold carbene complexes through Friedel–Crafts reactions [916] and cyclopropane ring opening [917] were also reported. Gold carbene complexes (*e.g.* **1162**) were similarly generated from the coupling of ynamine derivatives (*e.g.* **1161**) with gold(I) chloride [918]. Formation of ketone **1163** involves a pinacol-type rearrangement of carbene complex **1161** followed by demetallation. The reaction led to a mixture of enyne metathesis and [2 + 2]-cycloadducts using a terminal alkyne. A similar copper catalyzed reaction involving a propargylic alcohol was also reported [919]. Cycloisomerization reactions that feature silicon in the tether were also reported [920]. The reaction employing an enamine–propargyl alcohol afforded a cyclopropanation product. A cyclobutene (**1166**) was obtained from the reaction of allene–yne **1164** with platinum(II) chloride [921]. A mechanism involving

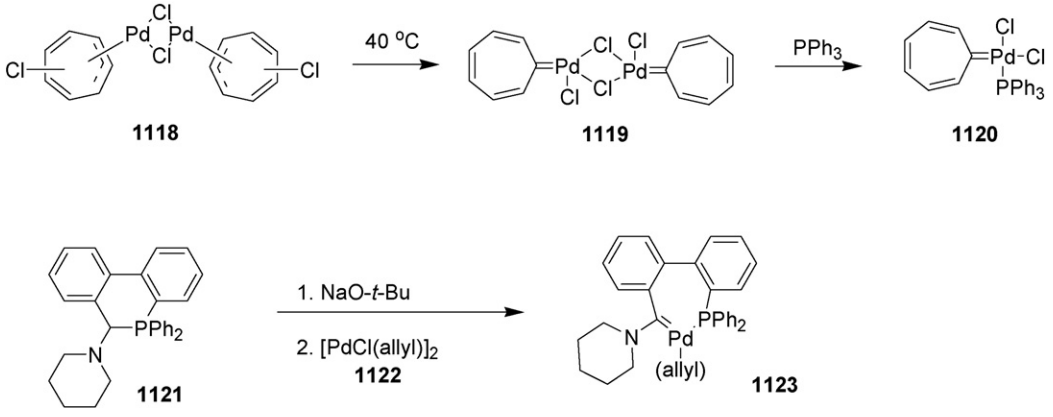


Scheme 115.

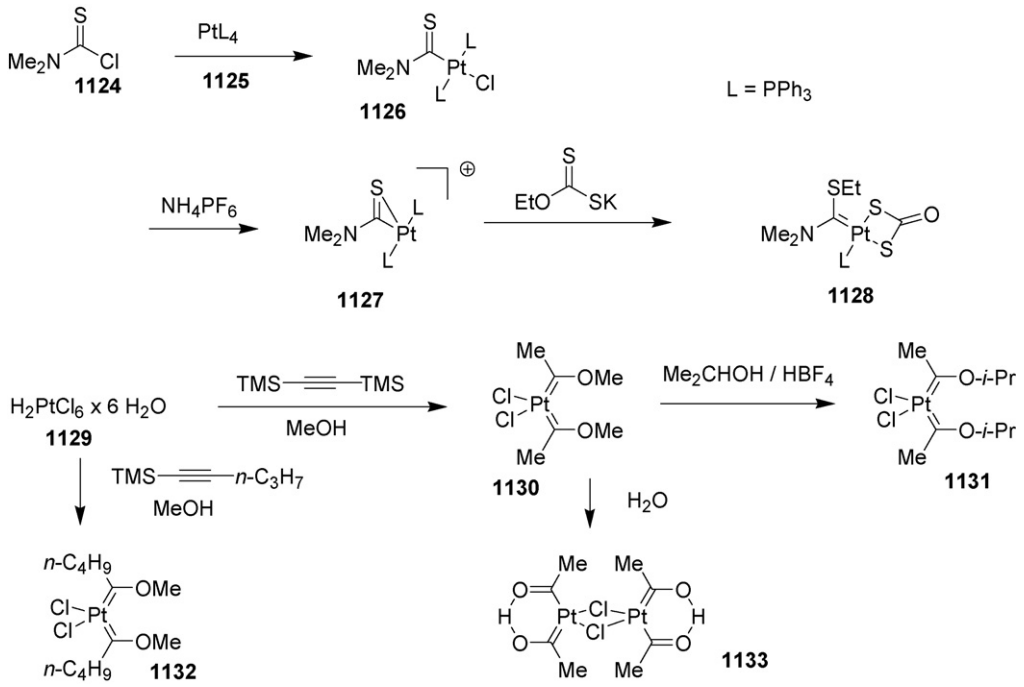
formation of the cyclopropylcarbene complex (**1165**) followed by ring expansion was proposed. Alkoxy–enynes (*e.g.* **1167**) converted to cyclohexadienes (*e.g.* **1169**) upon treatment with gold or platinum catalysts [922]. In this case a complex series of alkyl shifts from cyclopropylcarbene complex **1168** leads to the eventual reaction products. Hydrative platinum-catalyzed enyne cycloisomerization was studied computationally [923]. Gold carbene complexes were suggested as intermediates in the cycloisomerization of bis(propargyl) ethers [924].

Gold carbene complexes (*e.g.* **1171**, Scheme 120) were proposed as intermediates in gold-catalyzed intramolecular cyclization of furan–propargyl ethers (*e.g.* **1170**) [925]. A related cyclization process employing an amine tether between the furan and alkyne groups (*e.g.* **1173**) was also reported [926–928]. A DFT study of gold- and platinum-catalyzed conversion of 2-alkynylbiphenyl derivatives (*e.g.* **1177**) to phenanthrenes was reported [929]. Cyclopropylcarbene–gold complexes (*e.g.* **1178**) were intermediates in reactions employing electron-rich aromatic rings.

Gold– and platinum–carbene complexes were also generated from various heterocyclization processes; representative examples are depicted in Scheme 121. A novel cyclization/cycloaddition cascade reaction process occurred when diyne–dicarbonyl compound **1181** was treated with gold(III) chloride [930]. The major product type is steroidal derivatives (*e.g.* **1185**), and this ring system is formed through conversion of the initially formed gold–carbene complex/carbonyl ylide **1182** to carbene complex **1183**, which undergoes intramolecular 1,3-dipolar cycloaddition and 1,2-vinyl shift to afford the bridged structure **1184**, which aromatizes to form the eventual product **1185**. A similar heterocyclization-1,3-dipolar addition sequence was reported for platinum-catalyzed reactions of benzaldehyde–diynes [931]. Platinum– and gold–carbene complexes (*e.g.* **1188**, **1189**) were proposed as intermediates in a tandem cyclization/cycloaddition reaction involving *o*-alkynylaniline imines (*e.g.* **1186**) and monosubstituted alkenes [932]. In this process, the initial reaction of the alkyne with the catalyst affords the 1,3-dipolar species **1188**, which then undergoes 1,3-dipolar cycloaddition followed by



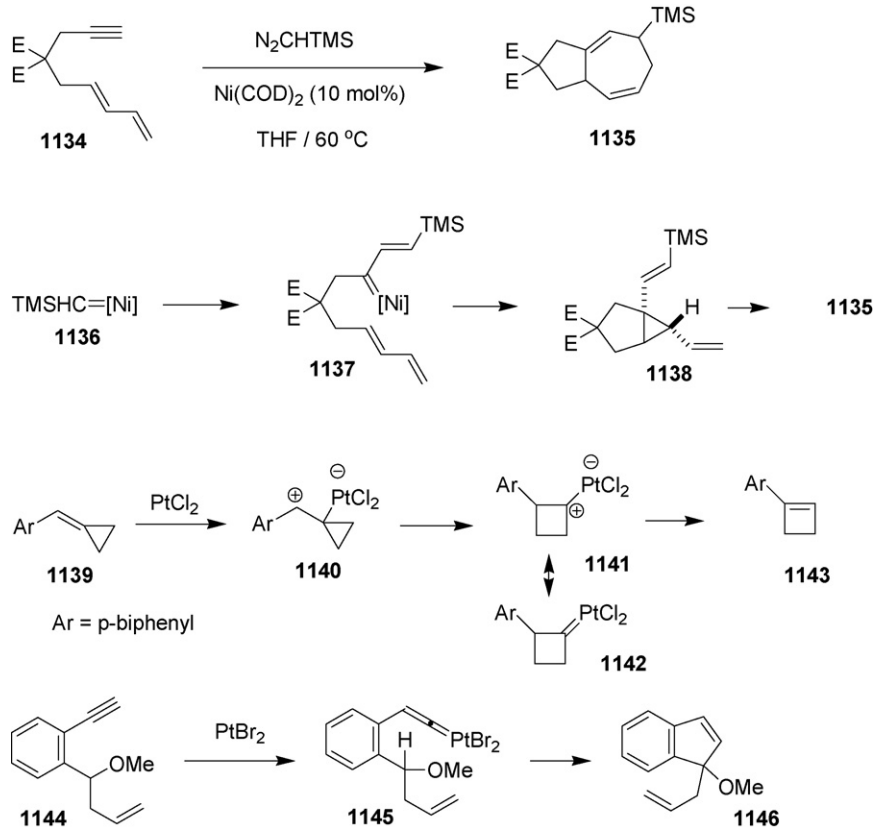
Scheme 116.



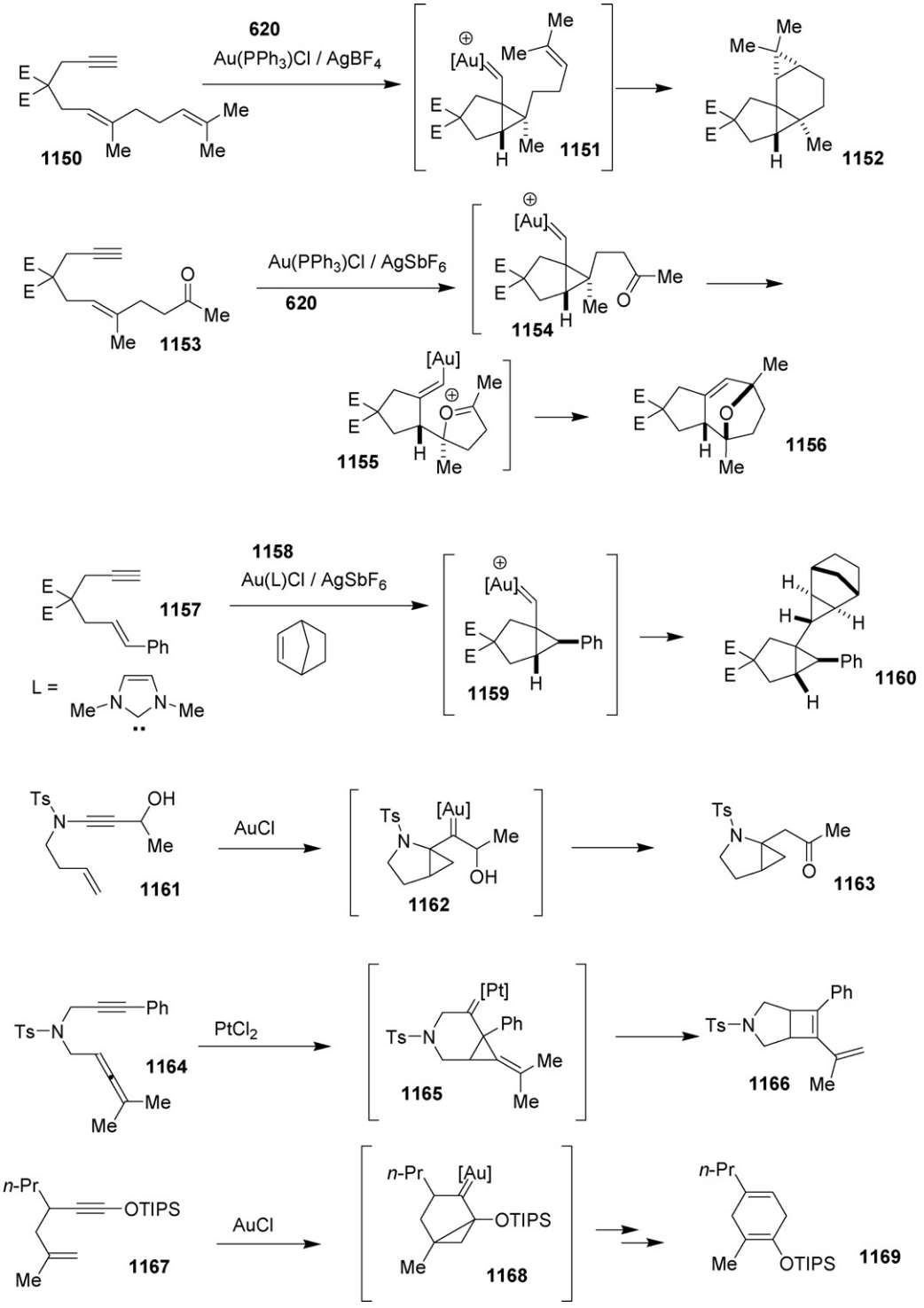
Scheme 117.

1,2-alkyl shift to afford the observed products **1191**. Platinum carbene complexes are similarly likely intermediates in platinum catalyzed conversion of 4-azoalkynes to pyrroles [933].

Carbene complexes were also generated from 1,2-shift of propargyl ester derivatives in the presence of platinum and gold complexes (see Scheme 122). Reaction of propargyl ester **1195** with platinum, silver, or copper salts led to the tri-

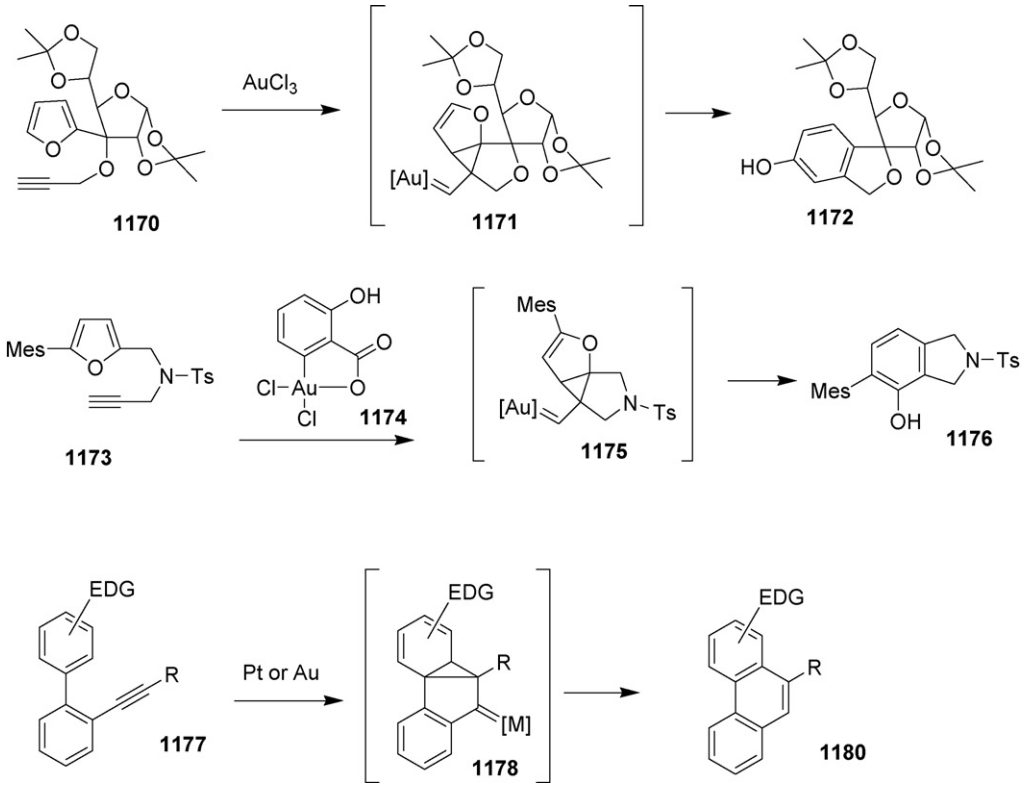


Scheme 118.

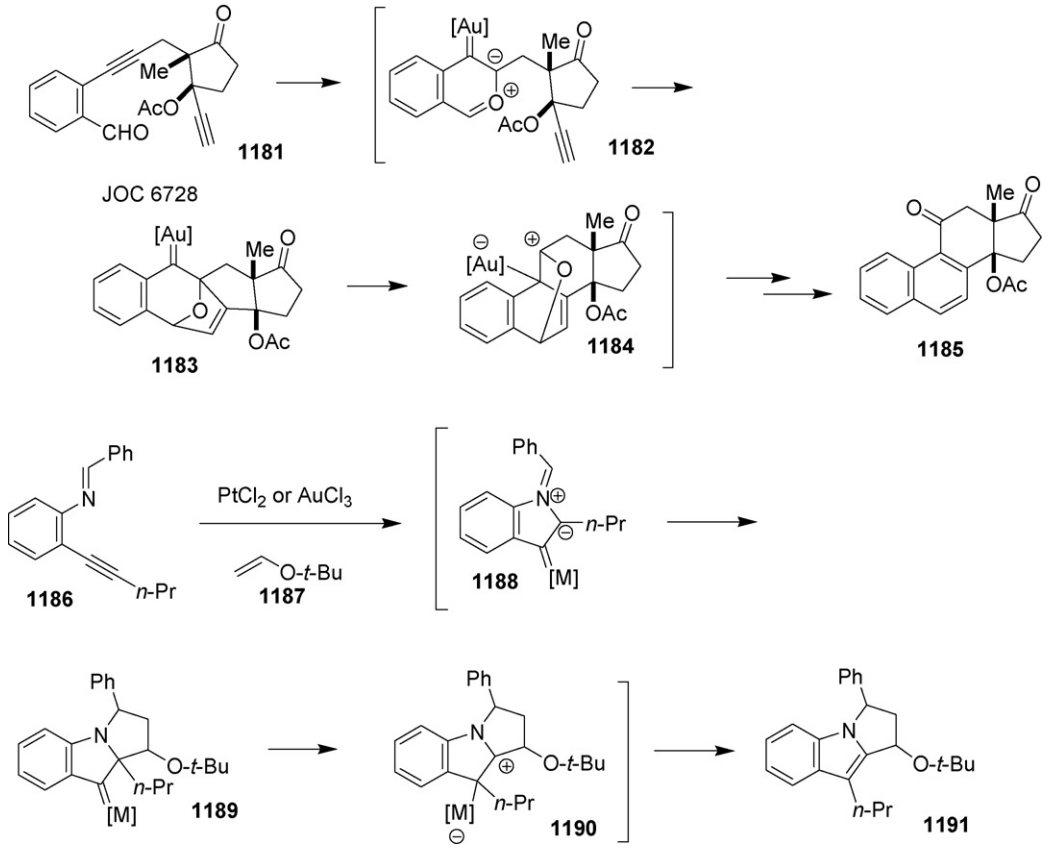


Scheme 119.

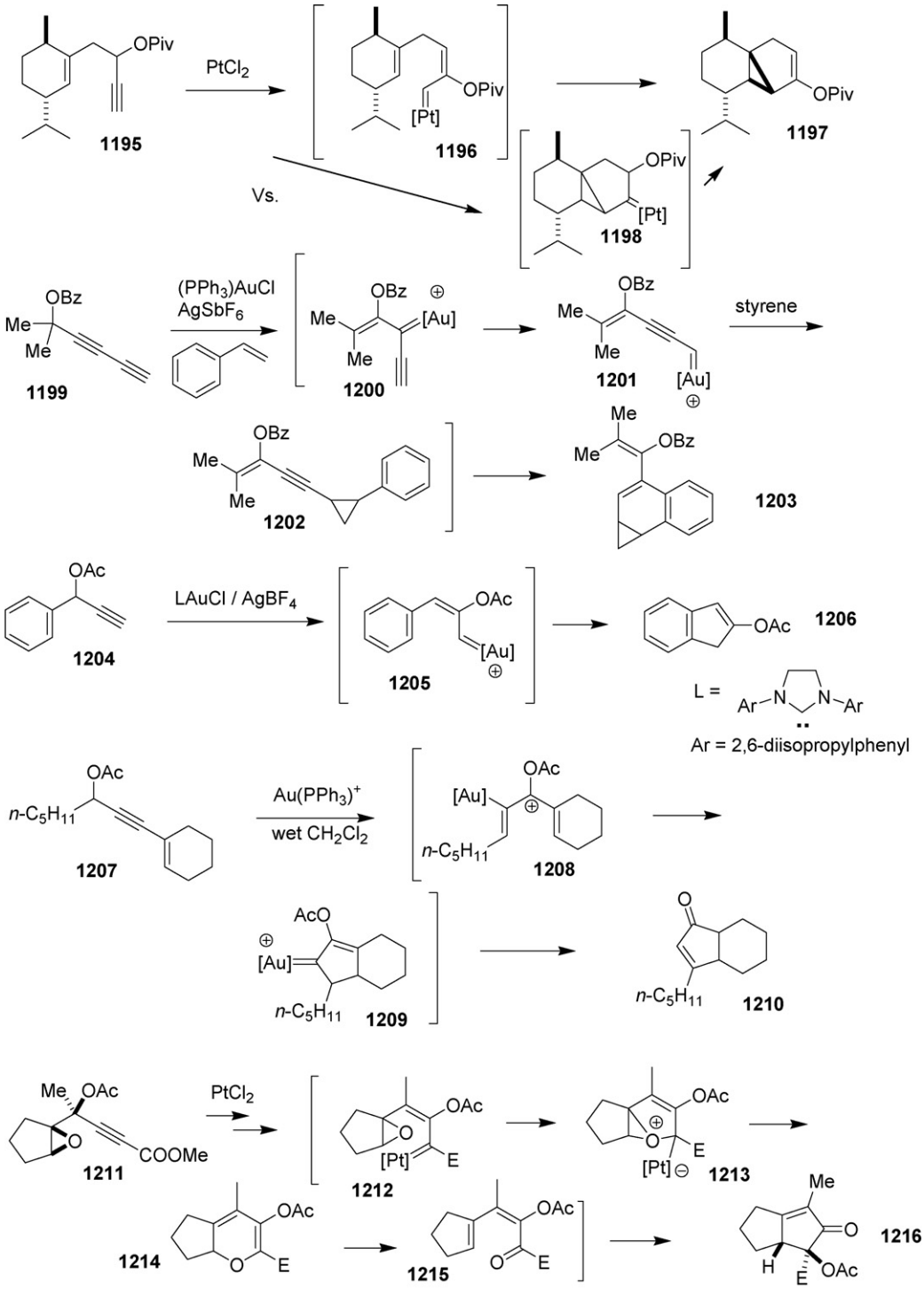
cyclic compound **1197**, which was used for total synthesis of cubebol [934,935]. A mechanism involving cyclopropanation prior to 1,2-shift of acetate (via intermediate **1198** and not **1196**) was proposed to account for the retention of stereochemistry. Similar processes were reported in the synthesis of bicyclo[3.1.0]hexenes from 1-acetoxy-5-ene-2-yne derivatives [936] and in gold-catalyzed cyclization of propargylic esters containing two alkene groups [937]. The coupling of bis(alkyne) ester **1199** with styrene in the presence of a gold catalyst led to net cycloaddition/cyclopropanation product **1203** [938]. A novel process was proposed involving 1,2-ester shift to afford carbene complex **1200**, followed by a propargylcarbene shift and cyclopropanation to afford alkynylcyclopropane **1202**, which then undergoes cyclization to afford the product **1203**. A related intramolecular bis(cyclopropanation) process employing a diene–diyne system was also reported [939]. Reaction of



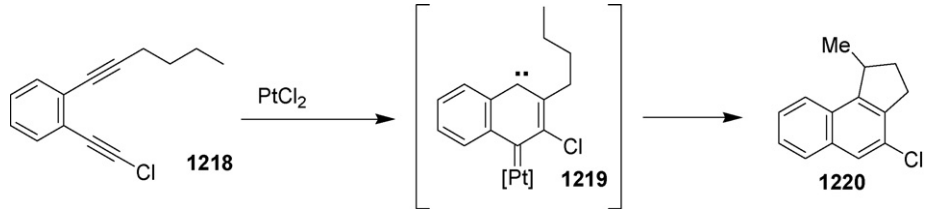
Scheme 120.



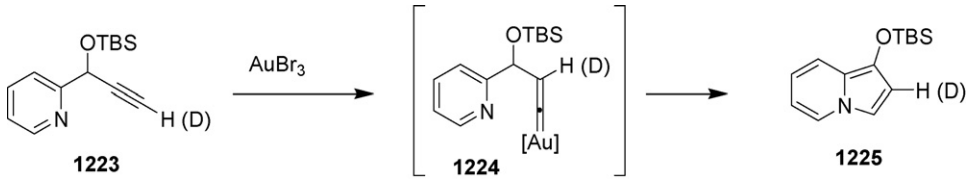
Scheme 121.



Scheme 122.



Scheme 123.

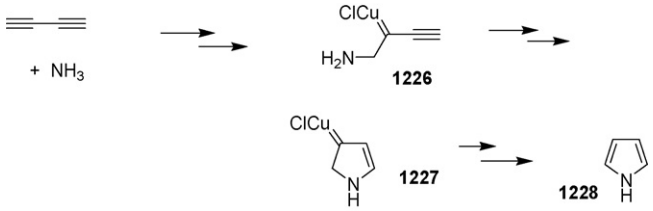


Scheme 124.

phenylpropargyl acetate (**1204**) with cationic gold complexes led to indene **1206** [940]. Gold carbene complexes were also suggested as intermediates in five-membered ring forming reactions involving enynyl propargyl acetates (*e.g.* **1207**), however the initial role of gold in this case is to induce a 1,3-shift of acetate to afford intermediate **1208**, which then undergoes a Nazarov-like process to afford the cyclopentenylidene–gold species **1209**, which gives rise to the product **1210** after hydration of the carbene complex functionality. An alternative five-membered ring forming cyclization was observed in the treatment of epoxy alkyne–esters (*e.g.* **1211**) with platinum chloride [941]. In this case, the initially formed carbene complex (**1212**) affords an ylide (**1213**), which affords the pyran (**1214**) after demetallation, which rearranges to the observed cyclopentenone (**1216**). Gold-catalyzed reactions of propargyl acetates were studied computationally [942].

Several other reaction processes were reported from the reaction of alkynes and platinum or gold complexes that might involve carbene complex intermediates (see Scheme 123). Platinum-catalyzed enyne metathesis (see Scheme 1) likely involves metal–carbene complex intermediates. Use of a bis(gold) complex for catalysis of enyne metathesis was reported [943]. Platinum carbene complexes (*e.g.* **1219**, Scheme 123) were suggested as intermediates in platinum-catalyzed cycloaromatization of conjugated enediynes (*e.g.* **1218**) [944]. A mechanism involving initial formation of the platinum carbene-free carbene complex **1219** followed by C–H insertion and demetallation was proposed.

Platinum carbene complexes were identified in the ion trap mass spectra of cationic platinum(II)–alcohol complexes [945]. Potential processes were evaluated by DFT calculations. Bimetallic carbene complexes involving platinum and another metal and their reaction with ammonia resulting in dehydrogenation were evaluated computationally [946]. Carbene resonance contribution to a *p*-platina–pyridine complex was discussed [947].

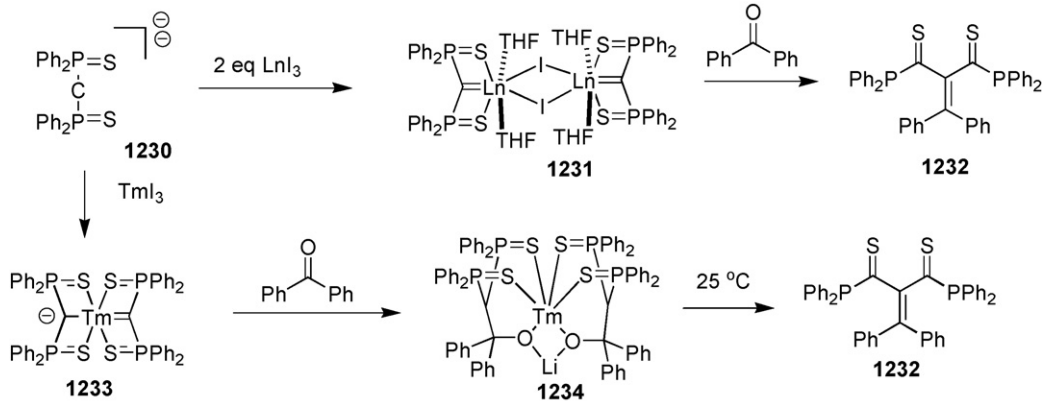


Scheme 125.

2.3.8. Group 11: carbene complexes

Gold vinylidenes (*e.g.* **1224**, Scheme 124) were suggested as intermediates in the isomerization of pyridine–alkynes (*e.g.* **1223**) to indolizines (*e.g.* **1225**) [948]. The intermediacy of a vinylidene complex was supported by deuterium labeling studies. The generation of copper carbene complexes through reaction of isothiocyanates with copper(I) phenoxide in the presence of a Lewis acid was reported [949]. These complexes were also studied computationally.

Several papers report on mechanistic aspects of copper–carbene complex intermediates generated from organic diazo compounds. A DFT study of copper–Tp complex catalyzed C–H insertions of diazo-esters was reported [950]. Stereoselectivity models were proposed for asymmetric cyclopropanation catalyzed by copper salts featuring chiral bidentate pyridine ligands [951]. Use of copper salts for copper catalyzed cyclopropanation was studied computationally [952,953].



Scheme 126.

Additional computational studies of Group 11 carbene complexes were reported in 2006. Copper carbene complexes were studied computationally with a focus on the degree of Cu–C double bond character [954]. The structure of various gold carbene complexes was studied through DFT calculations [955]. A DFT study of the addition of amines to diacetylenes to form pyrroles was reported (Scheme 125) [956]. The reaction was proposed to occur through copper carbene complex intermediates (**1226**, **1227**).

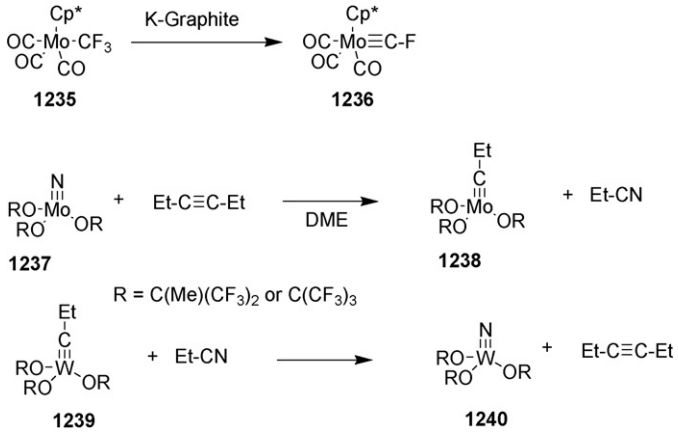
2.3.9. Lanthanide/actinide carbene complexes

The formation of pincer carbene complexes of lanthanum (*e.g.* **1231**, Scheme 126) and thulium (*e.g.* **1233**) was reported [957]. In both cases, the carbene complexes exhibited nucleophilic behavior, and coupled with benzophenone to afford the carbonyl olefination product **1232**. Polynuclear methylene–lanthanum and methylene–yttrium complexes were reported [958]. Although no actual metal-double bonded species was isolated, Schrock carbene-like behavior was demonstrated for the complexes. Reaction with ketones or aldehydes led to carbonyl olefination products.

3. Metal–carbyne or metal–alkylidyne complexes

3.1. Review articles

Review articles featuring metal–carbyne complexes which appeared in 2006 include: (1) perspectives on metal carbides, nitrides, and phosphides [959]; and (2) alkyne metathesis catalysts [960]. Reviews on titanium and vanadium carbene and carbyne complexes were noted in the carbene complex section [88,89].



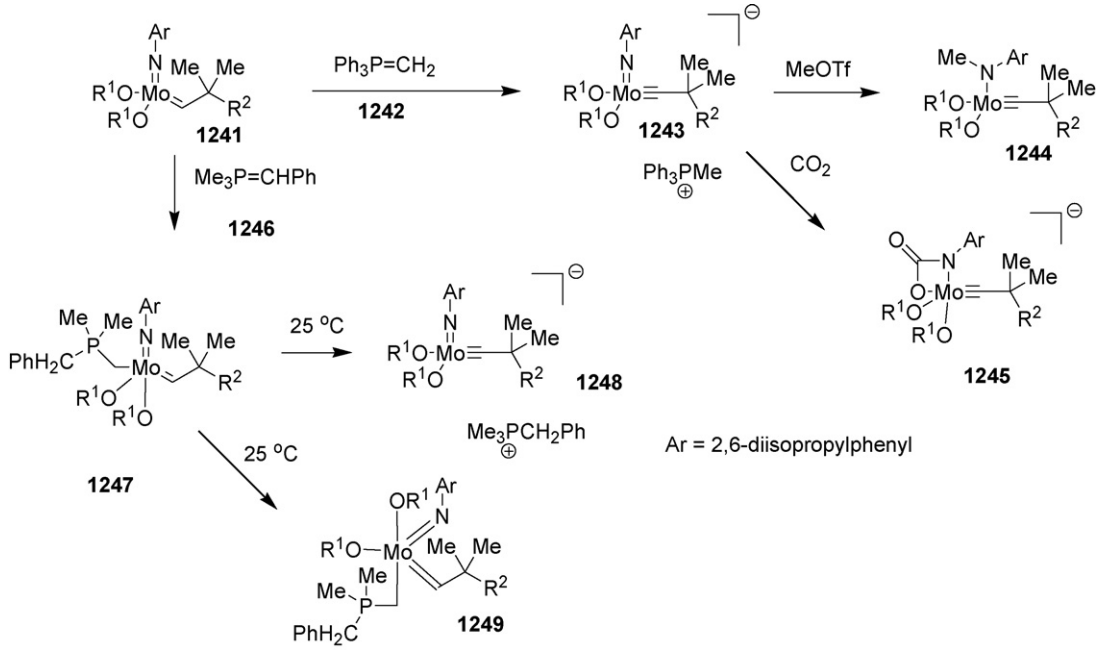
Scheme 127.

3.2. Synthesis and/or generation

Some papers in the carbene section feature minor segments on carbyne chemistry. These studies include Refs. [686,690,715,718,791,867,872,874,875].

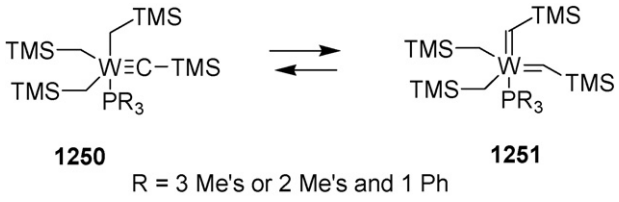
A molybdenum carbyne complex (**1236**, Scheme 127) was produced upon treatment of trifluoromethylmolybdenum complex **1235** with potassium graphite [961]. The formation of molybdenum carbyne complexes (*e.g.* **1238**) through the reaction of molybdenum nitrides (*e.g.* **1237**) with 3-hexyne was reported [962]. Nitriles were formed as by-products. The analogous tungsten reaction proceeded in the opposite direction. Tungsten carbyne complex **1239** reacts with nitriles to afford alkynes and tungsten nitride **1240**.

Anionic molybdenum carbyne complexes (*e.g.* **1243**, **1246**, Scheme 128) were prepared through reaction of the Schrock



Scheme 128.

carbene complex and derivatives with Wittig reagents [963]. Alkylation (silylation) of anionic carbyne complex **1243** occurred at nitrogen. Reaction with carbon dioxide led to the C=N [2 + 2]-cycloadduct **1245**. Reaction with the less hindered Wittig reagent **1246** led initially to the adduct **1247**, which transformed to a mixture the anionic carbyne complex (**1248**) and the isomeric adduct **1249**.



Scheme 129.

3.3. Reactivity

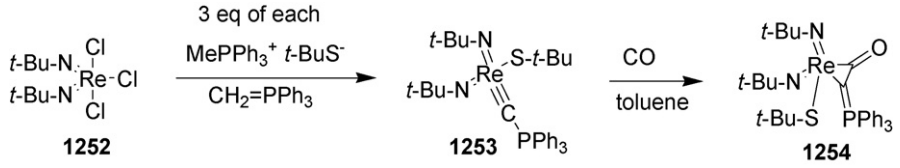
3.3.1. Addition reactions of metal–carbyne complexes

Tris(trimethylsilylmethyl)tungsten carbyne complexes (**1250**, Scheme 129) featuring various phosphine ligands were prepared [964]. The complexes exhibited fluxional behavior and were in equilibrium with the bis(carbene) complex structure. **1251**

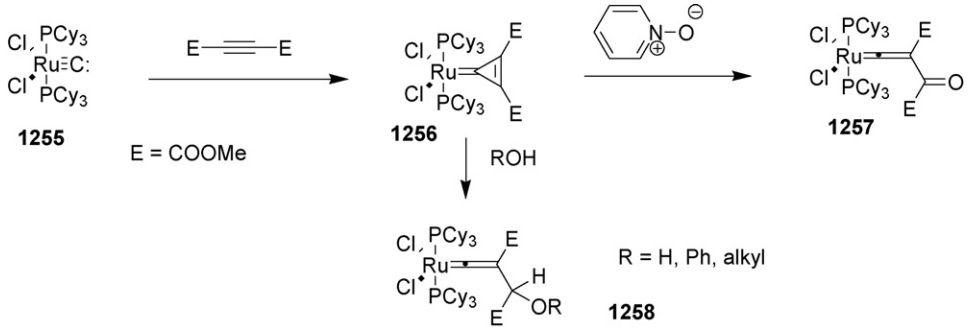
The synthesis of bis(imido)rhenium carbyne complexes (*e.g.* **1253**, Scheme 130) and subsequent reaction with CO was reported [965]. Reaction of bis(imido)rhenium trichloride complex **1252** with excess methylenetriphenylphosphorane and *t*-butyl thiolate led the carbine–phosphonium salt **1253**. Further reaction with CO led to the ketene complex **1254**.

Coupling of ruthenium carbide complex **1255** (Scheme 131) with dimethyl acetylenedicarboxylate led to the cyclopropenylidene complex **1256** [966]. Subsequent reaction with pyridine *N*-oxide led to the vinylidene complex **1257**. Reaction with alcohols, water or phenol led to the addition/ring-opening products (*e.g.* **1258**).

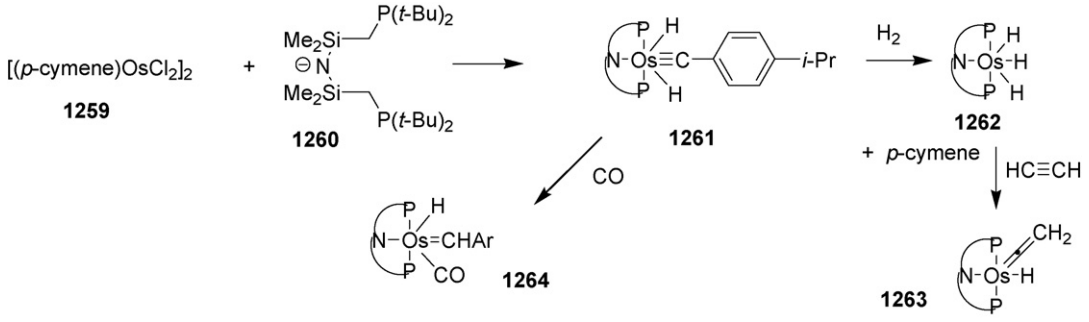
Osmium (dihydrido)carbyne complexes (*e.g.* **1261**, Scheme 132) were produced in the coupling of osmium complex **1259** with ligand additive **1260** [967]. A similar reaction with the analogous ruthenium complex afforded only a simple ligand substitution product. Reaction of the carbyne complex with CO led to the carbene complex **1264**. Reaction with



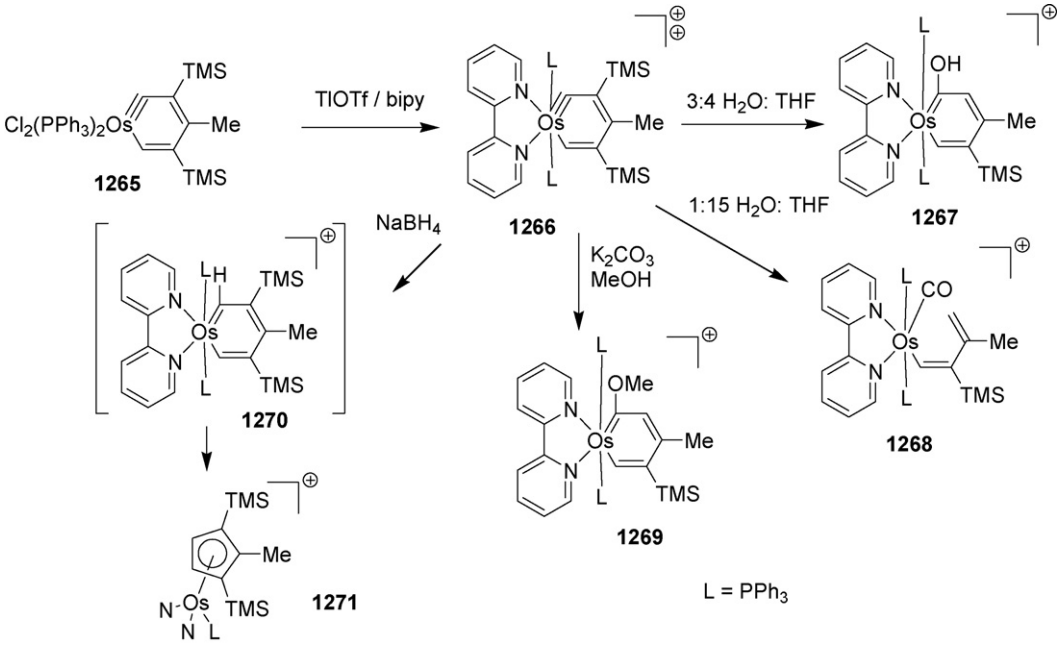
Scheme 130.



Scheme 131.



Scheme 132.



Scheme 133.

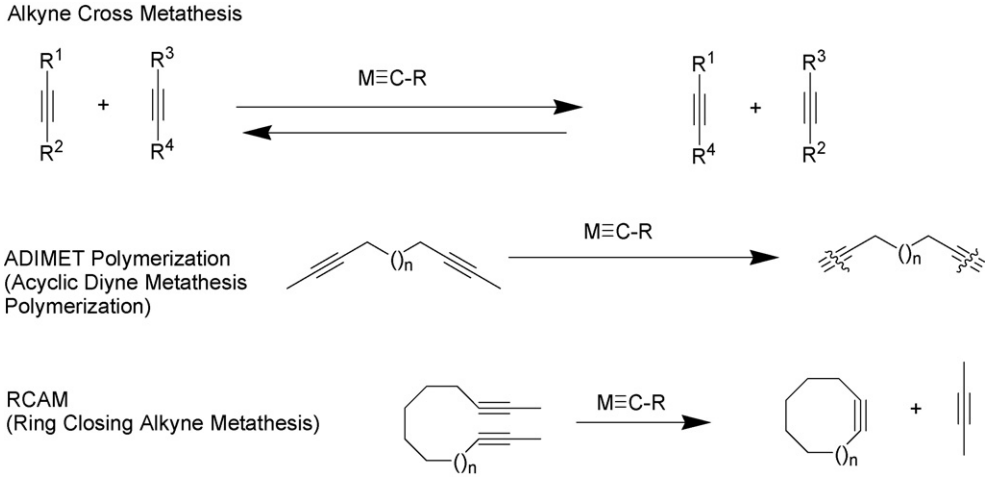
hydrogen led to the trihydride **1262** and *p*-cymene. Subsequent reaction of the trihydride with acetylene led to the vinylidene complex **1263**.

The synthesis and reactivity of osmabenzyne complex **1266** (Scheme 133) was reported [968]. Various nucleophilic reagents add to the carbene carbon. Reaction with water led to either the hydroxycarbene complex **1267** or the ring-opened CO complex **1268**, depending on the concentration of water in the medium. Both products were proposed to arise through the acyl complex, followed by either tautomerization or electrocyclic ring opening. Reaction with basic methanol led to the Fischer carbene complex (**1269**), while hydride addition led to the Cp complex **1271**. The Cp complex was proposed to arise from the carbene complex **1270**, which then undergoes alkyl migration to eventually afford the Cp complex.

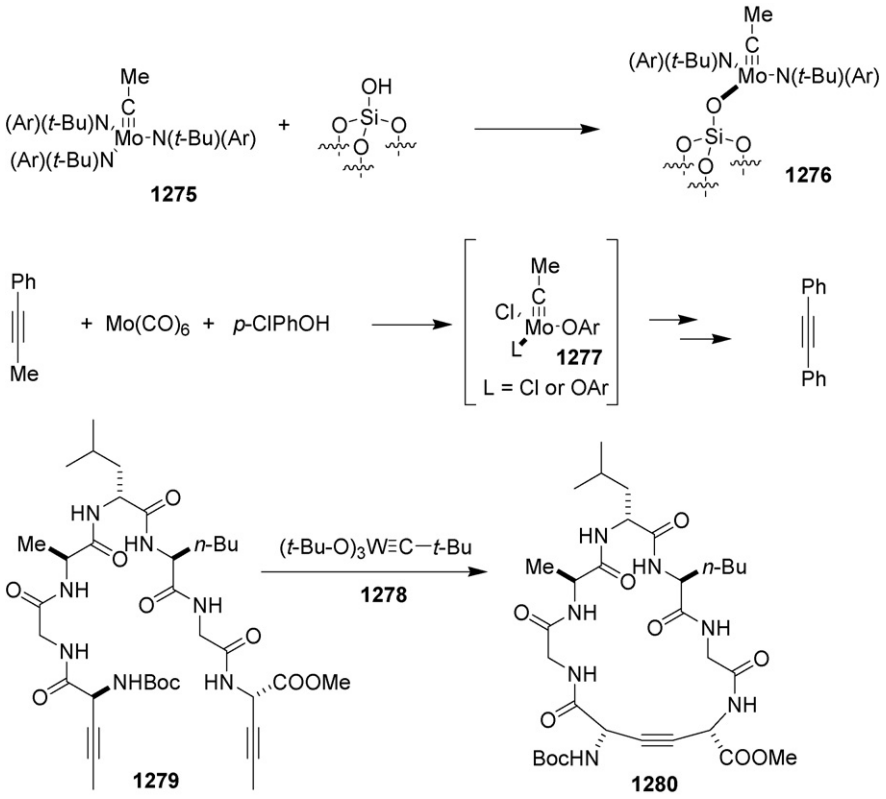
3.3.2. *Alkyne metathesis*

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 the mechanism and precedented modes are presented in Scheme 134.

Several examples of alkyne-cross metathesis were reported in 2006. Heterogeneous alkyne metathesis catalysts (*e.g.* **1276**, Scheme 135) were produced through reaction of a tris(amido)molybdenum carbyne complex **1275** with silica [969] or polyhedral oligomeric silsesquioxane [970]. A simple



Scheme 134.



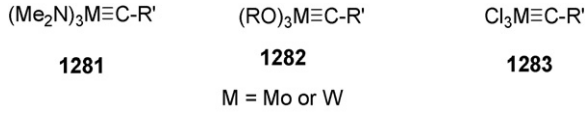
Scheme 135.

system for alkyne cross metathesis was developed employing molybdenum hexacarbonyl and 4-chlorophenol as catalyst [971]. DFT calculations of the hypothetical carbyne complex intermediate (**1277**) were also reported. Alkyne cross metathesis using terminal alkynes was reported using a $(t\text{-BuO})_3W{\equiv}C\text{-}t\text{-Bu}$ (**1278**)/quinuclidine system [972]. Macrocycle-bridged peptides (*e.g.* **1280**) were prepared through either ring-closing alkyne metathesis or though RCM [973].

A computational study of alkyne metathesis was reported [974]. In this manuscript the focus is the auxiliary ligands of the metal–carbyne complex catalysts (*e.g.* **1281**–**1283**, Scheme 136). Alkoxides are better ligands for the alkyne metathesis reaction since they have less π-donation ability and they result in stronger alkyne complexes. Tungsten complexes are generally better than molybdenum however use of electron-withdrawing alkoxide ligands increases the binding ability for the molybdenum complexes. Halide complexes lead to metallacyclobutadienes that are too stable and thus do not turn over.

3.3.3. Other processes involving metal–carbyne complexes

The reaction of $t\text{-BuC}{\equiv}W(CH_2t\text{-Bu})_3$ with phenols was reported [975]. The reaction with three or fewer equivalents of phenol leads to the analogs where the neopentyl ligands are replaced by aryloxide ligands (*e.g.* tungsten carbyne complex $t\text{-BuC}{\equiv}W(OAr)_3$ using three equivalents of phenol). The aryloxycarbene complex $t\text{-BuC(H)}{=}W(OAr)_4$ (Ar = 2,5-dimethylphenyl) was obtained when four equivalents of phenol was employed. The reaction of $Ph_3GeC{\equiv}W(O\text{-}t\text{-Bu})_3$ with three moles of $t\text{-BuCH}_2Li$ or $TMSCH_2Li$ resulted in the analogous tris(alkyl)tungsten carbene complexes [*e.g.* $Ph_3GeC{\equiv}W(CH_2t\text{-Bu})_3$] [976].

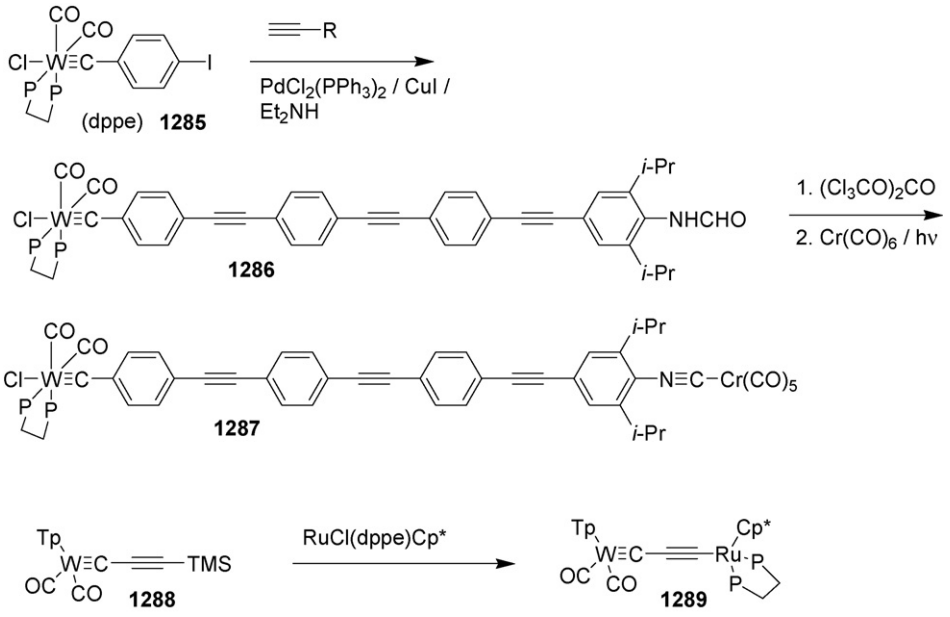


Scheme 136.

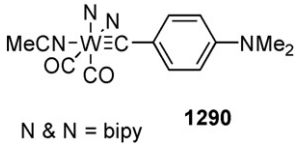
Synthesis and spectral properties were reported for tungsten–carbyne complexes conjugated to metal–isocyanide complexes (*e.g.* **1287**, Scheme 137) [977]. The synthetic route depicted in Scheme 137 transforms the iodophenylcarbyne complex **1285** into the formamide–carbyne complex **1286**. Dehydration of the formamide followed by complexation of the isocyanide group leads to the bimetallic carbyne complex **1287**. Formation of alkynylcarbyne complexes linked to additional metals (Ru, Co, Au) (*e.g.* **1289**) was also reported [978].

Electrochemical dimerization of cationic tungsten carbyne complexes (*e.g.* **1290**, Scheme 138) was reported [979]. It was suggested that the dimerization process upon electrochemical reduction is through the bipy ligands based on DFT calculations, which suggest that the negative charge is concentrated on the bipy ligands.

The reaction of laser-ablated titanium atoms with CCl_4 and CF_4 was reported [980]. The reaction leads to triply-bonded species $XC{\equiv}TiX_3$ which were isolated in an argon matrix. The structure was assigned through IR spectroscopy and comparisons with DFT-predicted spectra.



Scheme 137.



Scheme 138.

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