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Review

The chemistry of the carbon-transition metal double and triple bond: Annual survey covering the Year 2007☆

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Contents

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ABSTRACT

This is a review of papers published in the year 2007 that focus on the synthesis, reactivity, or properties of compounds containing a carbon-transition metal double or triple bond.

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Allenylidene complexes
Olefin metathesis
Alkyne metathesis

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 2007 [1,2]. 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 2007 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 [3,4]. This area was reviewed several times in 2007 [5–25]. The back donation issue for transition metal N-heterocyclic carbene complexes was evaluated computationally [26,27] and found to be present to a small extent in some cases. This survey has been divided into two sections, metal–carbene (or alkylidene) complexes and metal–carbyne (or alkylidyne) complexes; the carbene complex section represents the vast majority of this article. The metal–carbene section has been organized according to metal, starting from the left side of the Periodic Table. The Ionic Model [28] has been employed for the discussion of oxidation states and ligand electron count throughout this survey. A special section focusing on alkene metathesis has been included prior to the discussion of carbene complexes of individual metals. The metal–carbyne section has been organized according to reaction type. Articles from the journals *Angewandte Chemie International Edition*, *Chemistry*: *A European Journal*, *Tetrahedron*, and *Tetrahedron Letters* are restricted to volumes 46, 13, 63, and 48, respectively, which covers the period of December 2006–December 2007 according to some search engines.

Abbreviations (see also author instructions for the *Journal of Organic Chemistry* [29]).

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

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

2. Metal–carbene or metal–alkylidene complexes

2.1. Review articles, highlights, and comments

Several reviews/highlights/comments covering aspects of metal–carbene complex chemistry appeared in 2007. Many articles focusing on some aspect of carbene complex-initiated olefin metathesis were published, including the following specific subjects: (1) the history of alkene metathesis [30]; (2) the current status and outlook for alkene metathesis [31–34]; (3) catalytic olefin metathesis [35]; (4) development of olefin metathesis catalysts [36–38]; (5) search for improved metathesis catalysts [39]; (6) evolution and application of second-generation ruthenium olefin metathesis catalysts [40]; (7) immobilization of metathesis catalysts on silica [41,42]; (8) NHC's as ligands for metathesis catalysts [43]; (9) immobilized catalysts featuring chelating N,O-ligands [44]; (10) metathesis of amine-containing systems [45]; (11) sustainable concepts in olefin metathesis [46]; (12) chiral molybdenum carbene catalysts [47]; (13) formation of medium-ring heterocycles via ring closing and enyne metathesis [48]; (14) ruthenium vinylcarbene complexes in enyne metathesis [49]; (15) synthesis of natural products using enyne metathesis [50,51]; (16) olefin metathesis in organic synthesis [52]; (17) synthesis of heterocycles using enyne metathesis [53]; (18) synthesis of macrocyclic compounds by ring closing metathesis [54]; (19) metathesis of functionalized olefins [55]; (20) formation of polycyclic compounds via metathesis [56]; (21) synthesis of carbasugars via RCM [57]; (21) ring rearrangement metathesis [58]; (22) synthesis of nitrogen-containing organofluorine compounds using metathesis [59]; (23) recent developments in metathesis polymerization [60]; (24) living ring opening metathesis polymerization [61]; (25) olefin metathesis in dendrimers [62]; (26) synthesis of liquid crystal fill using metathesis [63]; (27) rhenium imido catalysts [64]; (28) metathesis-based commercial processes [65]; and (29) olefin metathesis in Brazil [66]. 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) tetrahydrofurans [67]; (2) α,β-unsaturated six-membered ring lactone natural products [68,69]; (3) tetrasubstituted alkenes [70]; (4) prostaglandins [71]; (5) marine polyether toxins [72,73]; (6) carbasugars [74]; (7) antitumor antibiotic FR900482 [75]; (8) fluorinated heterocycles [76]; (9) carbocycles from carbohydrate derivatives [77]; (10) antibacterial polymers [78]; (11) five-membered rings [79]; (12) resorcyclic acid lactones [80]; (13) lentiginosine [81]; (14) nicotine derivatives [82]; (15) multiporphyrins [83]; (16) polymers than contain phosphazene rings [84]; and (17) monolithic polymeric materials [85]. Additional review articles include some segments on carbene complex-initiated metathesis. Articles in this category focus on the following subjects: (1) transition metal-catalyzed reactions in non-conventional media [86]; (2) isotopic labeling in the study of organometallic reaction mechanisms [87]; (3) transition metal complexes as catalysts for double bond migration [88]; (4) NHC ligands in asymmetric catalysis [89]; (5) routes to NHC complexes [25]; (6) use of fluorinated alcohols as solvents in homogeneous catalysis [90]; (7) pattern recognition in retrosynthetic analysis [91]; (8) development of environmentally benign methods for drug discovery [92,93]; (9) catalytic processes in the manufacture of materials from natural oils and fats [94]; (10) microwave reactions under continuous flow conditions [95]; (11) automated solid phase oligosaccharide synthesis [96]; (12) divergent catalysis [97]; (13) supported ionic liquid catalysts [98]; (14) ruthenium catalysts in organic synthesis [99]; (15) iridium-catalyzed allylic alkylation [100]; (16) organic synthesis in ionic liquids [101]; (17) vanadium complexes for catalysis of olefin polymerization [102]; (18) nanocomposite materials from functional polymers [103]; (19) carbonic anhydrase inhibitors [104]; (20) APT quantum mechanical methods [105]; (21) recent progress in organic synthesis [106]; and (22) transition metal alkenyl complexes [107].

Several reviews on carbene complex chemistry featuring some aspect other that metathesis appeared in 2007. These reviews include the following subjects: (1) 1,3-metal shift processes in alkynylcarbene complexes [108]; (2) synthesis of nitrogen-

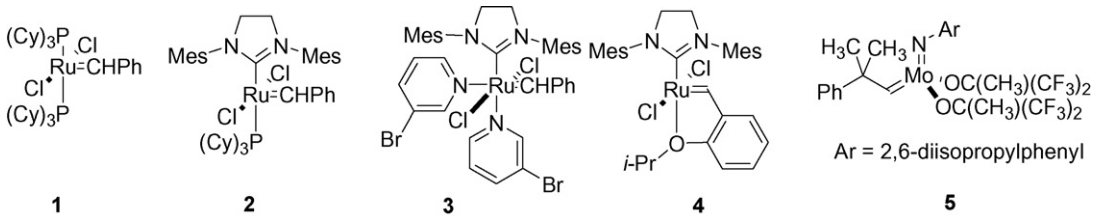
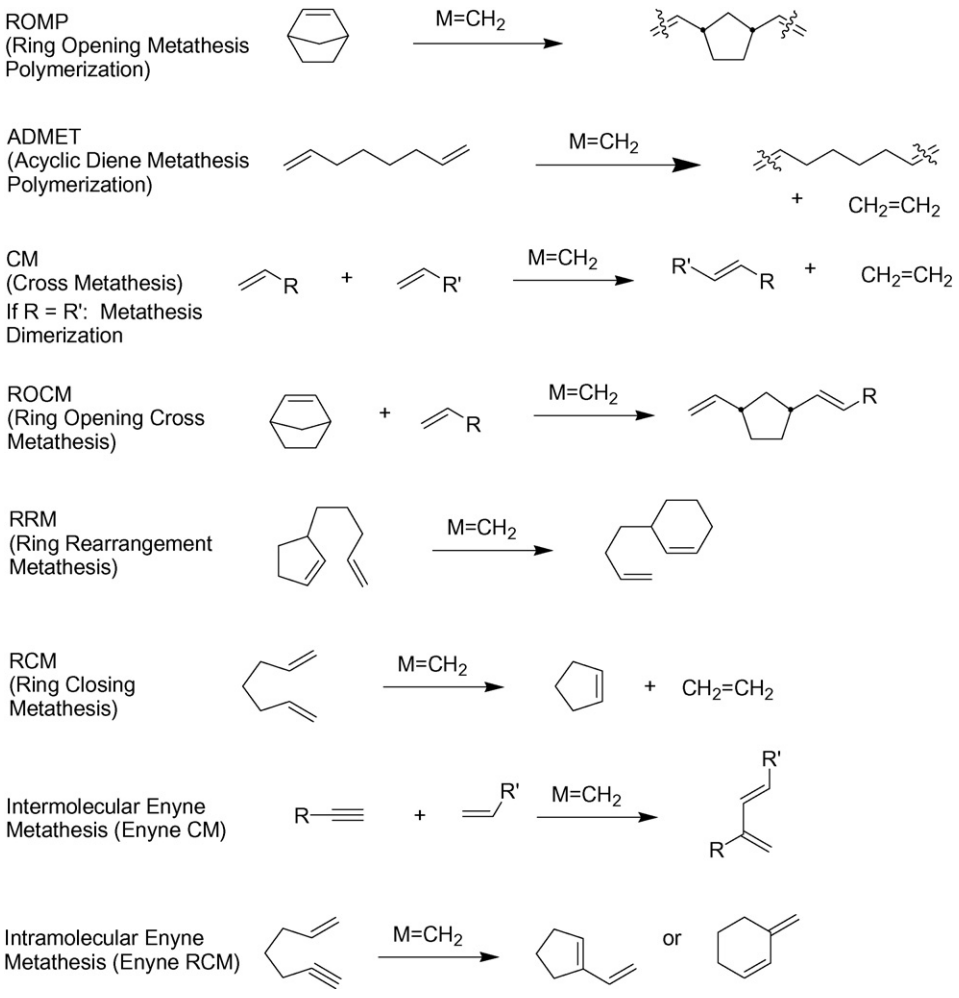


Fig. 1. Structures of alkene metathesis catalysts **1**–**4**.

containing compounds using Fischer carbene chromium complexes [109]; (3) Group 8 metal–carbon multiply bonded complexes [110]; (4) formation and reactivity of osmium–carbon double bonds [111]; (5) carbene complex radical anion chemistry [112]; (6) metal–carbene tautomerization processes [113]; (7) use of manganese vinylidene complexes in organometallic and organic synthesis [114]; (8) transition metal promoted reactions involving ruthenium vinylidene complexes [115]; (9) catalysis of propargyl substitution reactions using binuclear ruthenium complexes [116]; (10) titanium carbene complexes in organic synthesis [117]; (11) non-metathesis reactions of ruthenium carbene complexes [118]; and (12) annual survey of the carbon–metal double and triple bond for 2005 [119]. Although not specifically focusing on metal–carbene complexes, some review articles place some emphasis on this subject. Subjects reviewed in this category include: (1) synthesis of titanium imide complexes [120]; (2) titanium carbenoids [121]; (3) platinum and gold catalysis [122]; (4) gold-alkyne reactions [123]; (5) gold-catalyzed reactions of propargyl esters [124]; (7) acetate migration in gold- and platinum-catalyzed reactions of propargyl esters [125]; (8) oxygen templating approach to eight- and nine-membered ring carbocycles [126]; (9) characterization of coordination compounds via electrochemistry [127]; (10) anisotropically ordered conjugated polymers [128]; (11) rhodium catalyzed polymerization of ethyl diazoacetate [129]; (12) C–H activation reactions catalyzed by $ReBr(CO)_3(PPh_3)_2$ [130]; (13) complexes featuring the dianionic polydentate phosphino sulfides as ligands [131]; (14) compounds of Groups 8–10 that contain carbon–metal σ-bonds [132]; (15) Group 8 metal complexes [133]; and (16) silver and gold complexes [134].

2.2. Alkene metathesis

Alkene metathesis was the most common reaction process reported for metal–carbene complexes in 2007, and this special section is devoted to papers that focus on this process. Many examples of both polymerization [mostly ring opening metathesis



Scheme 1.

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 2007; some representative examples are depicted in Fig. 2. Several derivatives of the Grubbs and Schrock catalysts (see Fig. 1) were synthesized and tested in their ability to undergo various olefin metathesis processes, including: (1) analogs of Grubbs catalyst I featuring pyridinedicarboxamido ligand in place of the chlorides and one tricyclohexylphosphine [135]; (2) water soluble analogs of Grubbs catalyst I that feature a polar group attached the aryl ring [136]; (3) analogs of Grubbs catalyst I bound to a Merrifield resin [137]; () indenylidene analogs of Grubbs catalysts I and II [138]; (4) analogs of Grubbs catalyst II featuring a triphenylphosphine ligand and 2,6-dimethylphenyl groups in place of the mesityl groups [139]; (5) analogs of Grubbs catalyst II featuring pyridine ligands [140,141]; (6) a chiral analog of Grubbs catalyst II (*e.g.* **6**) [142]; (7) fluorous analogs of Grubbs catalyst II [143]; (8) analogs of Grubbs catalyst II featuring chelating phosphine–carboxylate ligands (*e.g.* **7**) [144]; (9) allenylidene analogs of Grubbs catalyst II and additional examples featuring either pyridine ligands of salicylimine ligands [145]; (10) a diphenylyclohexylphosphine analog of Grubbs catalyst II [146]; (11) trialkylphosphine analogs of Grubbs catalyst II [147]; (12) analogs of Grubbs catalyst II and/or the Hoveyda–Grubbs catalyst where one of the mesityl groups has been replaced by a 2,6-diisopropropylphenyl group [148] or a 2,6-difluorphenyl group [149]; (13) chelating pyridyl analogs of Grubbs catalysts I and II (*e.g.* **8**) [150]; (14) an analog

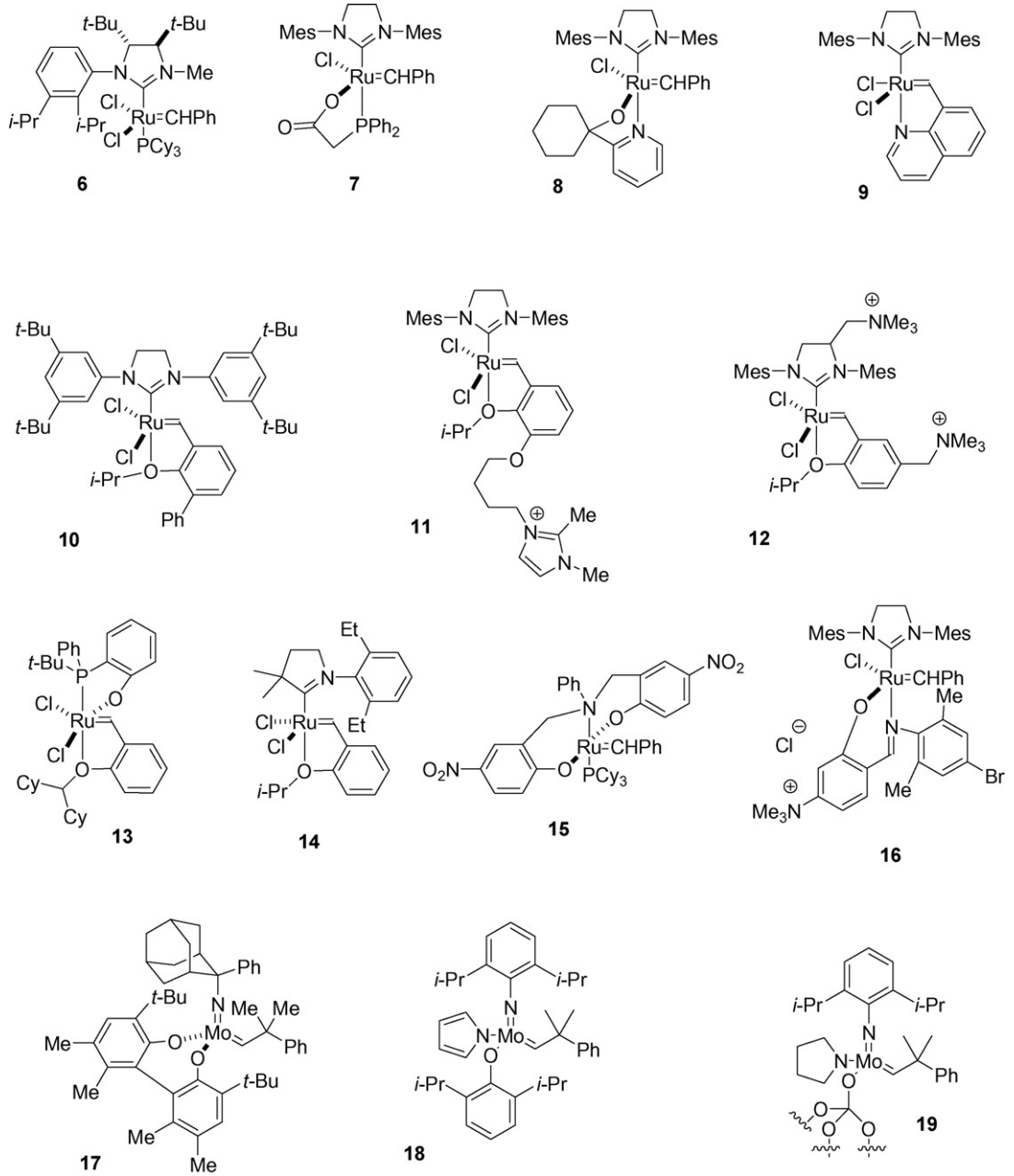


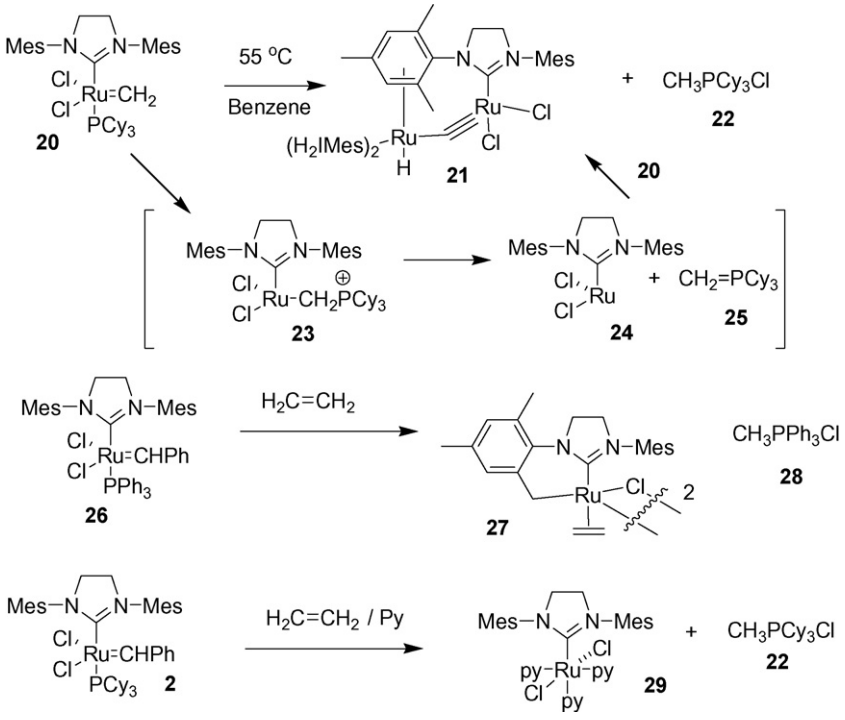
Fig. 2. Representative examples of new catalysts for alkene metathesis.

of Grubbs catalyst II where a chelating quinoline group replaces the phenylcarbene group (**9**) [151]; (15) fluoromethylene analogs of Grubbs catalysts II and III [152]; (16) polymethyldisiloxane-bound Grubbs catalyst II for metathesis reactions in a capillary gas chromatograph [153]; (17) a bis(3,5-di-*t*-butylphenyl) analog of the Hoveyda–Grubbs catalyst (**10**) [154] and a related bis(*o*-tolyl) analog that are effective catalysts for tetrasubstituted alkene formation [155]; (18) an analog of the Hoveyda–Grubbs catalyst featuring a chlorinated carbene aryl group [156]; (19) nitroarylcarbene analogs of the Hoveyda–Grubbs catalyst [157]; (20) analogs of the Hoveyda–Grubbs catalyst featuring different N-substituents in the NHC ligand [158]; (21) a heptane-soluble analog of the Hoveyda–Grubbs catalyst that features a long-chain alkyl group attached to the carbene–phenyl group [159]; (22) a polyphenylene-bound analog of the Hoveyda–Grubbs catalyst [160]; (23) analogs of the Hoveyda–Grubbs catalyst featuring modifications at the oxygen ligand [161]; (24) analogs of the Hoveyda–Grubbs catalyst featuring NHC ligands containing a long alkyl chain of perfluorocarboxylate ligands in place of halide ligands [162]; (25) analogs of the Hoveyda–Grubbs catalyst featuring chelating NHC ligands [163]; (26) analogs of the Hoveyda–Grubbs catalyst featuring imidazolium salts (*e.g.* **11**) [164] or pyridinium salts [165]for metathesis in ionic liquids; (27) analogs of the Hoveyda–Grubbs catalyst featuring tetraalkylammonium salts useful for aqueous phase metathesis reactions (*e.g.* **12**) [166]; (28) an analog of the Hoveyda–Grubbs catalysts where a chelating phosphine ligand replaces the NHC ligand (**13**) [167]; (29) analogs of the Hoveyda–Grubbs catalyst bound to silica through the carbene phenyl group [168]; (30) analogs of the Hoveyda–Grubbs catalysts linked to a carborane [169]; (31) a polyethylene-glycol bound analog of the Hoveyda–Grubbs catalyst that is easily removed by filtration [170]; (32) an analog of the Hoveyda–Grubbs catalyst bound to silaceous mesocellular foam through the carbene phenyl group [171]; (33) chelating carboxylate analogs of the Hoveyda–Grubbs catalyst that become active upon protonation [172]; (34) analogs of the Hoveyda–Grubbs catalyst featuring polymer-bound fluorinated carboxylate ligands [173]; (35) a Bertrand carbene analog of the Hoveyda–Grubbs catalyst (**14**) [174]; (36) a ruthenium carbene complex featuring a chelated proline ligand in place of the NHC and one chlorine ligand [175]; (37) ruthenium carbene complexes featuring chelating salicylamine ligands (*e.g.* **15**) [176,177] and salicylaldimine ligands (*e.g.* **16**) [178]; (38) chiral analogs of the Schrock catalyst (*e.g.* **17**) [179]; (39) silylmethylene analogs of the Schrock catalyst [180]; (40) tris(*t*-butyl)silyl and/or N-pyrrolyl analogs of the Schrock catalyst [181]; (41) alkoxy(N-pyrrolyl) analogs of the Schrock catalyst, useful for alternate regioselectivity enyne metathesis (*e.g.* **18**) [182]; (42) chiral analogs of the Schrock catalyst prepared from N-pyrrolyl(η^5-pyrrole)-molybdenum carbene complexes [183]; (43) silica-bound analogs of the Schrock catalyst (*e.g.* **19**) [184,185]; (44) a ruthenium carbene complex generated *in situ* through reaction of ruthenium nanoparticles with ethyl diazoacetate [186]; (45) *in situ* generation and solution characterization (NMR) of a methylenevanadium complex [187]; and (46) ruthenium carbene complexes that contain an NHC-ligand as the only carbene ligand [188–192]. A convenient large-scale preparation of the Hoveyda–Grubbs catalyst was reported [193]. Use of Simmons–Smith type reagents to prepare methylene analogs of Grubbs catalyst I, in addition to other metal–carbene complexes, was reported [194]. Use of 2-pentafluorophenyl-1,2-dimesityltetrahydromidazole as a source of the NHC ligands of Grubbs catalyst II and the Hoveyda–Grubbs catalyst was demonstrated [195]. Improved syntheses of ruthenium allenylidene catalysts and mechanistic investigations of their formation were reported [196]. Several patents were issued for the synthesis and development of metal–carbene containing olefin metathesis catalysts [197–203].

Potential decomposition pathways for methylene analogs of Grubbs catalyst II (*e.g.* **20**, Scheme 2), a hypothetical catalytic species in olefin metathesis, were reported [204]. All of the decomposition studies were accompanied by the formation of a methylphosphonium salt (**22**, **28**). Decomposition at 55 °C led to the dimeric complex **21** and methyltricyclohexylphosphonium chloride (**22**). A mechanism involving formation of the ruthenium-phosphinomethylene complex **23** followed by loss of the phosphorane ligand to form dichlororuthenium complex **24**, followed by proton transfer and dimerization was proposed. Reaction of Grubbs catalyst II (**2**) with ethylene in the presence of pyridine led to ruthenium complex **29** and the phosphonium salt. Reaction of the triphenylphosphine analog (**26**) led to a C–H activation product **27**.

The formation of stable ruthenacyclobutanes (*e.g.* **31**, Scheme 3) through reaction of 14-electron carbene complex-phosphonium salt **30** with ethylene was reported [205]. The metallacyclobutane features significant C–C agostic interaction, and undergoes rapid exchange with labeled ethylene via an associative mechanism. The ethylene exchange process was studied computationally [206]. Related complexes were involved in a study designed to better understand the reluctancy of vinyl chlorides to participate in cross metathesis [207]. Attempted metathesis of vinyl chlorides using Grubbs catalyst II led to the carbide complex **32** and the carbene complex/phosphonium salt **33**. The hypothetical metathesis intermediate **34** could be generated through treatment of the carbide complex with HCl at low temperature, which then rearranges to the carbene complex/phosphonium salt **33** upon warming. Additional syntheses of carbene complex/phosphonium salt **33** were also reported.

Other general studies of alkene metathesis where carbene complexes were discussed include: (1) a study of Grubbs catalysts I and II by K-edge X-ray absorption spectroscopy and subsequent attempts to equate ligand binding effects with relative reactivity in olefin metathesis [208]; (2) the stopping of a metathesis reaction through addition of amines (*e.g.* N-methylimidazole) and subsequent restarting through addition of phosphoric acid [209]; (3) a comparison of microwave vs. thermal conditions for cross metathesis reactions [210]; (4) an experimental study that shows that identical propagating species are involved in metatheses using the Noels catalytic system {[$RuCl_2$(*p*-cymene)(PCy_3)] and $TMSCHN_2$} and Grubbs catalyst I [211]; (5) a DFT study comparing various molybdenum (tungsten) imido carbene complexes and rhenium carbyne carbene complexes as alkene metathesis catalysts [212]; (6) use of MOG-L DFT to study the mechanism for alkene metathesis and to accurately predict the increased activity of Grubbs catalyst II relative to Grubbs catalyst I [213]; (7) a DFT study of various ligand combinations for analogs of Grubbs catalyst II and prediction of relative reactivity for various ligand combinations [214]; (8) a DFT study addressing the formation of carbene complexes in metathesis reactions initiated by ruthenium(II)-NHC complexes [215]; (9) a DFT study of hypothetical ring opening metathesis reactions involving cyclohexene [216]; (10) a DFT study of enyne metathesis favoring an associative mechanism for small phosphine-ligated ruthenium catalysts and a dissociative mechanism for NHC-ligated systems [217]; (11) a DFT study of metathesis reactions involving halogenated alkenes [218]; (12) a comparison of different quantum chemistry approaches to the study of ruthenium carbene-catalyzed olefin metathesis [219]; (13) a computational study of the reaction of ruthenium and osmium carbene-oxo complexes with ethylene [220]; (14) a computational study of metathesis using molybdenum-silica systems [221–223]; (15) RCM reactions in highly aqueous media [224]; (14) RCM reactions in an ionic liquid [225]; (16) generation of carbene complex intermediates in ROMP of norbornene initiated by a $RuCl_3$/Zn system [226]; (17) *in situ* generation of carbene complexes in ROMP reactions initiated through

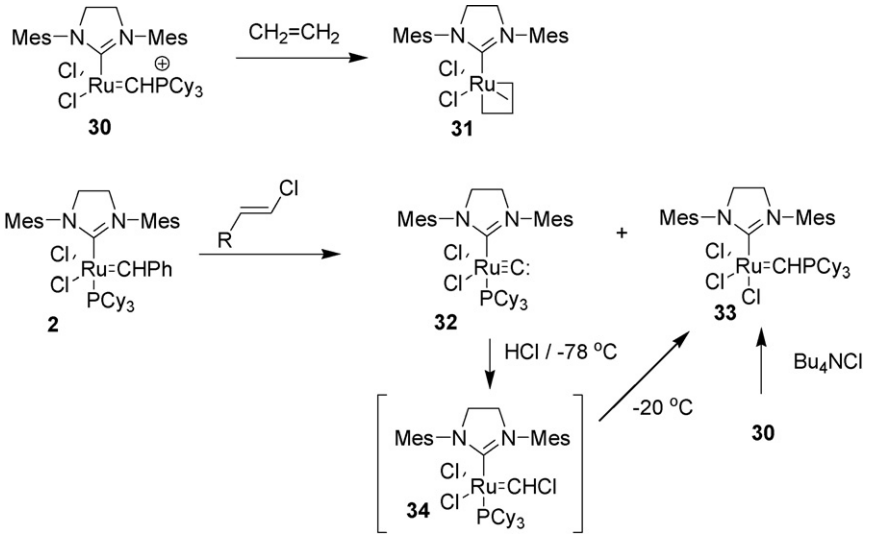


Scheme 2.

bis(tungsten) complexes [227]; (18) initiation steps for metathesis reactions employing rhenium(VII) oxide catalysts [228,229]; and (19) control of molecular weight and molecular weight distribution in ROMP reactions through use of triphenylphosphine [230]. A patent was awarded for development of a technique to remove metallic byproducts from ROMP reactions [231]. Undergraduate lab experiments involving the synthesis and evaluation of ruthenium metathesis catalysts were reported [232,233].

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 2007; 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) norbornenecarboxylic acid [234]; (2) O-hydroxysuccinimide esters of norbornenecarboxylic acid [235]; (3) various norbornenecarboxylate esters [236–238]; (4) norbornenedicarboxylate esters [239–242]; (5) norbornene-dicarboxylate esters, and subsequent hydrogenation, both processes catalyzed by Grubbs catalyst III [243]; (6) norbornene ester-nitriles [244]; (7) norbornenylmethyl esters or norbornenylmethylhydrazines [245]; (8) norbornenedimethanol esters [246,247]; (9) norbornene-azobenzenes (*e.g.* **35**) [248]; (10) norbornenecarboxamide derivatives of ethylenediamine [249]; (11) norbornenes connected to trithiocarbonates [250]; (12) norbornenes connected to pyrrolidinoxyl



Scheme 3.

radicals [251]; (13) norbornenes fused to succinimides [252–257]; (14) oxanorbornene [258]; (15) benzoxanorbornenes fused to succinimides [259,260]; (16) oxanorbornenes that contain groups capable of initiating radical polymerization (*e.g.* **36**) [261]; (17) norbornenes connected to protected ribonolactone [262]; (18) norbornenes connected to fluorine, carbazole, or pyrene [263]; (19) norbornenes connected to phenanthroimidazole rings [264]; (20) norbornenes connected to xanthine dyes [265]; (21) norbornenes connected to triazines [266]; (22) norbornenes linked to phosphazenes (*e.g.* **37**) [267]; (23) norbornenes attached to poly(methyl methacrylate) chains [268]; (24) norbornenes fused to cyclic sulfonium salts (*e.g.* **38**) [269]; (25) associated norbornenes (*e.g.* **40**) [270,271]; (26) norbornenes connected to ammonium salts or hydrogen-bonding motifs [272]; (27) norbornenes connected to oligothiophene units [273]; (28) norbornenes connected to peptides [274]; (29) norbornenes connected to dendrimer precursors [275]; (30) norbornenes connected to polystyrene-polylactate copolymers [276]; (31) norbornenes connected to polylactide chains [277]; (32) bis(norbornene derivatives (*e.g.* **39**), which function as cross-linkers for ROMP polymers [278]; (33) norbornenes connected to Ru-BINAP systems for preparation of immobilized hydrogenation catalysts (*e.g.* **41**) [279]; (34) norbornenes connected to iridium bis(carbazolyl) complexes [280]; (35) norbornenes connected to carboranes [281]; (36) dicyclopentadiene [282–285]; (37) norbornenes fused to fullerenes [286]; and (38) norbornenes connected to monoliths [287]. Copolymers prepared through ROMP reported in 2007 include: (1) a norbornene connected to a platinum(II) complex (**43**) and a norbornene connected to a highly conjugated aromatic system (**42**) [288]; (2) norbornenes fused to succinimides and norbornenes connected to ruthenium bipyridyl complexes [289]; (3) norbornene connected to piperidinoxyl radicals (**44**) with bis(norbornenes) (*e.g.* **45**) [290]; and (4) bridged calixarenes, cyclooctene, and norbornene [291]. Other ring systems that have been subjected to ROMP reactions include: (1) macrocycle-bridged disaccharides (*e.g.* **46**) [292]; (2) cyclooctene derivatives [293–295]; and (3) cyclooctenes connected

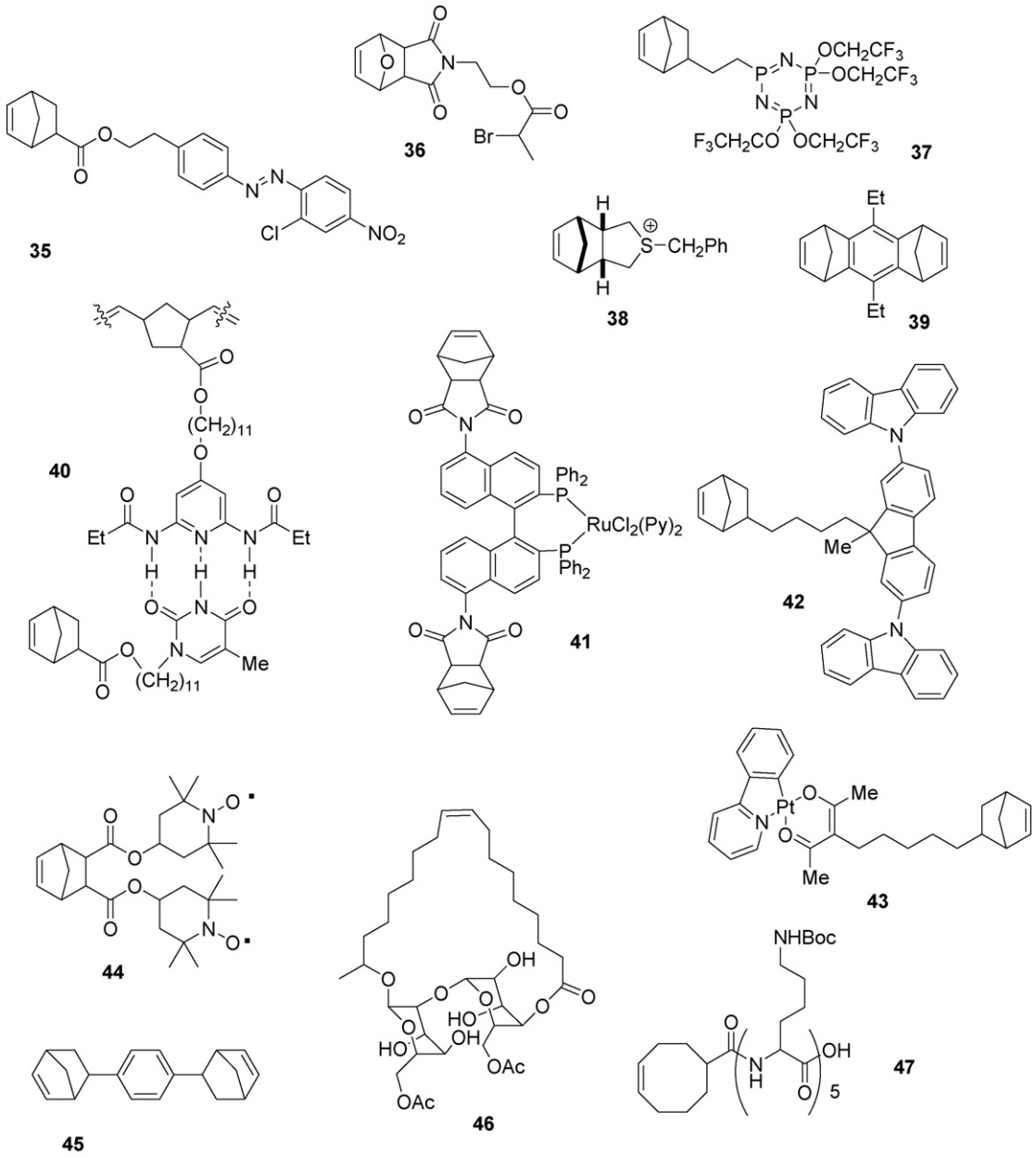


Fig. 3. Representative substrates for the ROMP reaction.

to polyamide groups (*e.g.* **47**) [296]. Polymerization of norbornene using the Schrock catalyst followed by quenching of the active carbene species with *p*-alkoxybenzaldehyde derivatives was reported [297]. *In situ* formation of a gold-bound carbene complex and subsequent ROMP of alkylnorbornenes was reported [298]. Similar studies were reported for alkenes bound to a silicon surface [299,300]. Mechanistic studies of the copolymerization of 1,5-cyclooctadiene and various norbornene derivatives were reported [301].

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) hexyl-substituted α,ω-dienes [302]; (2) chlorine-containing α,ω-dienes [303]; (3) bromine-containing α,ω-dienes (*e.g.* **50**) [304]; (4) ester-containing α,ω-dienes [305]; (5) acetal linked α,ω-dienes (*e.g.* **51**) [306]; (6) alkene-containing 2,7-diamido-1,8-naphthyridine derivatives (*e.g.* **52**) [307]; (7) *p*-divinylbenzenes in an aqueous emulsion (*e.g.* **53**) [308]; (8) α,ω-dienes connected through bis(biphenyl)fluorine system [309]; (9) a steroid-bridged α,ω-diene [310]; (10) ADMET polymerization of ester-linked α,ω-dienes and subsequent metathesis depolymerization reactions [311]; (11) ADMET polymerization of ester-containing α,ω-dienes (*e.g.* **54**) and ROMP of ester-containing cyclooctenes [312]; (12) synthesis of hyperbranched macromolecules through ADMET polymerization of a diester-triene (*e.g.* **55**) [313]; and (13) cyclic vs. acyclic ADMET/ROMP polymers/oligomers in the reaction of salen-cobalt-cyclooctene derivative **56** with Grubbs catalyst III [314]. Competition between ADMET polymerization and macrocyclic RCM was studied for various esters containing alkene groups at both ends of the chain (*e.g.* **57**) [315]. The course of the reaction was very concentration dependent. RCM was favored at high dilution, while ADMET polymerization was favored at high concentration. The ADMET polymer could be converted to the RCM product by secondary reaction with the metathesis catalyst.

2.2.3. Nonpolymer-forming ring opening metathesis reactions

Several examples of RO-CM (see Scheme 1) were reported in 2007. Representative examples are depicted in Scheme 4. Reaction of cyclopropenone acetal **61** with alkene **60** and a later RCM event were key steps in the total synthesis of spirofungin A [316]. Regioselective RO-CM reactions were reported for 7-aza-1-substituted norbornene derivatives (*e.g.* **63**) and monosubstituted alkenes [317]. Oxaborbornenedicarboxylic anhydride was also employed in RO-CM reactions with styrene derivatives [318]. Asymmetric co-metathesis of various 8-azabicyclo[3.2.1]octene derivatives (*e.g.* **65**) and styrene using chiral molybdenum or ruthenium catalysts was reported [319,320]. In this case the identity of the R group determined whether molybdenum or ruthenium catalysts provided higher ee's. Enantioselective RO-CM of 8-oxabicyclo[3.2.1]-octenes (*e.g.* **67**) was employed in a total synthesis of baconipyrone C [321]. Asymmetric RO-CM of cyclopropenes and monosubstituted alkenes was also reported [322]. Other studies include opening of the macrocyclic and medium-size rings of the natural product manzamine A through RO-CM with ethylene [323]. The RO-CM of peptide-bound norbornenes (*e.g.* **69**) with alkene-containing amino acids was reported [324].

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 2007. 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) resin-bound carbohydrates (through an alkene) and ethylene for resin release [325]; (2) fluorous-tagged oligosaccharides and ethylene in an effort to remove the fluorous tag [326]; (3) the natural product apicularen A and ethylene [327]; (4) vinyldihydroisobenzofuran derivatives and monosubstituted alkenes [328]; (5) nitro group-containing alkenes and monosubstituted alkenes [329]; (6) 2Z,4E-dienyl esters (*e.g.* **76**) and monosubstituted alkenes (*e.g.* **75**) [330]; (7) unsaturated amino acid derivatives and various monosubstituted alkenes [331]; (8) double metathesis of C_2 symmetric bis(tetrahydrofurans) (*e.g.* **77**) and monosubstituted alkenes (*e.g.* **78**) [332]; (9) O-allylcarbohydrate derivatives and monosubstituted alkenes [333–335]; (10) C-alkenyl glycosides and monosubstituted alkenes [336]; (11) 2-vinylthiazoles and various monosubstituted alkenes [337]; (12) steroidal alkenes and various monosubstituted alkenes [338]; (13) α-methylene lactones (*e.g.* **79**) and monosubstituted alkenes (*e.g.* **80**) [339,340]; (14) allylbenzenes and methyl acrylate [341]; (15) unsaturated fatty acid esters and methyl acrylate [342]; (16) a silyloxyheptenol and methyl acrylate [343]; (17) vinylcarbohydrate derivatives and methyl acrylate [344]; (18) alkene alcohols and methyl acrylate [345]; (19) a homoallylic ether and benzyl acrylate [346]; (20) a complex alkene and acrolein for preparation of a dictyostatin fragment [347]; (21) an allyloxymethyl indole and methacrolein [348]; (22) a 4-pentenol silyl ether with crotonaldehyde [349]; (23) α,β-unsaturated ketones (*e.g.* **81**) and homoallylic amine derivatives (*e.g.* **82**) followed by spontaneous Michael addition to afford substituted pyrrolidines (*e.g.* **83**) [350]; (24) an α,β-unsaturated ketone and a homoallylic ether [351]; (25) a homoallylic alcohol and a 4-pentenoate ester [352]; (26) a homoallylic alcohol and allyltrimethylsilane [353]; (27) homoallylic alcohols and lactone alkenes for synthesis of pyranones [354]; (28) a homoallylic alcohol and a vinylborane as part of a failed synthetic route to bruguierol C [355]; (29) homoallylic amines and cis 1,4-diacetoxy-2-butene [356]; (30) amine-dienes and diacetoxy-2-butene [357]; (31) a β,γ-unsaturated ester with 1,4-diacetoxy-2-butene [358]; (32) a homoallylic ester and 1-octadecene [359]; (33) allylnucleoside bases and vinylphosphonate esters [360]; (34) vinylnucleosides and 2-hydroxyvinylacetic acid esters [361]; (35) an allylic fluoride and 1-pentadecene [362]; (36) an allytetrahydrofuran derivative and a 4-pentenoic acid derivative [363]; (37) C-alkenyl glycosides and N-acryloyl peptides [364]; (38) imidazopyridine derivatives and styrene derivatives [365]; (39) borrorneates and styrene derivatives [366]; (40) alkenes linked to Bodipy chromophores (*e.g.* **84**) and densely functionalized alkene derivatives (*e.g.* **85**) [367]; (41) selective CM (no RCM) of electron-deficient alkenes and 4-sila-1,6-dienes [368]; (42) two different alkenes bound to a peptide chain [369]; (43) polymer-bound alkenes and quinine [370]; (43) crown ether-assisted CM of ferrocene-linked alkenes (*e.g.* **86**) and acrylate esters [371]; (44) a porphyrin-linked alkene and a fullerene-linked alkene [372]; and (45) styrene with molecular knots containing alkene groups [373]. Several publications appeared in 2007 where cross metathesis was employed in natural products synthesis. Examples of alkenes employed in CM for natural products synthesis include: (1) a γ,δ-unsaturated ester and the Weinreb acrylamide derivative for azaspiracid A total synthesis [374]; (2) Weinreb amide derivatives and monosubstituted alkenes for synthesis of amphidinolide fragments [375] and total synthesis of muricatacin [376]; (3) an allylic alcohol and a homoallylic amine for total synthesis of an indolizidine natural product [377]; (4) an allylic alcohol derivative and phenyl vinyl ketone for total synthesis of diospongin [378]; (5) an allylic alcohol and 11-methyl-1-dodecene for total synthesis of plusbacin A_3 [379]; (6) an allylic alcohol derivative and an allylamide derivative for total synthesis of andrachcinidine [380]; (7) 1-octen-3-ol and acrolein for total synthesis of 4-hydroxy-2-nonenal [381]; (8) an alkene–alcohol and methyl acrylate for eventual total synthesis of spirastrellolide [382]; (9) an allylic ether and styrene for total synthesis of crocacin [383]; (10) a vinylte-

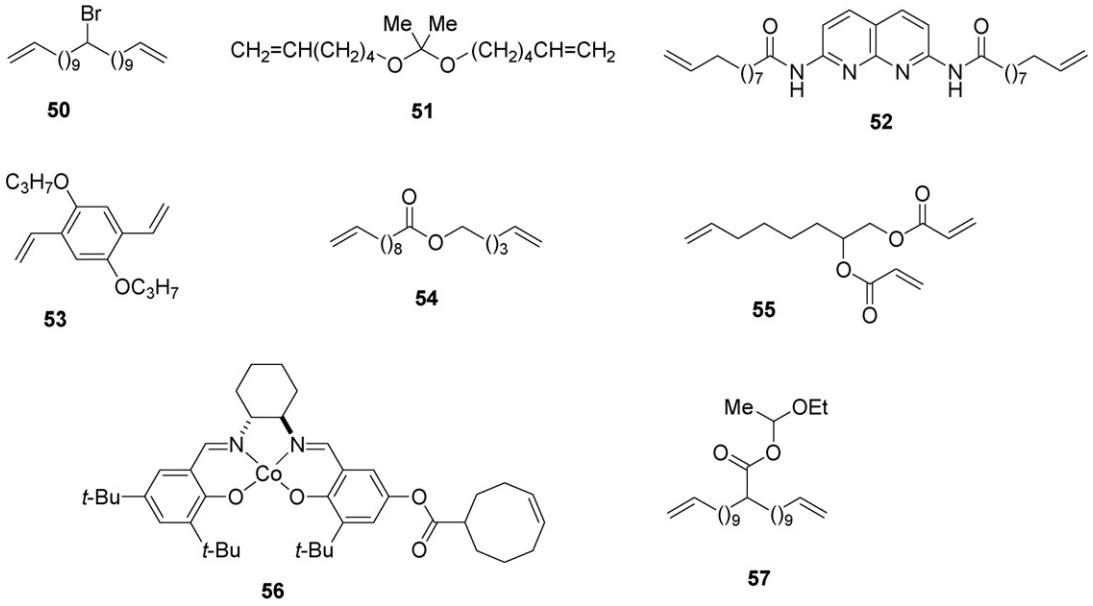
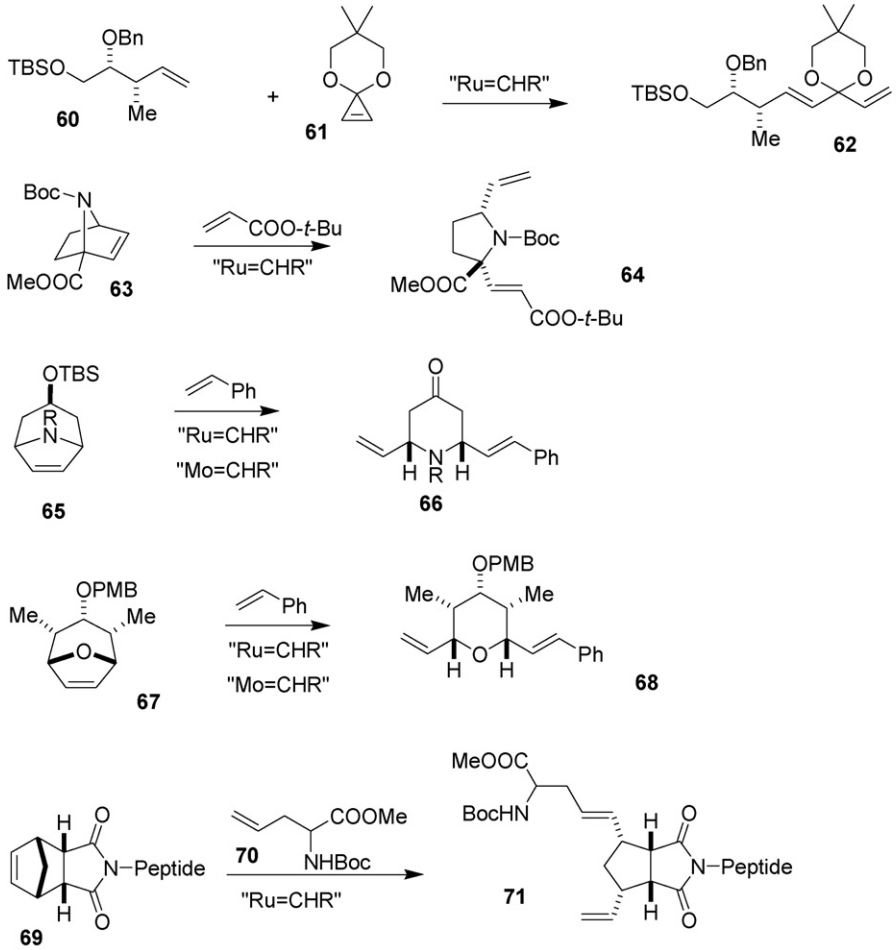


Fig. 4. Representative substrates for ADMET polymerization.

trahydropyran derivative with styrene for total synthesis of crocacin [384]; (11) a complex homoallylic alcohol (**87**) and acrolein for total synthesis of SCH 351448 [385]; (12) a homoallylic alcohol and *t*-butyl acrylate for total synthesis of cryptophycin A [386]; (13) 4-methyl-1-pentene and a homoallylic alcohol derivative for disparlure total synthesis [387]; (14) 1-octene-3-one and a complex cyclopentane-diene for total synthesis of prostaglandins [388]; (15) a polyoxygenated alkene and methyl acrylate for total syn-



Scheme 4.

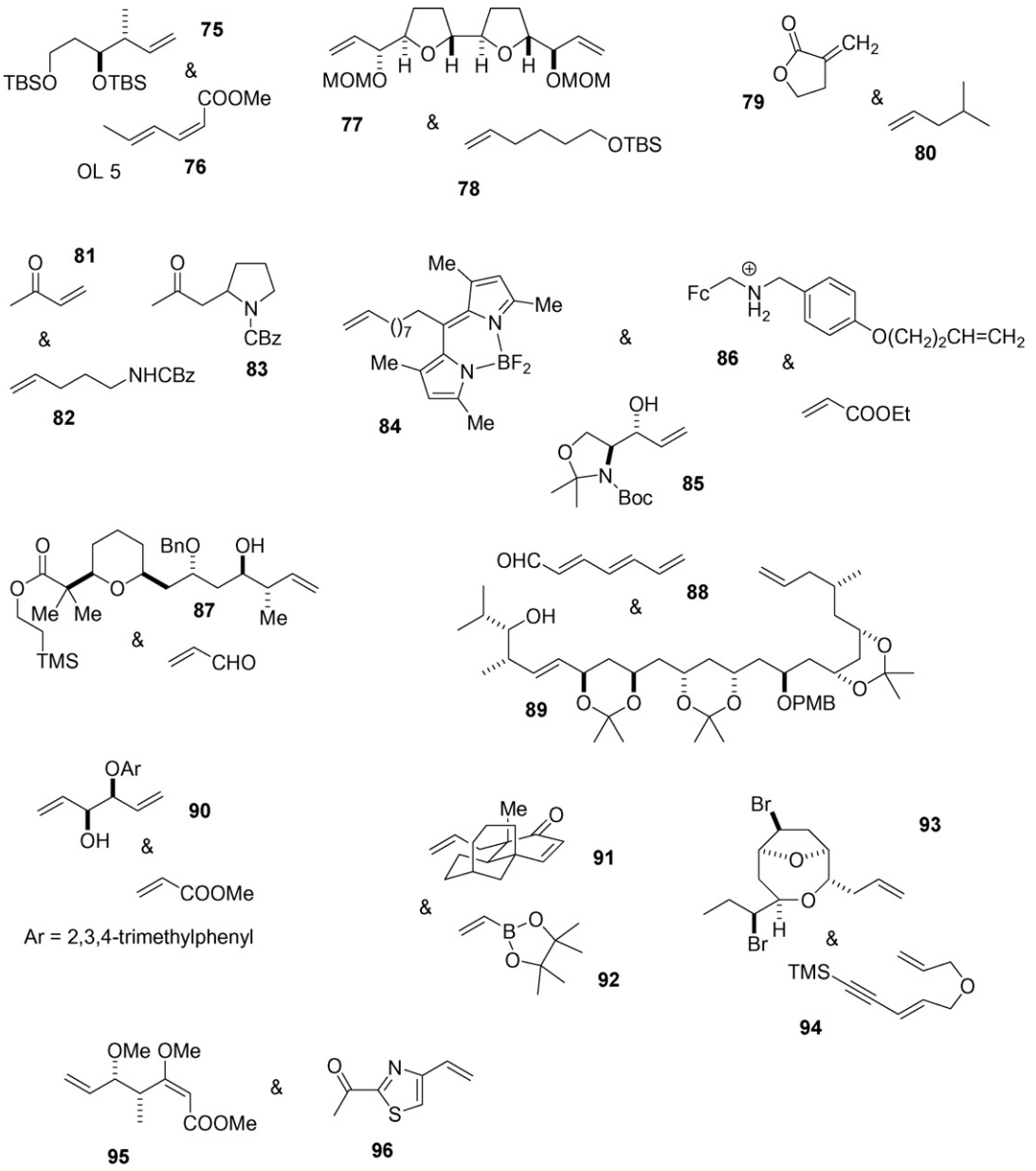
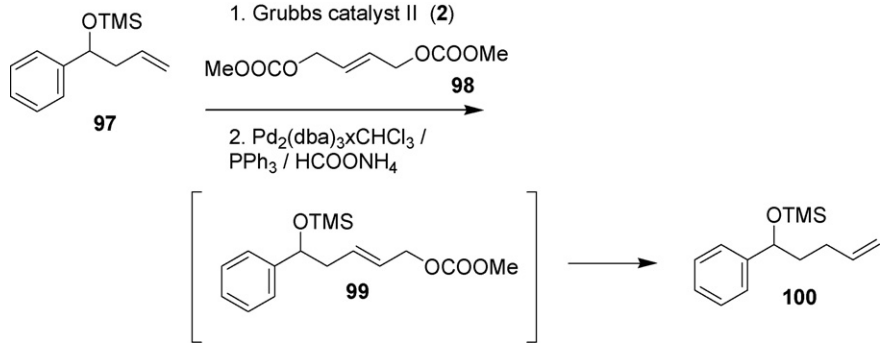


Fig. 5. Represent pairs of alkenes subjected to cross metathesis.

thesis of phytosphingosine [389]; (16) a protected polyol (**89**) and hexatrienal (**88**) for total synthesis of RK-397 [390]; (17) chemoselective metathesis of a chiral 1,5-hexadiene derivative (**90**) and ethyl acrylate for total synthesis of isoaltholactone [391]; (18) a protected aminoalcohol-alkene and a vinyl ketone derivative for total synthesis of prosophyllines [392,393]; (19) an amine-containing alkene and acrolein for total synthesis of sedamine [394]; (20) a vinyloxazole with 1-pentadecene for total synthesis of sphin-



Scheme 5.

gosines [395]; (21) a vinylpyrrole derivative and an enone for total synthesis of deoxycasuarine total synthesis [396]; (22) an oxanorbornene lactone and an amine–alkene for total synthesis of azabrefeldin A [397]; (23) 4-pentenamide and 1-undecene for total synthesis of cascarillic acid [398]; (24) a vinylthiazole and a monosubstituted alkene for total synthesis of mycothiazole [399,400]; (25) double CM involving a 4,4-bis(allyl)cyclohexa-2,5-dienone and 2-methylpropene for total synthesis of nemorosone [401]; (26) allylflavinoids and 2-methyl-2-butene for total synthesis of 6-prenylgenistein and 6-prenylquercitin [402]; (27) an allylhydroquinone and a terpenic alkene for total synthesis of vitamin E [403]; (28) a complex diene and excess 2-methyl-2-butene for total synthesis of garsubellin A [404]; (29) a complex alkene and an allylborane for total synthesis of carbaplatensimycin [405]; (30) a complex alkene and methyl acrylate for total synthesis of leucasandrolide A in a synthesis that also employs RCM in an early step [406]; (31) a complex alkene and methacrylic acid for total synthesis of pinnaic acid [407]; (32) a complex alkene and an allylic alcohol for total synthesis of leucasandrolide A [408]; (33) a complex alkene and methacrolein for total synthesis of stephacidins A and B and notoamide B [409]; (34) a complex alkene and a 6-hepten-1-ol derivative for installation of the side chain of pestalotiopsin A [410]; (35) a vinylboronate and a homoallylic ether for radicicol total synthesis [411]; (36) complex alkene (**91**) with a vinylboronate (**92**) for total synthesis of platensimycin [412]; (37) Z selective cross metathesis using a conjugated enyne (**94**) (employing a relay approach) for total synthesis of laureatin [413]; and (38) two complex alkenes for total synthesis of blepharocalyxin D [414,415], lasonolide A [416], melithiazole C (**95** and **96**) [417], epicalyxin (total synthesis and structural revision) [418], sorangiolide [419], fragments of tetanolide [420], marinomycins A–C (as part of a failed synthetic route) [421], mycalolide B fragments [422], isocassine [423], and the C1–C52 fragment of amphidinol 3 [424]. Cross metathesis was employed to effect polymer crosslinking [425–427]. A tandem one-pot procedure involving cross metathesis for form an allylic carbonate (*e.g.* **99**, Scheme 5) followed by palladium catalyzed reduction was reported [428].

Several examples of dimerization via metathesis (see Scheme 1) were reported in 2007. Compounds subjected to carbene complex-catalyzed metathesis dimerization are depicted in Fig. 6, and include: (1) iso phthalate-terminated poly-THF derivatives (*e.g.* **101**) [429]; (2) metathesis dimerization and tandem metathesis dimerization-RCM for alkene-linked cyclodextrins [430]; (3) allylquinoline derivatives (*e.g.* **102**) [431]; (4) a complex allylic alcohol (**103**) for total synthesis of intricatetraol [432]; (5) a rhodium(II) complex featuring pendant alkene groups (**104**) (also RCM reactions for analogs featuring two pendant alkene groups) [433]; (6) head-to-tail dimer formation using a complex diene derivative [434]; (7) unsaturated fatty acids [435]; (8) self-aggregating pyrimidines [436]; and (9) steroidal ethers (*e.g.* **105**) [437]. Metathesis dimerization-RCM (cyclodimerization) was reported for alkene-terminated pentaphenyl derivatives (*e.g.* **106**, Scheme 6) [438]. The cyclization process becomes more efficient if two molecules of **106** are treated with 4,4′-bis(formyl)biphenyl to make a bis(imine) followed by a macrocycle-forming RCM. A similar metathesis cyclodimerization was reported for tethered vinylcyclopentenone derivatives (*e.g.* **107**) [439]. Competition between RCM and cyclodimerization was studied for a variety of α,ω-dieneesters [440].

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 2007, 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 of carbocyclic rings constructed through RCM include: (1) cyclopentenes [441–445], including total syntheses of fommanosin [446]; cuparenone [447], herbertenediols [448,449], herbertenol [450], laurokumarene B [451], carbovir [452], neplanocin A [453], tashirinones [454], allosamizoline [455], carbagalactofuranose [456], abacavirs [457], novel carbocyclic nucleosides [458–463], agelastatin (see **115**) [464]; and sphingosine phosphate receptor agonists [465]; (2) cyclopentenes fused to bicyclo[2.2.2]octene ring systems [466]; (3) a cyclopentene featuring a tetrasubstituted alkene for spirotenuipesines A and B total synthesis (see **116**) [467]; (4) cyclopentenones [468], including total synthesis of litseaverticillols [469]; (5) spiro-fused cyclopentenes for total syntheses of sequosempervirin [470] and acorenol [471]; (6) five- and six-membered rings containing a 1,2-arrangement of silyl and ether groups [472]; (7) five- and six-membered rings spiro-fused to a diquinane ring [473]; (8) five- to seven-membered ring gem diester derivatives [474]; (9) a cyclopentene ring for total synthesis of hirsutic acid C as well as bridged eight-membered ring carbocycles [475]; (10) cyclohexenes [476–480] including total syntheses of merrilactone A (see **117**) [481], tetrahydrocannabinol [482], aphanorphine [483], cuparene [484], ovalicin total synthesis [485], and fumagillins [486]; (11) a cyclohexene in the presence of several other alkene groups for total synthesis of ottelione B (see **118**) [487]; (12) cyclohexenone derivatives [488]; (13) a six-membered ring bridging the 2- and 4-positions of a nucleoside [489]; (14) cyclohexene rings fused to benzofuran ring systems [490]; (15) dihydronaphthalenes followed by oxidative aromatization to afford naphthalenes [491]; (16) a highly oxygenated hexahydronaphthalene derivative (**119**) [492]; (17) direct formation of phenols via RCM of 1,6-dienes linked through a β-hydroxyketone followed by dehydration [493]; (18) direct formation of benzene or naphthalene rings (*e.g.* **121**) using trienols (*e.g.* **120**) [494,495]; (19) a cyclohexenol ring fused to a thiophene ring system for total synthesis of thiohalenquinone [496]; (20) highly oxygenated six- and seven-membered rings [497]; (21) six- to eight-membered rings featuring allylic amine groups [498]; (22) cycloheptenes [499]; (23) an α,β-unsaturated cycloheptenone derivative (**122**) for sundiversifolide total synthesis [500]; (24) the seven-membered ring system of thapsigorgins (see **123**) [501]; (25) a lactone-fused seven-membered ring for total synthesis of arglabin [502]; (26) seven-membered ring cyclic amino acid derivatives [503]; (27) the seven-membered ring of 13-oxyingenol (see **124**) [504]; (28) spiro-fused seven-membered ring systems through a double RCM of a tetraene (see **125**) [505]; (29) indole-fused cyclooctenes [506]; (30) fluorinated cyclooctenones [507]; (31) 10-membered rings fused to γ-lactones [508]; (32) 10-membered ring fused to benzene rings [509]; (33) benzo analogs of the sarcodictyin ring system (*e.g.* **126**) [510]; and (34) eleutherobin analogs [511]. Some more complex examples of carbocycle-forming RCM are depicted in Scheme 7. A relay RCM was employed in the conversion of **127** to **128**, a key step in the total synthesis of dihydro-epi-deoxarteannuin B [512]. Double RCM was employed to form the five-and six-membered ring of the elisabethan diterpenes from a tetraene precursor (**129**) [513]. Initially the spiro compound **129** was formed which transforms to the final product **130** upon extended reaction time.

Numerous examples of the formation of nitrogen heterocycles using the RCM reaction (Fig. 8) were reported in 2007, including formation of the following ring systems: (1) dihydropyrroles [514–519], including total syntheses of azasugars (see **135**) [520,521], indolizidine 167B [522], and swainsonine [523];

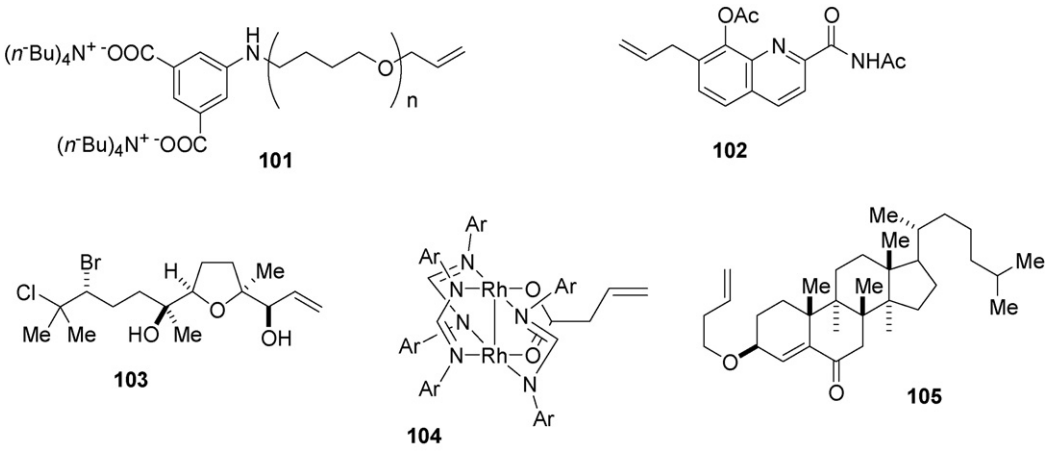
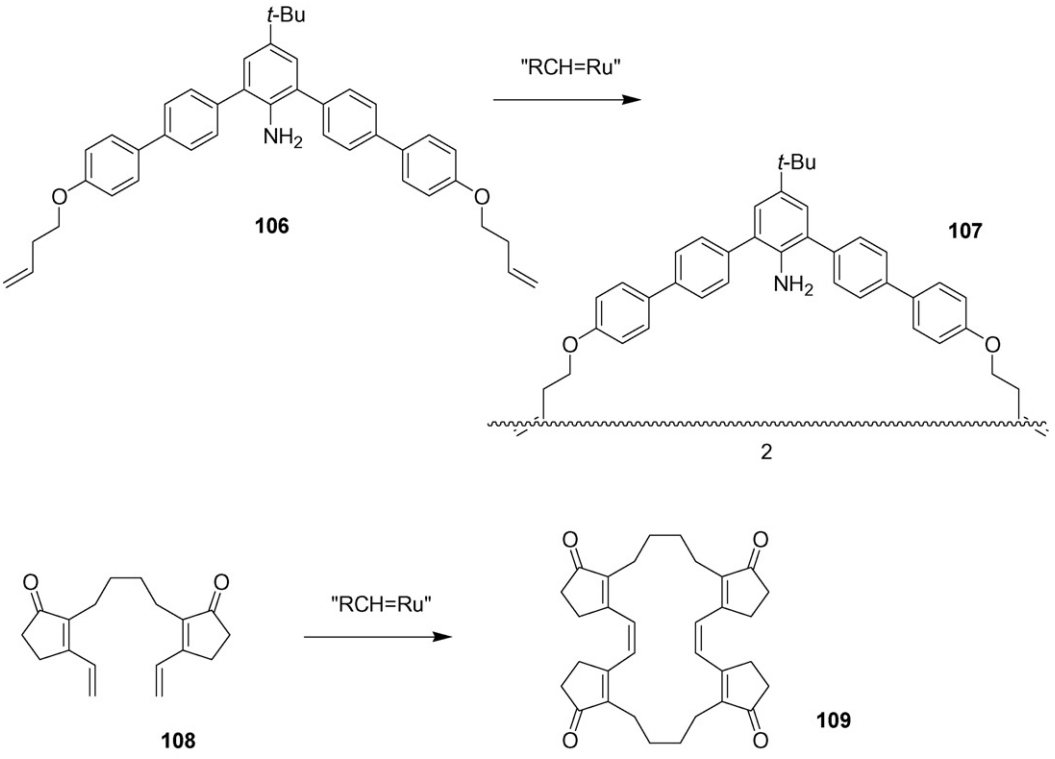


Fig. 6. Representative alkenes subjected to metathesis dimerization.

(2) five-membered ring α,β-unsaturated lactams [524]I including the total synthesis of pulchellalactam [525]; (3) pyrrolidinones (*e.g.* **136**) [526]; (4) five- and six-membered rings fused to imidazole rings as N-heterocycles or carbocycles [527]; (5) five- to seven-membered rings fused to isoindolone rings [528]; (6) five- to eight-membered ring nitrogen heterocycles containing a fluorinated alkene that was constructed in the metathesis event [529]; (7) tetrahydropyridines [530–536], including total syntheses of 3-methylpipecolic acid [537], adenophorine [538], 2-epi-lentiginosine [539], 8-episwainsonine [540], lasubine II [541], and deoxynojirimycins [542,543]; (8) tetrahydropyridines and dihydropyrans [544]; (9) six-membered ring α,β-unsaturated lactams [545], including total syntheses of methylphenidate derivative [546], poison frog alkaloids [547], HDAC inhibitors [548], and phlegmarine alkaloids [549]; (10) a six-membered ring β,γ-unsaturated lactam for total synthesis of α-conhydrine [550]; (11) a six-membered ring N-aminolactam for total synthesis of coniine (see **137**) [551]; (12) six-membered ring amines fused to the benzazepinedione ring system formed through either RCM or enyne metathesis [552]; (13) six-membered ring pyridinium cations [553]; (14) six-membered ring cyclic amino acid analogs [554]; (15) spiro-fused six-membered ring nitrogen heterocycles [555]; (16)f six- to seven-membered ring cyclic amines [556–558]; (17) six- to seven-membered nitrogen heterocycles fused to benzene rings [559]; (18) six- to seven-membered rings containing a benzimidazole group [560]; (19) six- to seven-membered ring cyclic hydroxylamine derivatives (*e.g.* **138**) [561]; (20) six- to eight-membered ring amines fused to benzene rings [562]; (21) six- to eight-membered ring β,γ-unsaturated lactams [563]; (22) six- to nine-membered rings fused to triazoles formed through either RCM or enyne metathesis (*e.g.* **139**) [564]; (23) an N-trityloxy seven-membered ring lactam for cobactin T total synthesis [565]; (24)



Scheme 6.

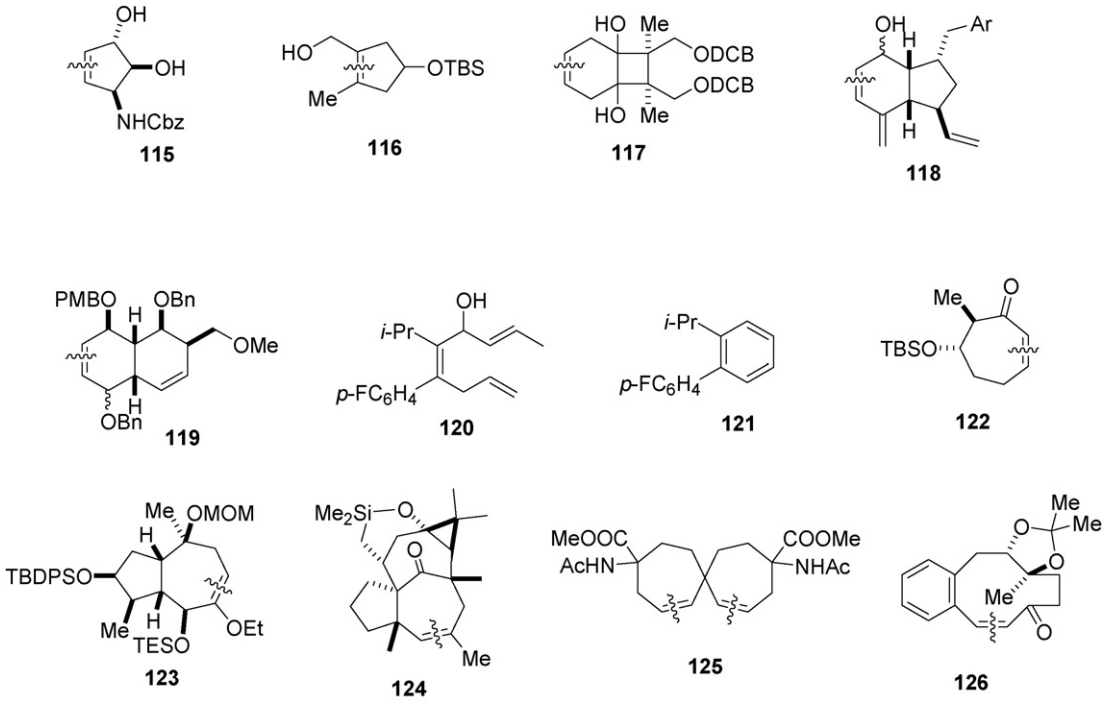
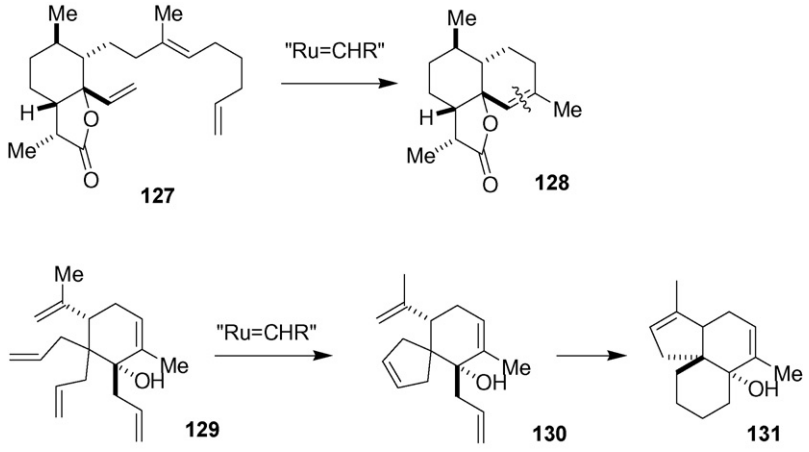


Fig. 7. Representative carbocycles produced through an RCM reaction (bond constructed through RCM indicated).

a seven-membered ring amide fused to a pyrrolidinone ring for stemoamide total synthesis (**140**) [566]; (25) seven-membered ring lactam-bromoalkenes [567]; (26) seven-membered ring cyclic amino acids [568]; (27) seven-membered rings bridging the 7-azabicyclo[4.2.1]nonane ring system (*e.g.* **141**) [569]; (28) seven-membered rings fused to indole ring systems through the indole nitrogen [570]; (29) seven-membered rings fused to pyrimidine rings [571]; (30) 8-azabicyclo[4.3.1]nonane derivatives (*e.g.* **142**) [572]; (31) seven- to eight-membered ring cyclic amines [573] and amino acids [574]; (32) seven- through nine-membered ring amino acid analogs (*e.g.* **143**) [575]; (33) eight-membered ring lactams (*e.g.* **144**) [576]; (34) eight-membered ring N-tosylamines fused to benzene rings [577]; (35) eight-membered rings fused to indole rings [578]; (36) eight-membered ring amine derivative for synthesis of buflavine analogs [579]; (37) eight-membered ring diamines formed through either RCM or intramolecular enyne metathesis [580]; (38) eight-membered ring diamines fused to the purine ring system (*e.g.* **145**) [581]; (39) eight- to 10-membered ring amines fused to a quinone ring [582]; (40) eight- to 10-membered ring diamines fused through the nitrogens to the indolocarbazole ring system [583]; (41) nine-membered ring bridged peptides [584]; and (42) failure to form a nine-membered ring cyclic guanidine [585].

Many diverse oxygen heterocycles were synthesized via the RCM reaction in 2007 (Fig. 9), including the following ring systems: (1) dihydrofuran-containing amphidinolide segments [586]; (2) direct formation of furans (*e.g.* **147**) through RCM and ethanol elimination using a diene-acetal starting material (*e.g.* **146**) [587]; (3) five-membered ring α,β-unsaturated lactones [588,589], including total syntheses of squamostolide [590], swainsonine [591], butenolide natural products [592], rubrolide E [593], umbelactones [594], and andirolactone [595]; (4) five-membered ring α,β-unsaturated lactones where the alkene formed in the metathesis reaction contains two electron-withdrawing groups [596]; (5) five- and six-membered ring ethers and/or lactones spiro-fused to β-lactam derivatives (including intramolecular enyne metathesis [597]; (6)



Scheme 7.

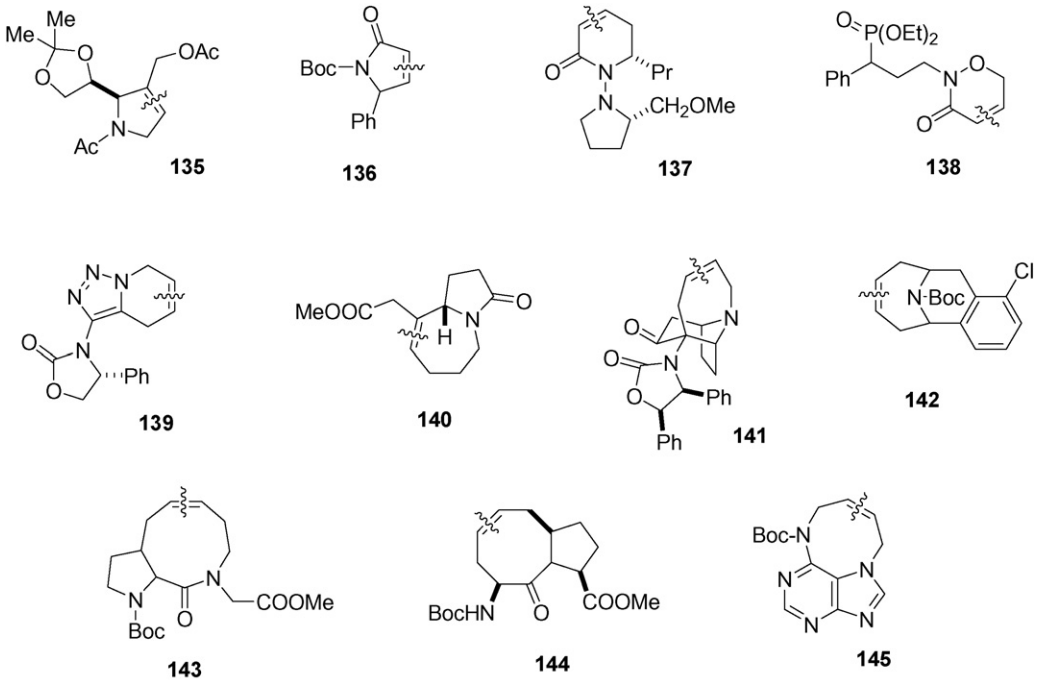
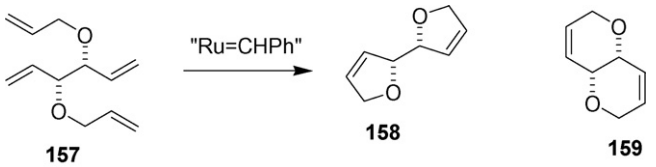


Fig. 8. Representative N-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).

competitive formation of either five- or six-membered ring α,β-unsaturated lactones in metathesis reactions of triene-esters [598]; (7) five- to eight-membered ring lactones and macrocyclic lactones fused to the five-membered ring of vitamin D_3 [599]; (8) six-membered ring enol ethers [600]; (9) a six-membered ring cyclic ether fused to a benzene ring [601]; (9) formation of six-membered ring ethers for total syntheses of FR901464 (see **148**) [602], jerangolid D [603], methyl sarcophytoate [604], cacospongiolide B analogs [605], 6-acetoxyhexadecanolide [606], methyl dihydrotrioxacarcinoside B [607], and scyphostatin [608]; (10) six-membered ring allylic ethers followed by isomerization to the cyclic enol ethers in the same reaction pot [609]; (11) α,β-unsaturated six-membered ring lactones [610,611], including total syntheses of kainic acid [612], herbimycin A (synthesis also employs CM) [613], attenol A (see **149**) [614], leucascandrolide A [615], pironetin [616], cytostatin [617], synrotolide diacetate [618], migrastatin [619], goniopyrones [620], goniodiol [621], epothilone segments [622], and tarchonanthuslactone [623]; (12) α,β-unsaturated six-membered ring lactones fused to a four-membered ring [624]; (13) diastereoselective formation of a six-membered ring ether (**150**) for total synthesis of didemniserinolipid B in a synthetic route that also employs cross metathesis [625]; (14) a spirocyclic six-membered ring (**151**) for total synthesis of aigialospirol [626]; (15) six-to-eight-membered ring cyclic ethers through either RCM or double metathesis reactions using norbornene-diethers [627]; (16) six- to eight-membered ring lactones and lactams [628]; (17) six- to eight-membered ring ethers fused to benzene rings [629]; (18) the six- to nine-membered ring ethers of brevitoxin and related marine toxins (see **152, 153**) [630–637]; (19) diastereoselective formation of a bridged seven-membered ring ether for total synthesis of thromboxane B_2 [638]; (20) a seven-membered ring lactone for total synthesis of goniopyrone and related natural products [639]; (21) seven-membered ring ethers fused to benzene rings [640], including total syntheses of heliannuols [641,642]; (22) seven- and eight-membered ring cyclic acetals [643]; (23) seven- and eight-membered ring oxygen heterocycles and carbocycles fused to naphthalene rings [644]; (24) eight-membered ring lactones [645]; (25) eight-membered ring ethers for total synthesis of microcladallene B (**154**) [646] and heliannuol A [647]; (26) eight-membered ring diethers and RO-CM reactions of the products with ethylene [648]; (27) eight- to 10-membered ring cyclic ethers [649]; (28) eight- to eleven-membered ring lactones [650]; (29) an oxygen-bridged 10-membered ring for total synthesis of eremantholide A (**155**) [651]; (30) a 10-membered ring lactones for total syntheses of Sch 642305 [652], an antimalarial noneolide [653]; microcarpolide (see **156**) [654,655], herbariums [656,657], and aspinolide B [658]. The RCM of various triene and tetraenes derivatives was reported [659]. Reaction of tetraene **157** (Scheme 8) with Grubbs catalyst I led exclusively to the dumbbell-type structure, while Grubbs catalyst II afforded both fused-ring and dumbbell structures. The selectivity of related systems with respect to double bond substitution pattern and oxygen protecting groups was also probed.

Heterocyclic compounds involving elements other than N and O were also constructed via the RCM reaction (Fig. 10). Examples include: (1) azaborines (*e.g.* **160**) [660]; (2) a cyclic allylboronate (**161**) for total synthesis of an ansatrienol derivative [661]; (3) bis(sila)cycloalkenes (*e.g.* **162**) [662]; (4) cyclic siloxanes (*e.g.* **163**) [663]; (5) a cyclic siloxane for synthesis of cornexistins [664]; (6) diastereoselective formation of cyclic siloxanes (*e.g.* **165**) using compounds containing diastereotopic vinyl groups (*e.g.* **164**) [665]; (7) six-membered ring cyclic sulfides (*e.g.* **166**) [666]; (8) cyclic sulfides fused to a pyrazole ring [667]; and (9) cyclic sulfones [668].

Numerous macrocyclic compounds (rings with ≥11 atoms) were synthesized using the RCM reaction in 2007 (Fig. 11), including: (1) a macrocyclic triene for total synthesis of terpestacin (**170**) [669]; (2) a 12-membered cyclic enyne (or diene) resem-



Scheme 8.

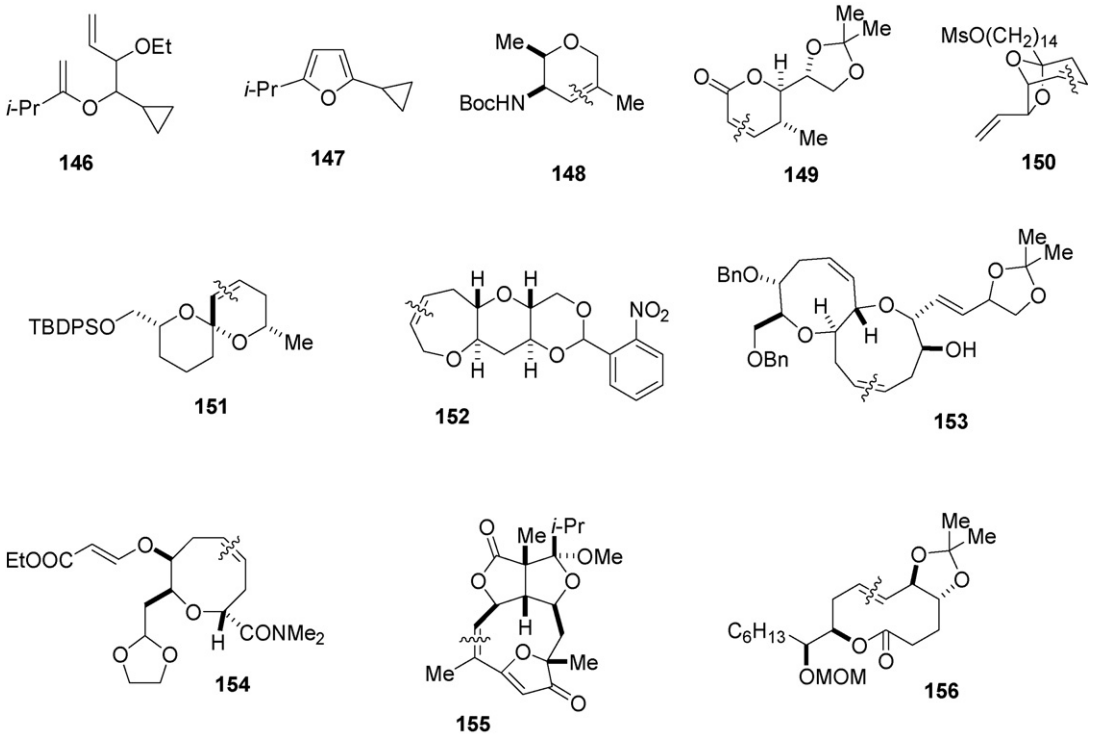


Fig. 9. Representative oxygen-heterocycles produced through an RCM reaction (bond constructed through RCM indicated).

bling the ring system of taxol [670,671]; (3) 11-membered ring ketones [672]; (4) an oxygen-bridged macrocyclic ketone for total synthesis of okilactomycin [673]; (5) a macrocycle-bridged tricyclic amine for sarain A total synthesis [674]; (6) macrocyclic diethers [675]; (7) a macrocyclic polyether for total synthesis of ring-expanded analogs of spirastrellolide A [676]; (8) the macrocyclic lactam ring of mazamanine A [677]; (9) a macrocyclic ketone–amide for total synthesis of proansamitocin [678]; (10) macrocyclic keto-lactams [679]; (11) a macrocyclic carbamate fused to a C-glycoside [680]; (12) macrocycle-bridged peptide derivatives (*e.g.* **171**) [681–697]; (13) a macrocyclic lactam–ether for synthesis of ansamycin analogs [698]; (14) macrocyclic amine–lactams [699]; (15) macrocyclic bis(lactams) [700]; (16) a macrocyclic lactam–diamine [701]; (17) a macrocyclic lactam–diamine for isooncinotine total synthesis [702] (18) macrocyclic urea Chk1 inhibitors (*e.g.* **172**) [703]; (19) macrocyclic urea-*m*-cyclophanepyrazines [704,705]; (20) macrocyclic lactone-lactams for total synthesis of spongidepsin (*e.g.* **173**) [706,707], (20) formation of 10- to 12-membered ring fluorinated lactones [708]; (21) formation of alkyne-containing macrocyclic lactones [709]; (22) formation of macrocyclic lactones [710], including total syntheses of archazolid B [711], lejimalide D [712], brefeldin A (see **174**) [713], an insect-derived natural product [714], apicularen A [715], pladienolides B and D [716], isomigristatin [717], both the erroneous and revised structures of palmerolide A [718], mycolactone core [719], aspercyclide C [720], amphidinolide E [721], and salicylihalamide [722]; (22) macrocyclic lactone-lactam breast cancer cell migration inhibitors [723], (24) macrocyclic spiro-fused bis(imines) [724]; (24) a macrocyclic polyene-lactone that immediately undergoes transannular Diels–Alder reaction [725]; (25) a pyran-bridged macrocyclic lactone [726]; (26) a macrocyclic keto-lactones for total syntheses of dehydrocurvularin [727], amphidinolides H and G (see **175**) [728], aigialomycin D [729], and sporiolide B [730]; (27) macrocyclic aza-keto-lactones for preparation of epothilone analogs [731]; (28) furan-bridged macrocycles [732,733]; (29) a macrocycle-bridged monosaccharide for total synthesis of ipo-

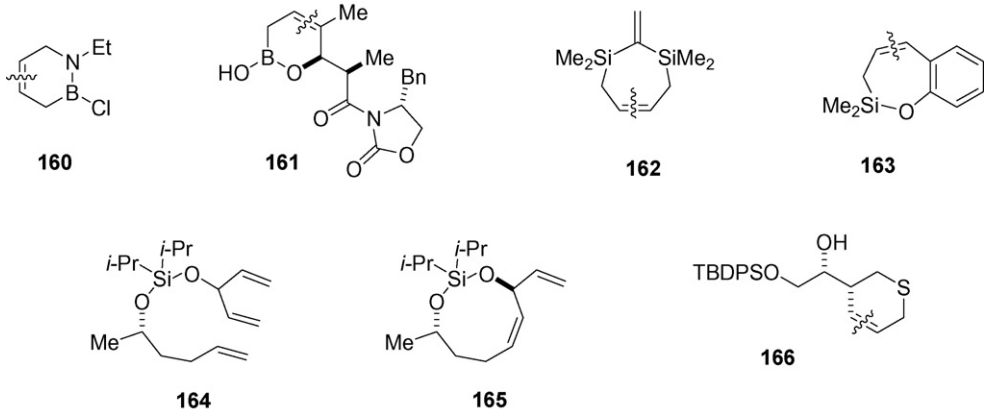


Fig. 10. Representative examples of other heterocycles prepared via the RCM reaction (bond constructed through RCM indicated).

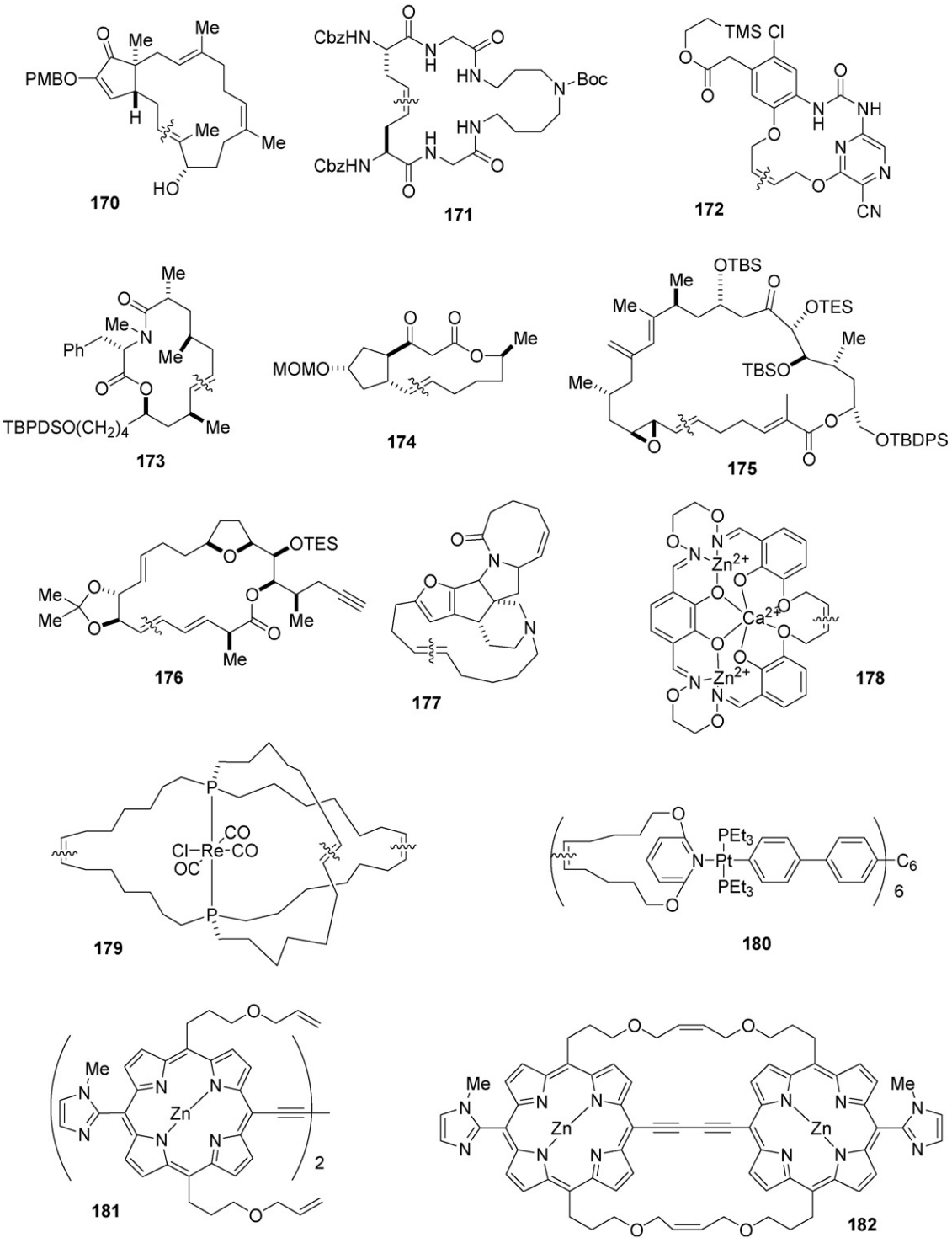
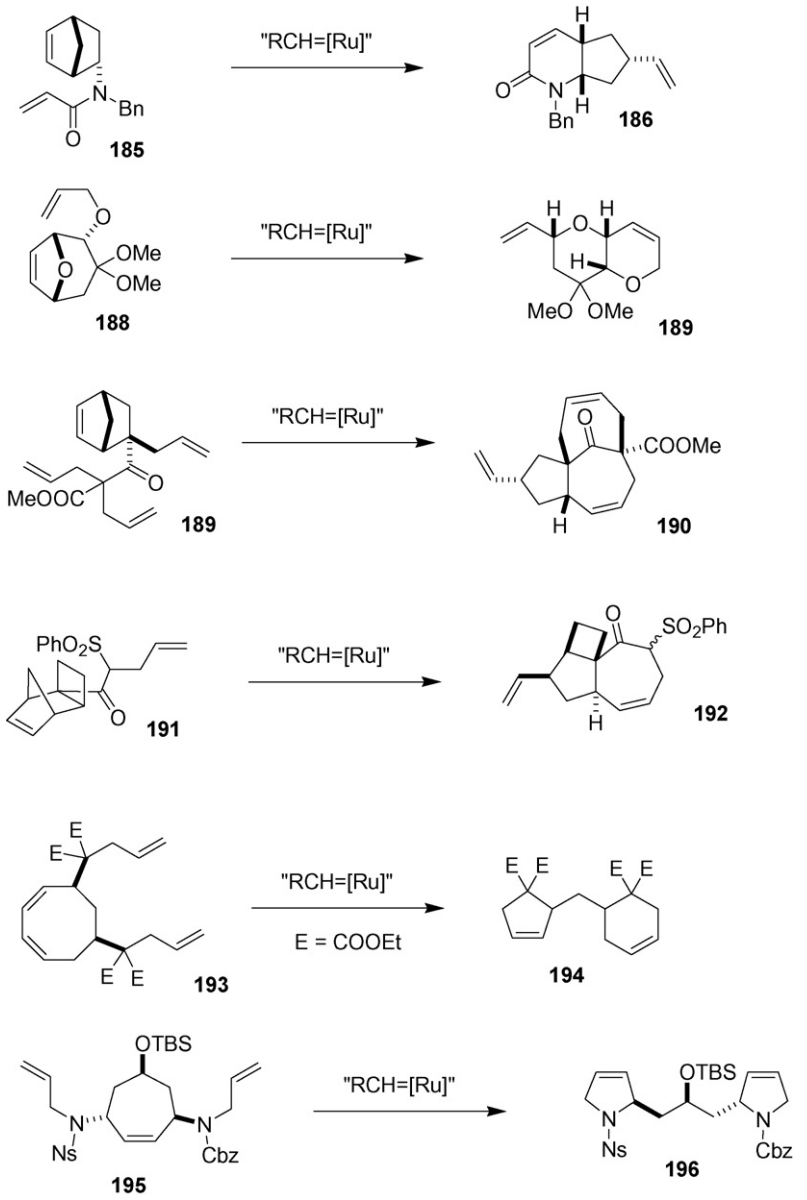


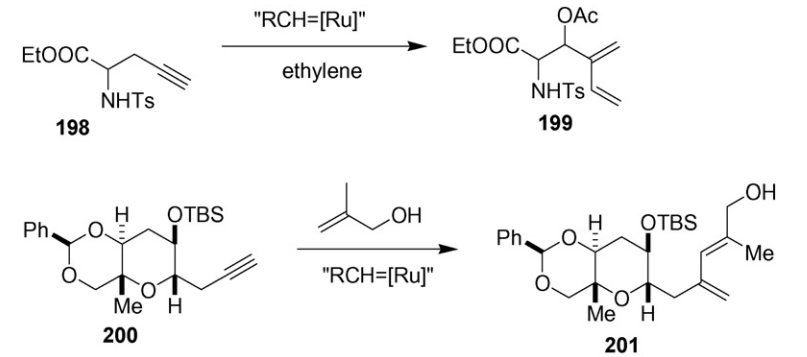
Fig. 11. Representative macrocycles (ring size >10) prepared using the RCM reaction (bond constructed through RCM indicated).

moeassin B and E [734]; (30) macrocycle-bridged disaccharides [735]; (31) macrocycle-bridged oligosaccharide derivatives [736]; (32) macrocycle-bridged deoxysugars [737]; (33) macrocyclic bis(lactones) derived from monosaccharides [738]; (34) macrocyclic bis(lactones) featuring two steroid units as part of the ring [739]; (35) a macrocyclic tris(lactone) for total synthesis of macrosphelides [740]; (36) macrocyclic tetralactones [741]; (37) an oxygen-bridged macrocyclic lactone for total synthesis of 2-epi-amphidinolide A (*e.g.* **176**) [742] and amphidonolide Y [743]; (38) a macrocyclic lactone- tetraether for synthesis of bryostatin analogs [744]; (39) a furanophane (**177**) for total synthesis of nakadomarin A (the synthesis also employs an RCM step) [745]; (40) a macrocyclic triene-siloxane [746]; (41) macrocycle-bridged tris(naphthalenes) [747]; (42) macrocycle-bridged taxol analogs [748]; (43) bridged calixarenes [749]; (44) bis(pyridinium salt)-based *m*-cyclophanes [750]; (45) *m*-pyridinophane derivatives [751]; (46) macrocycle-bridged phosphate esters [752]; (47) macrocycle-bridged cyclodextrins [753]; (48) catenanes [754–756]; (49) polyrotaxanes [757,758]; (50) interlocked daisy chains [759]; (51) interligand-bridged bis(phosphine)–rhenium



Scheme 9.

complexes (*e.g.* **179**) [760]; (52) inter- vs. intra-ligand metatheses for all-carbon bridged bis(platinum) diphosphine complexes [761,762]; (53) platinacycloalkenes through RCM of bis(alkyl)platinum(II) complexes where the alkyl groups have an alkene at the end of a long chain [763]; (54) formation of *m*-pyridinophane derivatives in the coordination sphere of platinum (*e.g.* **180**) [764]; (55) silicon-containing *p*-cyclophanes [765]; (56) macrocycle-bridged terpyridine–ruthenium(II) complexes [766];



Scheme 10.

(57) macrocycle-bridged tetraoxime ligands (*e.g.* **178**) [767]; (58) crosslinking of dendrimers via RCM [768–770]; (59) macrocyclic polymers [771]; and (60) macrocycle-bridged bis(alkyne)-linked porphyrin derivatives (*e.g.* **182**) featuring pendant vinyl groups (*e.g.* **181**) [772].

Several examples of ring rearrangement metathesis (RRM) were reported in 2007 (see Scheme 9). These examples include: (1) synthesis of cyclopentane-fused N-heterocycles (*e.g.* **186**) from norbornene-acrylamides (*e.g.* **187**) [773]; (2) synthesis of cyclic N-alkoxyamides [774]; (3) synthesis of pyranopyrans (*e.g.* **189**) through rearrangement of allyloxybicyclo[3.2.1]octane derivatives (*e.g.* **188**) [775]; (4) synthesis of bridged seven-membered ring systems (*e.g.* **190**) through a tandem RRM–RCM sequence employing norbornene-trienes (*e.g.* **189**) [776]; (5) synthesis of six- to seven-membered ring lactams through RRM of unsaturated amides of 7-azanorbornenes [777]; (6) synthesis of cyclobutene-fused hydroazulenes (**192**) from a tricyclic compound (**191**) [778]; (7) double RRM of a 1,3-cyclooctadiene derivative (**193**) resulting in RRM product **194** in preference to macrocyclic RCM [779]; and (8) double RRM for cycloheptene-diamine derivatives (*e.g.* **195**) for total synthesis of dendrochrysine [780].

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 2007. Examples of intermolecular enyne metathesis are depicted in Scheme 10 and include: (1) reaction of C-alkynyl glycosides with ethylene [781]; (2) reaction of alkyne-containing amino acid derivatives (*e.g.* **198**) with ethylene [782]; (3) reaction or propargylamine derivatives with various monosubstituted alkenes [783]; and (4) reaction of pyranopyran–alkyne **200** with methallyl alcohol to afford dien-ol **201** [784]. Examples of intramolecular enyne metathesis reactants and products are depicted in Fig. 12 and include the following processes: (1) synthesis of alkenylcyclopentenols [785]; (2) synthesis of silicon heterocycles [786,787]; (3) synthesis of 3-vinyl-2,4-dihydropyrroles [788]; (4) synthesis of vinyldihydrofuran derivatives [789]; (5) preparation of dienylboronates [790]; (6) preparation of vinylcyclohexenes (*e.g.* conversion of enyne **202** to diene **203**) [791–794], including vinylcyclohexenes used in total syntheses of galanthamine [795] and antibiotic YM-181741 [796]; (7) formation of vinyl-dihydropyran derivative (**205**) from an alkynyl ether (**204**) as part of a synthesis of ciguatoxin (the synthesis also employs multiple RCM steps) [797]; (8) double intramolecular enyne metathesis for synthesis of two oxygen heterocycle rings fused to a single benzene ring [798]; (9) formation of divinyl-dihydropyrans using 2-propargyloxy-1,5-hexadiene [799]; (10) synthesis of a benzo-fused cycloheptene derivative for preparation of allocolchicines [800]; (11) synthesis of benzoxepines [801]; (12) synthesis of a variety of spiro-fused seven-membered ring N- and O-heterocycles through either intramolecular enyne metathesis or RCM [802]; (13) synthesis of eight-membered rings (*e.g.* conversion of enyne **206** to vinylcyclooctene **207**) [803]; (14) synthesis of *p*-cyclophane derivatives (*e.g.* conversion of enyne **208** to cyclophane **209**) [804]; and (15) direct synthesis of vinylpyrroles (*e.g.* **211**) from N-allyl-N-propargyl amines (*e.g.* **210**) using Grubbs catalyst II and tetrachlorobenzoquinone as an oxidant [805]. Ring rearrangement intramolecular enyne metathesis reactions were

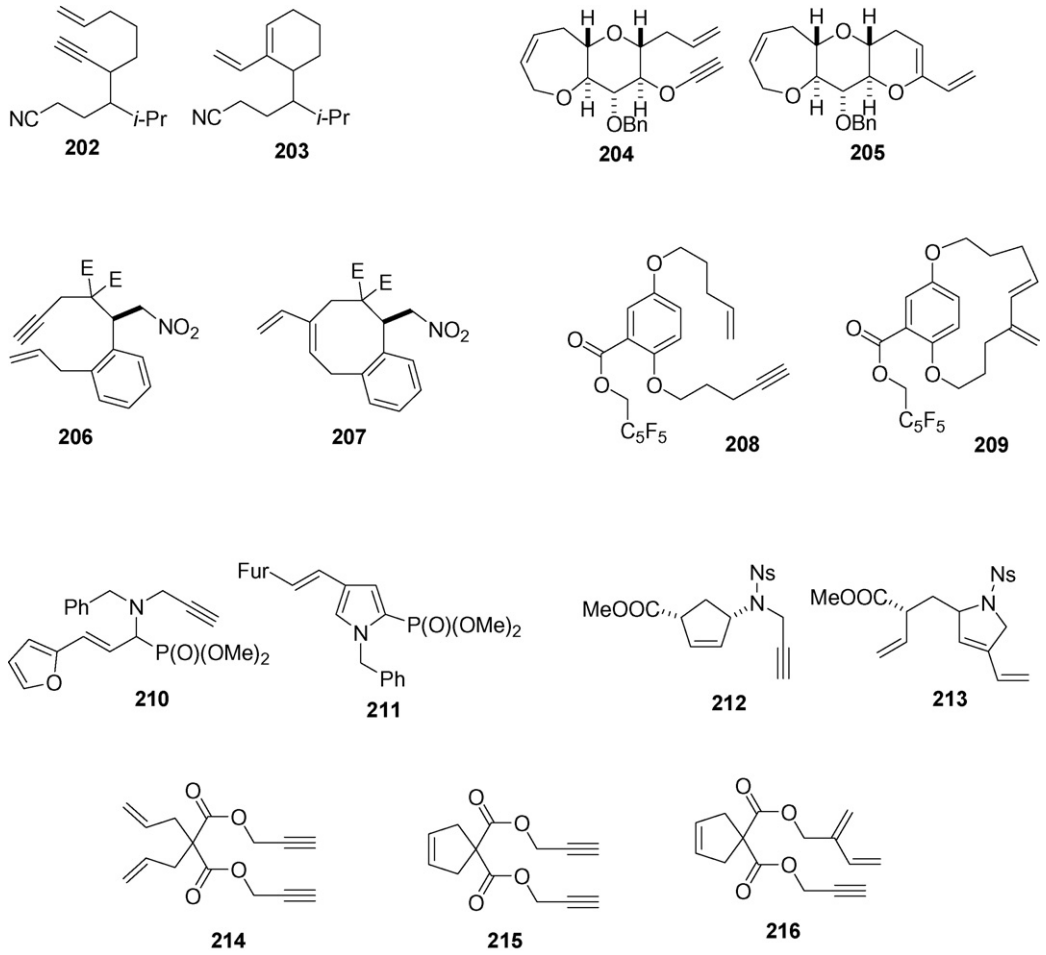
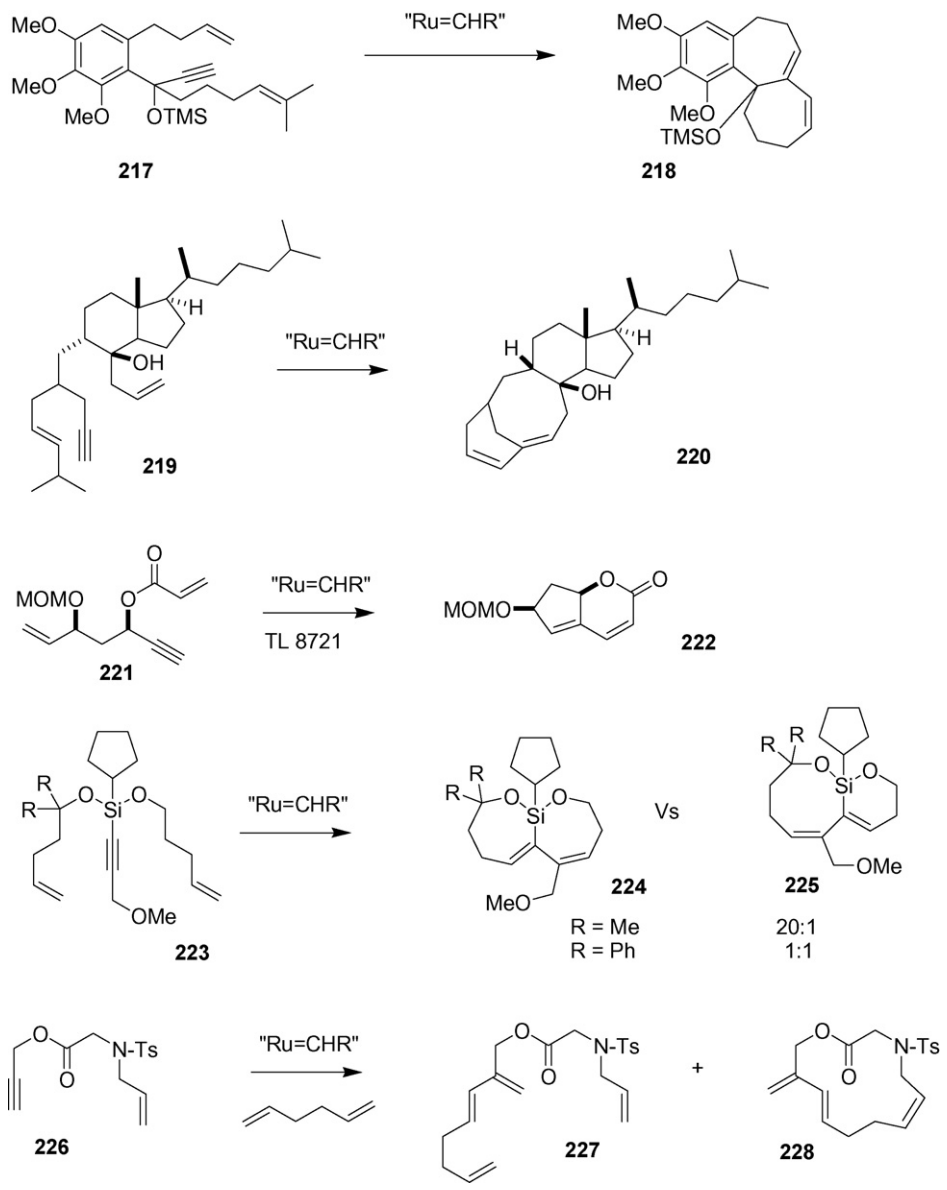


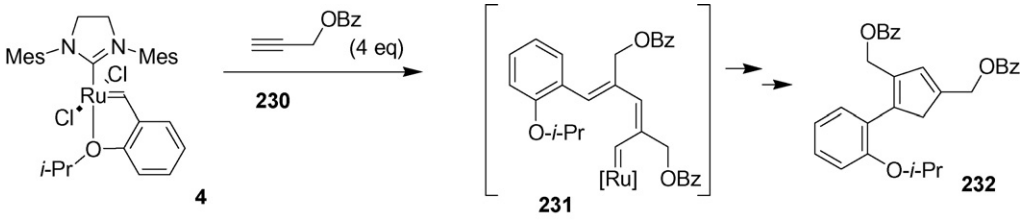
Fig. 12. Representative reactants and products from intramolecular enyne metathesis.



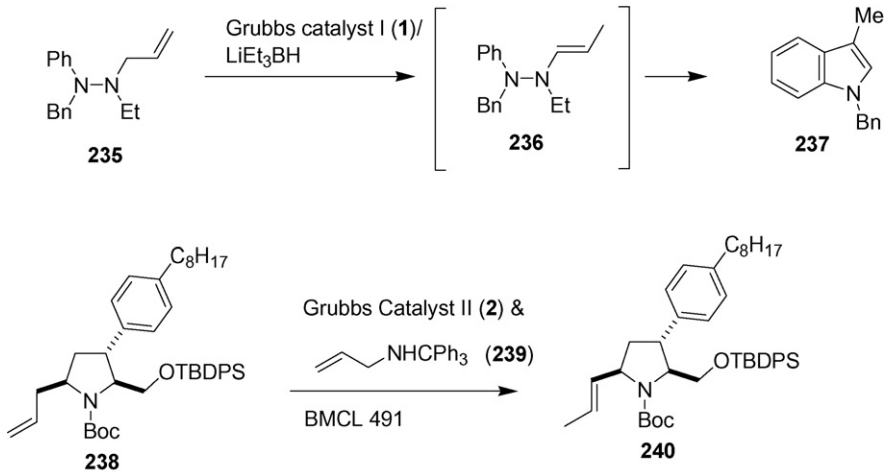
Scheme 11.

also reported (*e.g.* conversion of enyne **212** and ethylene to triene **213**) [806]. Preferences for RCM vs intramolecular enyne metathesis were reported for diene-diyne derivative **214** [807]. RCM leading to **215** is favored however the ethylene evolved could be captured in an intermolecular enyne metathesis resulting in triene-yne **216**.

Examples of enyne metathesis occurring in tandem with some other metathesis mode are depicted in Scheme 11. The conversion of triene **217** into tricyclic diene **218** via tandem enyne metathesis–RCM was employed for the construction of the colchicine ring system [808]. Tandem enyne metathesis–RCM was employed in an approach to steroidal taxanes (*e.g.* **220**) [809]. Tandem enyne metathesis–RCM of acrylic ester **221** was employed in a synthetic approach to ilexalactone [810]. Thorpe-Ingold controlled tandem enyne metathesis was observed in the treatment of silyl-tethered dienynes (*e.g.* **223**) with ruthenium carbene complexes, which leads to either of the cyclized products **224** or **225** [811]. Co-metathesis of enyne **226** and 1,5-hexadiene led to a mixture of the simple intermolecular enyne metathesis product **227** and the tandem enyne metathesis–RCM product **228** [812].



Scheme 12.



Scheme 13.

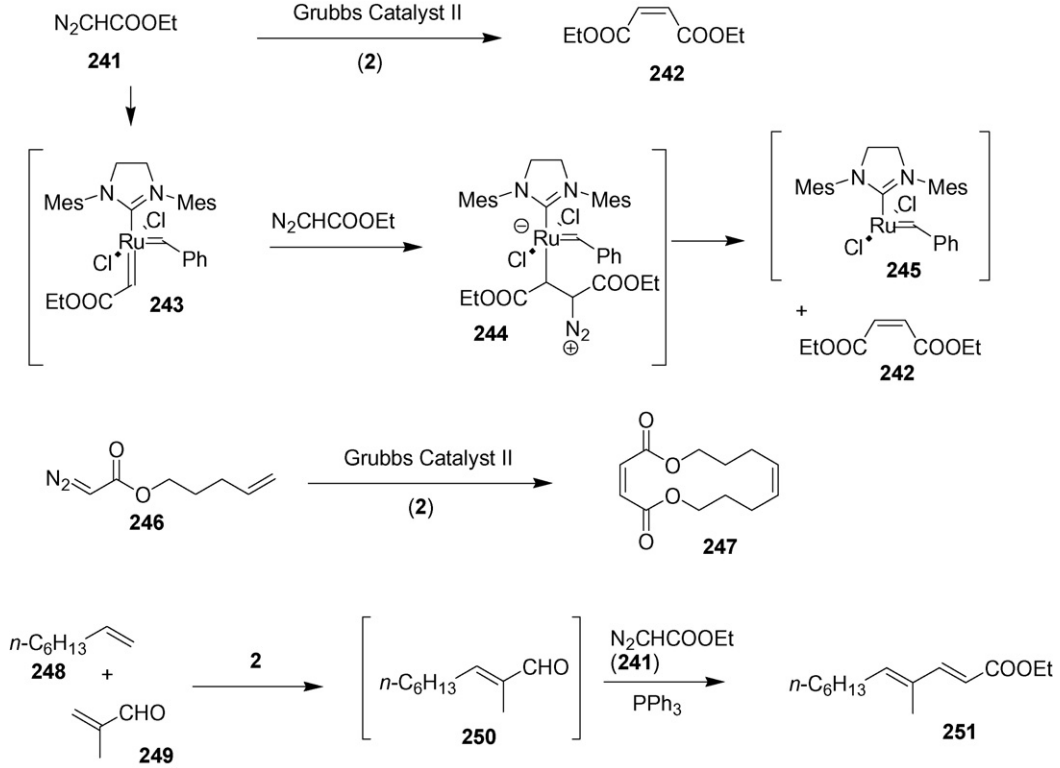
The reaction of the Hoveyda–Grubbs catalyst (**4**, Scheme 12) with a large excess of terminal alkynes (*e.g.* propargyl ester **230**) was reported [813]. The reaction affords arylcyclopentene derivatives (*e.g.***232**) in a process involving sequential alkyne insertions to afford a dienylcarbene complex intermediate (*e.g.* **231**) followed by cyclization.

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

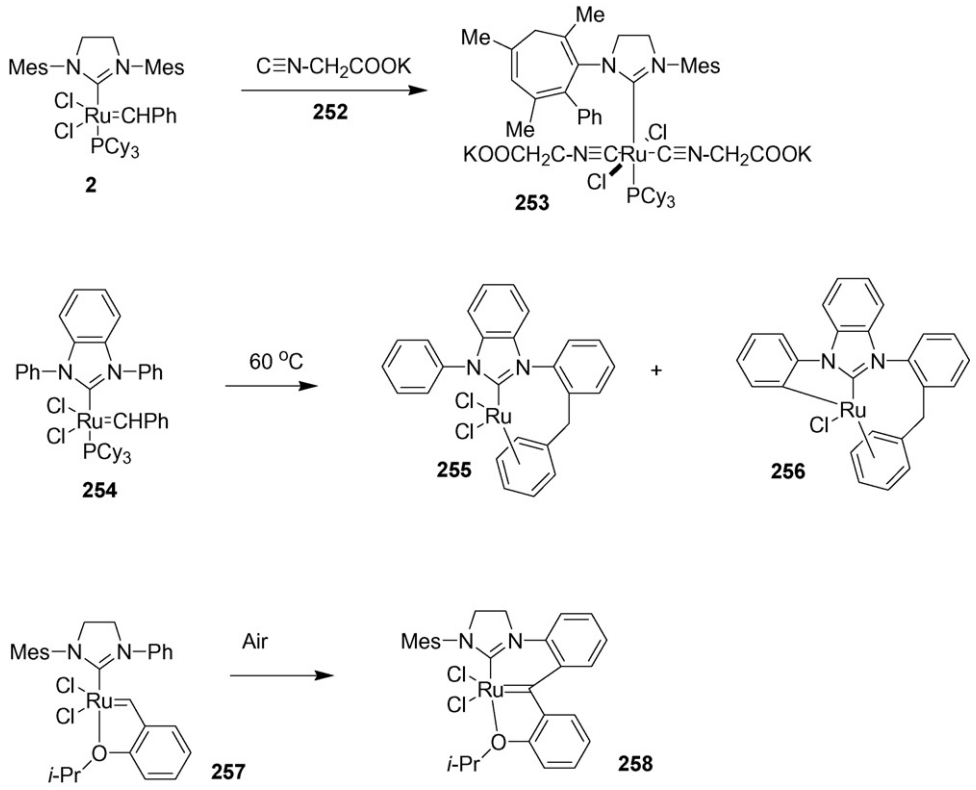
Several publications in 2007 report on processes unrelated to metathesis that are initiated by ruthenium carbene complex catalysts **1–4** and structurally related carbene complexes. Transformation of derivatives of Grubbs catalyst I to carbyne complexes is discussed in the carbyne complex section; see Scheme 88.

A frequent side reaction during metathesis is alkene isomerization; representative examples are depicted in Scheme 13. This side reaction has been attributed to the formation of metal hydride complexes under the conditions necessary for metathesis. Treatment of N-allyl-N′-phenylhydrazines (*e.g.* **235**) with Grubbs catalyst I/lithium triethylborohydride led to indoles (*e.g.* **237**) in a process involving alkene isomerization to afford an N-alkenyl-N-phenylhydrazine (*e.g.* **236**), a key intermediate of the Fischer indole synthesis [814]. A mixture of Grubbs catalyst II and N-allyltritylamine (**239**) was employed to effect an alkene isomerization of allylpyrrolidine derivative **238** [815]. The use of phenylphosphoric acid as an additive to prevent the isomerization of allylamides to enamides was also reported [816].

Use of Grubbs catalyst II in the formation of diethyl maleate (**242**, Scheme 14) from ethyl diazoacetate was reported [817]. The key step in the proposed mechanism involves nucleophilic addition of diazoacetate to the carbene carbon of bis(carbene) complex intermediate **243**, followed by elimination of ruthenium



Scheme 14.

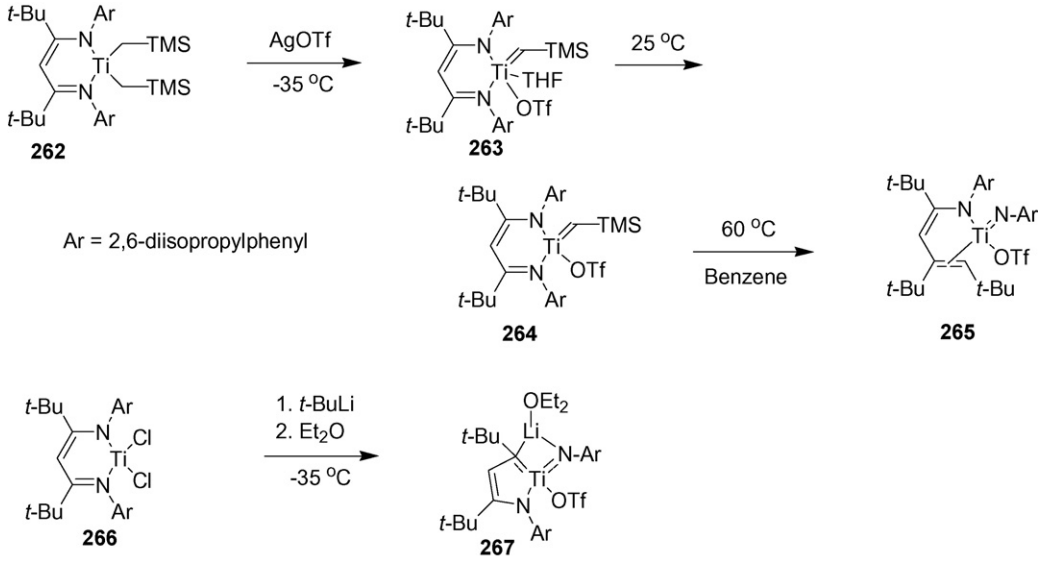


Scheme 15.

and nitrogen to afford the diester **242** and phosphine-dissociated Grubbs catalyst (**245**). Tandem diazo coupling-RCM was demonstrated in the formation of macrocyclic bis(lactones) (*e.g.* **247**) from alkene-diazoesters. Use of Grubbs catalyst II to catalyze a one-pot sequential process involving cross metathesis of an alkene with an α,β-unsaturated aldehyde (*e.g.* **249**) followed by carbonyl olefination using a diazo ester and triphenylphosphine was also reported [818].

Several stoichiometric transformations of ruthenium carbene complex metathesis catalysts were reported in 2007 (Scheme 15). The reaction of Grubbs catalyst II with isocyanides (*e.g.* **252**) leads to the ruthenium complex **253**, where the original carbene complex substituent has inserted into one of the mesityl rings [819]. This dianionic complex is easily removed from metathesis reaction mixtures. Thermolysis of Grubbs catalyst II analog **254** leads to the carbene ligand C–H insertion product **255** and the corresponding C–H activation product **256** [820]. A C–H activation product (**258**) was observed during air oxidation the of Hoveyda–Grubbs catalyst analogs featuring simple phenyl rings (*e.g.* **257**) [821].

Other studies of ruthenium carbene complex metathesis catalysts include: (1) the use of Grubbs catalysts I and II for the Friedlaender quinoline synthesis [822]; (2) the use of Grubbs catalyst I for Heck-type reactions of alkenes and aryl chlorides [823]; and (3) a study of the electronic absorption spectra for the indenylidene analog of Grubbs catalyst I [824].



Scheme 16.

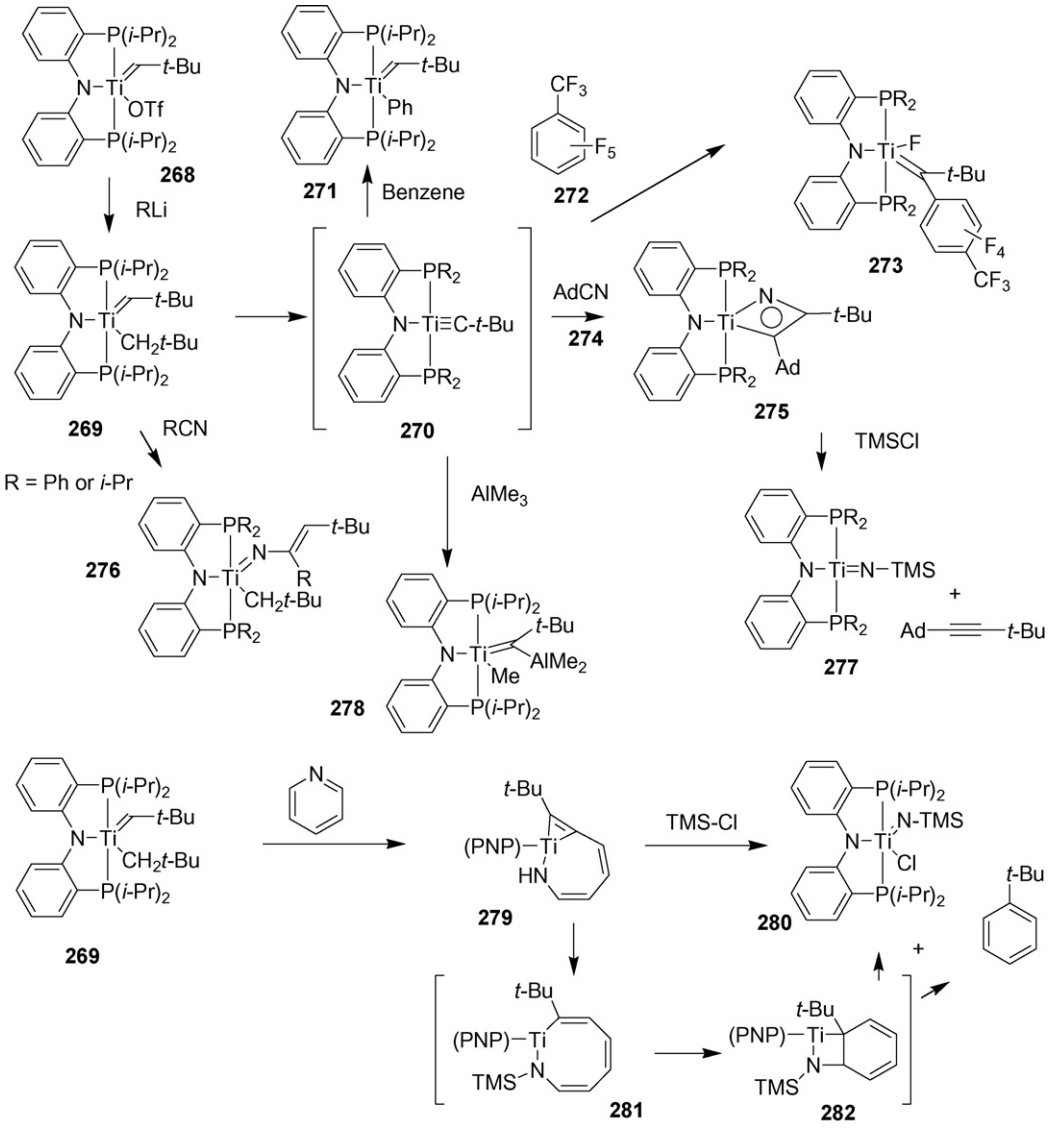
2.3. Individual carbene or alkylidene complexes classified according to metal

Several general studies of carbene complexes covering a wide variety of transition metals were presented in 2007. A structural analysis of several metal carbonyls, including the degree of double bond character in the metal–carbon bond, was reported to a variety of transition metal complexes [825]. The binding of the CH_2^+ fragment to all of the metals in the top row of the transition block was evaluated computationally [826]. The structure of various metal–methylene complexes was evaluated computationally, with emphasis on agostic interactions [827] and electronic structure [828]. The degree of aromaticity in metallabenzene derivatives featuring Group 8–10 metals was evaluated computationally [829]. It was noted that metallabenznes are 10π-electron systems and that 5d metal complexes display a greater degree of aromatic stabilization than 4d complexes.

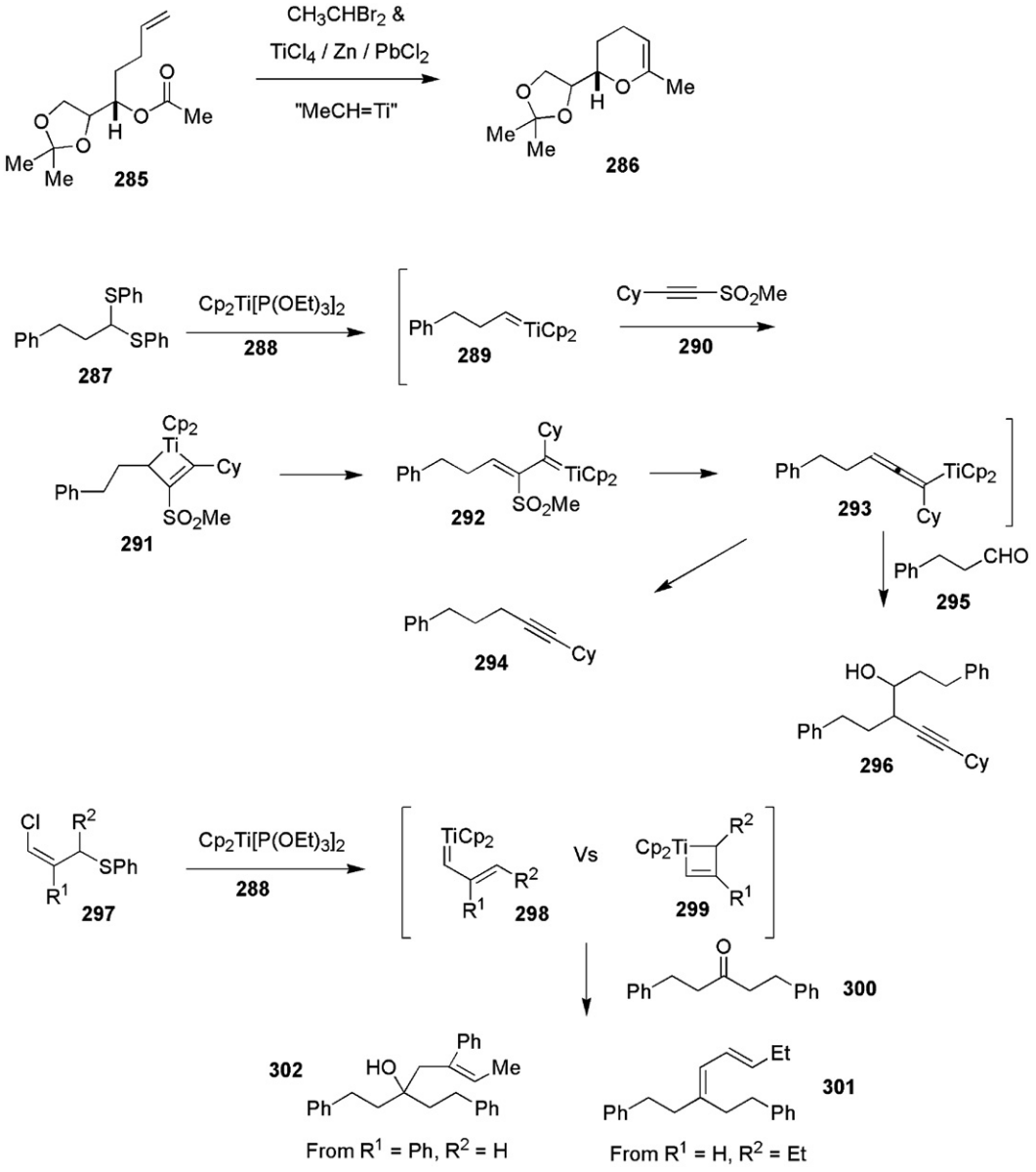
2.3.1. Group 4 metal–carbene complexes

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

A titanium carbene complex (**263**, Scheme 16) was produced from one-electron oxidation of bis(alkyl)titanium(III) complex **262** at low temperature [830]. Upon exposure to a vacuum, complex **263** looses THF to afford complex **264**, which transforms to internal metathesis product **265** upon heating. A related titanium carbenoid (**267**) was obtained through reaction of complex **266** with *t*-butyllithium [831]. Formation and C–H activation reactions of titanium carbene complexes featuring pincer P–N–P ligands (*e.g.* **269**, Scheme 17) were reported [832]. The carbene complexes undergo C–H activation reactions with arenes upon warming to room temperature to afford aryltitanium carbene complexes (*e.g.* **271**). Formation of the C–H activation products likely involves carbyne intermediates (**270**) according to DFT calculations. Thermolysis of the carbene complex in the presence of perfluorobenzene derivatives led to formation of C–F activation products (*e.g.* **273**) in a mechanistically similar process [833]. The reaction of titanium P–N–P carbene complexes with nitriles results in either azametallacyclobutadienes (*e.g.* **275**) or to the metathesis products, metal-imido complexes (*e.g.* **276**), depending upon the structure of the nitrile [834]. Formation of the azamaetallacycle involves formation of a carbyne complex followed by [2+2]-cycloaddition. Treatment of the azametallacycle

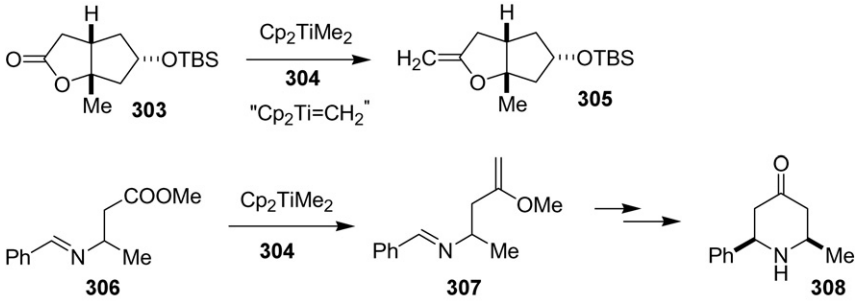


Scheme 17.



Scheme 18.

with either TMS-Cl or Me_3Al leads to formation of the alkyne and a Ti–N multiply bonded species (*e.g.* **277**). Use of a similar system to effect ring opening reactions of pyridines was reported [835]. Reaction of titanium carbene complex **269** with Me_3Al leads to the carbene complex product **278**, which results from addition of Me_3Al to the carbyne complex intermediate **270** [836]. Reaction of P–N–P carbene complex **269** with pyridine initially produces ring-opened product **279**. Subsequent reaction with chlorotrimethylsilane leads to imidotitanium complex **280** and *t*-butylbenzene. The mechanism for *t*-butylbenzene formation involves initial formation of the azametallacyclooctatriene (**281**), followed by electrocyclic ring closure and retro-[2+2]-



Scheme 19.

cycloaddition. The trimethylaluminum adduct **278** also induces ring opening of pyridine derivatives.

Several examples employing *in situ*-generated titanium-carbene complexes in synthetic organic chemistry were demonstrated in 2007; representative examples are depicted in Scheme 18. Titanium carbene complexes were generated *in situ* from the reaction low-valent titanium with 1,1-dibromoethane [837]. The carbene complex can promote olefin metathesis and tandem carbonyl olefination/olefin metathesis (*i.e.* conversion of alkene–ester **285** to cyclic enol ether **286**). Titanium carbene complexes (*e.g.* **289**) were generated *in situ* from the reaction of dithioacetals with titanium(II) complex **288**. Reaction of dithioacetals with sulfonylacetylenes and aldehydes was reported [838]. The reaction of ditioacetals with carbene complex **288** followed by an aldehyde leads to homopropargyl alcohol derivatives (*e.g.* **296**) in a process that involves formation of a titanium carbene complex (**289**), followed by [2+2]-cycloaddition, ring, opening, and elimination of the tosyl group to afford the allenyltitanium complex (**293**), which adds to the aldehyde as a propargyl anion equivalent. If the aldehyde coupling step was eliminated, the protonation product, alkyne **294**, is obtained. Titanium vinylidene complexes were suggested as intermediates in the titanium complex **288**-mediated addition of alkenyl sulfones to alkynes [839,840]. The reaction of chloroallylic sulfides (*e.g.* **297**) with titanium(II) complex **288** leads to a titanium species that could react as either a vinylcarbene equivalent (**298**) or a metallacyclobutene equivalent (**299**) [841]. In general, derivatives where $R^2 \neq H$ react as carbene equivalents and couple with ketones to afford carbonyl olefination products (*e.g.* **301**). Derivatives where $R^1 \neq H$ react as titanacyclobutene equivalents and afford carbonyl group addition products (*e.g.* **302**) upon treatment with ketones. The reaction of bis- and tris(thioacetals) with titanium(II) complex **288** and carbonyl compounds to afford the corresponding carbonyl olefination products was also reported [842]. A similar reaction using 1,1-dichlorocyclopropanes, titanium(II) complex **288**, and ketones led to alkylidenecyclopropanes [843].

Various carbonyl olefination reactions were accomplished using traditional *in situ*-generated titanium carbene complexes; examples are depicted in Scheme 19. Olefination of carbonyl compound **303** using the Petassis reagent (**303**) was reported [844]. Olefination of nine-membered ring lactones using the Tebbe reagent was instrumental in a synthesis of the core structure of eunicellins [845]. Titanium carbene complexes were instrumental in a stereoselective synthesis of piperidinone derivatives (*e.g.* **308**) from β-aminoester-derived imines (*e.g.* **306**) [846]. Carbonyl olefination using the Peatssis reagent affords enol ether–imine **307**, which affords the final product **308** after a cyclization reaction.

Other studies of Group 4 metal–carbene complexes are depicted in Scheme 20. A hafnium–vinylidene complex (**311**) was suggested as an intermediate in the conversion of hafnacene-dichloride (**309**) into silahafnacyclopentene derivative **312** [847]. Laser-ablated hafnium atoms generated in the presence of dichloromethane afforded a carbene complex, $H_2C{=}HfCl_2$, that could be isolated in an argon matrix and observed by IR spectroscopy [848]. DFT calculations of this carbene complex revealed no agostic C–H interactions, and the calculated IR spectrum was compared with the observed IR spectrum. Reaction of hafnium atoms with chloroform lead to a triplet carbyne complex. Similar studies were reported for laser-ablated Group 4 metal atoms reacting with difluoromethane [849]. Zirconium alkylidene complexes were suggested as likely intermediates in the surface decomposition neopentylzirconium complexes [850].

2.3.2. Group 5 metal–carbene complexes

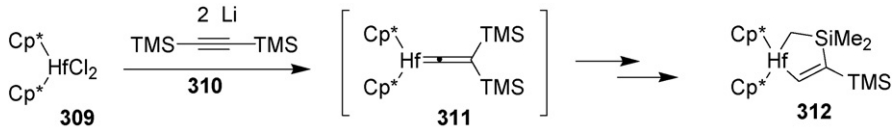
Niobium carbene complexes (*e.g.* **316**, Scheme 21) were prepared via the reaction of pincer-ligated niobium(IV) complex **315** (Scheme 21) with alkyllithium derivatives devoid of β-hydrogens [851]. A paramagnetic niobium(IV) carbene complex (**317**) could be prepared if only 2 equiv. of the alkyllithium reagent was employed. A related tantalum bis(carbene) complex featuring a pincer ligand (**320**) was prepared through thermolysis or photolysis of pincer-ligated tetramethyltantalum complex **319** [852]. The reaction of tantalum carbene complex **321** with zirconium hydroxide-bound silica (**322**) afforded an immobilized carbene complex (**323**) [853]. Hydrogenation of the carbene complex led to a tantalum hydride species (**324**) capable of alkane metathesis. Tantalum methylene complexes were identified from the reaction of tantalum atoms with methane in an ion-bean mass spectrometer [854].

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

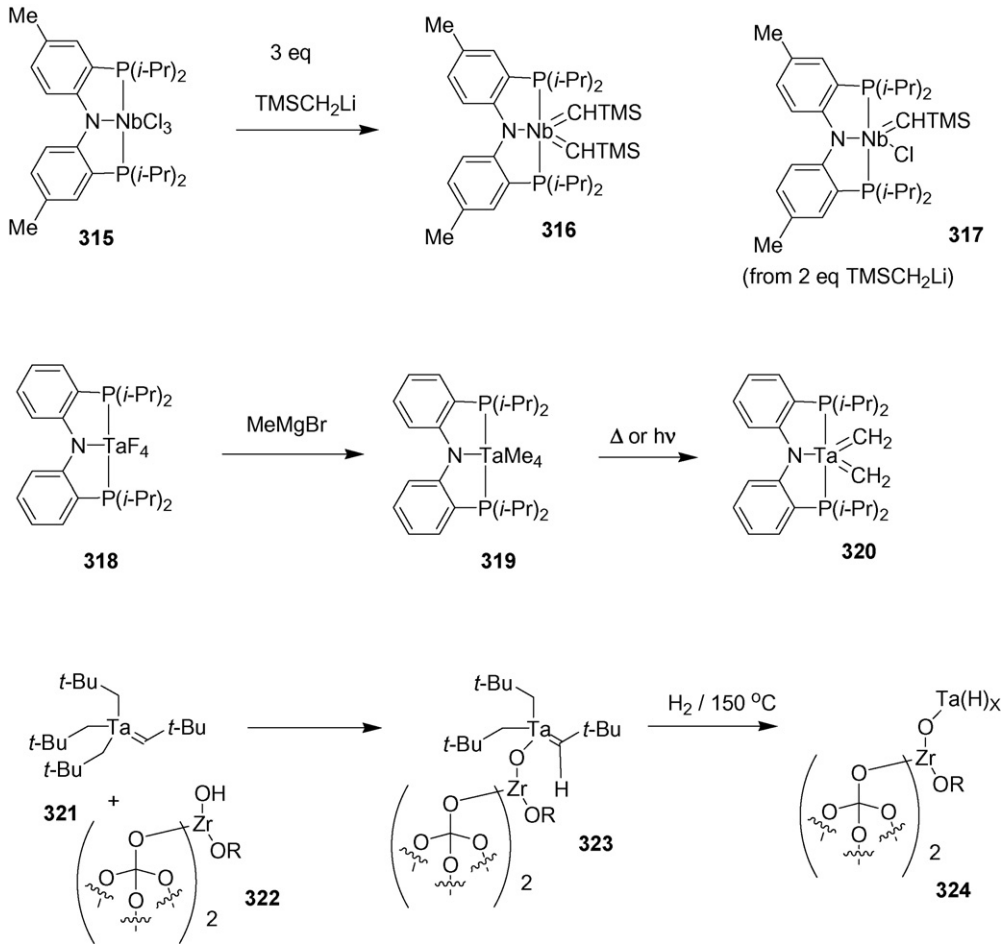
2.3.3.1. Schrock-type carbene complexes. A significant portion of this subject material has already been presented in the alkene metathesis section; the Schrock catalyst (**5**) belongs to this class of compounds.

The synthesis of tungsten analogs of the Schrock carbene complex (*e.g.* **331**, Scheme 22) followed by studies of their reaction with various alkenes was reported [855]. Reaction of the fluorinated alkoxytungsten species with ethylene led to the stable metallacyclobutane **332**. A metathesis reaction was observed with 3-hexene, resulting in carbene complex **333**, while formation of a non-carbene complex **334** occurred upon treatment with 2-methyl-2-butene. Reaction of the chelating aryloxytungsten complex **336** also afforded a stable metallacyclobutane upon reaction with ethylene. Reaction of metallacyclobutane **337** with THF led to propene and the ethylene complex **338**, which reacted with additional ethylene to afford an equilibrium mixture of the metallacyclopentane derivative **339** and ethylene complex **338**. Nitrogen-ligated analogs (*e.g.* **340, 341**) were also prepared from carbene complex **330** [856]. Thermolysis of carbene complex **340** led to the carbene transfer-C–H activation product **342**. Reaction of the cationic carbene complex **341** with ethylene resulted in the ethylene complex **343**. Formation of pyrrole ligated analogs of the tungsten complexes in Scheme 22 and their reactions with ethylene were also reported [857].

Protonation of tris(sulfide)-bridged dimolybdenum alkynyl complexes (*e.g.* **345**, Scheme 23) with acid led to the corresponding vinylidene complexes (*e.g.* **348**) [858]. Reaction in dichloromethane led to the reduced vinylidene **346** however reaction in ether led to mostly the hydroxyvinylidene complex **348**, accompanied by minor amounts of alkenylvinylidene (**349**) and allenylidene (**347**) complexes, accompanied by a vinylidene complex formed through acetone loss (**350**). A radical mechanism was proposed for formation of the reduction product. Similar studies were reported for diphenyl analogs. The complexes were studied by electrochemistry. Protonation of neutral bridging



Scheme 20.



Scheme 21.

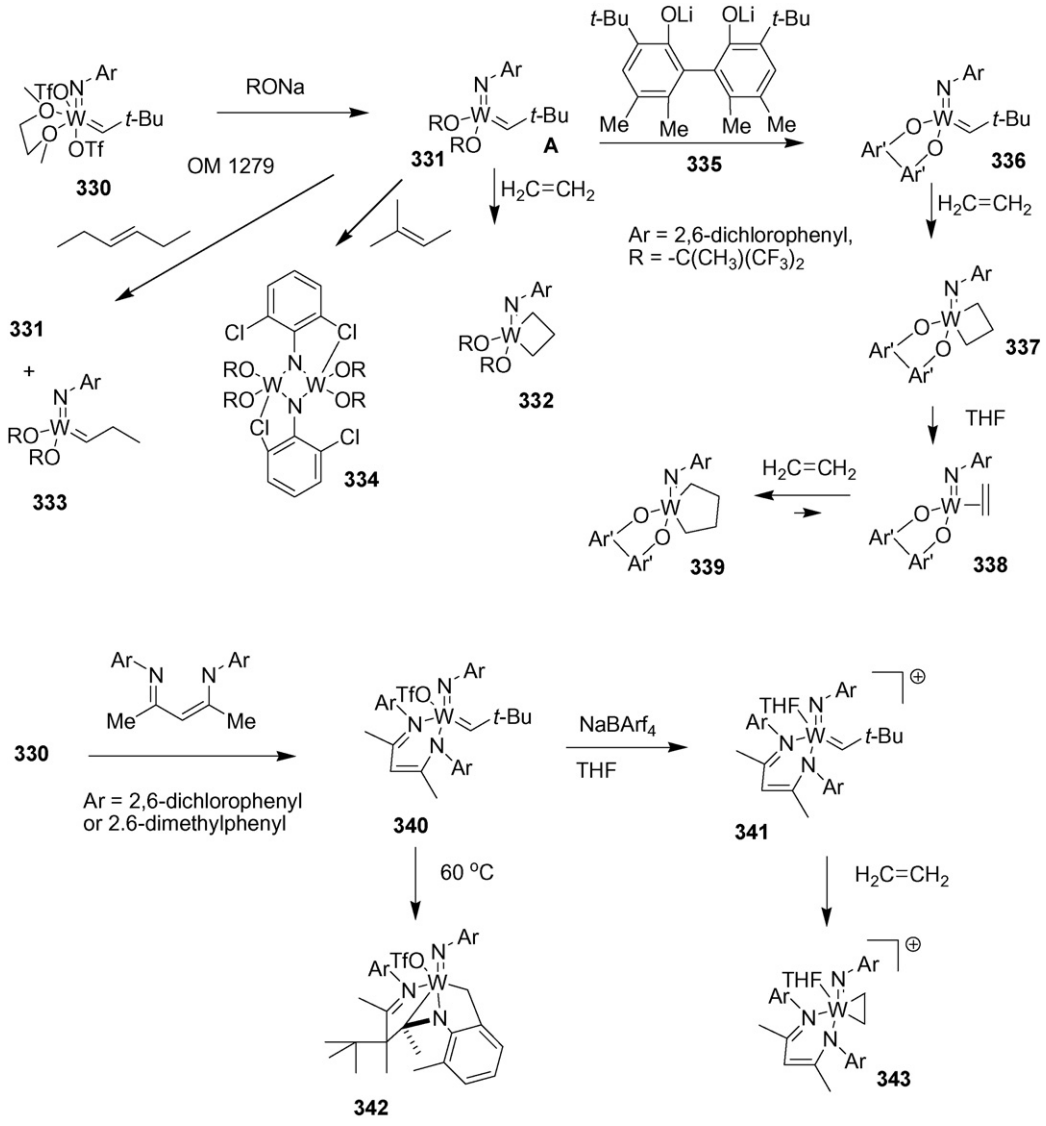
carbyne-dimolybdenum complexes led to cationic bridging carbene complexes [859].

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. Representative examples are depicted in Scheme 24. The synthesis of azo group-containing arylcarbene complex **353** using the Fischer synthesis was reported [860]. Conversion to the aminocarbene complex **355** followed by acetonide deprotection to afford polyhydoxylated carbene complex **366** was also reported. Photoisomerization and aggregation studies were reported for the resultant aminocarbene complexes. Heterocycle-linked bis(carbene) complexes (*e.g.* **368**) were prepared according to the Fischer synthesis from dilithioheterocycle derivative (*e.g.* **367**) [861].

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

Cyclopropanation or [4+1]-cycloaddition using alkenylcarbene-chromium complexes (*e.g.* **370**, Scheme 25) and electron-deficient ketones leads to either the cyclopropanation product (*e.g.* **376**) or the [4+1]-cycloadduct (*e.g.* **372, 377**), depending on the substrate and reaction time [862]. In most cases the cyclopropanation product rearrange to the [4+1]-cycloadduct upon heating. The [4+1]-cycloadducts undergo alcohol loss during purification to afford the alkenylfurans (*e.g.* **373**). Double addition of dihydrofuran to alkynylcarbene complexes (*e.g.* **378**) to afford complex cyclopropane derivatives (*e.g.* **381**) was reported [863]. The proposed process involves [2+2]-cycloaddition to afford cyclobutenylcarbene complex (**380**) followed by cyclopropanation using an additional equivalent of the alkene. A one-pot 1,3-dipolar addition of diazomethane to the triple bond followed by cyclopropanation was also demonstrated. Reaction of alkenylcarbene complexes (*e.g.* **370**) with 1,1-dimethylallene (**382**) led to 2-alkoxy-1,3-diene derivatives in a net metathesis reaction [864].

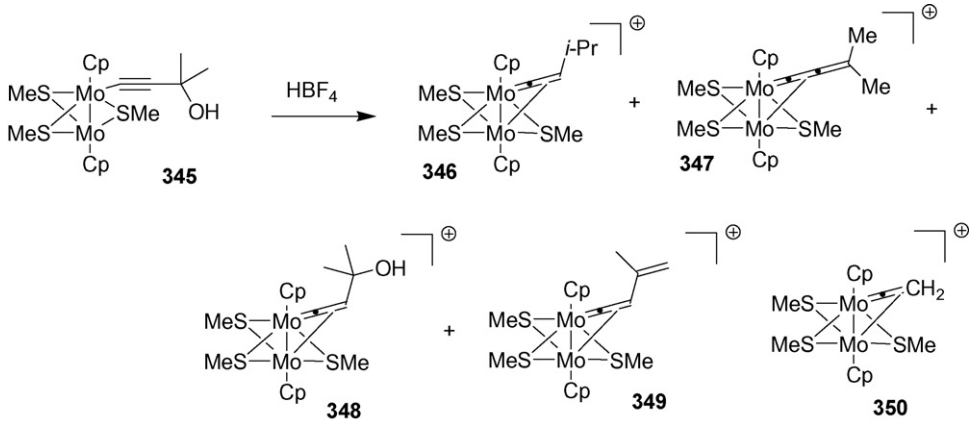
2.3.3.4. Reaction of Group 6 metal–carbene complexes with alkynes—benzannulation. Many examples of benzannulation using α,β-unsaturated chromium–carbene complexes (Scheme 26) and alkynes (commonly known as the Dötz reaction) were reported in 2007. Examples include: (1) formation of *m*-cyclophanes (*e.g.* **386**) in an intramolecular benzannulation as models for a potential synthesis of phomactin and dependence of the reaction success on ring size [865]; (2) use of a similar reaction process to form a related *m*-cyclophane (**388**) for total synthesis of phomactin B2 [866]; (3) enhanced yields for benzannulation reactions (and other



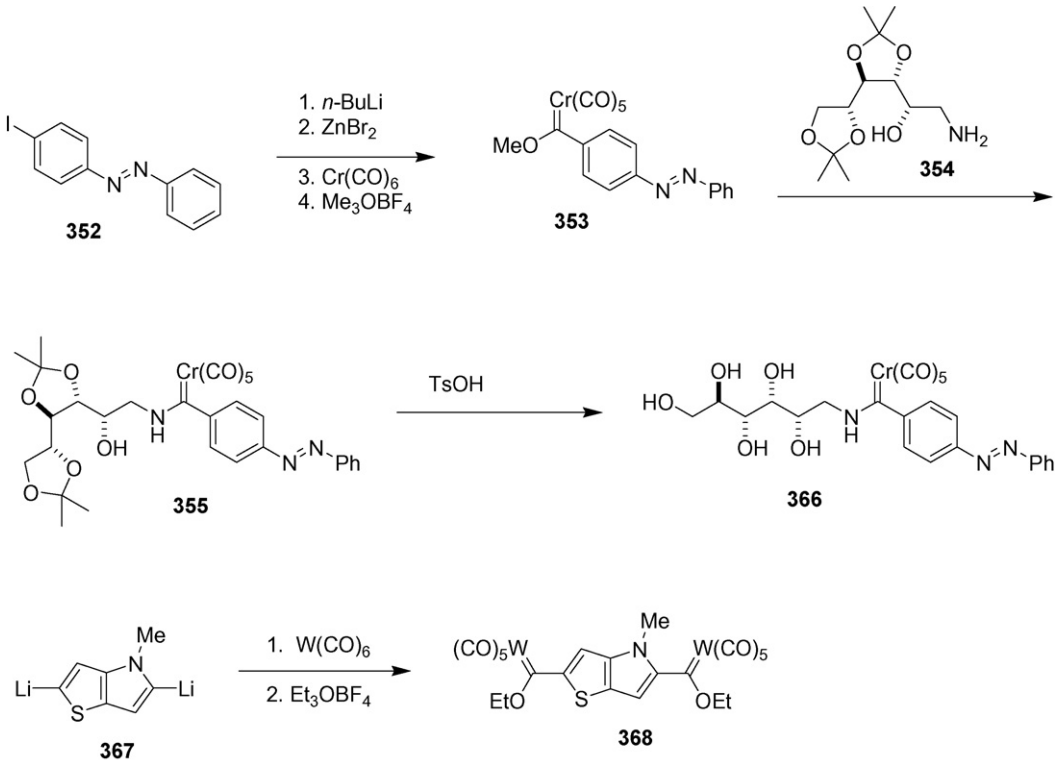
Scheme 22.

carbene-alkyne couplings) conducted in an ionic liquid solvent [867]; and (4) formation of a *m*-cyclophane (**390**) for total synthesis of arnebinol [868]; A DFT study of benzannulation and subsequent haptotropic rearrangements of the arene–$Cr(CO)_3$ complexes was reported [869].

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 2007.



Scheme 23.



Scheme 24.

A net [5+2+1]-cycloaddition process was observed in the coupling of dienylcarbene complexes (*e.g.* **391**, Scheme 27) with alkynes [870]. The eight-membered ring forming reaction was limited to systems where the α,β-alkene was embedded in a cyclobutene ring, likely due to the ring strain in the unobserved benzannulation product. Hypothetical mechanistic scenarios were studied by DFT calculations.

The coupling of ferrocenylalkynes (*e.g.* **394**, Scheme 28) with cyclopropylcarbene–Group 6 metal complexes was reported. Thermal reaction of alkyne **394** with the cyclopropylcarbene-molybdenum complex (**395**) leads to mixtures of cycloheptadienone isomers (**396–398**) accompanied by minor amounts of cyclobutenones in some of the examples [871]. The analogous reaction with the chromium carbene complex (**399**) leads to cyclopentenone derivatives (**400**) [872].

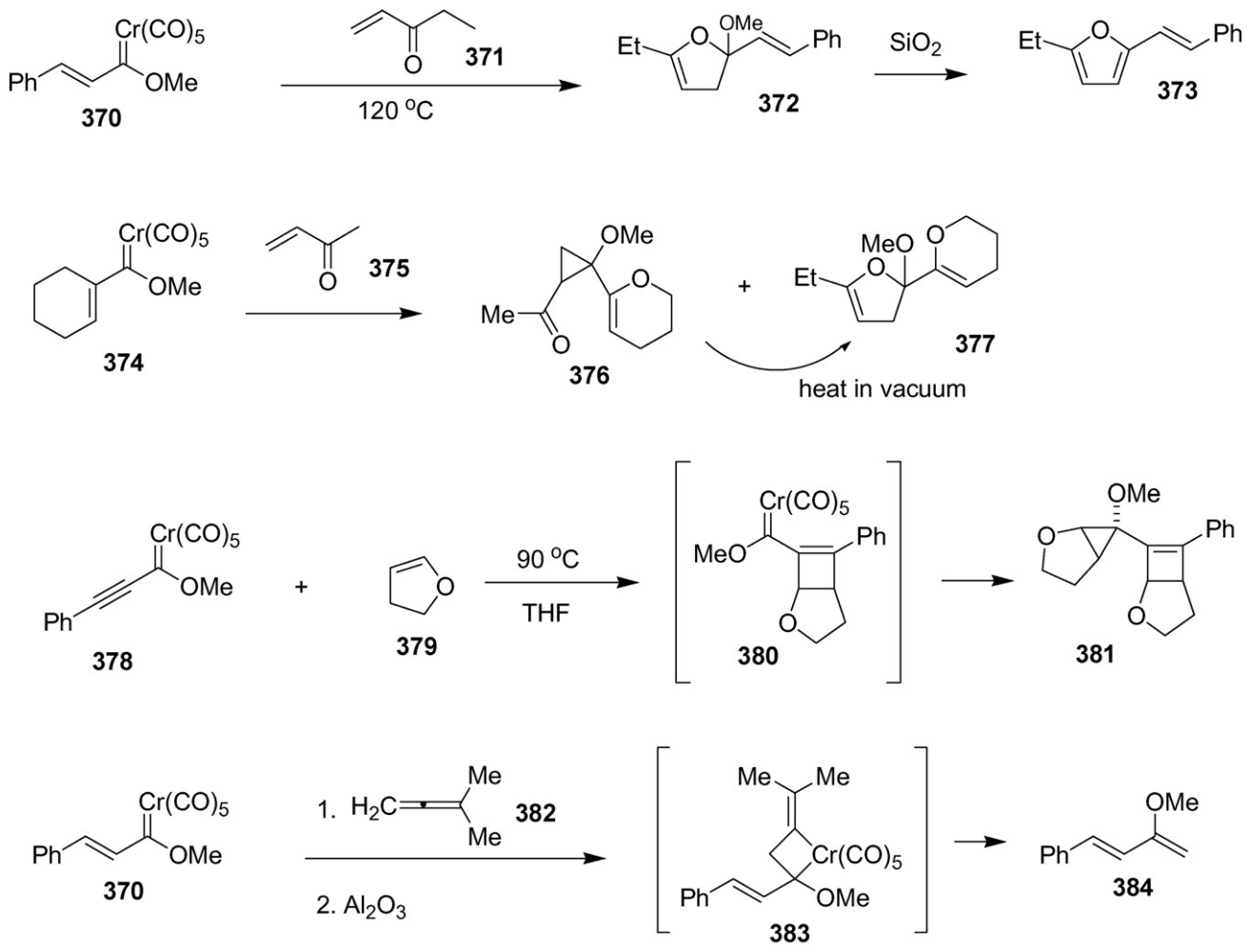
Several papers in 2007 report on the generation of isobenzofurans (*e.g.* **403, 409**, **412, 415**, Scheme 29) from the coupling of carbene complexes with 2-alkynylbenzoyl derivatives (*e.g.* **401, 407, 414**), followed by transformation of the isobenzofuran to a stable organic compound. The generation of dienylisobenzofurans (*e.g.* **403**) through the coupling of 2-alkynylbenzophenone derivatives (*e.g.* **401**) with α,β-unsaturated carbene complex **402** was reported [873]. The isobenzofurans react with dimethyl acetylenedicarboxylate (**404**) to form either furan Diels–Alder products (*e.g.* **405**) or [8+2]-cycloaddition products (*e.g.* **406**). In some cases, the Diels–Alder adducts thermally rearrange to [8+2]-cycloadducts. Related alkenylisobenzofurans (*e.g.* **409**) convert to alkylidenephthalans (*e.g.* **410**) when generated in aqueous solution [874]. Reaction of alkynylbenzaldehydes (*e.g.* **407**) with prenylated carbene complexes (*e.g.* **411**) lead to hydrophenanthrenes (*e.g.* **413**) in a net [5+5]-cycloaddition process [875]. The generation and trapping of pyridine analogs of isobenzofurans (*e.g.* **415**) was also reported [876]. After generation of the isobenzofuran *in situ*, treatment with electron-deficient alkenes (*e.g.* N-phenylmaleimide, **416**) leads to the aza-naphthalene derivatives (*e.g.* **417**).

The coupling of silylated alkynes featuring sterically bulky silyl groups (*e.g.* **418**, Scheme 30) with Fischer carbene chromium complexes (*e.g.* **408**) was reported [877]. The reaction initially affords stable α-silyl-vinylketene complexes (*e.g.* **419**), which subsequently react with carbene equivalents to generate cyclopentenone derivatives (*e.g.* **420, 421**).

The unusual multicomponent coupling of alkynes and alkynylcarbene complexes (*e.g.* **378**, Scheme 31) was reported [878]. Thermolysis with 3-hexyne affords a mixture of cyclopentadienyl compounds **422** and **423**. Compound **422** couples with additional moles of acetylene to afford spirocycles (*e.g.* **424**). Compound **422** can be converted to a bicyclic compound (**425**) via treatment with acid or via exposure to sunlight. Reaction of complex **378** with diphenylacetylene led directly to a bicyclic compound (**426**). Key steps in the mechanism are alkyne insertion to afford an enynylcarbene complex intermediate (**427**), followed by intramolecular addition of the organochromium species to the alkyne to afford a cyclopentadienylchromium complex (**429**) and CO insertion to afford an acylchromium species (**430**), which reacts with additional moles of alkyne to afford the observed products.

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 2007 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").

Several examples of diastereoselective additions to chiral alkenylcarbene complexes were reported (Scheme 32). Diastereoselective Diels–Alder reactions were reported for chiral α,β-unsaturated carbene complex **435** [879]. A novel cyclopropanation reaction was observed in the coupling of chiral α,β-unsaturated tungsten carbene complexes (*e.g.* **437**) with methoxyfuran, leading to alkenylcyclopropylcarbene complexes (*e.g.* **440**) [880]. A mechanism involving Michael addition to afford a zwitterionic intermediate (**439**) followed by intramolecular S_N2 reaction on the



Scheme 25.

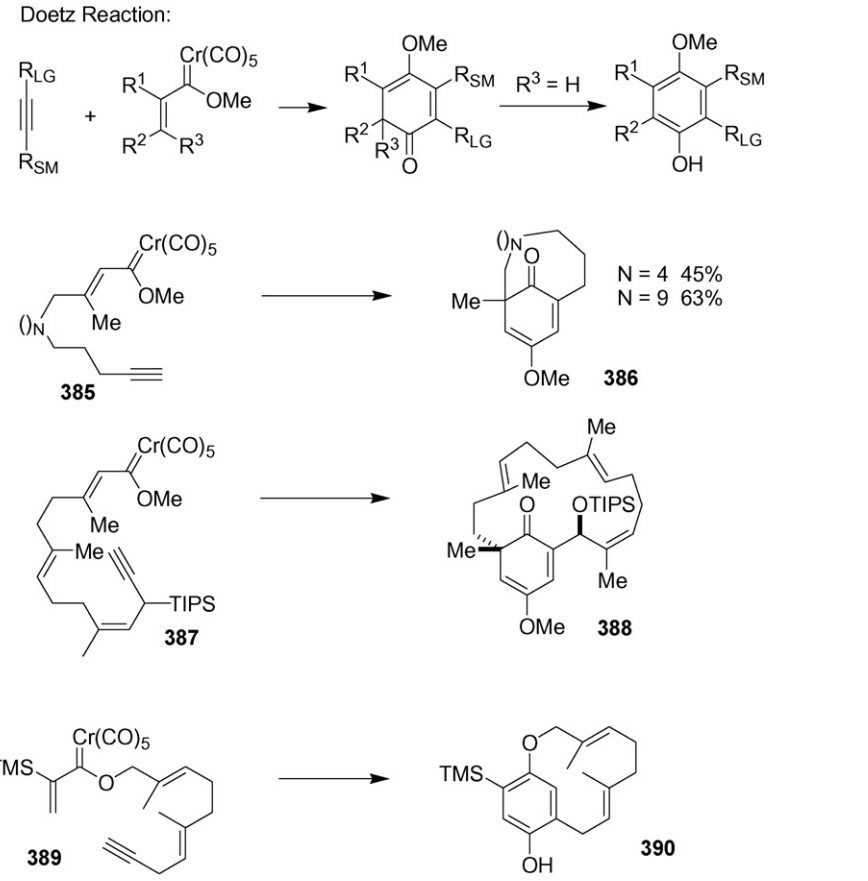
oxonium ion intermediate was proposed. The overall process was highly stereoselective.

Reaction of *o*-styrylalkynylcarbene complexes (*e.g.* **441**, Scheme 33) with various species that add to alkynylcarbene complexes (*e.g.* nitrones, enol ethers, or dienes) was reported [881]. Reaction with nitrones (*e.g.* **442**) leads to cyclopropane-fused dihydronaphthalenes (*e.g.* **444**) in a process involving cycloaddition to afford carbene complex **443**, which then undergoes intramolecular cyclopropanation. Related compounds (*e.g.* **446, 447**) were obtained through [2+2]-cycloaddition reactions with enol ethers followed by intramolecular cyclopropanation. Reaction of the stilbenyl analog led to the fused naphthalene derivative **447** in a process involving cycloaddition to afford an intermediate carbene complex (**445**) followed by metathesis with the pendant alkene group. Similar processes involving initial Diels–Alder reactions between **441** and dienes were also reported. The cyclopropanation process was suggested to involve an electrophilic attack on the alkene followed by ring closure based on DFT studies of the reaction.

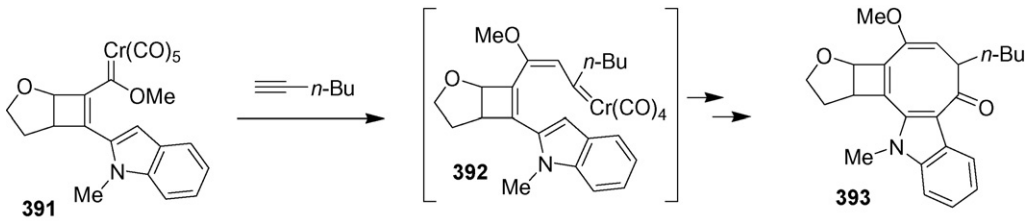
The reaction of urea derivatives (*e.g.* **449**, Scheme 34) with alkynylcarbene complexes (*e.g.* **448**) led to uracil-type carbene complexes [882]. If unsymmetrical ureas were employed, predominantly the regiochemistry depicted by **451** was observed. The key step in the proposed mechanism is Michael addition of the less substituted nitrogen to the alkyne group of the carbene complex to afford zwitterionic intermediate **450**, which leads to **451** after proton transfer and intramolecular aminolysis. Microwave and thermal heating methods were compared. The complexes were oxidized to the corresponding uracils (*e.g.* **452**) through treatment with amine oxides or fluoride ion in air [883].

2.3.3.7. Physical organic chemistry of Group 6 Fischer carbene complexes. The rate constants for the exchange of aryl groups in complexes of general structure **453** (Scheme 35) and hydrolysis reactions were reported [884]. The aryl exchange process is a stepwise process, which is in contrast to the results obtained with electronically related esters. The difference was attributed to the enhanced stability of the tetrahedral intermediate in the case of carbene complexes. Similar studies were reported for the conversion of aryloxycarbene complexes to thiocarbene complexes (**457**) [885]. In this case the rate determining step was dependent on the pKa of the thiol precursors. The rate determining step is k_2 for low pK_a thiols (*e.g.* PhSH) and k_1 for higher pK_a thiols (*e.g.* *n*-C_3H_7SH). The rate constants for the reaction of imidazolide anions (and benzimidazolide) (*e.g.* **459**) with Group 6 carbene complexes of general structure **458** was determined using methanol as the solvent [886]. No significant difference in reaction rates between imidazolide and benzimidazolide derivatives was observed.

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

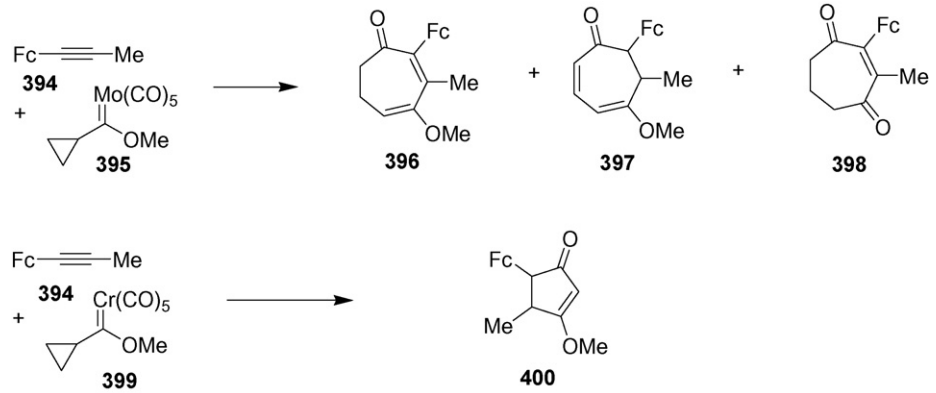


Scheme 26.

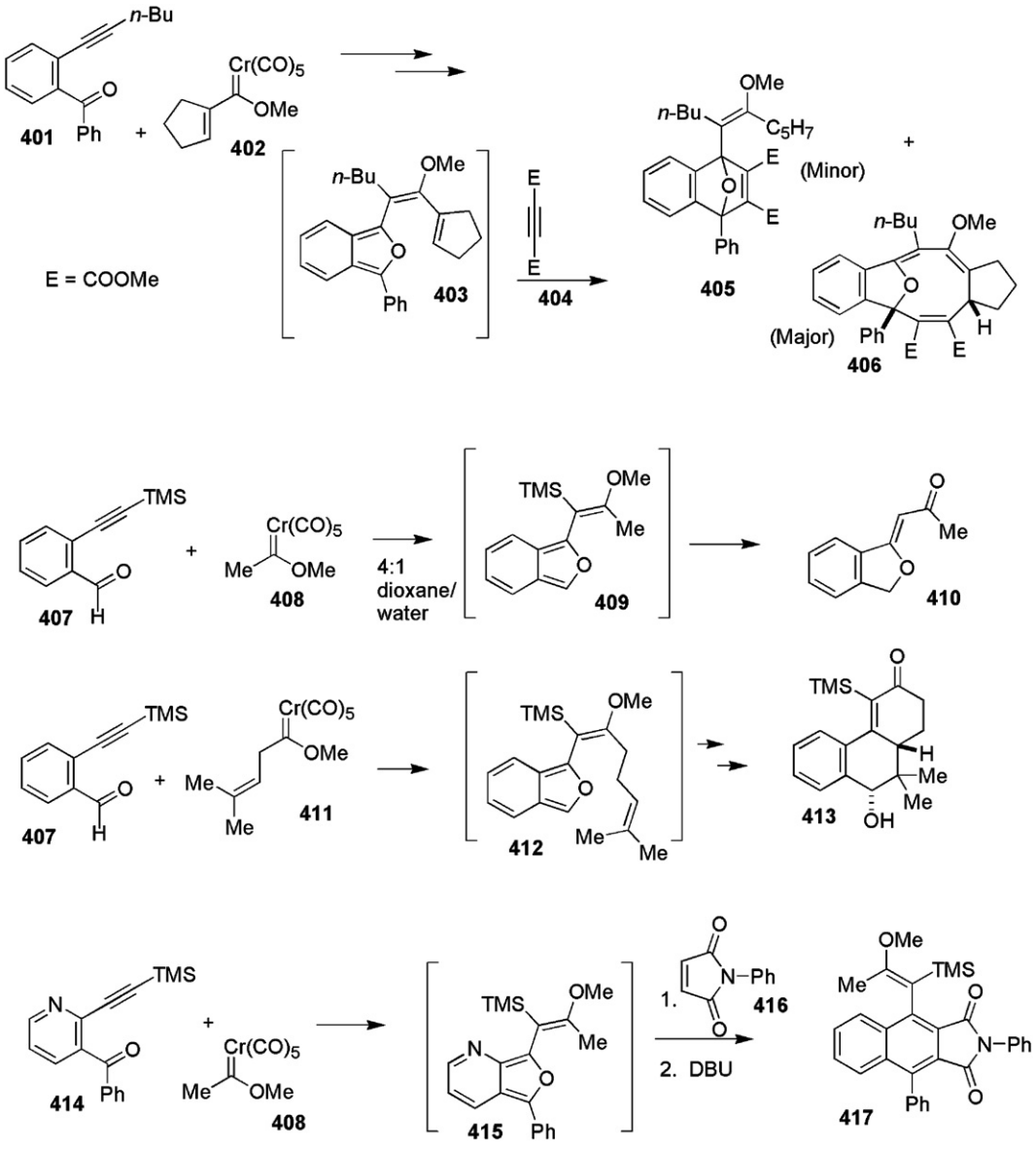


Scheme 27.

The reaction of tungsten vinylidene complexes (*e.g.* **464**, Scheme 36) with alkynyl ethers was reported [887]. The reaction afforded the azulene–carbene complex **466** in a process involving nucleophilic addition to afford the zwitterionic complex **467**, followed be electrophilic addition to the aromatic ring, followed by cyclopropane formation to afford **469**, which undergoes electrocyclic ring opening to afford the carbene complex. The synthesis and reactivity of heptapentaenylidene–chromium and –tungsten complexes (*e.g.*

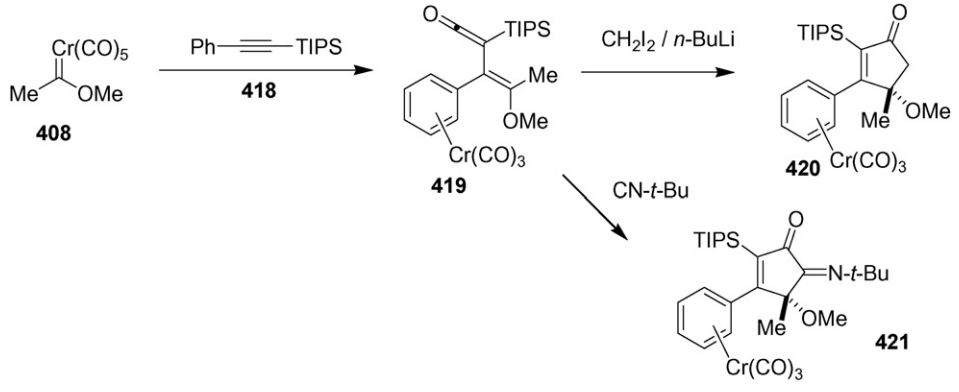


Scheme 28.

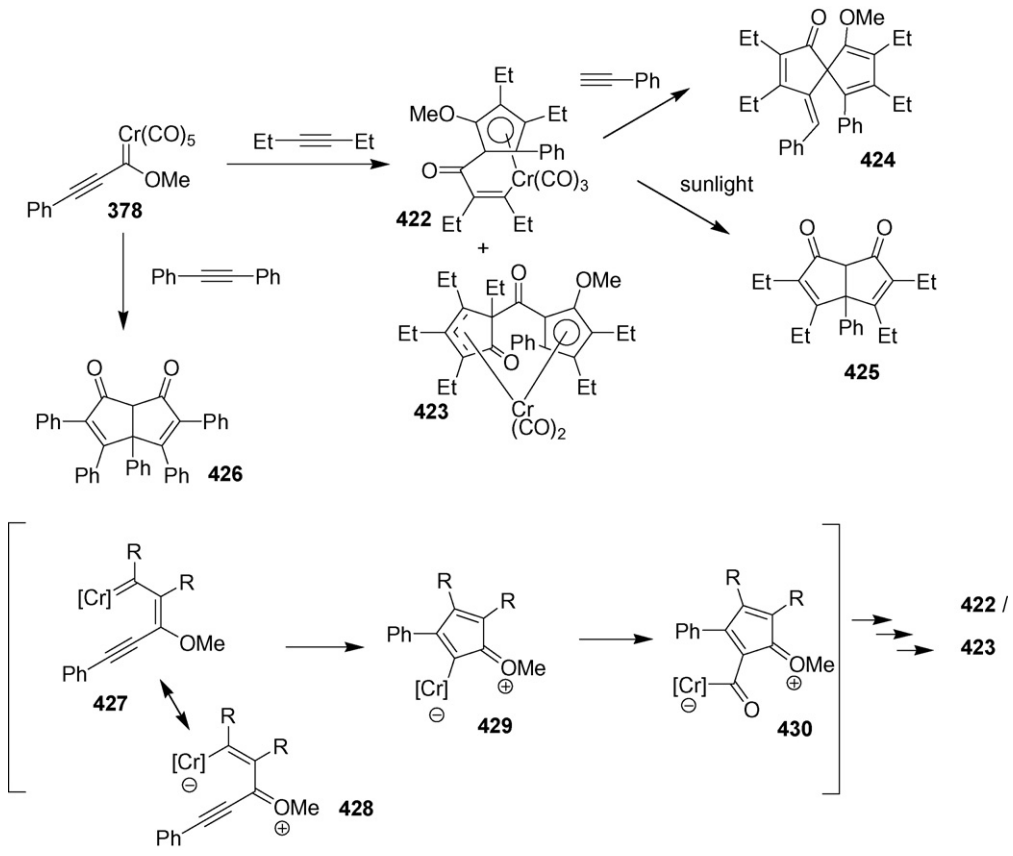


Scheme 29.

471) was reported [888]. The carbene complex was synthesized by reaction of a dialkynyl anion with a metal pentacarbonyl source, which forms the cumulenylidene complex upon elimination of one amine group. The bis(dimethylamino)heptapentaenylidene complex **471** reacts with nucleophiles at either the 5- or 7-positions to form substituted cumulenylidene complexes (*e.g.* **472–474**). The reaction of tungsten vinylidene complexes (*e.g.* **475**) with phosphinidenes (*e.g.* **476**) was reported [889]. The reaction leads to



Scheme 30.

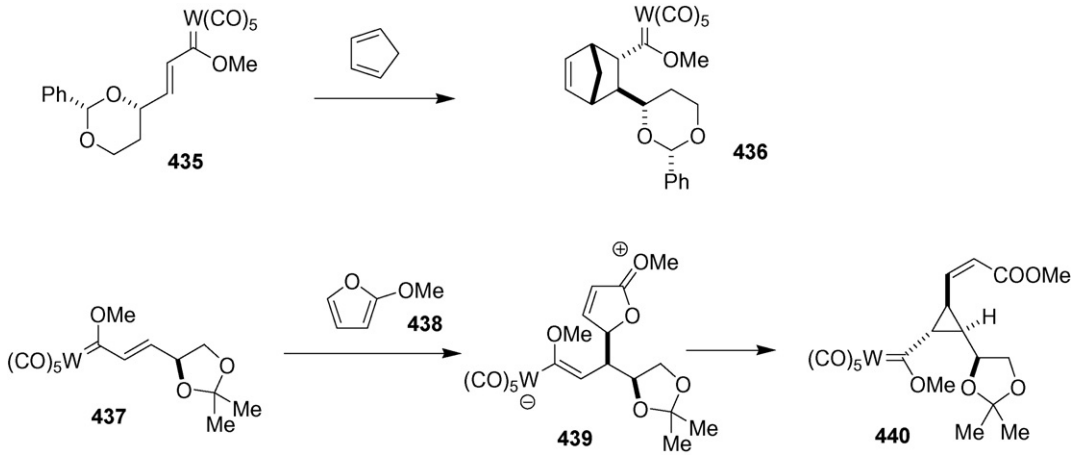


Scheme 31.

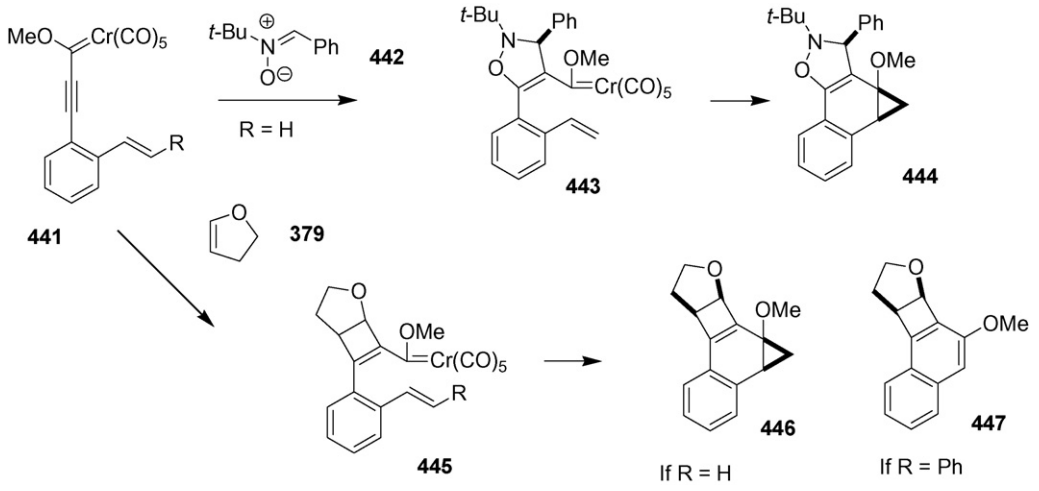
a mixture of the tungsten phosphido complexes (*e.g.* **477**) and aminocarbene complexes (*e.g.* **478**).

Tungsten(0) vinylidene complexes (*e.g.* **480, 484**, Scheme 37) were suggested as intermediates in several processes involving the treatment of terminal alkynes with tungsten pentacarbonyl sources. Glycal derivatives (*e.g.* **481**) were synthesized through treatment of alkynols (*e.g.* **479**) with tungsten hexacarbonyl in the presence of DABCO and photoirradiation was reported [890]. Synthesis of disaccharide glycal derivatives (*e.g.* **485**) through treatment of glycosylated alkynols (*e.g.* **482**) with a catalytic amount of tungsten carbene complex (**483**) was reported [891]. In this reaction the tungsten carbene complex serves as a tungsten(0) source which reacts with the alkyne functionality to afford the vinylidene complex intermediate (**484**).

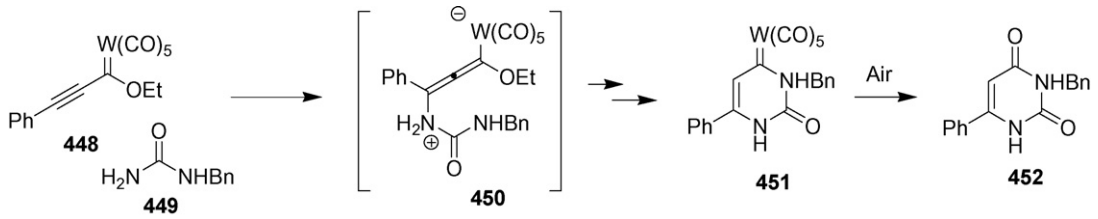
2.3.3.9. Reactions involving carbanions derived from deprotonation of Group 6 metal–carbene complexes. The reaction of carbene complexes with iminoyl halides (*e.g.* **489**, Scheme 38) was reported [892]. Alkoxycarbene complexes led to di-heteroatom stabilized carbene complexes (*e.g.* **491**) in a net metalladi-π-methane rearrangement from carbene iminoacylation product **490**. Thiocarbene complexes led to pyrroles (*e.g.* **493**) through formation of a vinylidene intermediate (**492**) followed by intramolecular hydrogen transfer and cyclization. Condensation with α,β-unsaturated iminoyl chlorides (*e.g.* **494**) was also reported [893]. In this case the thiocarbene complex afforded predominately the cyclopentadienimine **497**, while the alkoxycarbene afforded the amino(alkoxy)carbene complex analogous to **491**. The key steps in formation of the cyclopentadiene complex is conversion of imi-



Scheme 32.



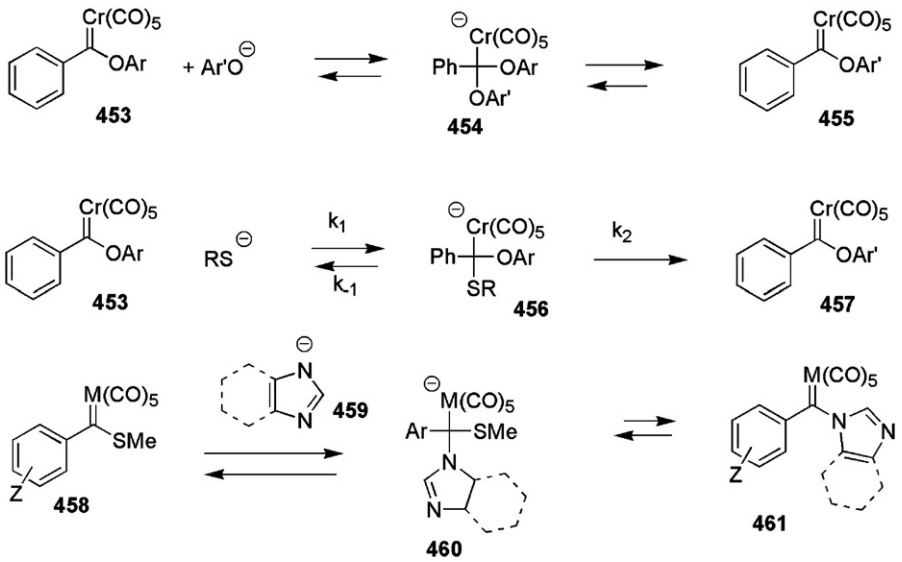
Scheme 33.



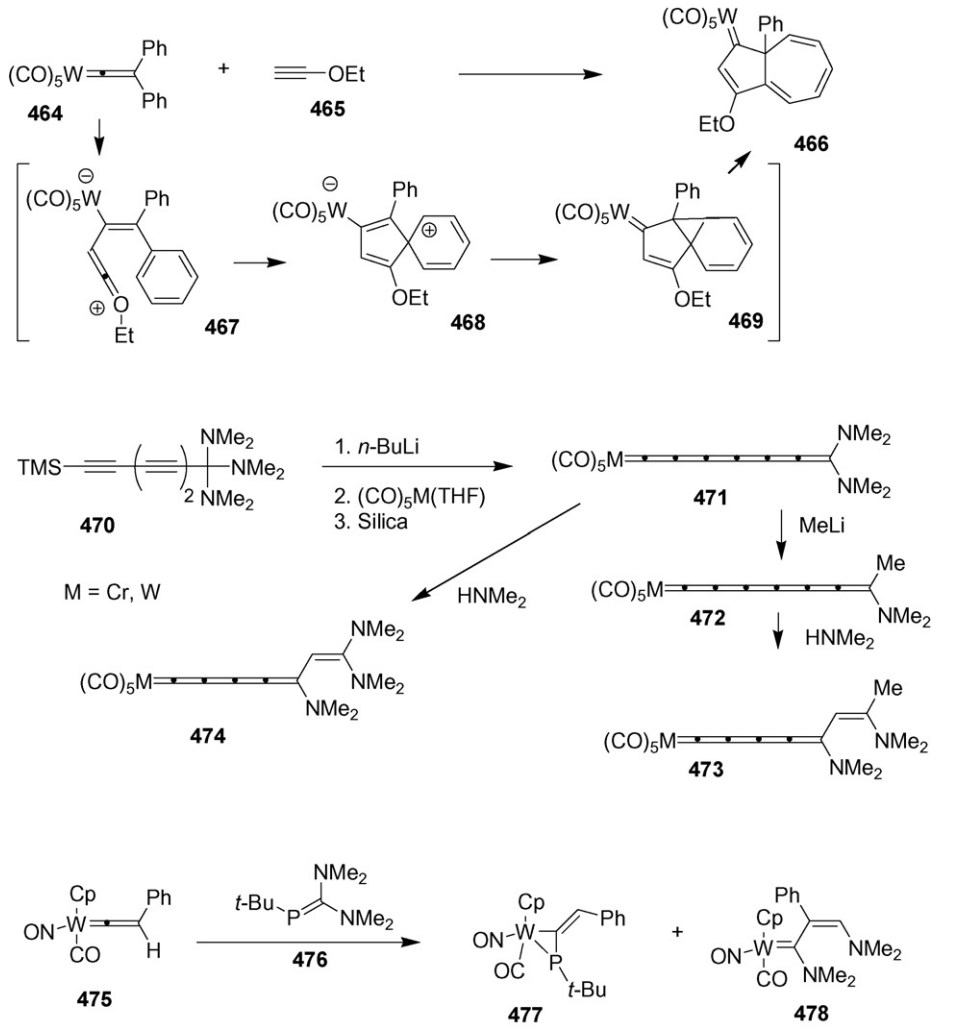
Scheme 34.

noacylation product **495** to the enamine followed by cyclization of intermediate β-amino-α,β,γ,δ-unsaturated carbene complex **496**. The reaction of cyclic carbene complexes (*e.g.* **498**) with Villsmeyer reagents was also reported [894]. This reaction afforded the alkenylcarbene complex **499**, which is stabilized through resonance interaction with the amine group. Condensation with β-aminoacrolein derivatives afforded dienylcarbene complexes (*e.g.* **501**) exhibiting similar resonance interactions. Analogs featuring additional conjugated alkenes were also prepared. The complexes were subjected to hyper-Rayleigh scattering studies.

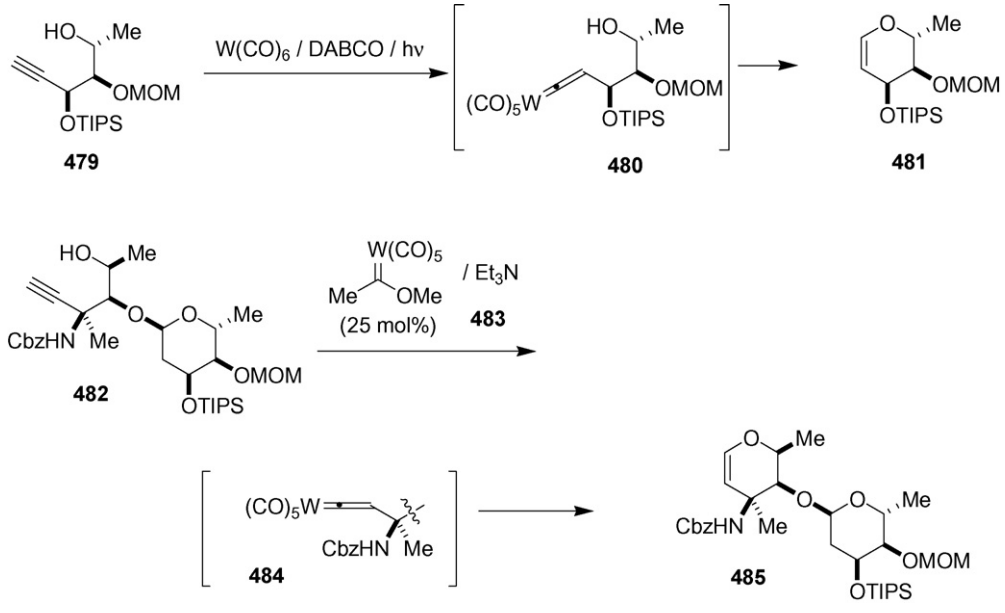
The coupling of Fischer carbene complex-derived anions with DMTSF ($Me_2SSMeBF_4$) in acetonitrile was reported (Scheme 39) [895]. In this reaction, both the product from condensation of activated nitrile **505** with the carbene complex-stabilized anion (**506**) and the sulfenylation-condensation product **507** were obtained. If trimethyloxonium salt was used to activate the nitrile, only the simple nitrile condensation product **506** was obtained. The coupling of carbene complex-stabilized anions with benzopyrilium salts (*e.g.* **508, 511**) was reported [896]. If the salt was substituted at the β-position, the simple extended conjugation products (*e.g.* **510**) were obtained. Analogs unsubstituted at the β-position (*e.g.* **511**) led to products derived from attack at this position (*e.g.* the heterocyclic ring opening product **512**).



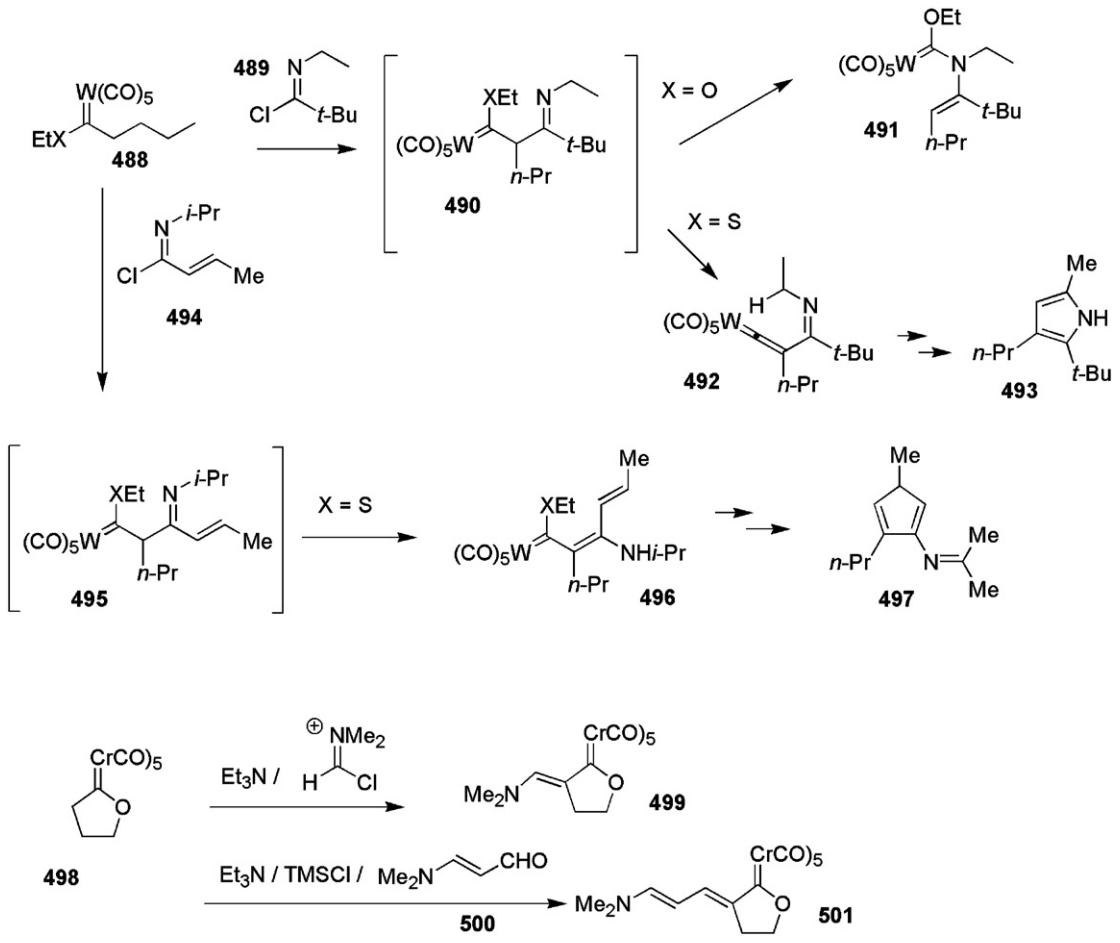
Scheme 35.



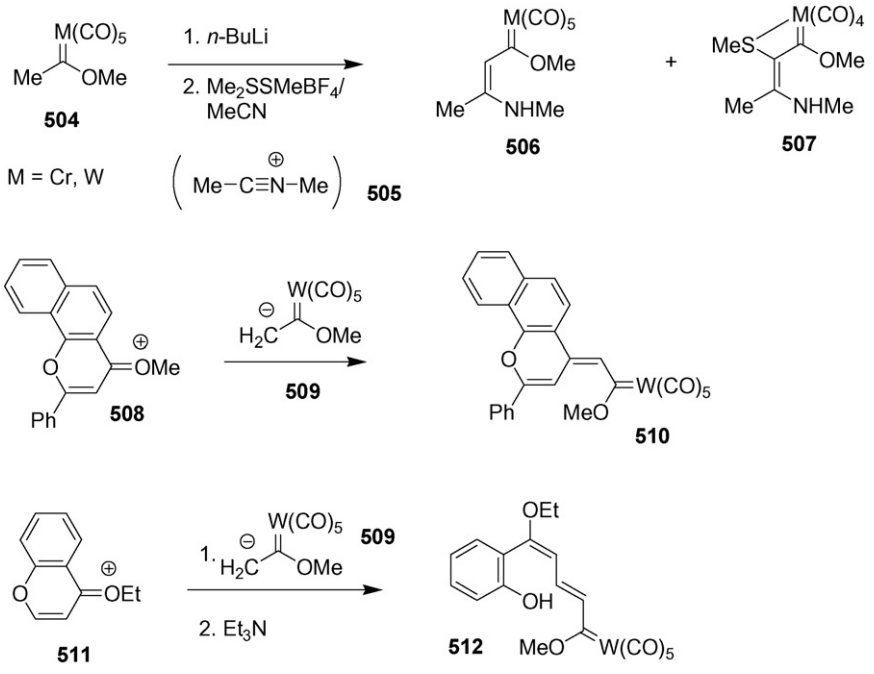
Scheme 36.



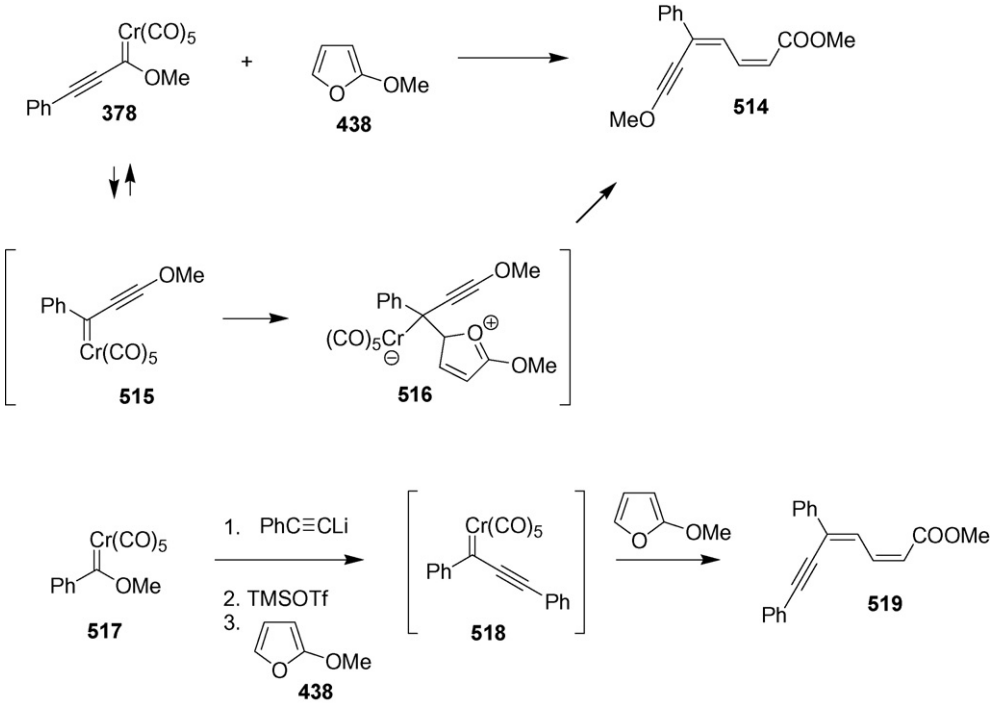
Scheme 37.



Scheme 38.



Scheme 39.



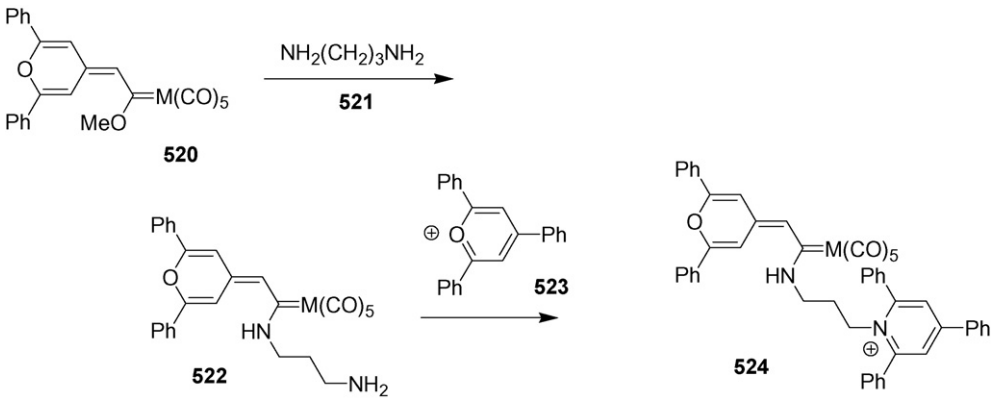
Scheme 40.

2.3.3.10. Reactions involving the addition of nucleophiles to the carbene carbon. The reaction of alkynylcarbene complex (*e.g.* **378**, Scheme 40) with methoxyfurans led to alkoxyalkyne-dienyl esters (*e.g.* **514**) [897]. This reaction proceeds through 1,3-shift of the carbene complex to afford intermediate **515**, followed by addition of methoxyfuran to the carbene carbon to afford zwitterionic complex **516**, which affords dienyne ester **514** through demetallation with ring opening. An *in situ*-generated unstabilized alkynylcarbene complex (**518**) could similarly be trapped by methoxyfuran.

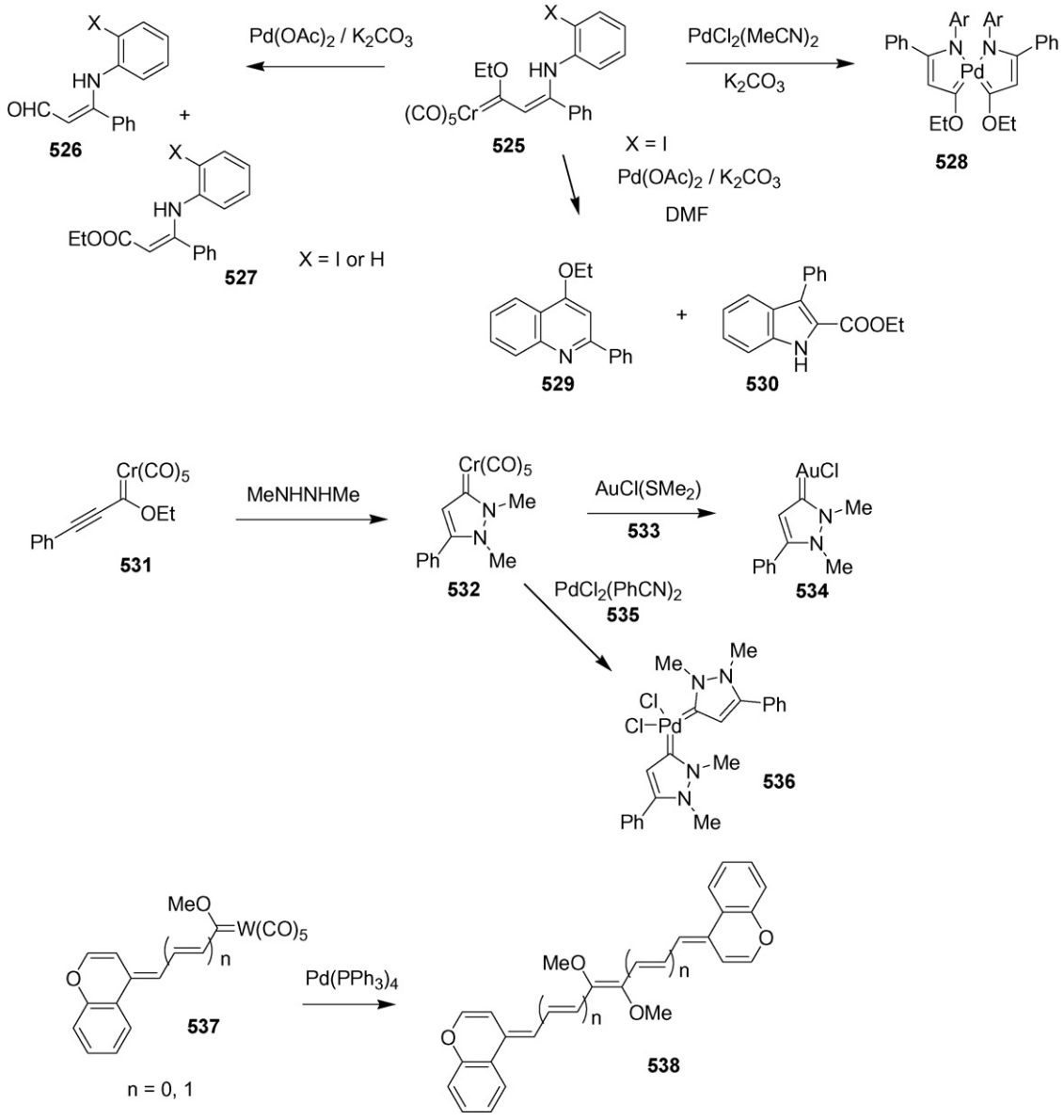
The coupling of diamines (*e.g.* **521**, Scheme 41) with pyrone-carbene complexes (*e.g.* **520**) was reported [898]. Coupling with the amine initially affords the aminocarbene complex (**522**), which subsequent reacts with pyrilium salts (*e.g.* **523**) to afford the pyridinium-substituted carbene complexes (*e.g.* **524**). Various NH_2-substituted carbene complexes were also coupled with 2,4,6-trichlorotriazene.

2.3.3.11. Reactions that involve transfer of a Fischer carbene ligand to another metal. The reaction of β-amino-α,β-unsaturated carbene-chromium complexes (*e.g.* **525**, Scheme 42) with various palladium salts was reported [899]. Reaction with bis(acetonitrile)palladium(II) chloride led to carbene transfer products [*e.g.* bis(carbene) complex **528**]. Reaction with palladium(II) acetate and potassium carbonate in solvents other than DMF led to carbene demetallation products (*e.g.* **526, 527**). In cases where the aryl X group is I, reaction with palladium(II) acetate and potassium carbonate in DMF led to cyclization products (*e.g.* **529, 530**). Palladium nanoparticles were generated through the reaction of tungsten carbene acylates with palladium(II) salts in water [900]. The addition of N,N′-dialkylhydrazines to alkynylcarbene complexes (*e.g.* **531**) to afford pyrazolylidene–chromium complexes (*e.g.* **532**), followed by transfer of the carbene unit to gold, silver, palladium, or platinum was reported [901]. Saturated heterocyclic ring systems were obtained through addition of dialkylhydrazines to allenylidene-chromium complexes. The palladium complexes (*e.g.* **536**) were useful as catalysts for the Suzuki coupling reaction. A palladium-catalyzed carbene dimerization of pyranylidene-carbene complexes (*e.g.* **537**) was also reported [902].

The nickel-catalyzed coupling of α,β-unsaturated Fischer carbene complexes (*e.g.* **539**, Scheme 43) and alkynes to afford



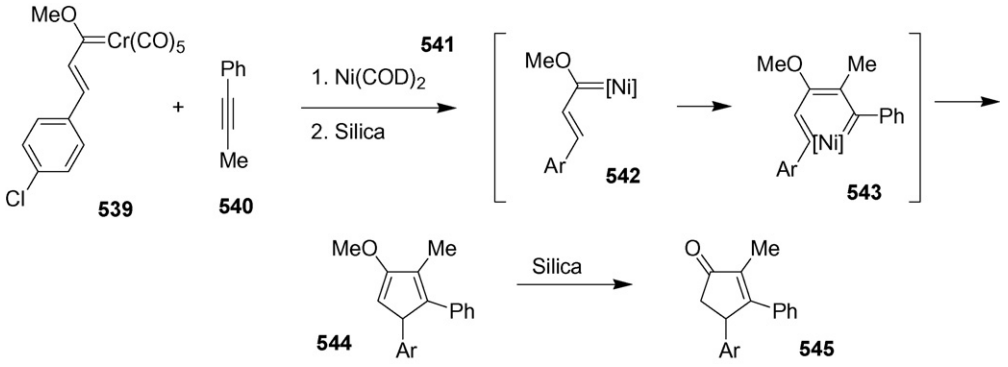
Scheme 41.



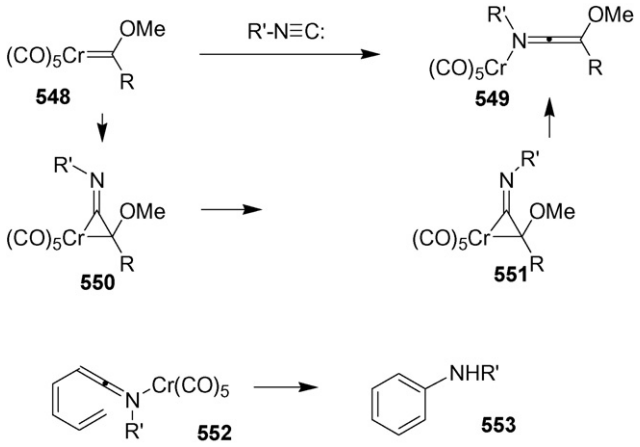
Scheme 42.

cyclopentenones (*e.g.* **545**) in a net [3+2]-cycloaddition process was reported [903]. The key step in the proposed mechanism is transfer of the carbene ligand from chromium to nickel to afford a nickel carbene complex intermediate (**542**), which undergoes alkyne insertion followed by cyclization to afford cyclopentadiene-enol ether derivatives (*e.g.* **544**). Enol ether hydrolysis on silica gel leads to the cyclopentenone products.

2.3.3.12. Other reactions of Group 6 metal–carbene complexes. A DFT study of the reaction of Fischer carbene complexes with iso-



Scheme 43.



Scheme 44.

cyanides to afford ketenimine derivatives (*e.g.* **549**, Scheme 44) was reported [904]. The most energetically reasonable mechanism involves addition to the carbene to afford the metallacyclopropane imine (**550**), followed by E/Z isomerization of the imine, followed by conversion to the N-ligated complex **549**. Metal coordination appears to have no effect on a commonly employed reaction of carbene complex derived ketenimine derivatives, the cyclization of dienylketenimines (**552**) to aminobenzene derivatives (**553**).

Other experimental studies of Group 6 metal–carbene complexes are depicted in Scheme 45 and include: (1) crystal structures for dppe-ligated Fischer carbene complexes [905]; (2) electrochemical studies of Fischer carbene complexes of Group 6 and iron [906]; (3) carbene ligand exchange reactions of Fischer carbene complexes with stable carbene complexes to afford NHC–$Cr(CO)_5$ complexes (*e.g.* **556**) [907]; (4) deprotonation of ethynylcarbene complexes (*e.g.* **557**) and conversion to the tungsten-gold bis(alkyne) complexes (*e.g.* **559**), and subsequent slow isomerization of the initially formed tungsten carbene complexes to the gold carbene complexes (*e.g.* **560**) [908]; (5) reduction of η^4-alkyne–molybdenum(II) complexes (*e.g.* **561**) using KFp, which leads to the η^2-alkenylmolybdenum complexes (*e.g.* **563**) via the molybdenum(I) η^4-alkyne complexes (**562**); and subsequent reoxidation of complex **563** to the original starting material **561** [909]; (6) contribution of a carbene resonance form (**565**) to a alkyne–thiolate–molybdenum complex (**564**) [910]; (7) formation of cationic bridging alkoxycarbyne-bis(molybdenum) and tungsten complexes (*e.g.* **567**) through protonation or methylation of the corresponding bring carbonyl compounds (*e.g.* **566**) [911] and reactions of neutral analogs with CO [912]; (8) ligand substitution processes in cationic bridging carbyne-bis(molybdenum) complexes [913]; (9) formation of tungstimidazole complexes (*e.g.* **569/570**) and discussion of carbene resonance contributions [914]; (10) possible involvement of chromium carbene complexes in chromium(II) chloride-induced cyclopropanation of alkenes using di- and trihaloalkanes [915]; (11) possible involvement of chromium carbene (*e.g.* **573**) or carbyne complexes in chromium(II) mediated coupling of 1,1,1-trichloroalkanes with aldehydes to afford allylic alcohols (*e.g.* **574**) [916] and conversion of 1,1,1-tricholoroalkanes to acetylenes or alkenes in the absence of a carbonyl additive [917]; (12) reaction of $Cr_2(CO)_5(C_5Me_5)$ with diazomethane to afforded coordinated ketene mononuclear complexes $Cr(CO)_2(CH_2{=}C{=}O)(C_5Me_5)$, which may involve carbene complexes [918]; and (13) identification of carbene and carbyne complexes from laser ablation of Group 6 metals in the presence of halogenated methane derivatives via comparison of transient IR spectra with DFT-calculated spectra [919]. A DFT comparison of conformational effects for alkoxy- and thiocarbenes and their respective Group 6 metal pentacarbonyl complexes was also reported [920].

2.3.4. Group 7 metal–carbene complexes

Carbene–dimanganese complexes (*e.g.* **581**, Scheme 46) and rhenium analogs were prepared using the Fischer synthesis applied to decacarbonyldimanganese (**580**) [921]. The initially formed alkoxycarbene–manganese complexes were of the axial orientation (carbene ligand trans to other metal), however ammonia-derived aminocarbene complexes (*e.g.* **582**, R = H) adopted the equatorial orientation, while propylamine-derived complexes (*e.g.* **582**, R = Pr) were of the original axial geometry. In the rhenium series all of the carbene complexes were of the equatorial geometry. The formation of unstabilized manganese carbene complexes (*e.g.* **585**) through borylene metathesis was also reported [922]. At low temperature, reaction of the borylene complex (**583**) with ketones leads to the observable oxaboramanganacycle (**584**), which converts to the carbene complex (**585**) at room temperature.

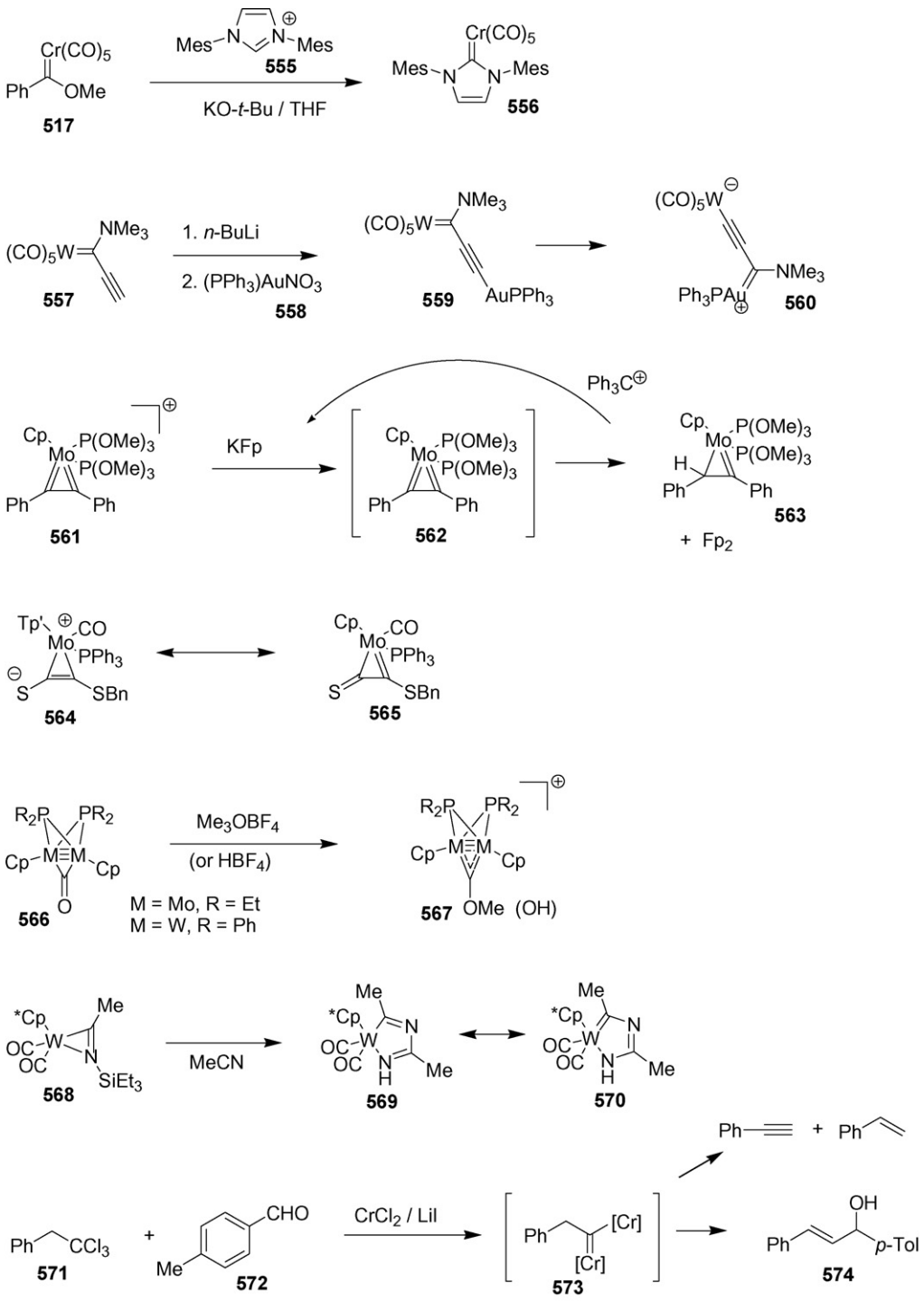
The formation of rhenium carbene complexes (*e.g.* **588**, Scheme 47) through reaction of rhenium hydride complex **587** with various cycloalkenes was reported [923]. The carbene complexes react with additional moles of cycloalkene to afford various dehydrogenation products, depending upon the ring size. Smaller rings favor η^4-cycloalkadiene complexes (*e.g.* **589**) while larger rings afford cyclic alkyne complexes (*e.g.* **590**). Reaction of hydride complex **587** with cyclododecene led directly to the π-allyl complex (**592**) instead of the carbene complex. Reaction of hydride complex **587** with norbornene led to norbornylidene complex **594**, which is a fluxional compound in equilibrium with agostically coordinated norbornene complex **593**. Thermolysis of the **594/594** mixture led to ring-opening product, carbyne complex **596**, which exists in equilibrium with the norbornene coordinated complex **597**.

Additional studies of Group 7 metal–carbene complexes are depicted in Scheme 48. The formation of manganese–NHC complexes (*e.g.* **600, 601**) through reaction of isocyanide complexes with propargylamines or propargyl alcohols was reported [924]. The non-aromatic NHC complexes (*e.g.* **600**) were initially obtained; however the aromatic NHC derivatives (*e.g.* **601**) were obtained using longer reaction times. Shorter C–Mn bonds were noted for the N,O-heterocycles vs the N,N-heterocycles. Manganese carbene complexes are likely intermediates in the reaction of cyclomanganated arylmanganese complexes with 1,1-diphenyldiazomethane, which leads to Mn–C insertion products [925]. The barriers to intramolecular α-hydrogen transfer were calculated for rhenium carbene imido complexes [926]. The one-electron oxidation of manganese vinylidene complexes (*e.g.* **603, 606**) was studied computationally [927]. Replacement of the Cp ligand by the dianionic dicarbollyl ligand (Dcarb) was predicted to result in a more facile one-electron oxidation and subsequent dimerization to the bis(carbyne–metal) complex (*e.g.***605, 608**).

2.3.5. Group 8 metal–carbene complexes

2.3.5.1. Cationic metal–carbene complexes that are not metallacumulenes. Osmium carbene complexes (*e.g.* **611**-**613**, Scheme 49) were generated through protonation of neutral alkenylosmium complexes [928]. Neutral or anionic carbene complexes could be prepared using HCl as the proton source, depending upon the amount of HCl added. Coordinatively unsaturated cationic complex **611** was obtained if HBF_4 was used as the proton source. The reaction of osmium carbene complex **614** with 2,6-diacetylpyridine (**615**) led to the carbene complex **616** in a net C–H activation/carbene exchange process [929].

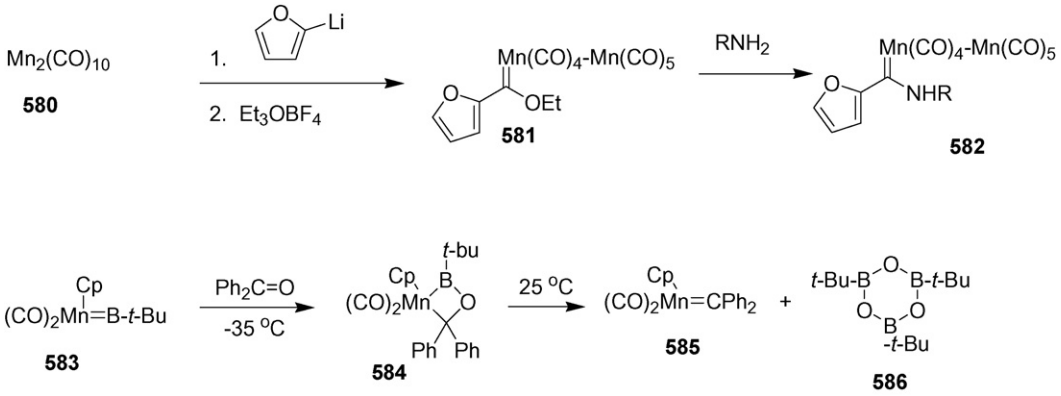
Additional studies of cationic Group 8 carbene complexes are depicted in Scheme 50 and include: (1) involvement of cationic ruthenium carbene complexes (*e.g.* **619–621**) in a diyne cyclization reaction that eliminates a carbon atom (*e.g.* conversion of diyne



Scheme 45.

617 to alkylidenecyclopentane **623**) [930]; (2) potential involvement of cationic ruthenium carbene complex intermediates in the ruthenium-catalyzed coupling of dialkynes with alkenes [931]; (3) involvement of carbene complex intermediates (*e.g.* **627**) in the conversion of sulfur-containing allenic esters (*e.g.* **624**) into alkoxyfuran derivatives (*e.g.* **628**) [932]; (4) involvement of cationic ruthenium carbene complexes in the coupling of benzoxanorbornenes and alkynoate esters to afford cyclopropane-fused norbornenes [933]; (5) generation of cationic ruthenium carbene complexes through electrochemical oxidation of phenylene-bridged diruthenium complexes [934]; (6) DFT studies of $[Cp(CO)_2Fe{=}CH_2]^+$ and comparison with boron analogs [935]; and (7) DFT studies of cationic iron carbene complexes $Cp(CO)(L)Fe{=}CHR^+$ [936].

2.3.5.2. Bis(carbene)ruthenium complexes from coupling of two alkynes and a ruthenium complex. Several reaction processes invoke cyclic ruthenium bis(carbene) complexes as reactive intermediates (see Scheme 51). Ruthenium bis(carbene) complexes (*e.g.* **632**, Scheme 51) were proposed as intermediates in the reaction of acetylene with cationic complex **630** to afford ruthenium–cyclopentadiene complex **631** [937]. The mechanism was studied by DFT calculations. The most reasonable path involves



Scheme 46.

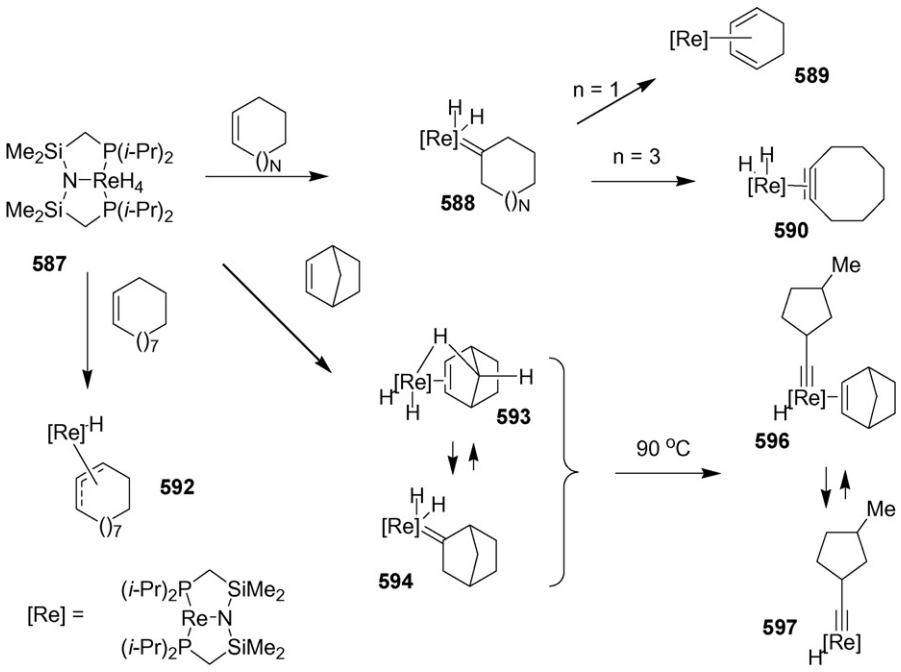
complexation of the alkyne to the bis(carbene) complex to afford metallacyclopentadiene **633**, followed by addition of the NHC to the alkyne ligand to afford the η^2-alkenyl complex **634**, followed by alkyl migration and intramolecular alkene insertion. Several examples of the hydrative cyclization of bis(alkynes) (*e.g.* **636**) to afford dienyl aldehydes (*e.g.* **638**) using cationic ruthenium complex **618** were reported [938]. A key step in this transformation is the addition of water to cationic bis(carbene) complex intermediate **637**.

Bis(carbene) ruthenium complexes (*e.g.* **642**, Scheme 52) are proposed as intermediates in various alkyne trimerization processes, which afford aromatic rings after [2+2]-cycloaddition of the third alkyne component, alkyl migration to the carbene carbon, and demetallation. Examples include: (1) highly regioselective solid-phase alkyne trimerization employing polymer-bound diynes (*e.g.* **639**) [939]; and (2) trimerization of carbohydrate bis(alkynes) (*e.g.* **644**) with acetylene [940].

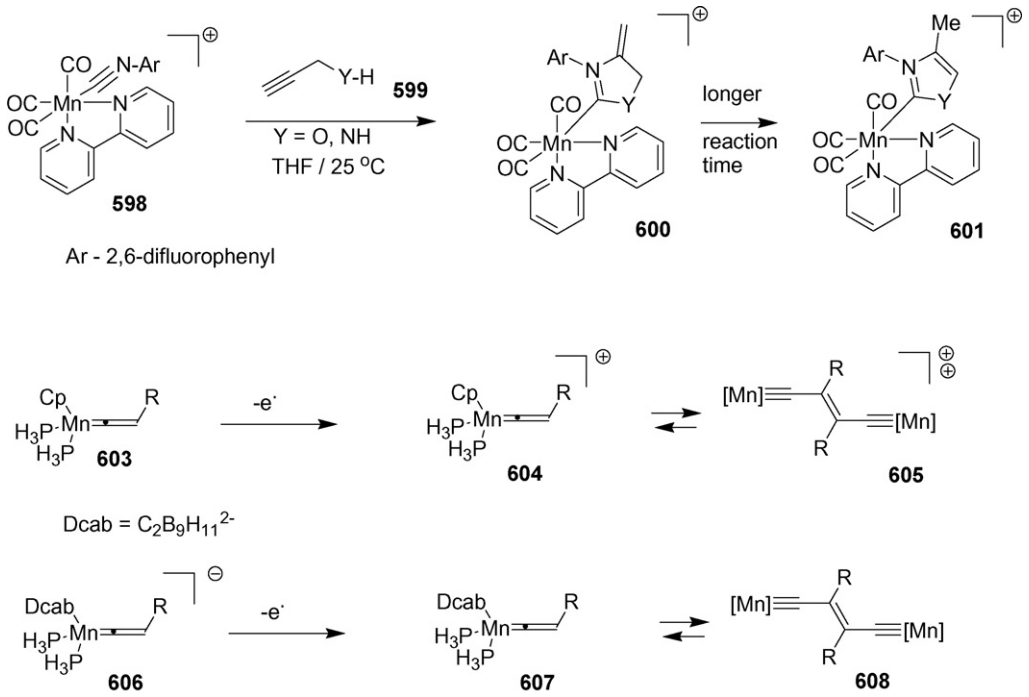
2.3.5.3. Neutral nonheteroatom-substituted metal–carbene complexes that are not cumulenes. Numerous additional examples of the synthesis and reactivity of this class of compounds have been presented in the alkene metathesis section. The Grubbs catalysts fall into this classification.

The formation and reactivity of ruthenabenzene derivatives (*e.g.* **653**, Scheme 53) was reported [941]. Ruthenabenzenes were formed in the coupling of diethynylmethanol (**650**) with ruthenium chlorides (**651** or **652**) in the presence of triphenylphosphine. Various ligand substitution reactions were reported for ruthenabenzene complex **653**. Oxidation with silver(I) led to the dimeric complex **657**, which affords the dicationic ruthenabenzene (**658**) upon treatment with trimethylphosphine. Various ligand substitution reactions were also demonstrated for ruthenabenzene **653**. Thermolysis led to the cyclopentadienyl-bis(phosphonium) salt **654**. The electrochemistry of the ruthenabenzene complexes was also studied.

Group 8 metal–carbene complexes were suggested as intermediates in several processes involving diazo compounds (see Scheme 54). Ruthenium carbene complexes (*e.g.* **661**) were suggested as intermediates in carbonyl olefination reactions using diazo compounds and triphenylphosphine [942]. Carbene complex **661** could be observed in the low temperature NMR when the aldehyde and triphenylphosphine were omitted from the reaction. Reaction of the carbene complex with triphenylphosphine led to the Wittig reagent (**663**), which can react with the carbonyl compound to afford the olefin. Ruthenium carbene complexes (*e.g.*



Scheme 47.

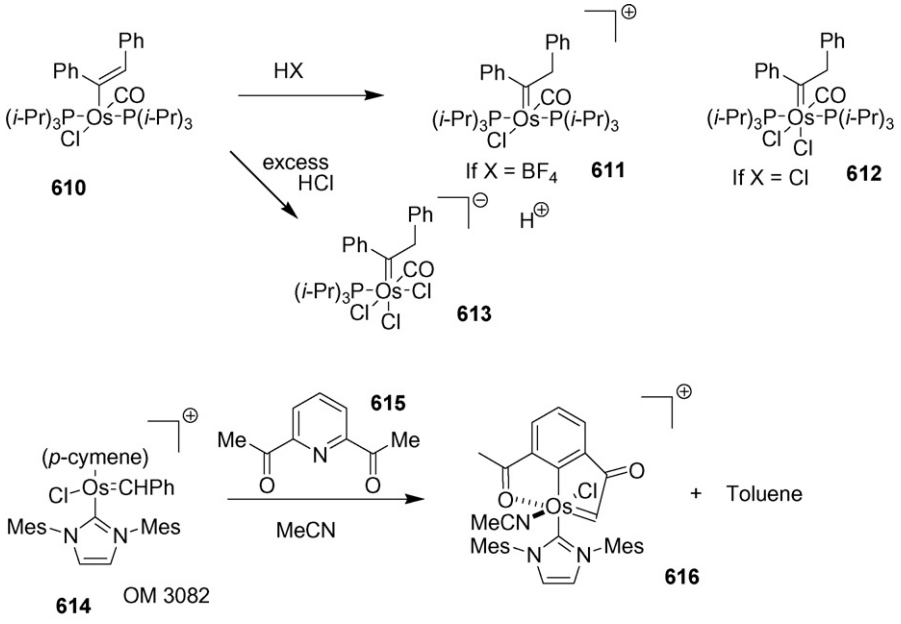


Scheme 48.

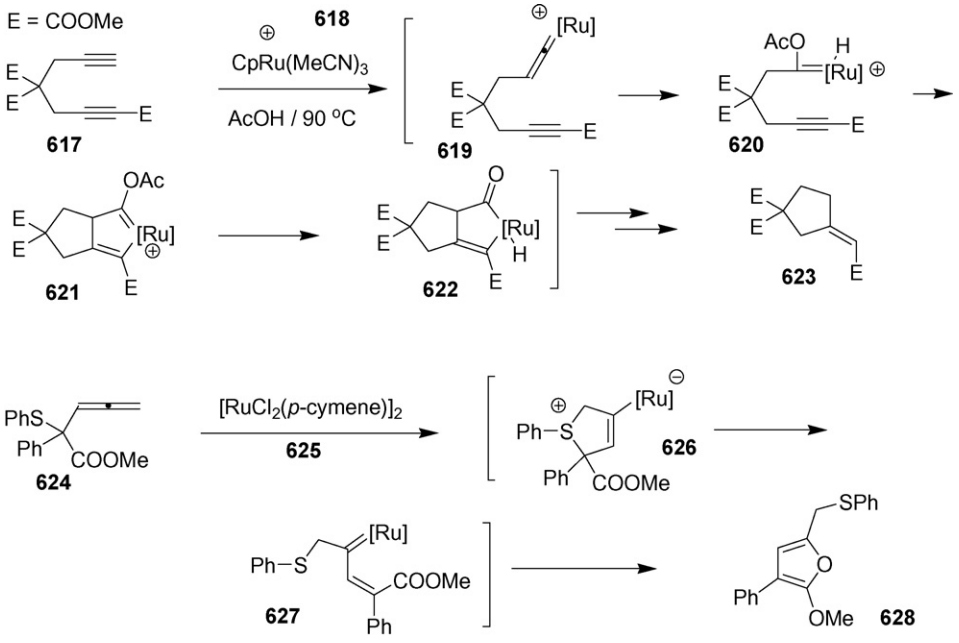
665, 666) were suggested as intermediates in the formation of ring-fused alkenylcyclopropanes (*e.g.* **667**) from enynes and diazo compounds [943]. Initially formed carbene complex **665** undergoes alkyne insertion to afford intermediate carbene complex **666**, which undergoes intramolecular cyclopropanation. Iron carbene complexes were suggested as intermediates in the iron-porphyrin catalyzed reaction of amines with diazo compounds to afford N–H insertion products [944].

Ruthenium carbene complexes have been suggested as intermediates in various ruthenium-catalyzed reactions of propargyl esters (Scheme 55). Ruthenium carbene complexes (*e.g.* **670**) were suggested as intermediates in the reaction of propargylic thioesters (*e.g.* **668**) with methoxyfurans to stereoselectively afford triene-esters (*e.g.* **672**) [945]. The key step in this transformation is formation of carbene complex **670**, which then reacts with methoxyfuran to afford cyclopropane **671**. Ring opening then affords the observed product **672**. Cyclization of propargyl acetates (*e.g.* **673**) to indenes (*e.g.* **675**) using ruthenium complex **669** likely proceeds through a similarly generated carbene complex intermediate (**674**) [946].

Other studies of Group 8 metal–carbene complexes in this category are depicted in Scheme 56, and include: (1) an extensive study of the bonding of carbon (and other elements) to various iron and ruthenium metal–ligand combinations [947]; (2) formation of bis(carbene)–diruthenium complex **677** from reaction of diruthenium–diiron complex **676** with diphenylacetylene



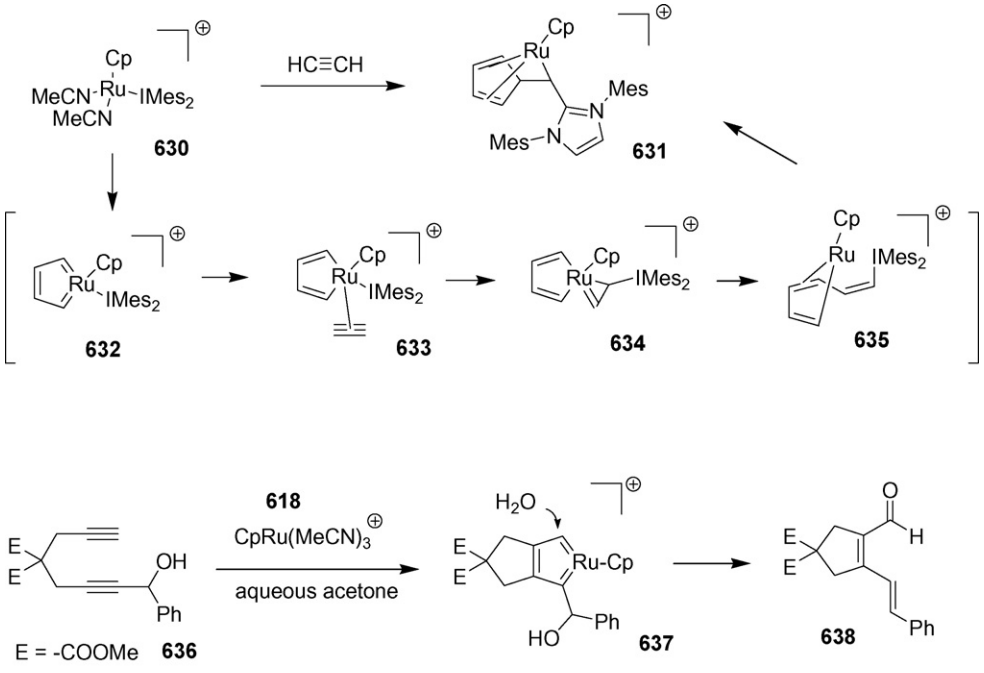
Scheme 49.



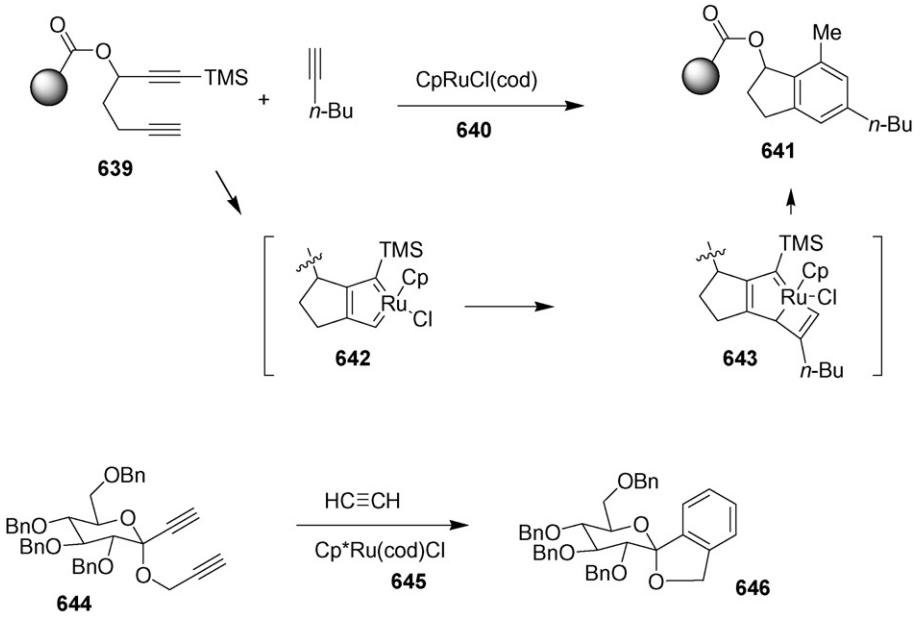
Scheme 50.

[948]; and (3) involvement of ruthenium carbene complexes (*e.g.* **680**) in the formation of bis(cyclopropanes) (*e.g.* **681**), which are minor products from ruthenium-catalyzed cyclization of dieneynes [949].

2.3.5.4. Heteroatom-substituted Group 8 metal–carbene complexes. Several processes involving bridging aminocarbyne-diiron complexes and bridging aminovinylcarbene were reported in 2007 (see Scheme 57). The reaction of bridging aminovinylcarbene iron complex **685** with diazo compounds led to the neutral diazo adduct **686** [950]. Methylation led to the cationic aminocarbene complex **687**. The coupling of bridging isocyanide complex **688** with alkynes led to diverse adducts, depending upon the electronic nature of the alkyne [951]. Reaction of methyl propiolate (**689**) with complex **688** led to complex **690**, which features resonance contribution from metalla-Cp complex structure **691**. Reaction with electron rich acetylenes (*e.g.* diphenylacetylene) followed by protonation led to bridging aminocarbene iron complexes (*e.g.* **693**). Related studies were reported for bridging thiocarbyne-diiron complexes [952]. The reaction of bridging aminovinylcarbene complexes (*e.g.* **694**) with alkynyl anions was reported to be highly dependent on the groups at nitrogen [953]. The N-methyl,N-xylyl complex **694** led to the dicarbene complex **696**, while the N,N-dimethyl complex **697** led to the carbene addition product **698**.



Scheme 51.

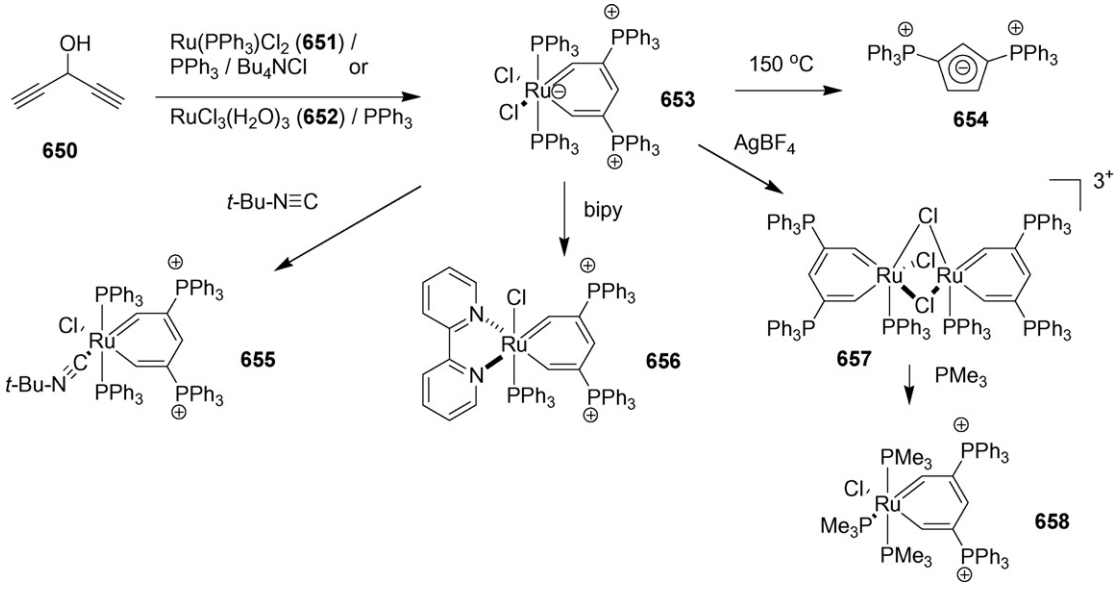


Scheme 52.

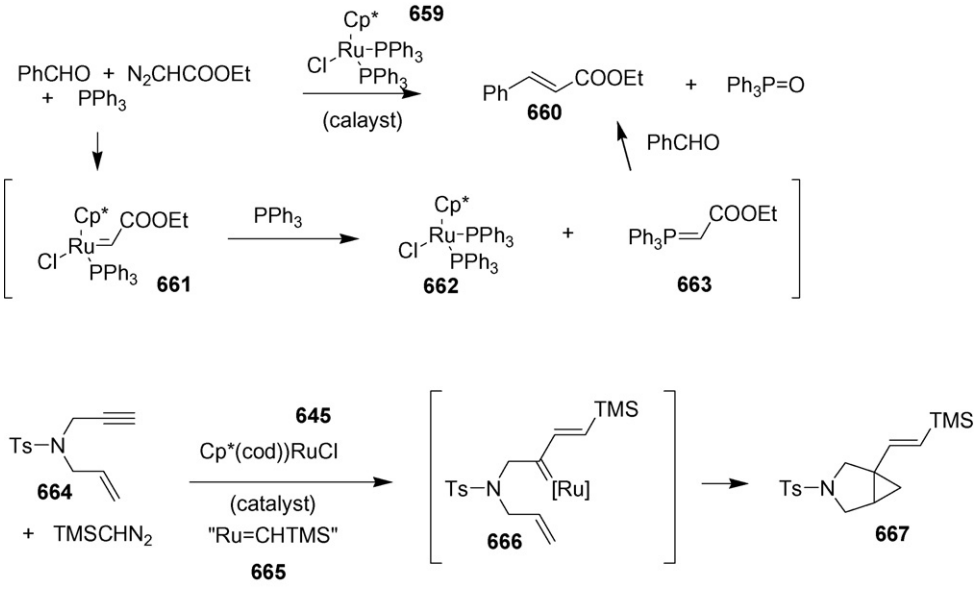
The formation and reactivity of chelating aminocarbene–ruthenium complexes (*e.g.* **700**, Scheme 58) was reported [954]. Reaction of the aminocarbene complex with dichloromethane followed by an amide base led to an equilibrium mixture of fluxional compounds, η^1-indenylcarbene complex **701** and phosphine-chelated aminocarbene complex **702**. Subsequent reaction of the **701/702** mixture with triphenylsilane led to the carbene silylruthenium complex **703**. Reaction with DMAP led to the coordinatively saturated carbene complex **704**.

Several papers report on the synthesis of aminocarbene complexes through C– H activation processes (see Scheme 59). Reaction of osmium trihydride complex **705** with HBF_4 led to the chelating carbene complex **706** through net formation of H_2 and α-hydride elimination [955]. Chelated osmium aminocarbene complexes (*e.g.* **709**) were also generated through reaction of bis(aminophosphine)-amine **707** with osmium complex **708** [956]. Further reaction with H_2 led to dihydrogen coordinated carbene complexes (*e.g.* **710**). Ruthenium– or osmium–carbene complexes (*e.g.* **714**) were synthesized through reaction of phenanthridine derivatives (*e.g.* **712**) with metal(II) dihydride complexes (**713**) in a net C–H activation process [957]. This process has been denoted as capturing the minor carbene tautomeric form of a pyridine ring. Initially the simple carbene(dihydrogen) complex (*e.g.* **714**) was obtained. Further heating in triethylamine led to the chelating phenanthridine complex (**715**). Similar formation of related carbene complexes through thermolysis of 2-vinylpyridine complexes was also reported [958].

Additional studies of Group 8 metal–carbene complexes in this category are depicted in Scheme 60. The iron-isocyanide complex **716** was reported to be the most extensively backbonded isocyanide complex ever recorded, and thus features extensive carbon–metal double bond character (*e.g.* resonance form **717**) [959]. The formation of boron NHC–iron complexes (*e.g.* **720**) was reported through reaction of borylene complex **718** with DCC (**719**) [960]. Carbon



Scheme 53.

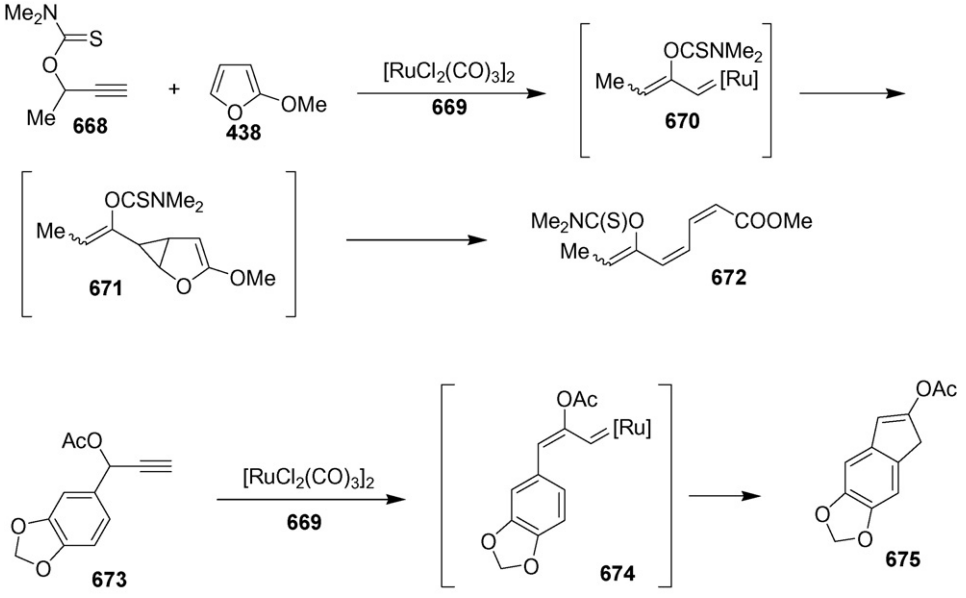


Scheme 54.

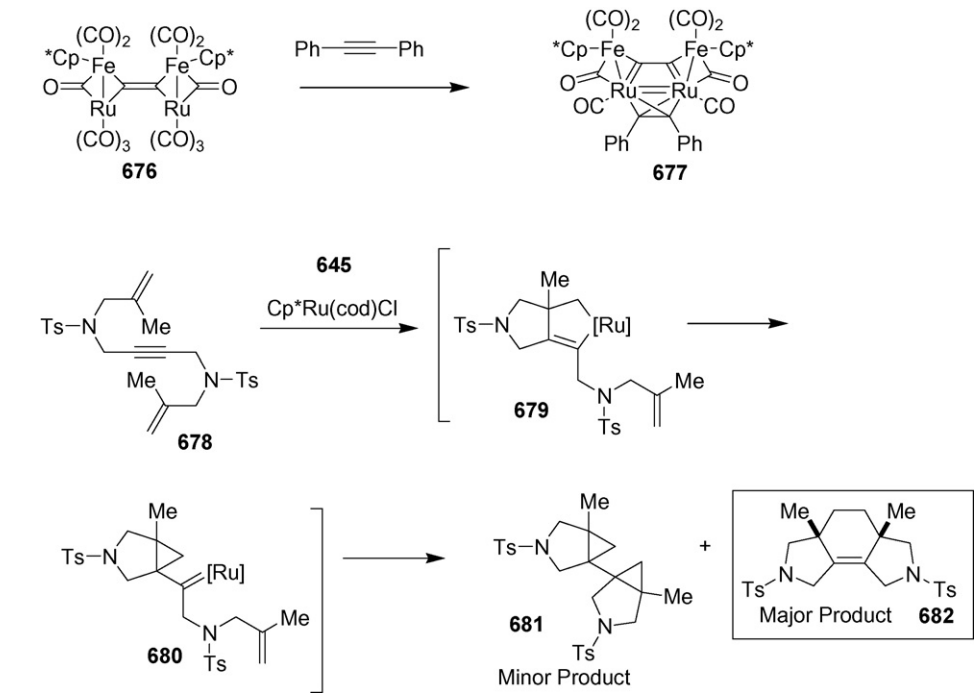
metal double bond character in the iron-carbon bond was minimal for this complex.

2.3.5.5. Group 8 metallacumulene complexes. Many examples of the formation of metal vinylidene complexes (**725**, Scheme 61) via coupling of coordinatively unsaturated Group 8 metal complexes with terminal or silylated alkynes were reported in 2007. Representative examples are depicted in Fig. 13. Common reaction pathways for these complexes include reaction with nucleophiles to form vinylmetal species (**728**), reaction with alcohols (or amines) to form Fischer carbene complexes (**729**) or water to form metal acyls (**727**), and deprotonation at the β-position to form alkynylmetal complexes (**726**). Other common synthetic routes to metal vinylidene include addition of electrophiles to metal acetylide complexes (*e.g.* the reverse of the reaction synthesizing **726**), and treatment of acylmetal complexes with dehydrating agents (*i.e.* the reverse of the reaction synthesizing **727**). Metal-higher cumulene complexes (**731, 736**) 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 (**735**). Common reaction pathways for these complexes include reaction with nucleophiles at the γ-position, resulting in alkynylmetal complexes (**733**), or attack at the γ-position, resulting in allenylmetal complexes (**734**). Reaction with alcohols or amines can lead to α,β-unsaturated Fischer carbene complexes (**732**). Representative examples of this class of compounds are depicted in Fig. 13.

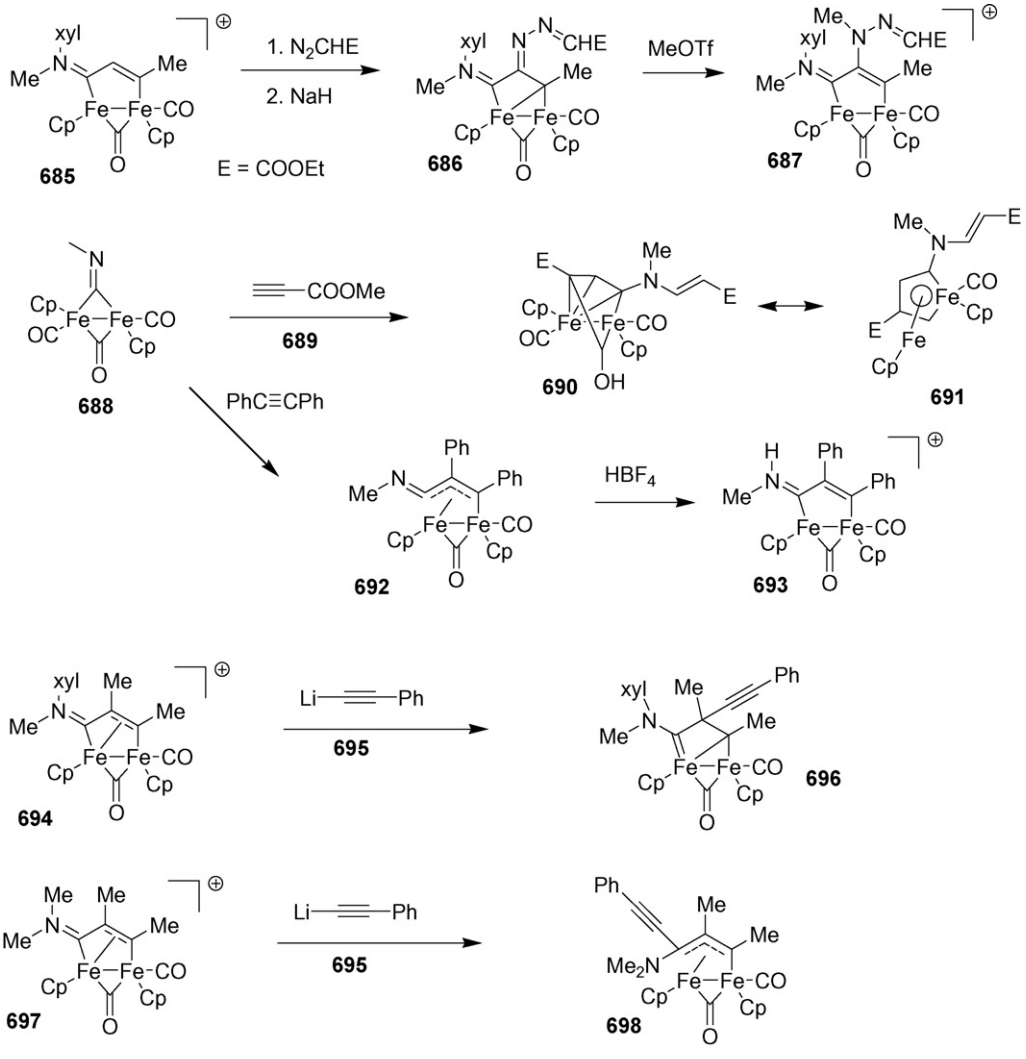
Specific reports which highlight the reaction pathways of Scheme 61 are depicted in Fig. 13 and include the following examples of vinylidene complexes: (1) formation and self-assembly of pyrimidine-ring-containing cationic vinylidene complexes (*e.g.* **737**) and their respective Fischer carbene complexes (*e.g.* **738**) [961]; (2) formation and electrochemistry of cationic iron vinyli-



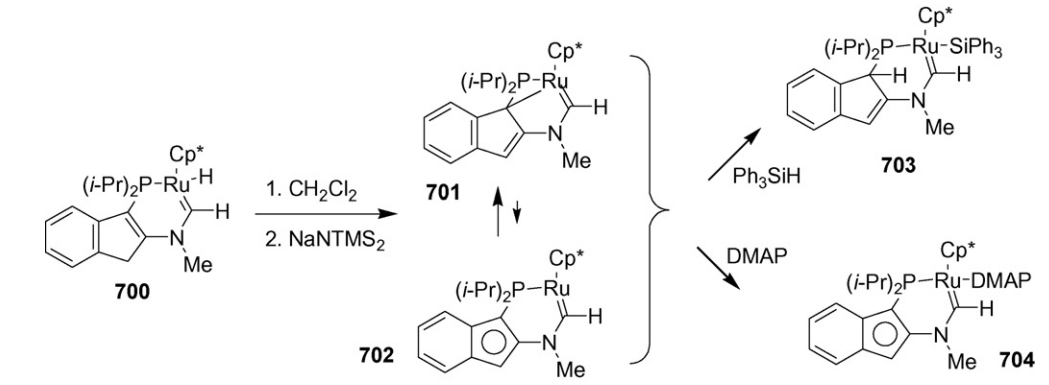
Scheme 55.



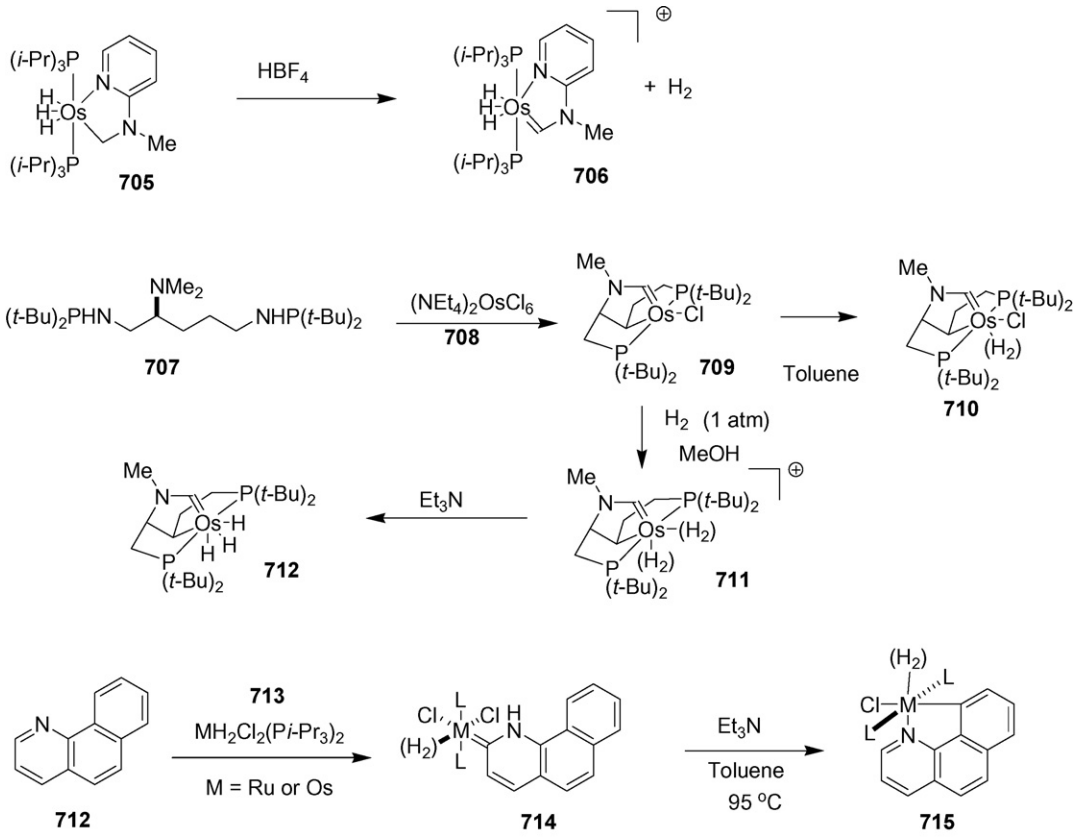
Scheme 56.



Scheme 57.

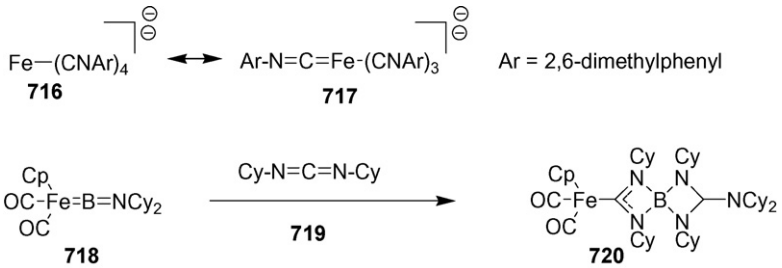


Scheme 58.

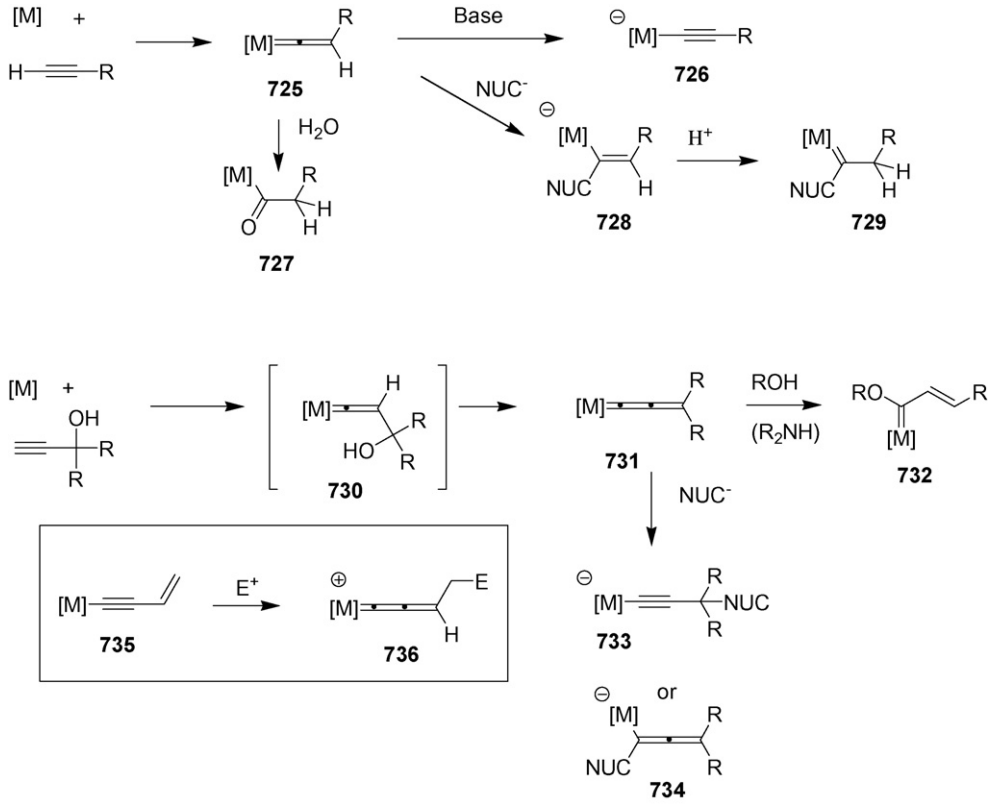


Scheme 59.

dene complexes and subsequent conversion to iron acetylides [962]; (3) formation of alkynylruthenium complexes [963,964]; (4) formation of fluxional osmium(II) vinylidene complexes (**739** and **740**) featuring chelating phosphinoether complexes [965]; (5) formation and UV/NLO studies of highly conjugated tetrakis(vinylidene-ruthenium) complexes (*e.g.* **741**) [966]; (6) formation of tetrakis(alkynyliron) complexes via the analogous vinylidene complexes [967]; (7) formation of fullerene-ligated



Scheme 60.



Scheme 61.

ruthenium allenylidene complexes (*e.g.* **742**) and subsequent addition of nucleophiles to the γ-carbon [968]; (8) formation of cationic ruthenium vinylidene, alkynyl, and allenylidene complexes featuring a chelating ferrocenylphosphine ligand (*e.g.* **744**) from the corresponding neutral ruthenium halide **743** [969]; (9) monokis- and bis(ruthenium–vinylidene) complexes conjugated to a ferrocene ring (*e.g.* **745**) [970,971]; and (10) formation of Group 8 metal-vinylidene complexes and subsequent conversion to the 1,2-dimetal-substituted alkyne derivatives [972]. Several processes likely involve metal vinylidene intermediates synthesized by these pathways, including: (1) acid-catalyzed hydration of bis(alkynyl)ruthenium complexes (*e.g.* **746**) to afford bis(acetyl)ruthenium complexes (*e.g.* **748**) via vinylidene intermediates (*e.g.* **747**) [973]; (2) ruthenium-catalyzed reaction of propargyl alcohols (*e.g.* **749**, Scheme 62) and isocyanates to afford α,β-unsaturated imines via formation of a vinylidene complex (**753**) and intramolecular nucleophilic addition of an amide nitrogen to the carbene carbon of the vinylidene complex [974]; (3) involvement of ruthenium vinylidenes in the cyclotrimerization of *t*-butylacetylene to form substituted Cp-ruthenium complexes [975]; (4) alkyne dimerization using a η^5-pentadienylruthenium complex [976]; and (5) alkyne dimerization using $[(p\text{-cymene})RuCl_2]_2$ [977]. Theoretical studies of the bonding in iron vinylidene complexes and comparisons with boron analogs were reported [978].

Several papers reported on the formation and reactivity of vinylidene complexes than contain pendant carbanion-stabilizing groups (see Scheme 63). Formation of cationic iron vinylidenes featuring a cyanomethyl group (*e.g.* **755**) and subsequent based induced cyclization to cyclopropenyliron complexes (*e.g.* **756**) was reported [979]. Formation of propargylvinylidene complexes (*e.g.* **759**) followed by their subsequent conversion to bis(vinylidene) complexes (*e.g.* **760**) was reported [980]. Several transformations for the bis(vinylidene) complexes were reported. Bis(vinylidene) complex **760** was transformed to a cyanomethylbis(vinylidene) complex (*e.g.* **761**) (or ester analogs, *e.g.* **762**) via deprotonation followed by alkylation of the intermediate acetylide complex. Subsequent treatment of the cyanomethyl complexes (**761**) with base provided either the cyclopentenylvinylidene complex (**763**) or the cyclopropenylvinylidene complex (**764**), depending upon the identity of the metal-ligand combinations. The ester analogs (**762**) afforded either the analogous cyclopentenylvinylidene complexes (**766**) or alkoxyfuryl-vinylidene complexes (**765**), depending upon the identity of the metal–ligand combinations.

The reaction of osmium complex **767** (Scheme 64) with excess phenylacetylene led to allenylcarbene complex **768** [981]. Formation of the allenylcarbene complex **768** involves carbene–alkene insertion from the initially formed vinylidene complex **769**. Reaction of complex **768** with phenylacetylide anion leads to the metallacycle derivative **770**. Conversion of the allenylcarbene complex to the metallacycle involves deprotonation to afford propatrienylosmium complex **771**, followed by 1,3-shift of osmium to afford the enynylosmium complex **772**, followed by C–H oxidative addition. The same metallacycle **770** forms via addition of acetylide anion to the vinylidene complex. The reaction of allenylcarbene–osmium complex **774** with acetylenes was also reported [982]. Reaction with alkynes led to isomeric η^6-fulvene complexes (*e.g.* **775** and **776**). A mechanism involving conversion to the metallacyclobutene (**777**), followed by alkyne insertion to afford either **778** or **779** and reductive elimination was proposed. Reaction with styrene led to a dihydrofulvene (**780**) and an inorganic osmium byproduct (**781**).

Additional examples of the formation and reactivity of Group 8 vinylidene complexes are depicted in Scheme 65. Formation of bimetallic ruthenium vinylidenes (*e.g.* **783**) from terminal alkynes was reported [983]. The analogous reaction with acetylene however led to the bridging carbide derivative **784**. A novel method to

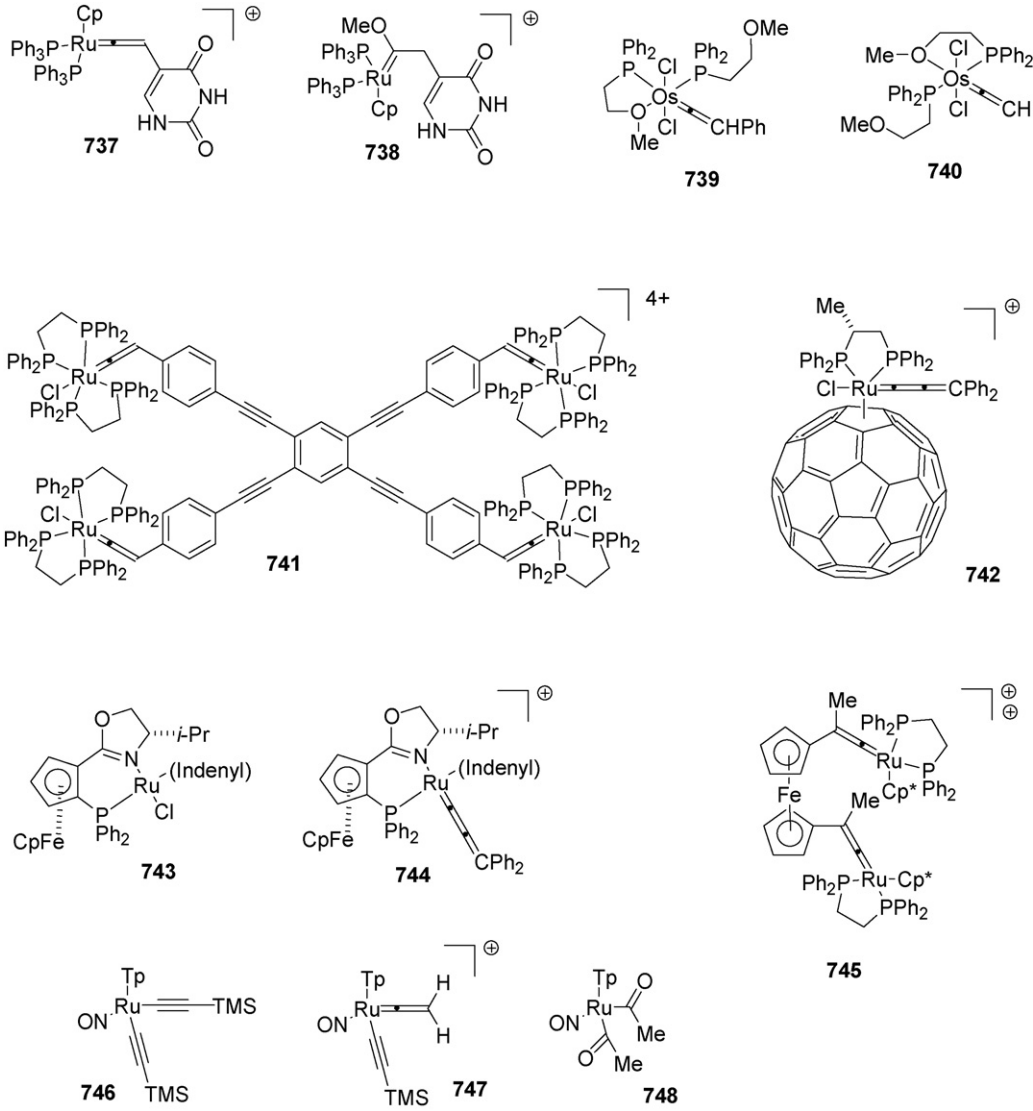
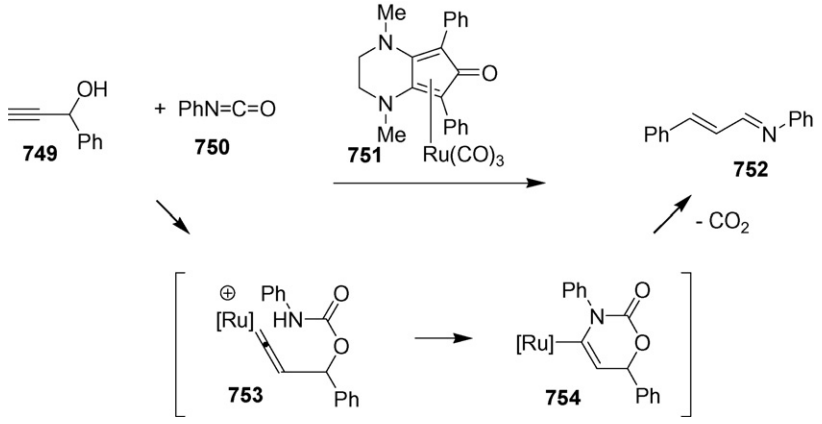


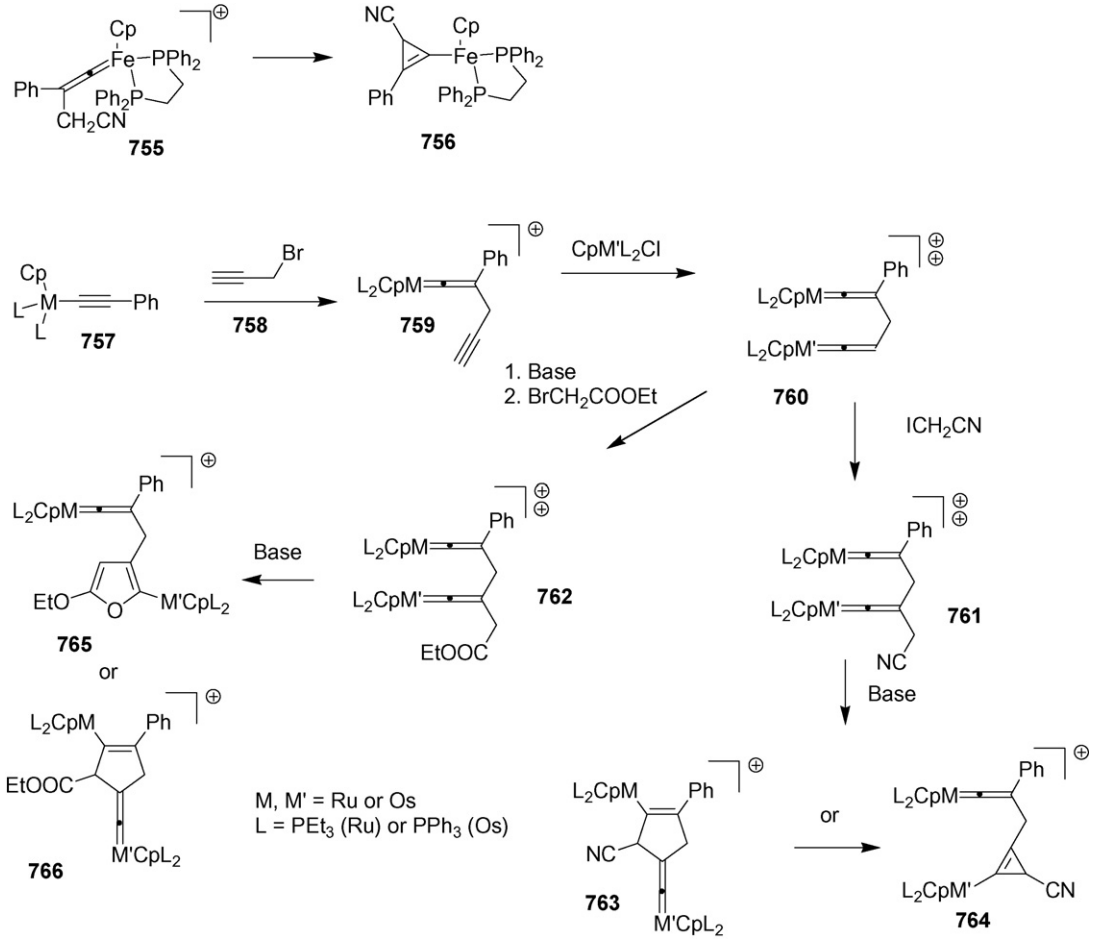
Fig. 13. Representative Group 8 metallacumulene complexes, precursors, and reaction products reported in 2007.

prepare ruthenium vinylidene complexes (*e.g.* **788**) from internal alkynes (*e.g.* **785**) was reported [984]. In this reaction, the alkyne ketone reacts with cationic ruthenium complex **786** to afford the oxygen-coordinated complex **787**, which rearranges to the phenyl-shifted vinylidene complex **788**. At lower temperature the O-bound ruthenium complex **787** could be characterized spectroscopically in solution.

Several cyclization reactions involving terminal alkynes invoke vinylidene intermediates; examples are depicted in Scheme 66. Reaction of 1,6-diynes (*e.g.* **790**) with carboxylic acids in the



Scheme 62.



Scheme 63.

presence of ruthenium catalyst **625** led to six-membered ring derivatives (*e.g.* **792**) [985]. They key step in this reaction is nucleophilic addition of carboxylate anion to an intermediate alkyne(vinylidene)–ruthenium complex **791**. Reaction of 2-alkynyldiphenylmethane derivatives (*e.g.* **793**) with ruthenium complex **794** led to indene derivatives (*e.g.* **797**) [986]. A key step in this transformation is the net 1,5H-transfer in the vinylidene complex (**795**) to afford the indene after cyclization of the vinylruthenium complex (**796**). The mechanism was supported through deuterium labeling studies. Treatment of alkynylruthenium complexes (*e.g.* **798**) with HBF_4 led to observable cationic vinylidene complexes (*e.g.* **799, 802**), which form cyclic compounds (*e.g.* **800, 801**) upon treatment with alcohols [987]. A very unusual C-13 label scrambling pattern was noted in the formation of the vinylidene complex in some of the cases, and was attributed to reversible formation of a cyclobutanylidene intermediate (**803**). The extent of label scrambling was highly dependent on the placement of the *gem* diphenyl groups relative to the alkyne and alkene groups.

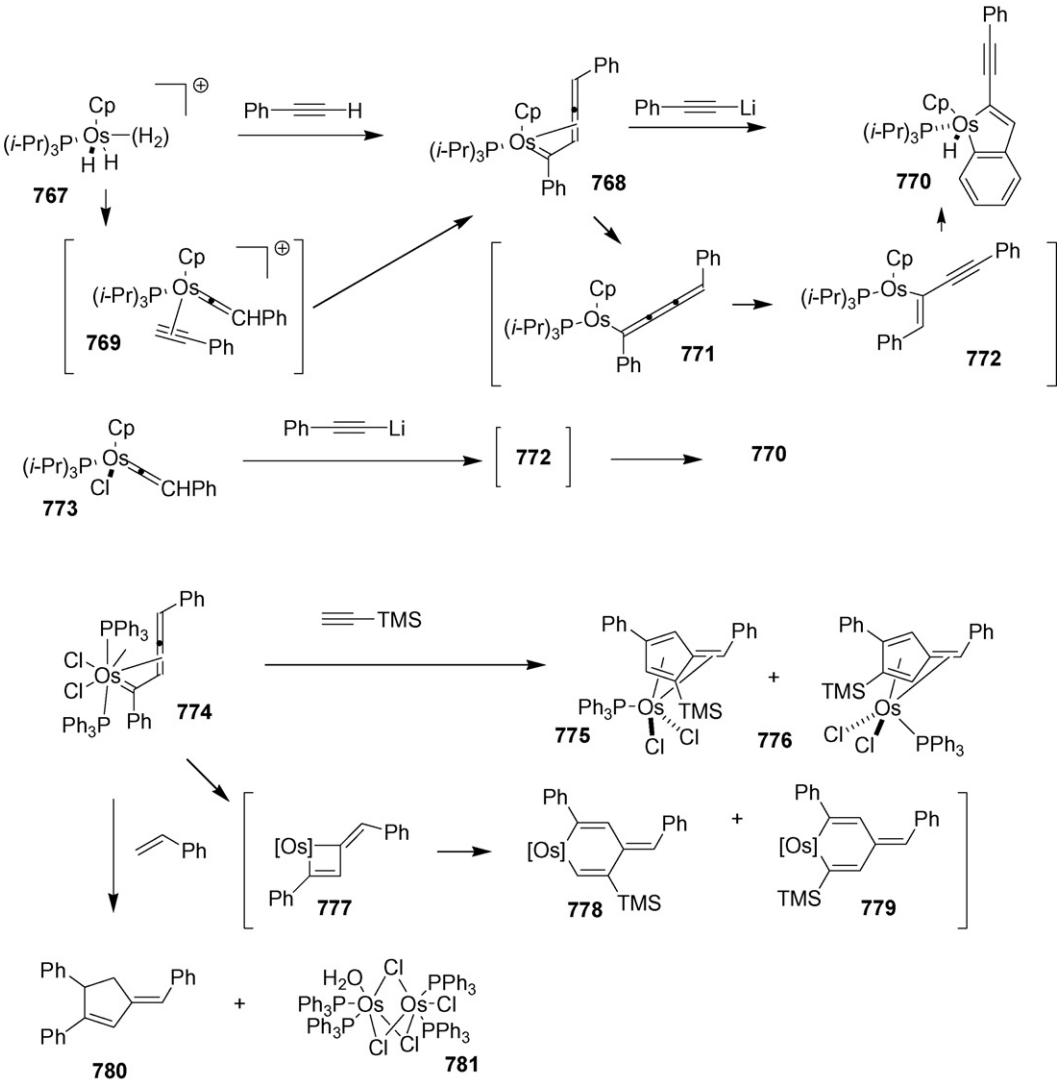
The formation of a complex ruthenium-bridged bis-(allenylidene–ruthenium) complex (**809**, Scheme 67) through coupling of bis(butadiynyl)ruthenium complex **805** with two eq of cationic ruthenium allenylidene complex **806** was reported [988]. This transformation likely involves proton transfer to afford allenylidene complex **807** and enynylruthenium complex **808**. Electrophilic addition to of the butatrienylidene-ruthenium unit of **807** to the alkene carbon of enynylruthenium complex **808** then occurs. Electrochemical studies were reported for the triruthenium complexes.

Ruthenium allenylidene complexes (*e.g.* **812**, Scheme 68) were also proposed as intermediates in nucleophile-induced ring opening reactions of cyclopropyl(alkynyl)methanol derivatives (*e.g.* **811**) [989]. Ruthenium allenylidene complexes were suggested as intermediated in the various propargyl substitution reactions using propargylic alcohols and various thiol-bridged diruthenium complexes. Examples include: (1) propargylation of indole derivatives using propargyl alcohols [990], (2) N-propargylation of carbazole derivatives [991], and (3) propargylation of furans (*e.g.* asymmetric conversion of propargyl alcohol **814** and methylfuran to furan-alkyne **818** via allenylidene complex **817** in 94% ee) [992,993]. Ruthenium allenylidene complexes (*e.g.* **820**) were also proposed as intermediates in the formation of ruthenacyclobutanones (*e.g.* **823**) via protonation of bis(alkynyl)ruthenium complexes (*e.g.* **815**) [994]. The proposed mechanism involves formation of the allenylidene complex (**820**) upon protonation, followed by alkynyl group migration to afford the allenylruthenium complex (**821**) followed by rearrangement of the propargyl alcohol to the enone (**822**), followed by complexation of the pendant alkene group to ruthenium.

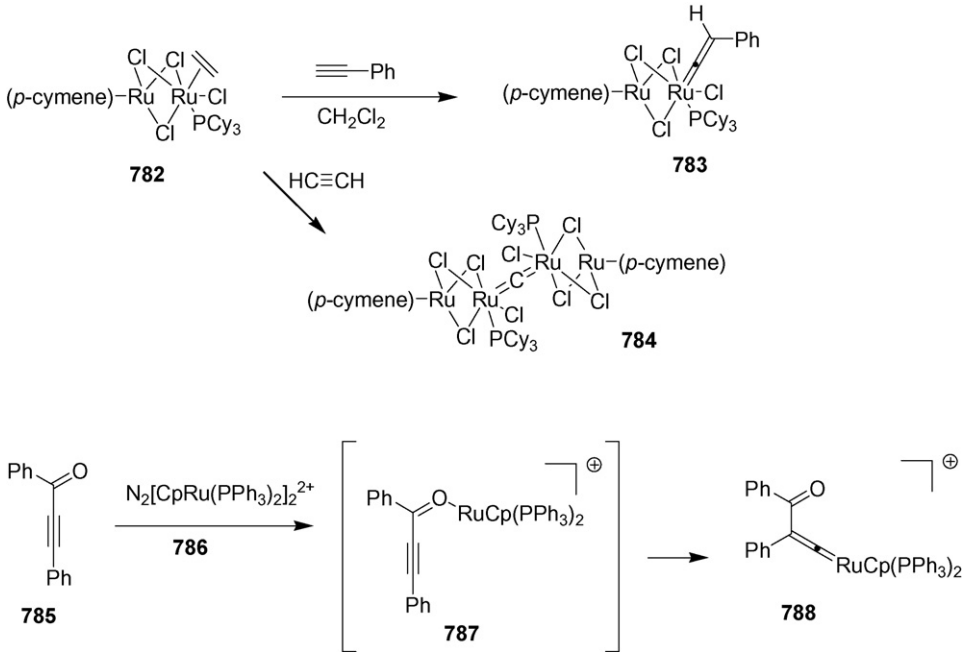
Electrochemical oxidation of butadiyne-bridged bimetallics led to two-electron oxidation products consistent with cumulenylidene resonance forms (*e.g.* **825**, Scheme 69) [995].

2.3.6. Group 9 metal–carbene Complexes

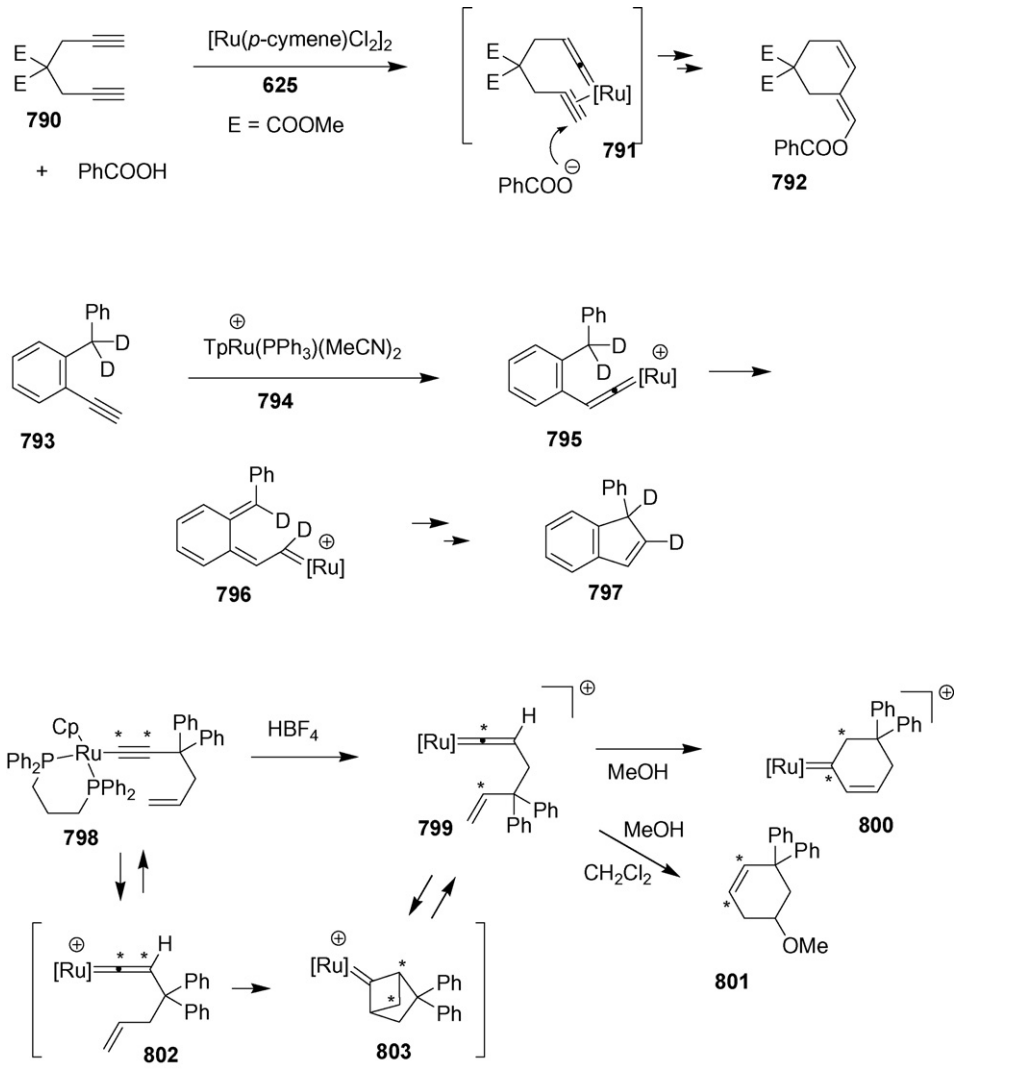
2.3.6.1. Simple carbene complexes. An iridium carbene complex (**831**, Scheme 70) was generated through reaction of pyridine with iridium complex **830**, which features a pentadentate ligand [996]. When pyridine-d_5 was employed in the preparation of **831**, the CH_2 arms of the pentadentate ligand were partially deuterium labeled. Related rhodium carbene complexes (*e.g.* **834**) were prepared through reaction of carbene precursors (*e.g.* **832** with $RhCl(cod)_2$ (**833**) [997]. A bis(trifluoromethyl) iridium carbene



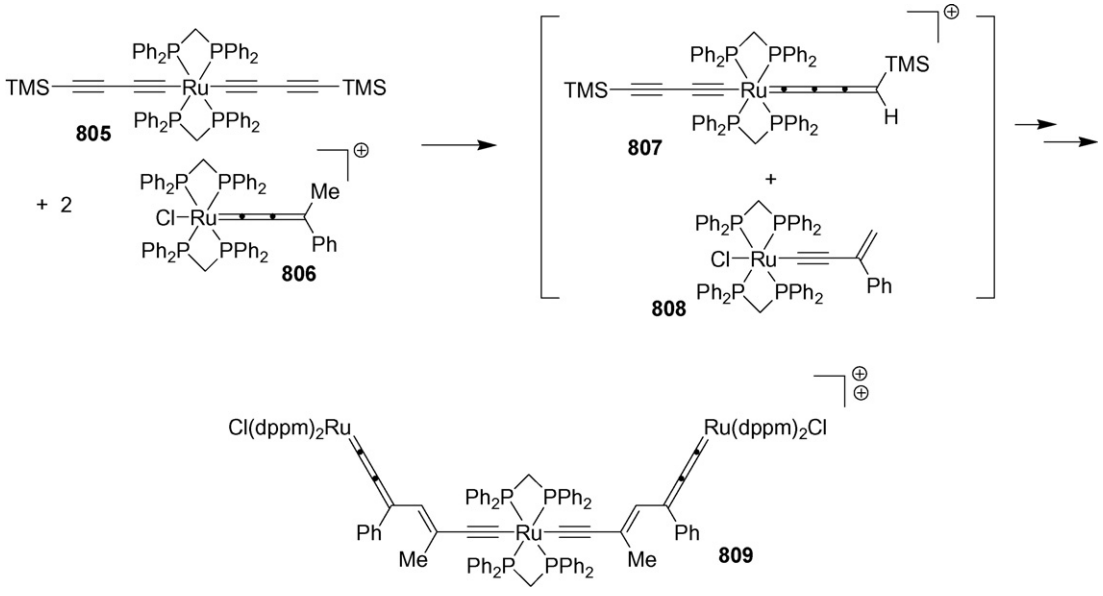
Scheme 64.



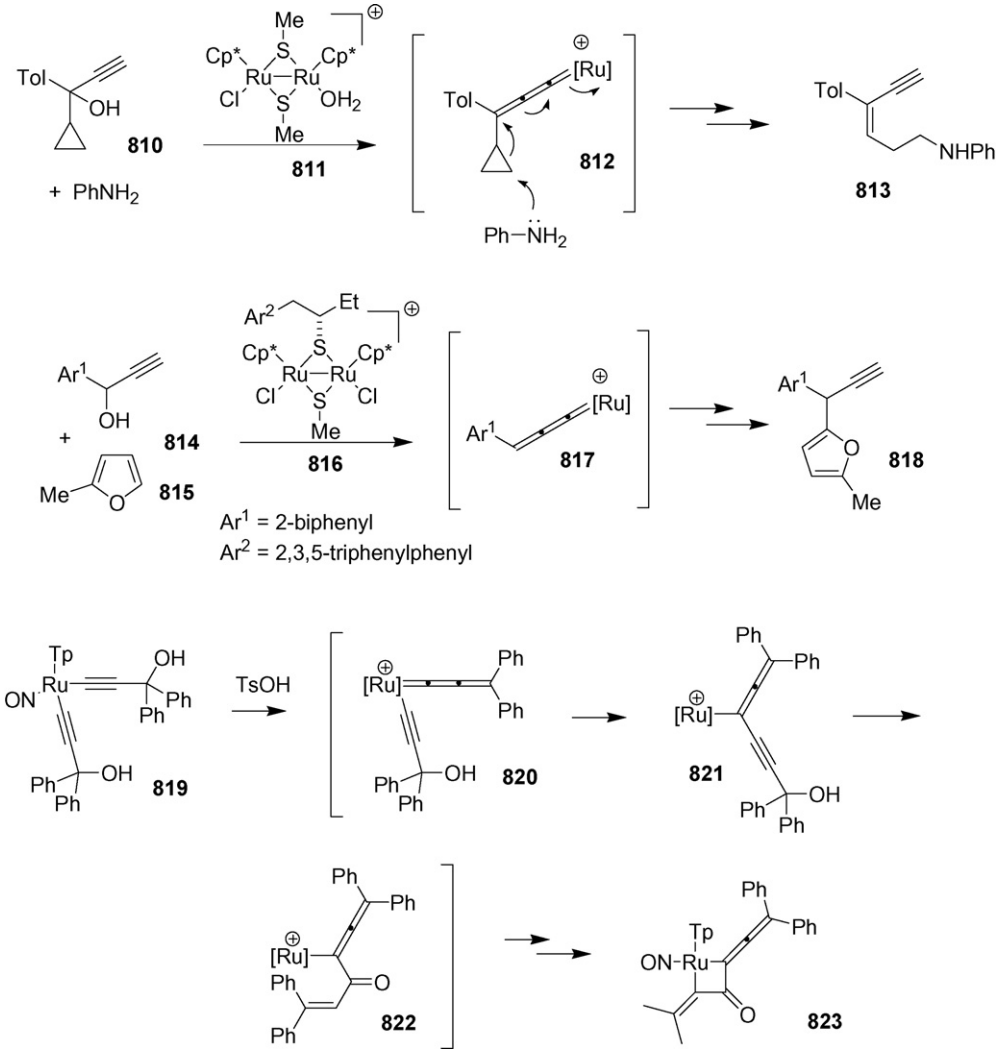
Scheme 65.



Scheme 66.



Scheme 67.

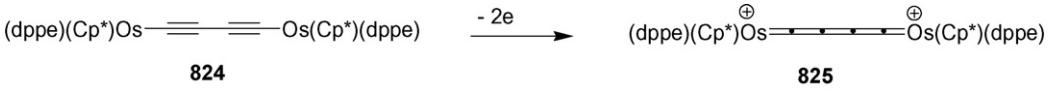


Scheme 68.

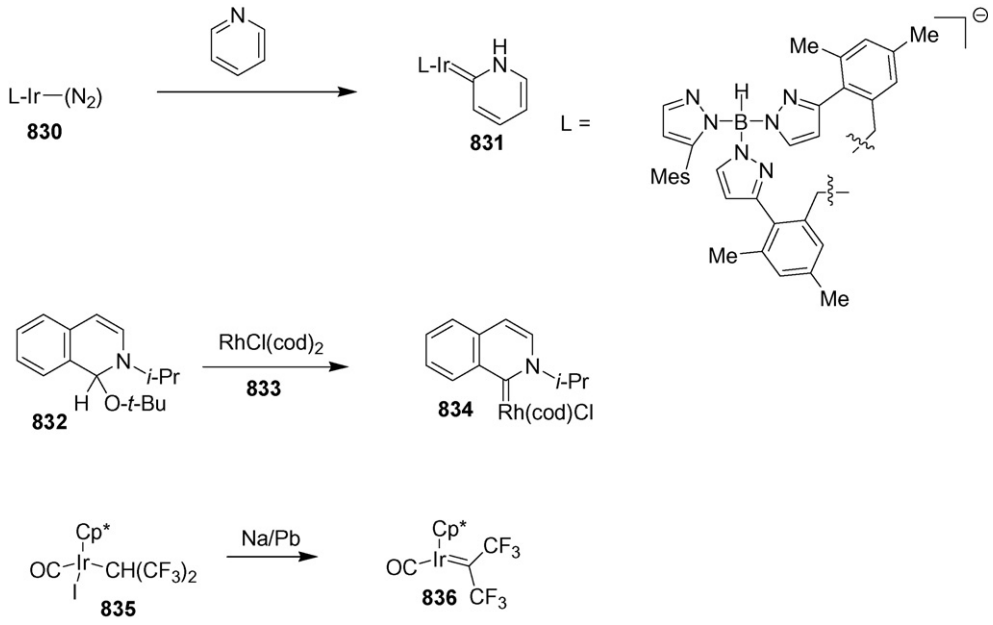
complex (**836**) was generated though reduction of the iridium(III) complex **835** [998]. Related carbene complexes derived from pyridine were suggested as intermediates in rhodium-catalyzed C–H activation reactions of pyridines [999].

The synthesis and reactivity of iridium alkoxycarbene complexes (*e.g.* **838**, Scheme 71) was reported [1000]. The carbene complex was initially generated through a C–H activation process. Refluxing of complex **838** in cyclohexane led to the alkoxymethylphenyliridium complex **839** in a process involving hydrogen migration to afford alkoxymethyl complex **840** followed by β-hydride elimination to form the iridium-benzyne complex **841**, which undergoes alkyne insertion to afford the observed product **839**. If acetonitrile was present the monodentate complex **842** was formed. Heating in benzene led to the alkoxymethylarene (**844**) and the carbene–bis(hydride) complex **843**, which upon further heating led the CO complex (**845**). Complex **845** is the apparent product of a 1,3-alkyl shift process from complex **843**.

The synthesis and reactivity of various metallabenzenes are depicted in Scheme 72. Thermolysis of iridabenzvalenes (*e.g.* **848**) led to either iridabenzenes (*e.g.* **849, 851**) or substituted Cp complexes (**850**) [1001]. Stirring of iridabenzvalene **848** (Y = Ph) at room temperature led to a mixture of **851** and Cp complex **850**. No stable iridabenzene was observed when both of the groups were TMS groups; only direct formation of the Cp complex **850** from the iridabenzvalene was observed. The processes were also evaluated computationally. Oxidation of metallacycloheptatrienes (*e.g.* **852**) results in iridabenzenes (*e.g.* **855**) [1002]. Initially the oxidation affords a metallacyclohexadiene complex (**854**), which upon further Bayer-Villiger-type oxidation leads to the iridabenzene. Formation of the iridacyclohexadiene (**854**) involves formation of an epoxide (**853**) at the distal alkene followed by rearrangement with alkyl migration. Reaction of iridacyclopentadienes (*e.g.* **856**) with alkenes results in the synthesis of iridabenzenes (*e.g.* **857**) accompanied by chelating allyl (*e.g.* **858**) and alkene (*e.g.* **859**) complexes [1003]. The proposed mechanism for iridabenzene formation



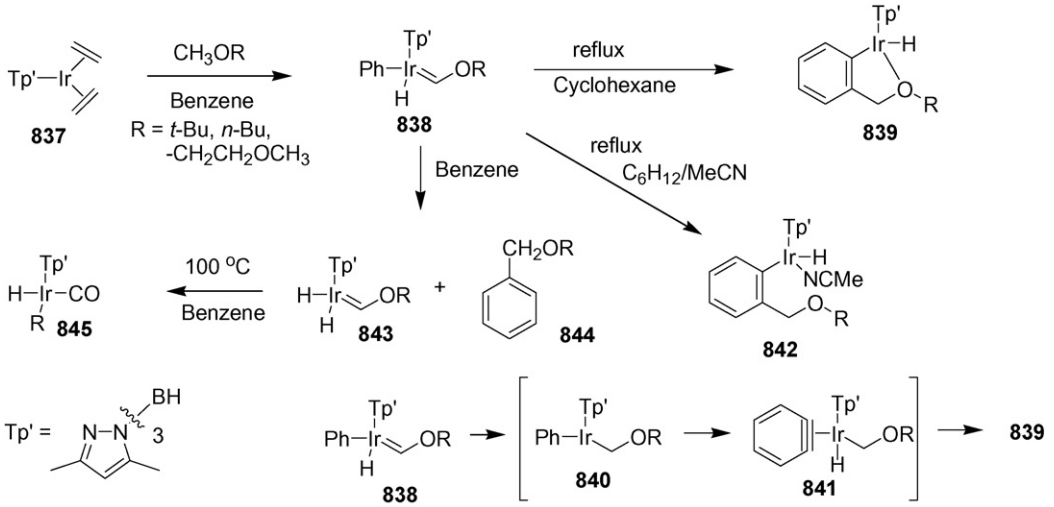
Scheme 69.



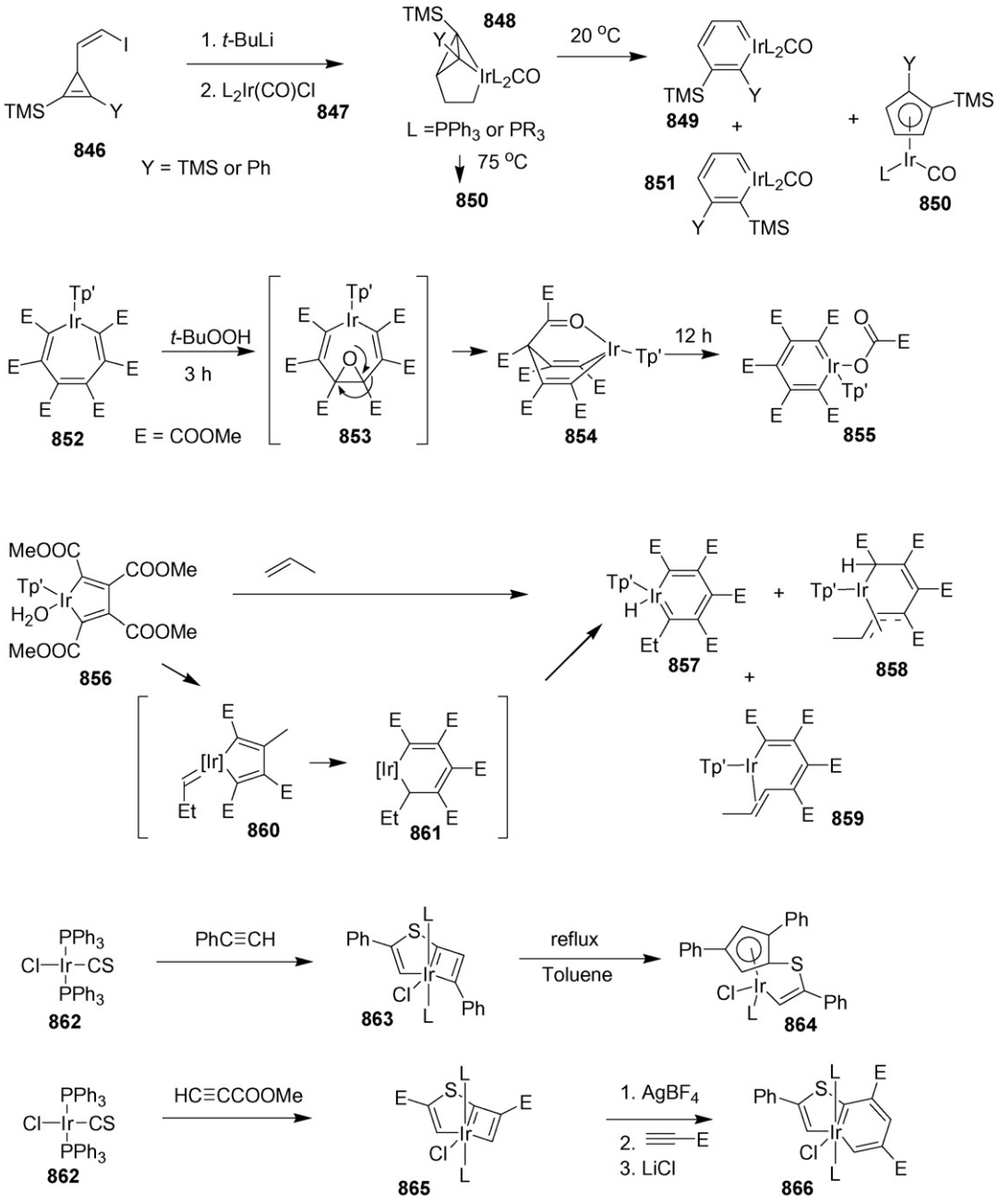
Scheme 70.

involves formation of the carbene complex (**860**) followed by alkyl shift to afford iridacyclohexadiene complex **861**, which undergoes α-hydride elimination. Treatment with acetonitrile leads to formation of the acetonitrile-coordinated iridacyclohexadiene complex. Iridathiophene (*e.g.* **863, 865**) and iridabenzothiophene (*e.g.* **866**) derivatives were also reported [1004]. Reaction of iridium thiocarbonyl complex **862** with terminal alkynes led to iridathiophene derivatives (*e.g.* **863, 865**). The phenyl derivative afforded the bridged Cp-complex **864** upon extended thermolysis. The ester derivative (**865**) afforded the iridabenzothiophene derivative **866** upon treatment with silver and addition of methyl propiolate. The structural properties of iridabenzenes and osmabenzenes was studied by DFT calculations [1005]. Nonplanaraity in the metallaaromatic ring systems was attributed to a fourth π-orbital involving the metal center and the adjacent ring carbons coupled with a steric effect due to bulky ligands and the metal.

Other studies of Group 9 metal–carbene complexes (excluding metallacumulenes) are depicted in Scheme 73 including: (1) a study of nitrogen kinetic isotope effects in rhodium-catalyzed cyclopropanation, which suggested that the loss of nitrogen to form a carbene complex is the slow step of the reaction [1006]; (2) cobalt porphyrin catalyzed cyclopropanation [1007]; (3) involvement of rhodium carbene complexes (*e.g.* **869**) in formation of dihydroazulenes (*e.g.* **871**) from alkynes and benzyldiazo compounds (*e.g.* **868**), including a significant discussion of the affect of substituents on carbene complex intermediate reactivity [1008]; (4) involvement of an iridium carbene complex (**873**) in the formation of an iridapyrrole (**874**) from the reaction of ruthenium–DMAD complex **872** with acetonitrile [1009]; (5) involvement of iridium carbene complexes (*e.g.* **876**) in the conversion of chelating phosphabenzyl ligands to phosphastilbene complexes (*e.g.* **877**) [1010]; (6) possible involvement of rhodium carbenes in carbonyl olefinations employing a diazoester, triphenylphosphine, and a rhodium catalyst [1011,1012]; (7) possible involvement of rhodium carbenoids (*e.g.* **879**) in the ring expansion of cyclopropenylpyridine derivatives (*e.g.* **878**) [1013]; (8) possible involvement of rhodium carbene



Scheme 71.



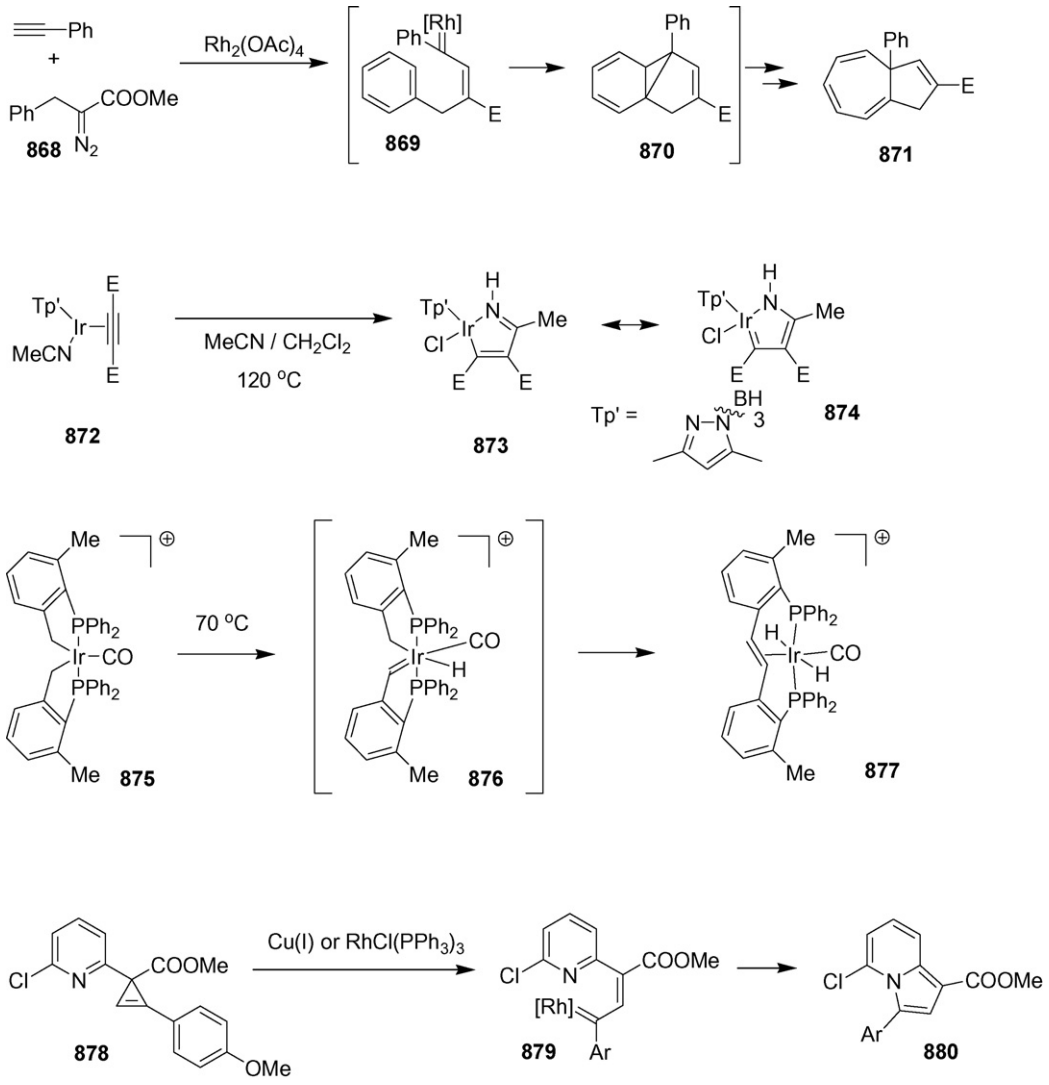
Scheme 72.

complexes in the dehydrogenation of hydroxymethylrhodium complexes to form acylrhodium complexes [1014]; (9) determination of the extent of π-backbonding in various rhodium-NHC complexes [1015] and acyclic analogs [1016]; and (10) polymerization of ethyl diazoacetate using rhodium complexes [1017]. Several papers reported computational studies of rhodium-catalyzed reactions of diazo compounds. Specific topics covered include: (1) rhodium-catalyzed cyclopropanation of ethylene [1018,1019]; (2) rhodium-catalyzed polymerization of ethyl diazoacetate to give polymaleate polymers [1020]; (3) rhodium-catalyzed insertion of carbenes into O–H bonds [1021] and S–H bonds [1022]; and (4) reaction of rhodium carbene complexes with ammonia [1023].

2.3.6.2. Metallacumulene complexes. Similar synthetic procedures and reactivity patterns were generally observed for Group 9 and Group 8 metallacumulene complexes (Scheme 61).

The interconversion of η^2-alkyne–rhodium and rhodium–vinylidene complexes was studied [1024]. Reaction of alkynes with complex **883** (Scheme 74) led to a spectroscopically observable π-alkyne complex (**884**) which converted to the vinylidene complex (**886**) at room temperature. The hypothetical hydrido-alkynyl complex **885** was not observed. Numerous kinetic studies and crossover experiments using labeled alkyne substrates were conducted, and the conversion of the alkyne complex to the vinylidene complex was determined to be a unimolecular process. The processes were also evaluated computationally.

Group 9 metal–vinylidene complexes were proposed as intermediates in several reaction processes involving terminal alkynes. Representative examples are depicted in Scheme 75. Rhodium–vinylidene complexes (*e.g.* **889**) were proposed as intermediates in the rhodium-catalyzed conversion of alkynylaniline derivatives (*e.g.* **888**) to indoles (*e.g.* **890**) [1025]. Formation of ben-

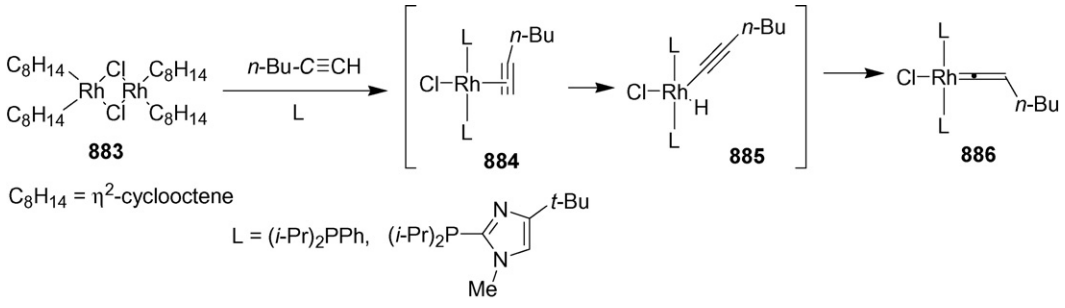


Scheme 73.

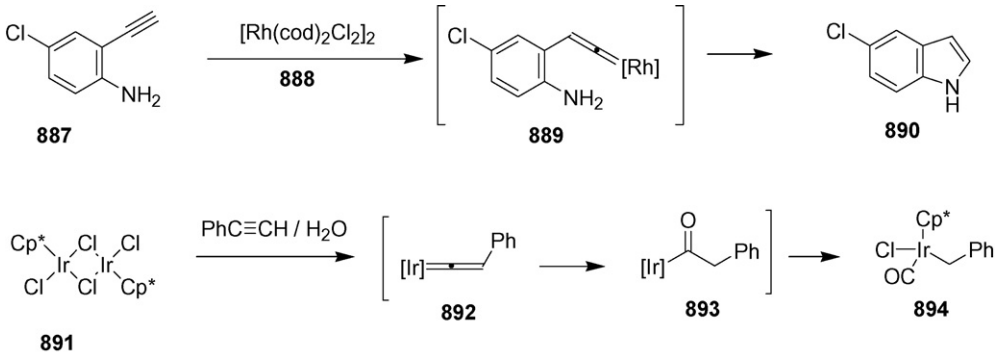
zyliridium complex **894** from complex **891** and phenylacetylene and water was proposed to occur through vinylidene formation (**892**), followed by addition of water to form the iridium acyl (**893**), followed by decarbonylation [1026]. A unimolecular mechanism for vinylidene formation was favored over a bimolecular mechanism. Rhodium-vinylidene complexes were suggested as likely intermediates in rhodium-catalyzed anti-Markovnikov addition of amines to terminal alkynes [1027]. Iridium vinylidenes were considered as possible intermediates in iridium catalyzed hydroalkynylation however other pathways were determined to be more favorable [1028]. A computational study of the conversion of ruthenium acetylide hydrides to vinylidene complexes was reported [1029].

2.3.7. Group 10 metal–carbene Complexes

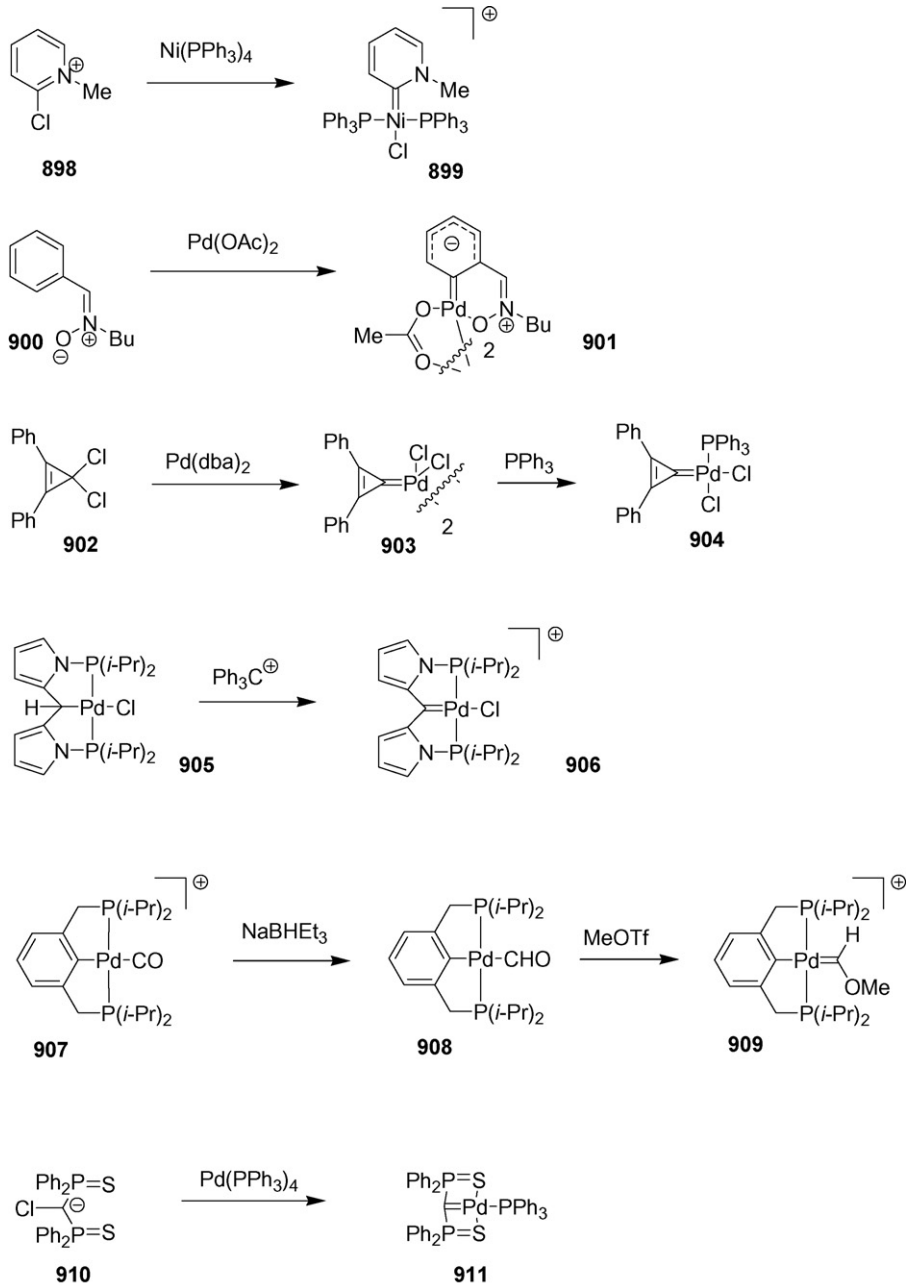
Several examples of the preparation of Group 10 metal–carbene complexes are depicted in Scheme 76. Nickel carbene complexes (*e.g.* **899**) were prepared through oxidative addition reactions of chloropyridinium salts (*e.g.* **898**) and subsequently employed in cross coupling reactions [1030]. Palladium carbene complexes (*e.g.* **901**) were prepared through C–H activation of aromatic nitrones



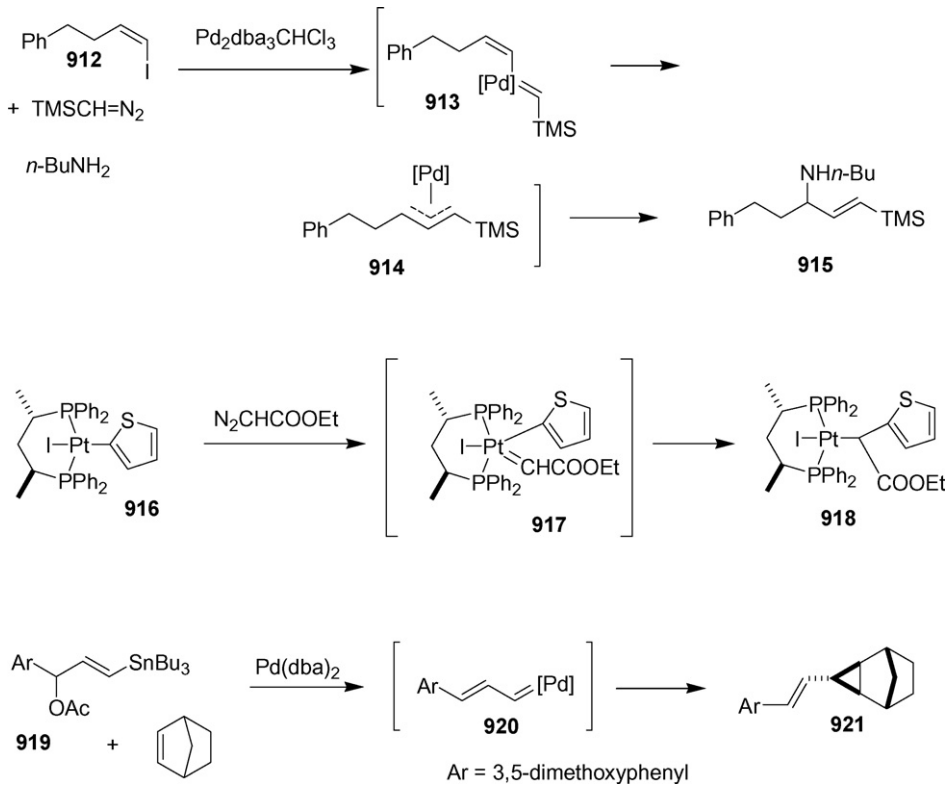
Scheme 74.



Scheme 75.



Scheme 76.

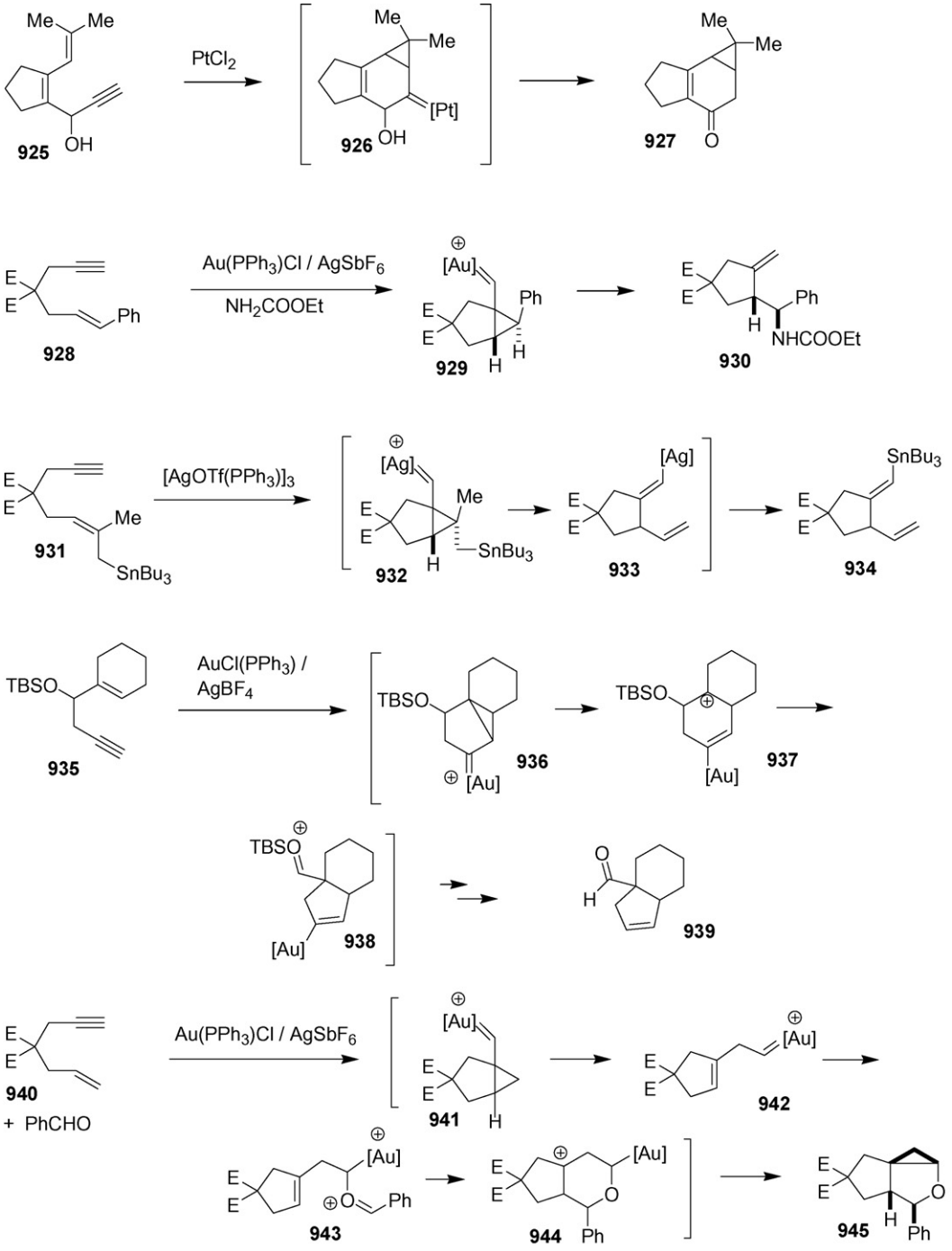


Scheme 77.

(*e.g.* **900**) and subsequently employed as catalysts for the Heck reaction [1031]. Cyclopropenylidene–palladium complexes (*e.g.* **903, 904**) were also prepared via an oxidative addition reaction used to catalyze C–N coupling reactions [1032]. Similarly prepared cyclopropenylidene–palladium complexes were employed as catalysts for the Suzuki coupling reaction [1033,1034]. A palladium carbene complex (**906**) was generated through hydrogen abstraction reactions of pincer-type bis(pyrrolyl)methylpalladium complex **905** [1035]. The palladium carbon bond showed only weak π-bonding behavior, which was attributed to low basicity of the metal–ligand system. A cationic pincer palladium carbene complex (**909**) was generated through methylation of palladium formyl complex **909** [1036]. A pincer carbene–palladium complex (**911**) was also prepared through reaction of bis(phosphine sulfide) **910** with palladium(0) [1037]. Related pincer carbene complexes featuring a bis(phosphino)methylene ligand were also reported [1038]. The bonding in palladium–carbene complexes was studied computationally [1039].

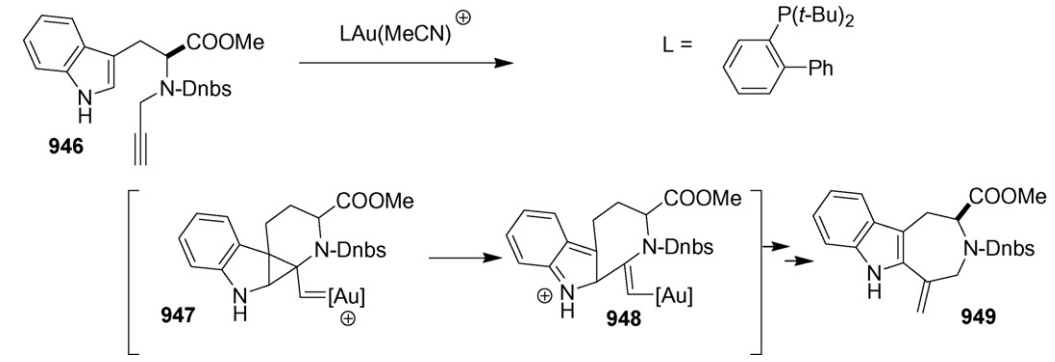
Several processes (other than cyclopropanation) involving diazo couplings were proposed to proceed through Group 10 metal–carbene complexes (see Scheme 77). The palladium-catalyzed synthesis of allylic amines (*e.g.* **915**) through three-component coupling of iodoalkenes (*e.g.* **912**), amines, and trimethylsilyldiazomethane was reported [1040]. A key step in this transformation is formation of carbene–alkenyl complex **913**, which transforms to a π-allyl complex (**914**). Addition of the amine to the allylpalladium complex leads to the final product **915**. Formation of palladium nanoparticles during cyclopropanations using diazomethane and a DFT study of the mechanism for palladium-catalyzed cyclopropanation was reported [1041]. Reaction of thiophenylplatinum complex **916** with ethyl diazoacetate leads to the net carbene insertion product **918** [1042]. The proposed mechanism involves formation of the carbene complex **917** followed by aryl group migration. Palladium carbene complexes were also generated from other precursors. Treatment of stannylated allylic acetates (*e.g.* **919**) with palladium(0) in the presence of norbornene led to cyclopropanation products (*e.g.* **921**) [1043]. The proposed mechanism for this process involves formation of vinylcarbene complex intermediate **920** followed by cyclopropanation. Various coupling reactions of 1,1-dihaloalkanes with nickel(0) complexes were proposed to proceed through nickel carbene or carbenoid complex intermediates [1044]. Platinum-catalyzed cyclopropanation was studied computationally [1045].

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 78. Platinum-catalyzed isomerization of diene-propargyl alcohols (*e.g.* **925**) led to cyclopropyl-fused cyclohexenone derivatives (*e.g.* **927**) [1046]. Formation and demetallation of the α-hydroxy platinum cyclopropylcarbene complex intermediate (**926**) were proposed as key steps in this transformation. Gold-catalyzed reaction of enynes (*e.g.* **928**) in the presence of ethyl carbamate resulted in diastereoselective preparation of cyclized amine derivatives (*e.g.* **930**) [1047]. In this reaction the cyclopropylcarbene complex intermediate (**929**) undergoes nucleophilic attack by the amine derivative with ring opening. Related trapping of cyclopropylcarbene complex intermediates through nucleophilic attack by various other nucleophiles was also reported. Examples of nucleophiles successfully employed include indoles [1048] and alcohols [1049–1051]. Reaction of alkyne-allylstannanes (*e.g.* **931**) with silver salts led to alkylidenecyclopentenes (*e.g.* **934**) in a process where an intermediate cyclopropylcarbene complex (**932**) undergoes loss of a tributyltin group [1052]. Cyclopropylcarbene complexes (*e.g.* **936**) are likely key intermediates in a related tandem cyclization-skeletal rearrangement of oxygenated enynes (*e.g.* **935**) to aldehyde derivatives (*e.g.* **939**) [1053]. After formation of the cyclopropylcarbene complex intermediate (**936**), ring opening and 1,2-alkyl shift occurs to afford the aldehyde product (**939**). Cyclopropylcarbene–gold complexes (*e.g.* **941**) were

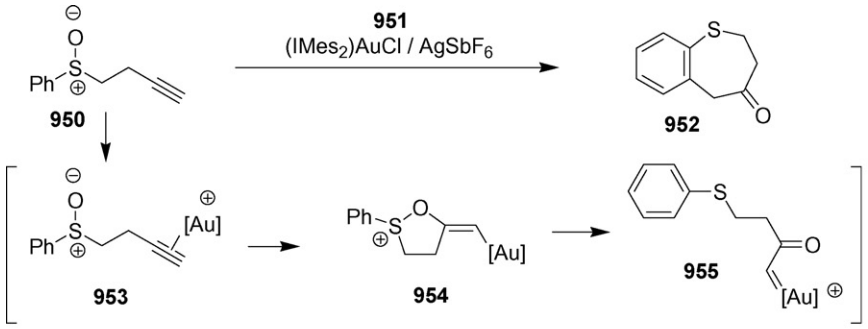


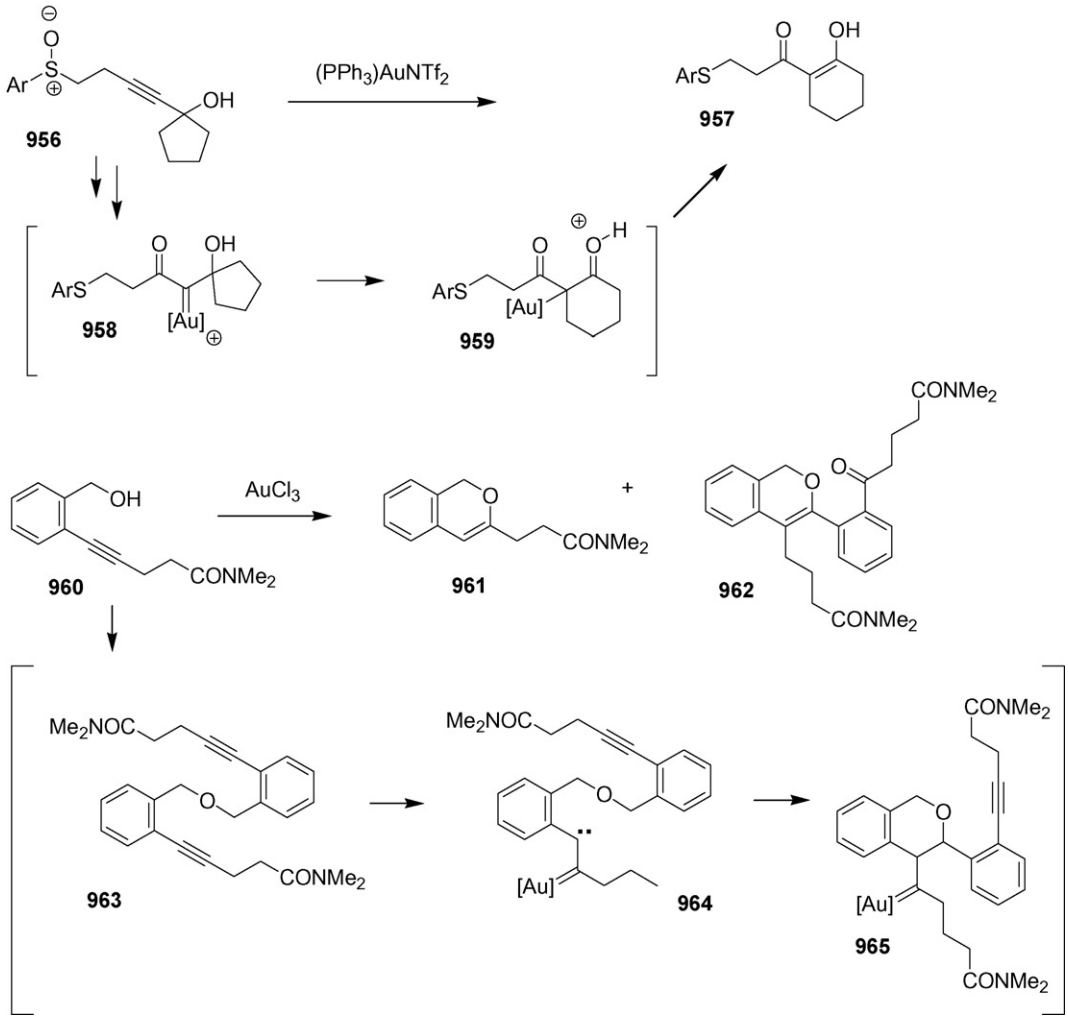
Scheme 78.

key intermediates in a novel net cyclization–cycloaddition reaction involving the coupling of enynes (*e.g.* **940**) with aldehydes to afford cyclopropane-containing cycloadducts (*e.g.* **945**) [1054]. In this case, the cyclopropylcarbene complex intermediate (**941**) undergoes ring opening to carbene complex **942**, which undergoes a nonconcerted cycloaddition process with the aldehyde to afford intermediate carbocation **944**. Coupling of the alkylgold unit with the carbocation leads to the observed product, cyclopropane derivative **945**. Additional cyclopropanation reactions were also observed through reaction of enynes with various late transition metal catalysts. Intramolecular cyclopropanation was observed upon treatment of dihydropyran-alkynes with platinum(II) chloride [1055], while a cyclopropane ring opening process was observed using gold catalysts A double cyclopropanation reaction was reported in the reaction of alkyne-cyclohexadienes with cationic gold complexes [1056]. Cyclopropylcarbene gold complexes were suggested as intermediates in the enyne cyclization of enynes containing an allylic ester functionality [1057]. Trapping of the gold cyclopropylcarbene intermediate through oxidation to the aldehyde via intramolecular oxygen atom transfer from a sulfoxide was also reported [1058]. Cyclopropylcarbene–gold complexes (*e.g.* **947**, Scheme 79) were proposed as intermediates in the gold-catalyzed cyclization of alkyne-containing indoles (*e.g.* **946**) to ring-fused indoles (*e.g.* **949**) [1059]. Cyclopropylcarbene complexes are likely intermediates in the conversion of alkyne–furans to phenol derivatives [1060] and other furan–alkyne couplings

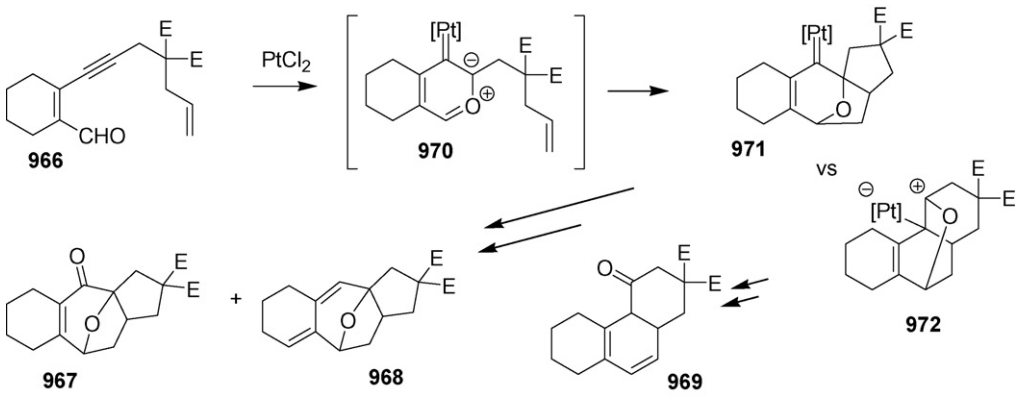


Scheme 79.





Scheme 80.



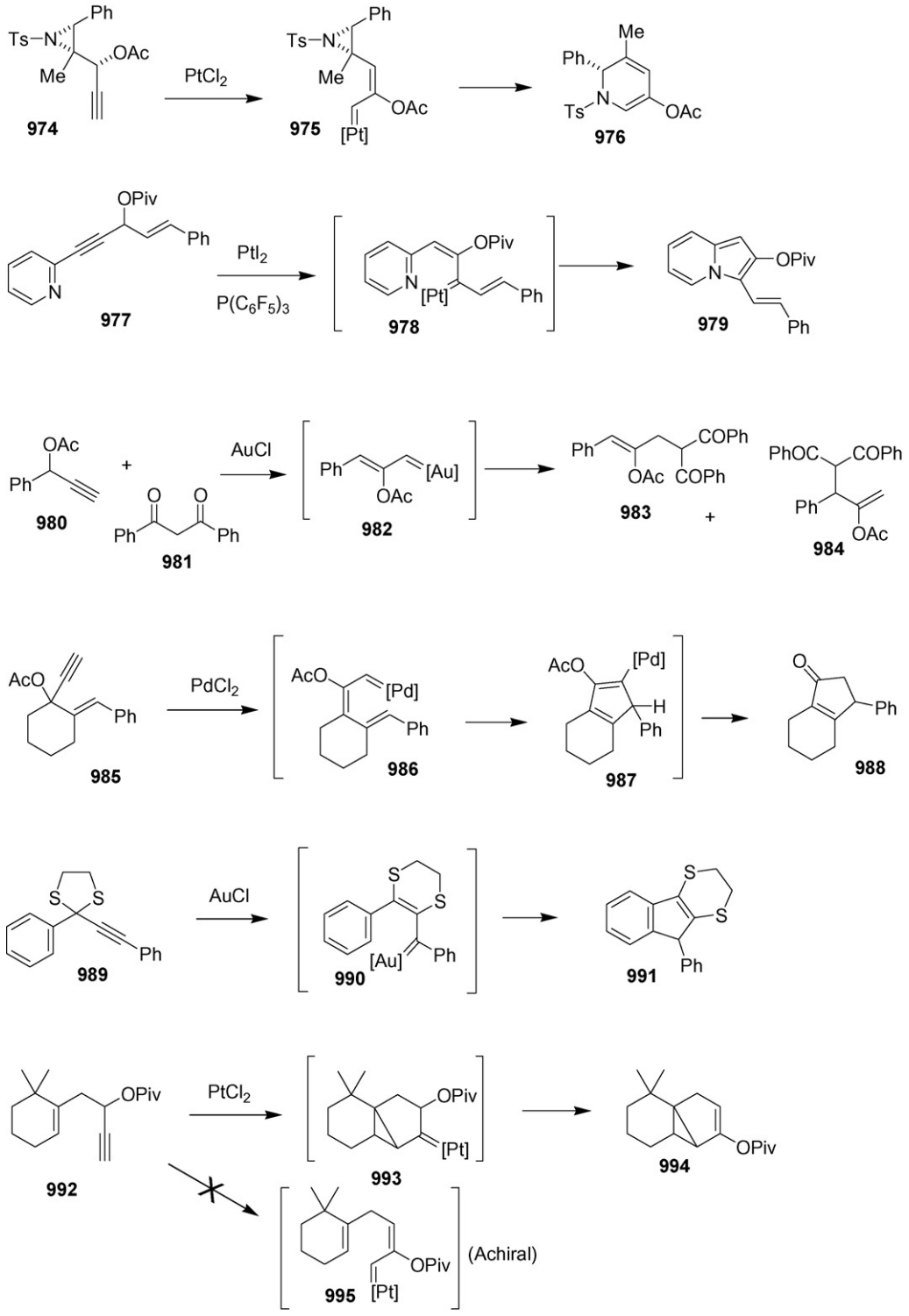
Scheme 81.

[1061]. Platinum-catalyzed intramolecular enyne metathesis (see Scheme 1), was employed for the synthesis of vinylcyclopentene derivatives [1062].The mechanism of gold- and platinum-catalyzed enyne metathesis which involves cyclopropylcarbene complex intermediates, was studied experimentally and computationally [1063].

Gold– and platinum–carbene complexes were also generated from various heterocyclization processes; representative examples are depicted in Scheme 80. The treatment of aryl propargyl sulfoxides (*e.g.* **950**) with gold(I) complex **951** led to the cyclization products (*e.g.* **952**) [1064]. A mechanism involving nucleophilic addition of the sulfoxide oxygen to the alkyne–gold complex to afford the vinylgold species (**954**), followed by formation of the α-ketocarbene complex (**955**), followed cyclization onto the aromatic ring was proposed. A similar process was reported for propargyl alcohol analogs (*e.g.* **956**), which ultimately led to ring-expanded products (*e.g.* **957**) through rearrangement of the carbene complex intermediate (**958**) [1065]. The reaction of *o*-alkynylbenzyl alcohol derivatives (*e.g.* **960**) with gold(III) chloride led to cyclization products (*e.g.* **961**) accompanied by dimerization products (*e.g.* **962**) [1066]. The proposed mechanism for dimer formation involves formation of the symmetrical ether (**963**), followed conversion of an alkyne unit to the α-carbenylcarbene complex (**964**), followed by C–H insertion and demetallation of the resulting carbene complex (**965**). A tandem heterocyclization–cycloaddition employing aldehyde–alkenes (*e.g.* **966**, Scheme 81) was reported [1067]. Initial formation of a carbene complex (**970**) is followed by either intramolecular dipolar cycloaddition or intramolecular Diels–Alder reaction. Intramolecular dipolar cycloaddition from intermediate **970** leads to oxygen-bridged hydroazulenes (*e.g.* **967, 968**) after demetallation. Intramolecular Diels–Alder reaction from **970** leads to hydronaphthalenes (*e.g.* **969**) after metallation. Gold carbene complexes were proposed as possible intermediates in the cyclization of O-propargyl hydroxylamines [1068]. Platinum–carbene complexes were also suggested as intermediates in the cycloaddition of 2-alkynylbenzaldehyde derivatives with enol ethers catalyzed by platinum [1069].

Carbene complexes were also generated from 1,2-shift of propargyl ester derivatives in the presence of platinum and gold complexes (see Scheme 82). Dihydropyridine derivatives (*e.g.* **976**) were prepared through platinum-catalyzed cyclization of propargyl esters that contain an aziridine group (*e.g.* **974**) [1070]. The reaction was proposed to involve intermediate carbene complex **975**, which subsequently cyclizes. Indolizines (*e.g.* **979**) were prepared through cyclization of propargyl esters containing pyridine groups (*e.g.* **977**) via carbene intermediates (*e.g.* **978**) [1071]. Tandem carbene formation (*e.g.* **982**) followed by nucleophilic addition was proposed as a key step in the gold-catalyzed formation of coupling adducts (*e.g.* **983, 984**) from propargyl acetates (*e.g.* **980**) and 1,3-diketones (*e.g.* **981**) [1072]. The **983:984** ratio was highly dependent on the identity of the gold catalyst. Acetoxyenynes (*e.g.* **985**) react with palladium complexes to afford cyclopentenones (*e.g.* **988**) [1073]. A mechanism involving carbene formation/acetoxy shift (**986**) followed by cyclization was proposed. In a related process, conversion of alkynyl thioacetals (*e.g.* **989**) to indenes (*e.g.* **991**), 1,2-shift of a sulfur group leading to a carbene intermediate (*e.g.* **990**) was also proposed as a key step [1074]. The platinum-catalyzed conversion of alkene-containing propargyl esters (*e.g.* **992**) to bicyclo[3.1.0]-hexane derivatives (*e.g.* **994**) was studied computationally [1075]. A mechanism where migration of the acetoxy group occurs after cyclization (**992** to **993** to **994**) was found to be more reasonable and is in agreement with the experimental results where stereochemical information is transferred to the product. Generation of a carbene complex (**995**) through 1,2-shift of an acetoxy group is not consistent with the conservation of stereochemical information since this intermediate is achiral. A similar process likely occurs in the gold-catalyzed isomerization of enyne-propargyl esters to aromatic ketones [1076]. Platinum-catalyzed conversion of 4-aryl-4-acetoxy-2-butynoate esters to either indenes or 4-aryl-2,4-dienoate esters was studied computationally [1077].

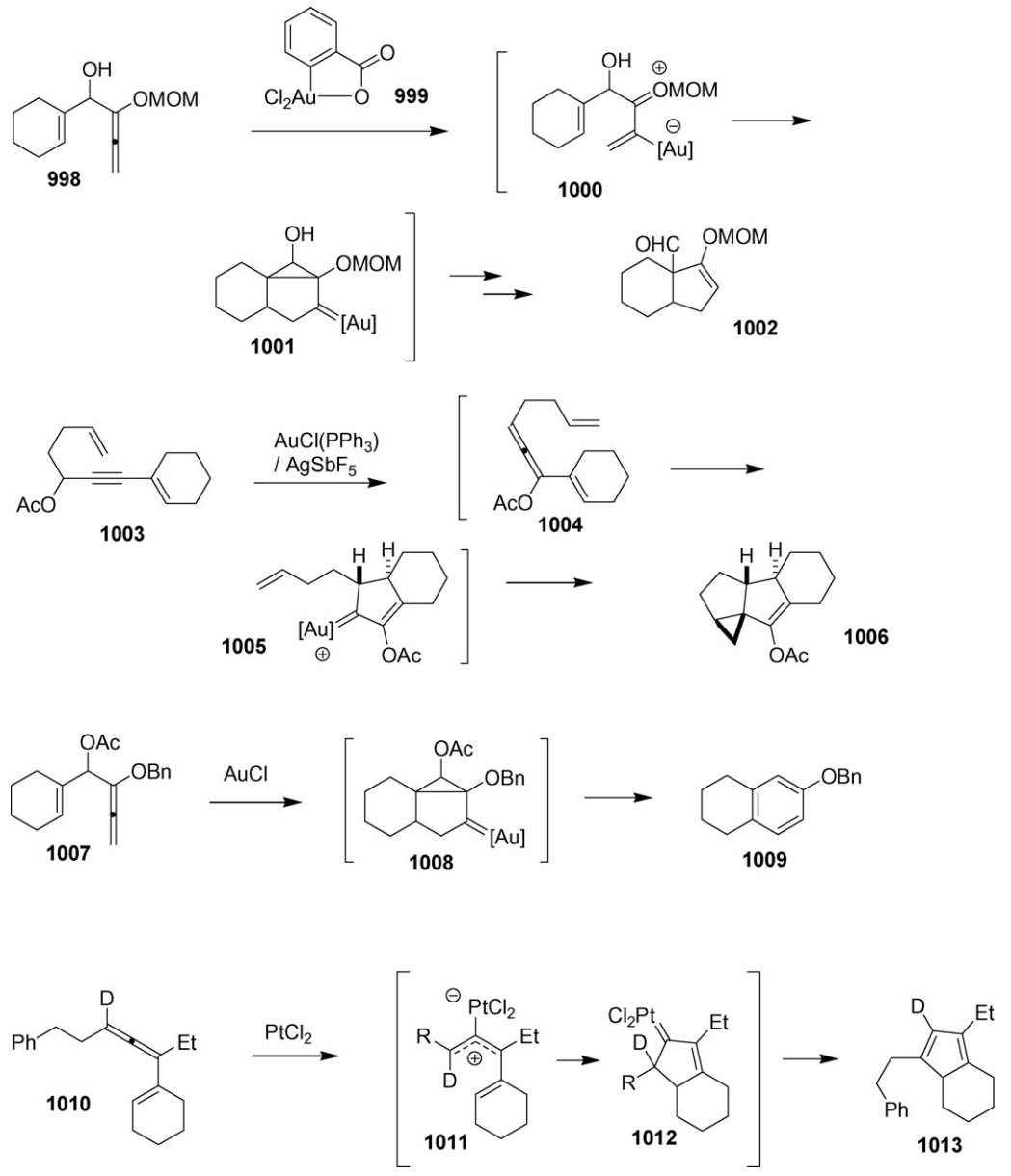
Related processes involving the formation of platinum– or gold–carbene complexes from allenyl esters or ethers were also reported (see Scheme 83). Cyclopropylcarbene–gold complexes (*e.g.* **1001**) formed through cyclization of an intermediate ylide (**1000**) were proposed as intermediates in the cyclization of ene-allene alcohols (*e.g.* **998**) [1078]. Another related [3+2]-cycloaddition process was reported that also involves ylide like structures similar to intermediate **1000** [1079]. Reaction of dienyne ester **1003** with gold(I) complexes led to the tetracyclic compound **1006** [1080]. Key events in this transformation include rearrangement to an allenyl ester (**1004**) and generation of a gold carbene complex (**1005**) through a metalla-Nazarov process, followed by intramolecular cyclopropanation. The synthesis of cyclopentenones through a Nazarov-like process involving gold-catalyzed cyclization of enyne-propargyl ester in aqueous systems was studied computationally [1081]. Reaction of allenyl ether **1007** with a gold(I) species led to the benzannulation product **1009** [1082]. A key step in this transformation is formation of cyclopropylcarbene complex **1008** followed by cyclopropane ring opening and elimination of both the acetate and gold groups. Gold- or platinum-induced cyclization of ene-allenes (*e.g.* **1010**) leads to cyclopentadienes (*e.g.* **1013**) [1083,1084]. A mechanism involving formation of a metallapentadienyl cation (**1011**) followed by electrocyclic ring closure



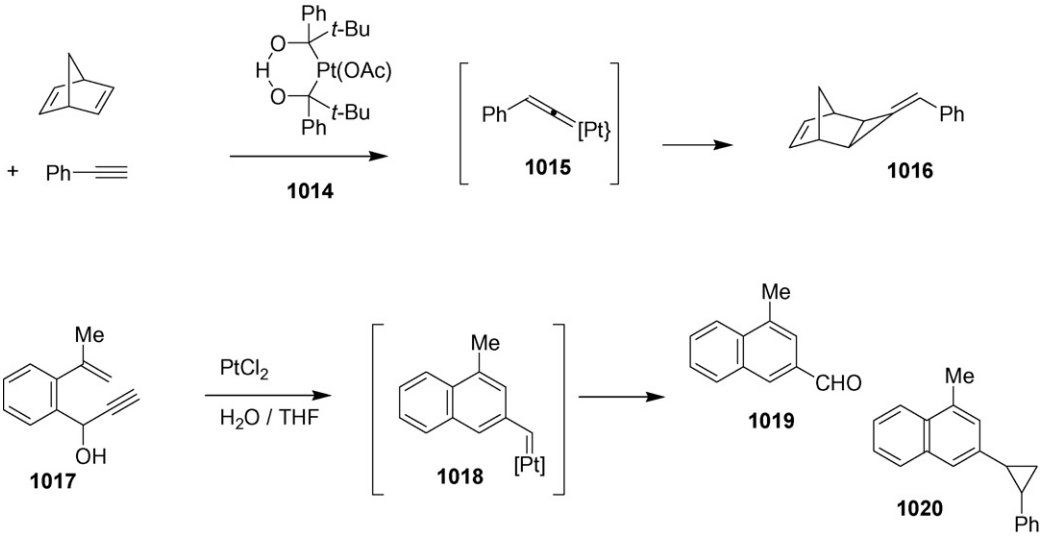
Scheme 82.

to generate a carbene complex (**1012**), followed by conversion to the alkene was proposed and supported through deuterium labeling studies. Cyclopropanation products were observed using tetrasubstituted allenes. Gold–carbene complexes were proposed as intermediates in the gold-catalyzed conversion of allenyl ketones to furans [1085].

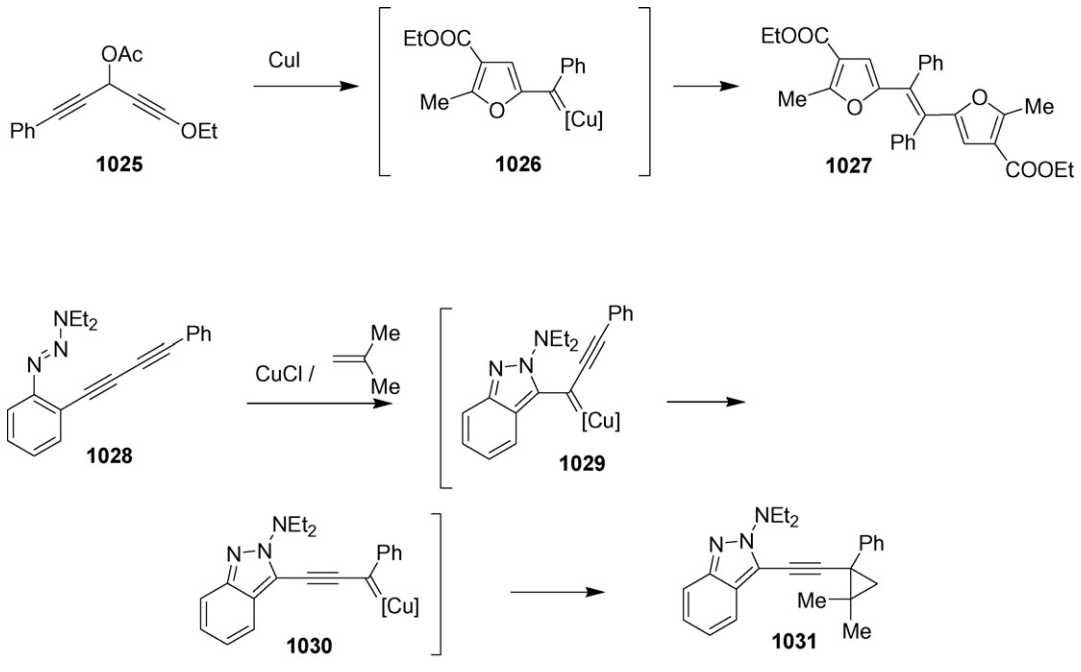
Several other reaction processes were reported from the reaction of alkynes and platinum or gold complexes that might involve carbene complex intermediates (see Scheme 84). Platinum–vinylidene complexes (*e.g.* **1015**) were suggested as intermediates in the alkylidenecyclopropanation of strained alkenes (*e.g.* norbornadiene) employing terminal alkynes and a platinum complex (**1014**) [1086]. Platinum–vinylidene complexes are also likely intermediates in the conversion of alkynylbiphenyls into the corresponding phenanthrenes [1087]. A platinum carbene complex (**1018**) was suggested as an intermediate in the cyclization of enyne-propargyl alcohols (*e.g.* **1017**) to naphthaldehydes (*e.g.* **1019**) [1088]. The intermediacy of carbenes was further supported through formation



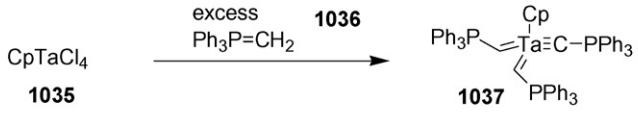
Scheme 83.

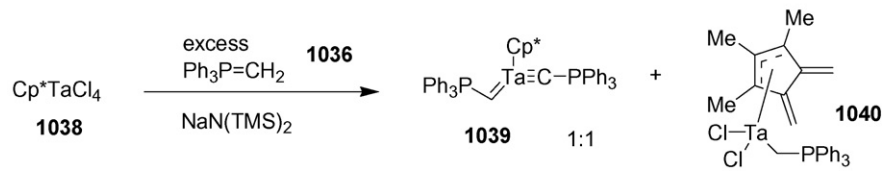


Scheme 84.

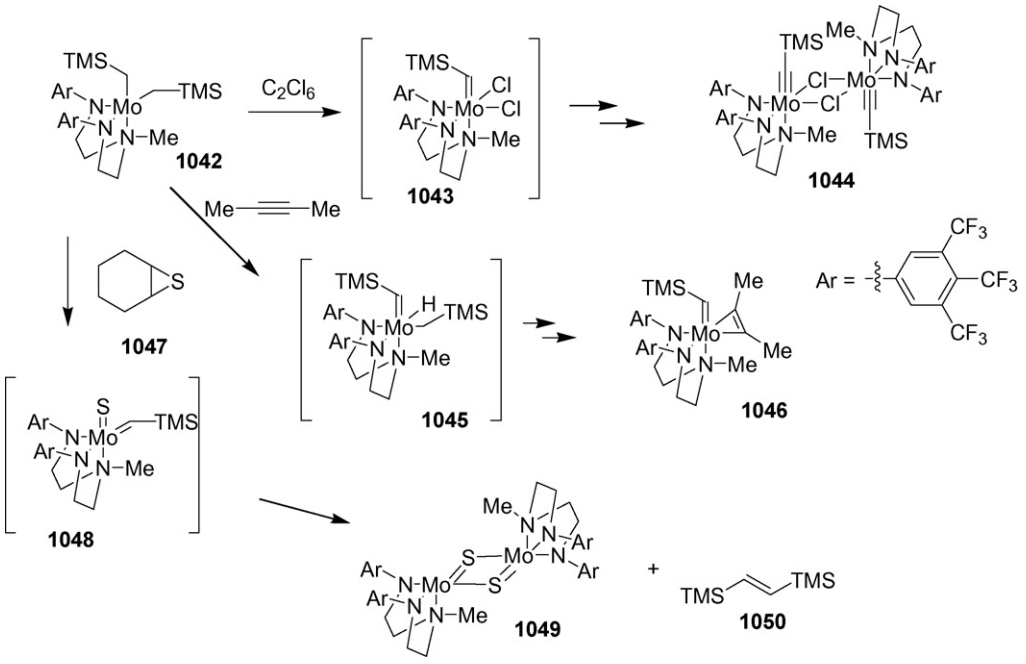


Scheme 85.

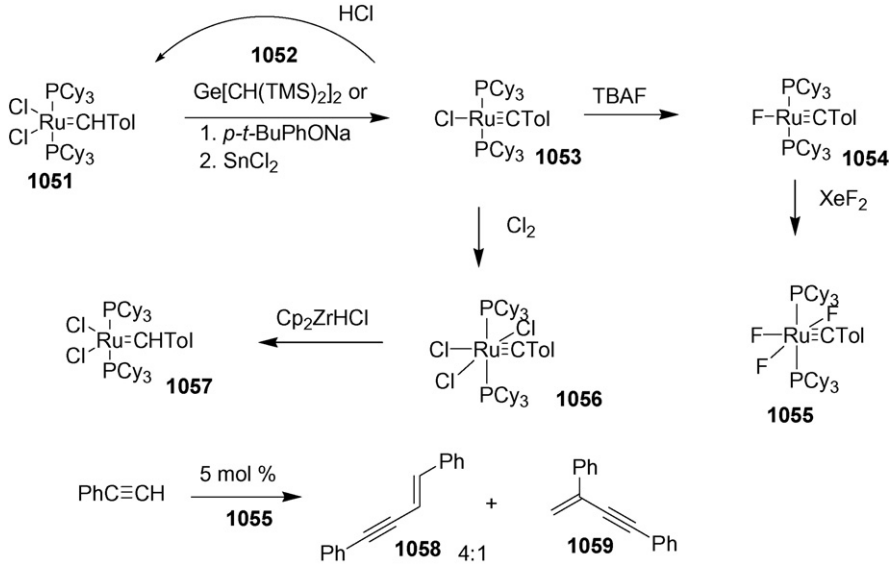




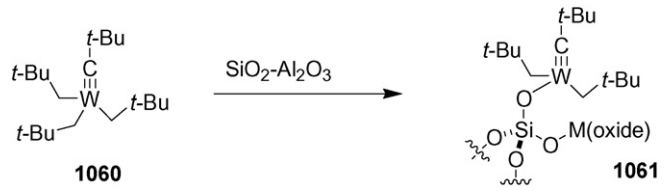
Scheme 86.



Scheme 87.



Scheme 88.

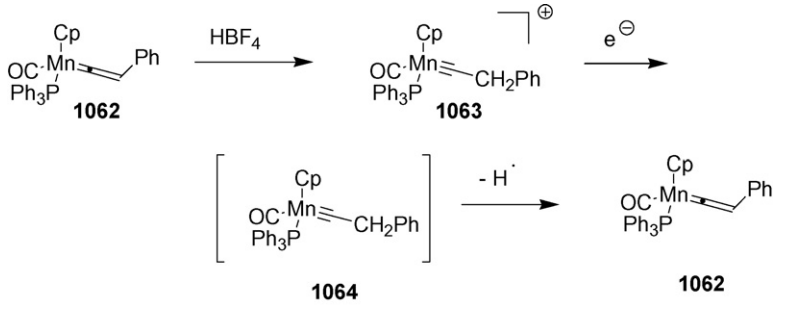


Scheme 89.

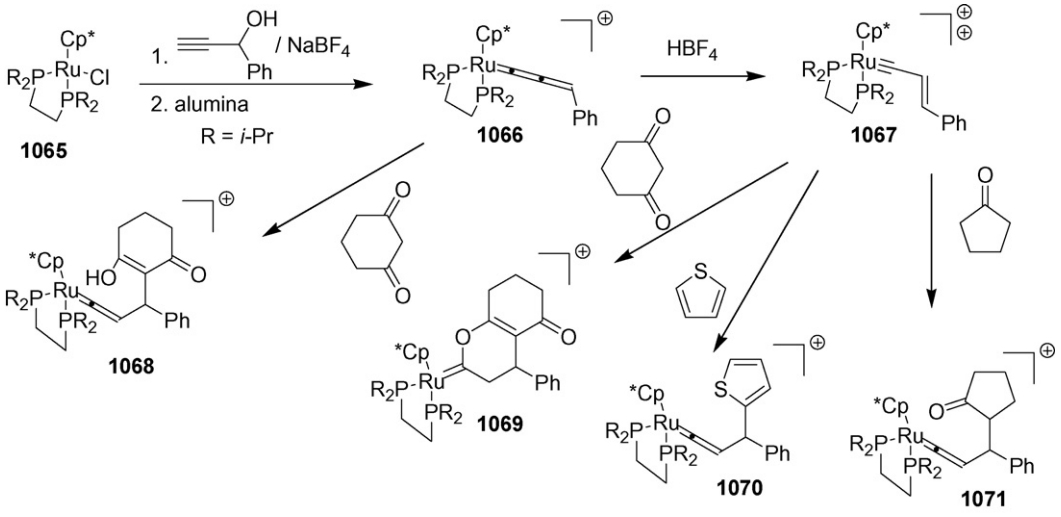
cyclopropanation products (*e.g.* **1020**) when styrene was added to the reaction mixture.

2.3.8. Group 11 carbene complexes

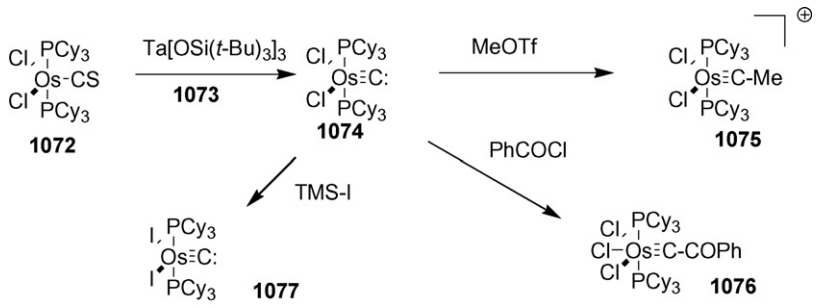
Several processes catalyzed by Group 11 metal–carbene complexes propose copper carbene complexes as intermediates (see Scheme 85). Copper carbene complexes (*e.g.* **1026**) were suggested as intermediates in the cyclization/dimerization of dialkyne-esters



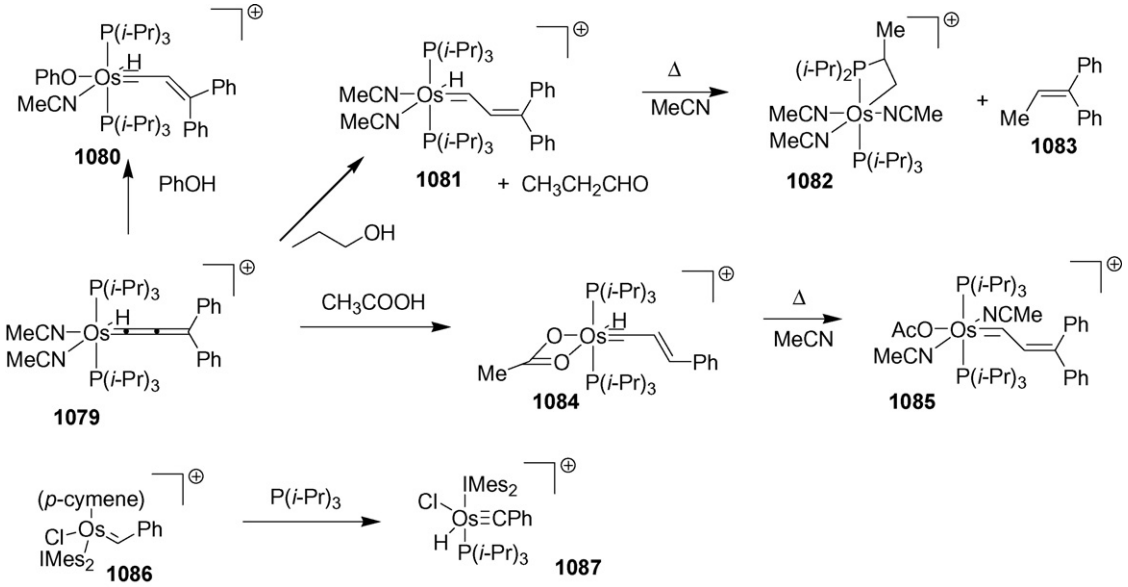
Scheme 90.



Scheme 91.

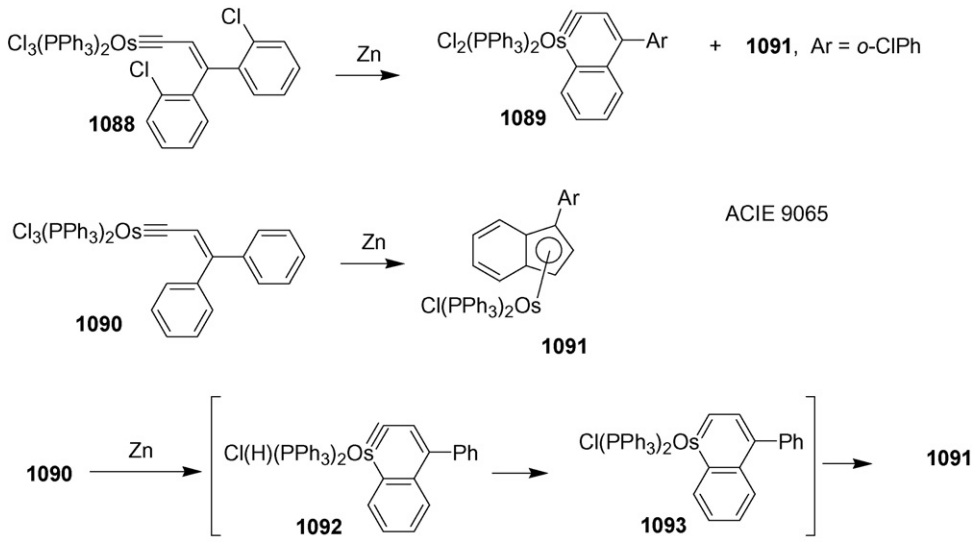


Scheme 92.

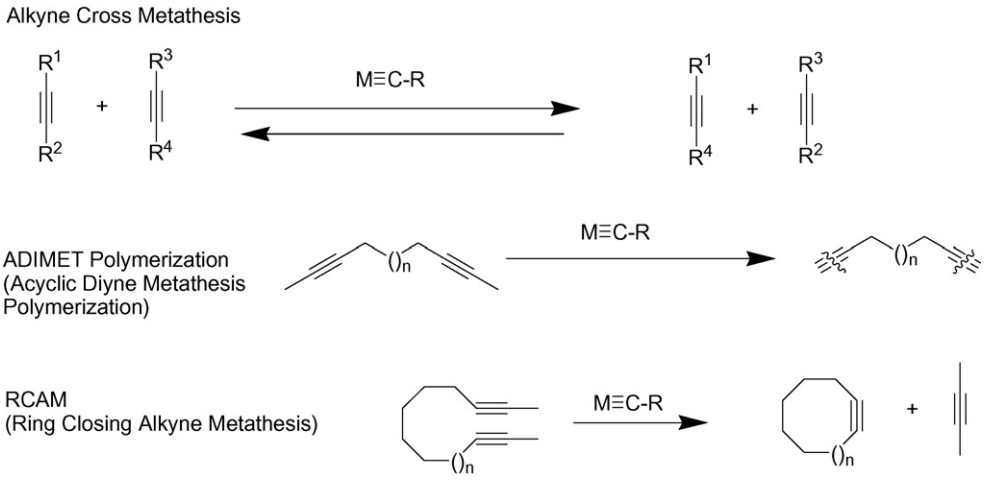


Scheme 93.

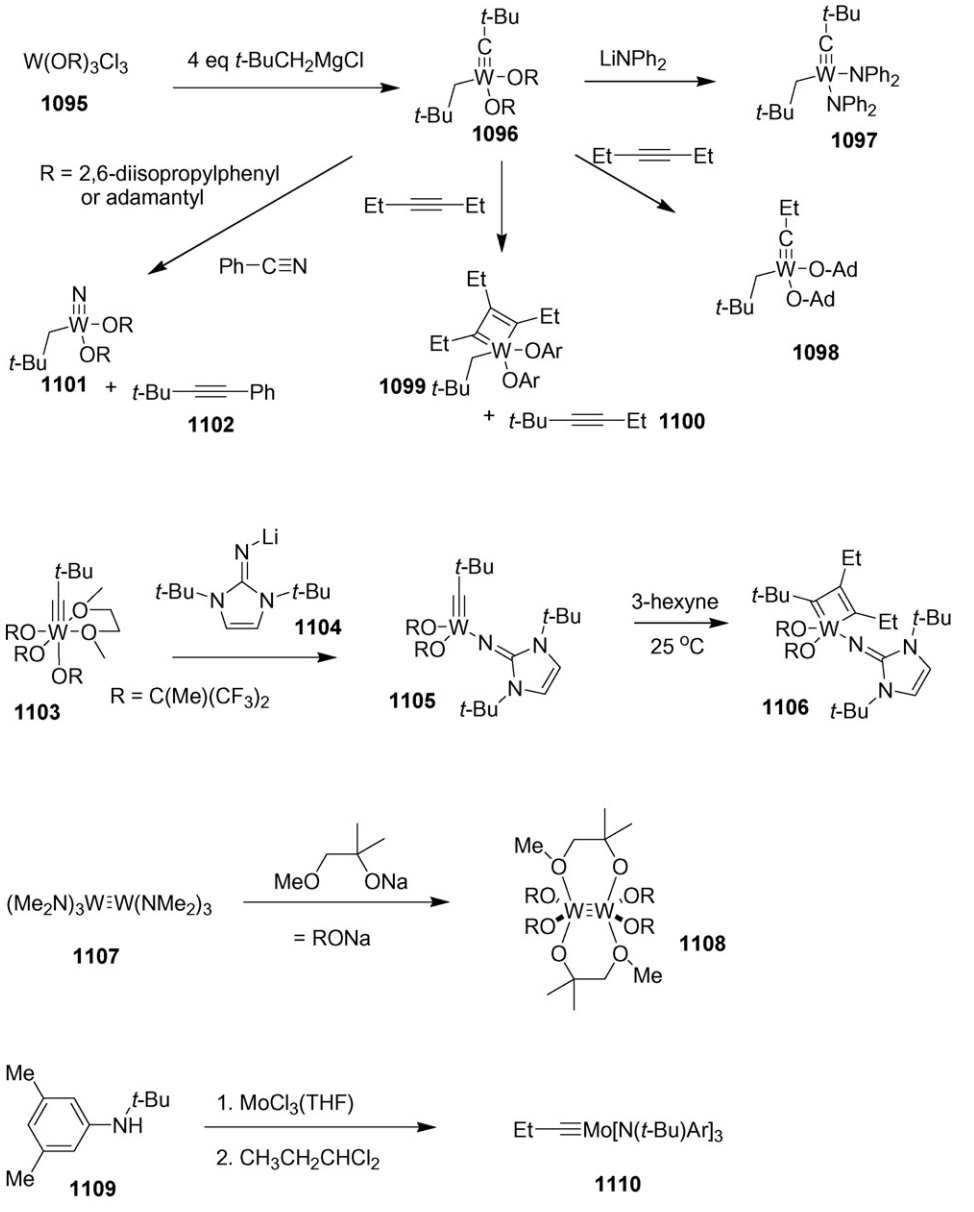
(*e.g.* **1025**) to afford bis(furanyl)alkenes (*e.g.* **1027**) [1089]. Group 11 metal–carbene complexes were proposed as intermediates in the metal-catalyzed cyclization of 4-acetoxy-2-alkyn-1-ones to 3-acetoxyfuran derivations, and conversion of the analogous imines into 3-acetoxypyrrole derivatives [1090]. Copper carbene complexes (*e.g.* **1029**) were suggested as intermediates in a novel cyclopropanation process involving conversion of dialkyne-azo derivatives (*e.g.* **1028**) into cyclopropanated indazole derivatives (*e.g.* **1031**) [1091]. Key events in the proposed mechanism involve tandem copper-catalyzed nucleophilic addition of nitrogen to the dialkyne group to form an alkynylcarbene complex (**1029**), which undergoes a 1,3-carbene shift to generate iso-



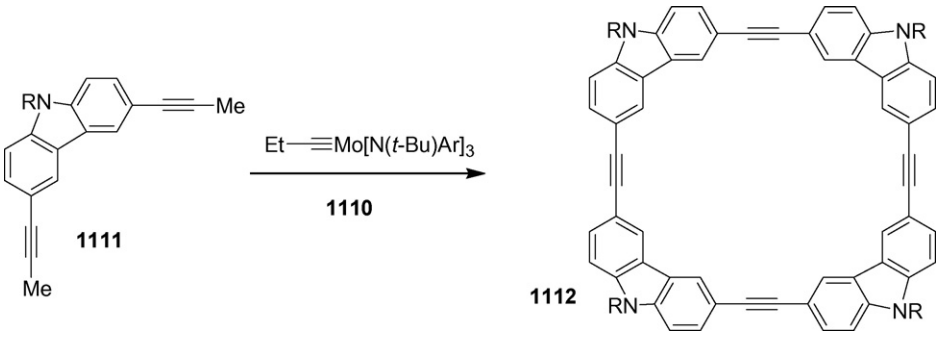
Scheme 94.



Scheme 95.



Scheme 96.



Scheme 97.

meric alkynylcarbene complex **1030** followed by cyclopropanation. Diaminocarbene–gold complexes were prepared through addition of amines to gold isocyanide complexes [1092].

Several papers report on mechanistic aspects of copper–carbene complex intermediates generated from organic diazo compounds. Studies reported in 2007 include: (1) an inconclusive attempt to spectrally characterize a copper carbene complex intermediate in copper-catalyzed asymmetric cyclopropanations using ethyl diazoacetate, and related DFT studies [1093]; (2) discussion of stereoselectivity models for copper-catalyzed asymmetric preparation of chrysanthemic acid esters [1094]; (3) a DFT study of copper–tris(3,5-dimethylpyrazolylmethane) complex catalyzed C–H insertions of diazo-esters [1095]; (4) C–H insertions of methane using metal–carbene complexes using DFT [1096]; and (5) use of copper–Tp complexes to catalyze cyclopropanation reactions [1097,1098].

2.3.9. Lanthanide/actinide carbene complexes

Thorium carbene complexes were generated from the interaction of laser-ablated thorium atoms with chlorofluoromethane [1099]. The product $H_2C{=}ThCClF$ was identified through comparison of the observed IR spectra with those calculated for the energy-minimized structure, which is chiral at thorium. Uranium carbene complexes were generated through interaction of laser-ablated uranium atoms with methane [1100]. Complexes were identified through comparison of observed IR spectra with DFT calculated spectra. Similar laser-ablation studies were reported for scandium atoms in the presence of methane, however the carbene complexes showed little metal–carbon double bond character according to DFT studies [1101]. Lanthanum carbene complexes are possible intermediates in lanthanum-medium cyclopropanation using 1,1-dibromoalkenes and styrene [1102].

3. Metal–carbyne or metal–alkylidyne complexes

3.1. Review articles

Review articles featuring metal–carbyne complexes which appeared in 2007 include: (1) development of alkyne metathesis catalysts [1103]; and (2) metallabenzynes [1104].

3.2. Synthesis and/or generation

Some papers in the carbene section feature minor segments on carbyne chemistry. These studies include references [207,832–836,916,919,923,927].

A tantalum carbyne-bis(carbene) complex (**1037**, Scheme 86) was generated in the reaction of tantalum complex **1035** with methylenetriphenylphosphorane [1105]. Formation of related carbyne complexes (**1039**) from Cp^*TaCl_4 (**1038**) was also reported [1106]. In this case, formation of carbyne complex **1039** was accompanied by bis(methylene)Cp complex **1040**. The yield of carbyne complex was enhanced by addition of the amide base, presumably to assist in the deprotonation of intermediate triphenylphosphinemethyl and methylene complex intermediates.

Several processes were reported for bis(trimethylsilylmethyl)-molybdenum complex **1042** (Scheme 87), including carbyne complex formation [1107]. A dimeric molybdenum carbyne complex (**1044**) was produced upon treatment of bis(trimethylsilylmethyl)molybdenum complex **1042** with hexachloroethane. A mechanism involving oxidation and elimination of tetramethylsilane to afford intermediate carbene complex **1043** followed by elimination of HCl was proposed. Treatment of the same complex with 2-butyne leads to the carbene(alkyne) complex **1046**. Treatment with cyclohexane sulfide leads to the dimeric sulfide complex **1049**. All of the above processes invoke α-hydride elimination and formation of the carbene complex and tetramethylsilane as key steps.

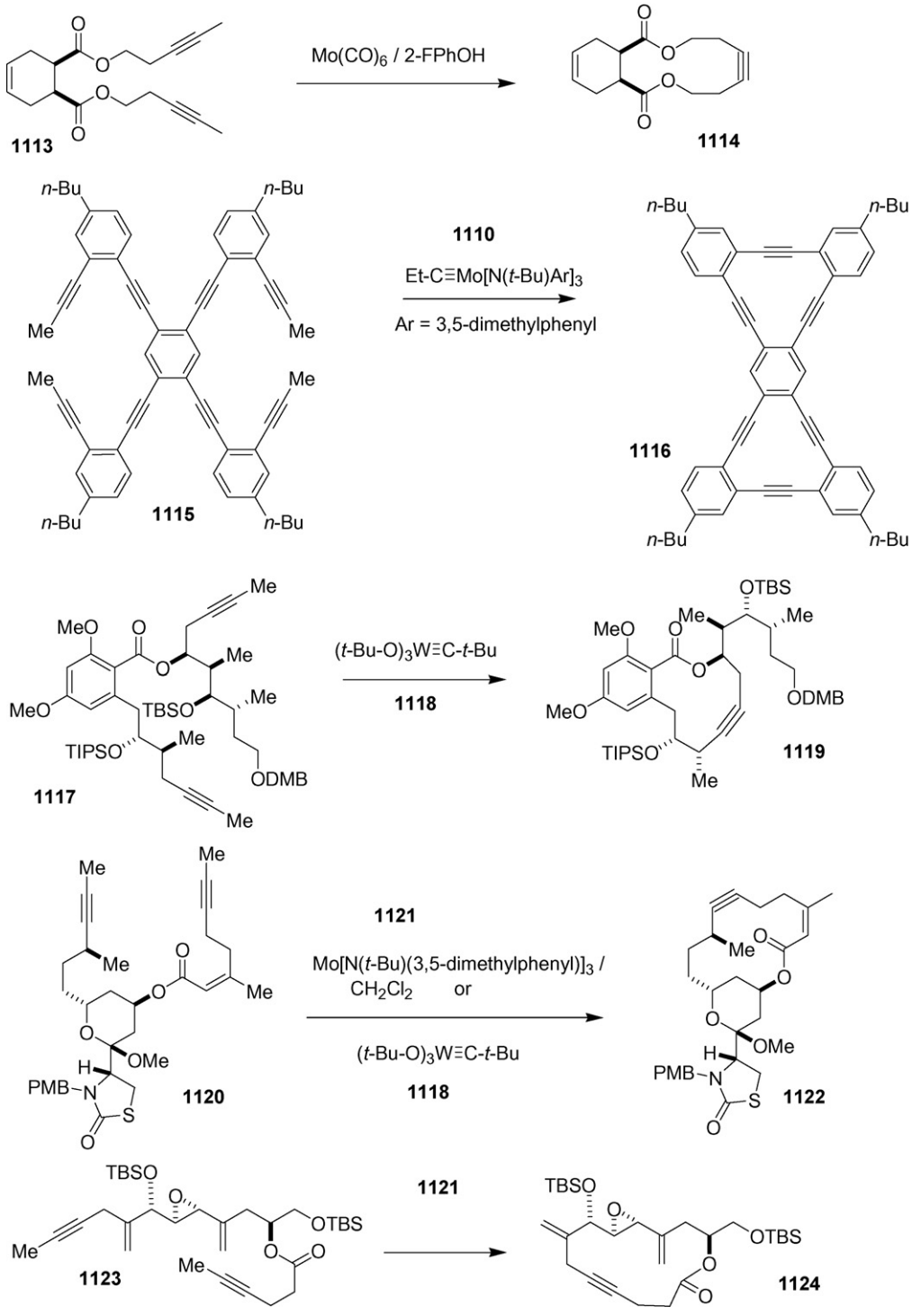
The preparation of ruthenium carbyne complexes (*e.g.* **1053–1056**, Scheme 88) from derivatives of Grubbs catalyst I (*e.g.* **1051**) was reported [1108]. Deprotonation of carbene complex **1051** led to carbyne complex **1053**, which could be transformed back to carbene complex **1051** by treatment with HCl. Various ligand substitution processes were reported for the carbyne complexes. Reaction of carbyne complexes (*e.g.* **1054**) with halogens led to the ruthenium(IV) carbyne complexes (*e.g.* **1055**). The carbyne complex **1055** serves as a catalyst for alkyne dimerization.

Several papers in 2007 reported on the generation of carbyne complexes from the interaction of methane or halomethanes with various metal atoms generated through laser ablation. The reaction of Group 4 metals with perhalomethanes (CF_4, CCl_4 or CF_2Cl_2) led to triplet state carbyne complexes XC-MX_3, which were identified through comparison of their transient IR spectra with those from calculated spectra [1109]. Related studies were reported for the reaction of Group 4 metals with CF_3Cl, $CFCl_3$, and CF_3Br [1110]. Rhenium carbyne complexes ($XC{\equiv}ReX_3$) were prepared through the interaction of laser-ablated rhenium atoms with perhalomethane or haloform derivatives (halogen=F or Cl) [1111]. The complexes were identified through their IR spectra and compared with optimal structures from DFT calculations. Carbyne species were generated from the reaction of laser ablation-generated rhenium atoms with methane [1112]. In this case, a highly characteristic alkylidyne C–H stretch was observed at 3102 cm^{-1}.

3.3. Reactivity

3.3.1. Addition reactions of metal–carbyne complexes

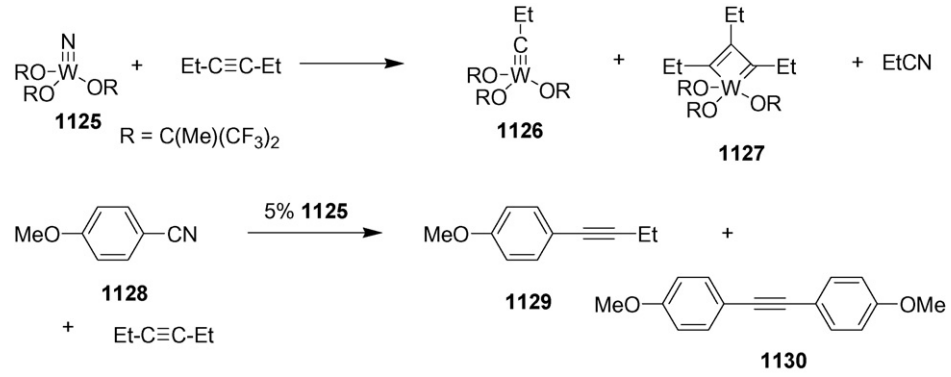
Tungsten carbyne complex **1060** (Scheme 89) was successfully grafted onto a silica–alumina surface [1113]. Reaction with hydrogen led to a surface-bound tungsten hydride species. Both the carbyne complex and the hydride functioned as alkane metathesis catalysts. The process was also examined computationally [1114].



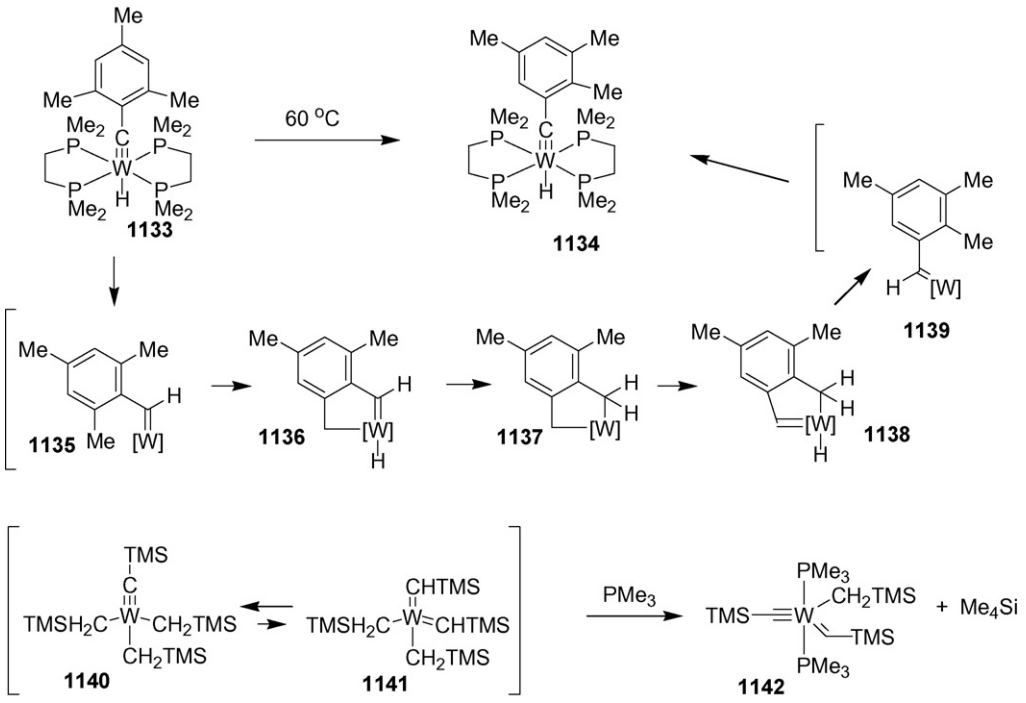
Scheme 98.

The formation and electrochemistry of manganese carbyne complexes (*e.g.* **1062**, Scheme 90) was reported [1115]. Protonation of the neutral vinylidene complex **1062** affords the cationic carbyne complex **1062**. Electrochemical reduction of the carbyne complex affords initially the 19-e radical **1064**, which undergoes net loss of a hydrogen atom to regenerate vinylidene complex **1062**. The process has been described as an electrochemical formation of hydrogen.

The synthesis and reactivity of cationic ruthenium allenylidene (*e.g.* **1066**, Scheme 91) and dicationic ruthenium carbyne complexes (*e.g.* **1067**) was reported [1116]. Ruthenium allenylidene complex **1066** was generated from ruthenium chloride **1065** and propargyl alcohols. Reaction of allenylidene complexes with 1,3-diketones led to substituted ruthenium vinylidene complexes (*e.g.* **1068**). Protonation of allenylidene complex led to dicationic carbyne complex **1067**. Reaction of this complex with 1,3-diketones led to cyclic carbene complexes (*e.g.* **1069**). Reaction of dicationic carbyne complexes with heteroaromatics (*e.g.* thiophene), electron rich aromatics, and simple ketones (*e.g.* cyclopentanone) to afford substituted vinylidene complexes (*e.g.* **1070, 1071**) was also reported.



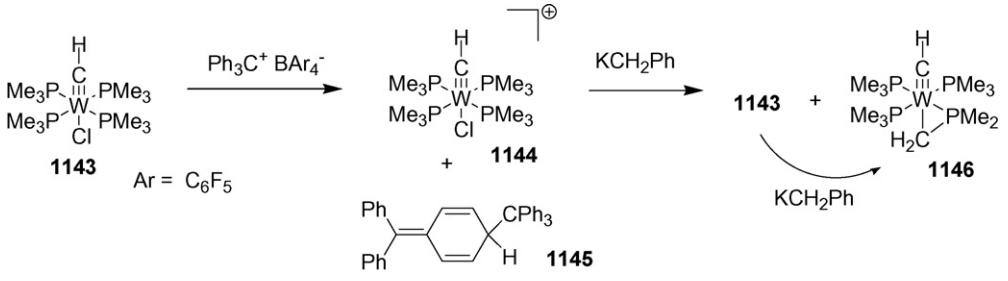
Scheme 99.



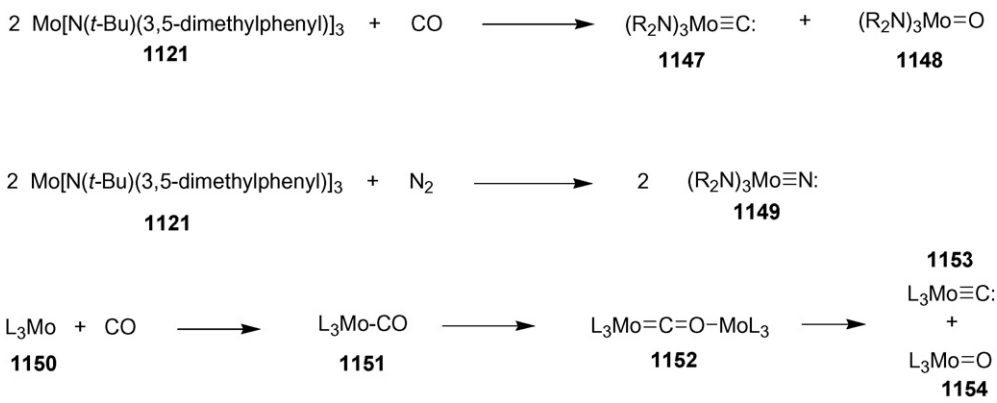
Scheme 100.

The synthesis and reactivity of osmium carbide complexes (*e.g.* **1074**, Scheme 92) was reported [1117]. The carbide complex was generated via desulfurization of thiocarbonyl complex **1072** using tantalum(III) alkoxide **1073**. This complex was subsequently treated with a variety of electrophiles to afford the cationic square pyramidal (or neutral octahedral) carbyne complexes (*e.g.* **1075, 1076**), depending upon the nucleophilicity of the electrophile counterion. Ligand substitution reactions (*e.g.* formation of carbide complex **1077**) were also demonstrated.

The synthesis and reactivity of several hydrido(carbyne)-osmium complexes was reported in 2007 (see Scheme 93). Cationic hydrido(carbyne)–osmium complexes (*e.g.* **1084**) were generated through protonation of a hydrido(vinylidene)–osmium complex (**1079**) [1118], which affords the vinylcarbene complex (**1085**) upon thermolysis. Treatment of allenylidene complex **1079** with phenol also leads to a hydrido(carbyne)–osmium complex (**1080**) containing a phenoxide ligand [1119]. The allenylidene complex can mediate the oxidation of primary and secondary alcohols. Treat-



Scheme 101.



Scheme 102.

ment of complex **1079** with alcohols (*e.g.* 1-propanol) leads to the alkenylcarbene complex (**1081**) and propanal. Thermolysis of carbene complex **1081** leads to the alkene **1083** and a phosphine ligand C–H oxidative addition product, complex **1082**. Synthesis of NHC-ligated hydride(carbyne)–osmium complexes (*e.g.* **1087**) from carbene complexes (*e.g.* **1086**) was also reported [1120].

The reduction and cyclization of osmium–alkenylcarbyne complexes (*e.g.* **1088, 1090**, Scheme 94) was reported [1121]. Reduction of the bis(*o*-chlorophenyl) complex **1088** led to osmabenzyne complex **1089** accompanied by indenyl complex **1091**. Reduction of the diphenyl analog led only to indenyl complex **1091**. The proposed mechanism for formation of the osmabenzyne involves reduction to the monochloroosmium–carbyne complex followed by either C–Cl or C–H oxidative addition. Formation of the indenyl complex occurs through hydride migration in osmabenzyne complex **1092** to form the osmanaphthalene complex (**1093**) followed by aryl group migration to afford the indenyl system. The mechanistic hypotheses were supported by DFT calculations.

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 95.

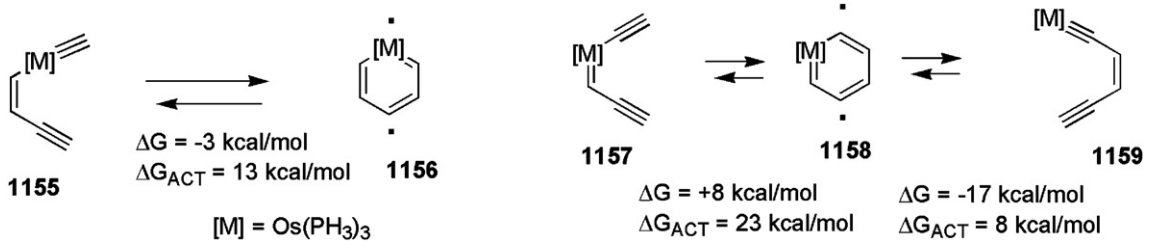
Several efforts to develop new alkyne metathesis catalysts were reported in 2007, representative examples are depicted in Scheme 96. Tungsten carbyne complexes featuring aryloxy or diarylamido ligands (*e.g.* **1096–1098**) were prepared from tungsten(VI) alkoxide **1095** [1122]. The initial carbyne complex **1096** afforded the analogous amido complex **1097** through ligand exchange and the nitride complex **1101** through reaction with a nitrile. Reaction of **1096** (R = aryl) with 3-hexyne led to the tungstacyclobutadiene complex **1099** and alkyne **1100**. The adamantyl analog reacted with 3-hexyne to afford the metathesized carbene complex **1098**. The tungsten carbyne complex (**1105**) can catalyze alkyne metathesis reactions at room temperature [1123]. The ditungsten complex **1108** can effect terminal alkyne metathesis [1124]. The development of a silica-bound bis(tungsten)pentakis(dimethylamido) complex for alkyne metathesis was also reported [1125]. An Organic Synthesis preparation for alkyne metathesis catalyst **1110** was reported [1126]. A patent was awarded for the development of alkyne metathesis catalysts [1127].

Several examples of alkyne-cross metathesis were reported in 2007. Tandem alkyne cross metathesis–alkyne ring-closing metathesis occurred in the conversion of a diyne (*e.g.* **1111**, Scheme 97) to a cyclic tetramer (**1112**) using metathesis catalyst **1110** [1128,1129]. Examples of ring closing alkyne metathesis reported in 2007 are depicted in Scheme 98 and include: (1) synthesis of bis(lactone) derivatives (*e.g.* **1114**) followed by subsequent use of the cyclic alkyne for enyne metathesis [1130]; (2) construction of cyclic triyne derivatives (*e.g.* **1116**) [1131]; (3) construction of a macrocyclic lactone (*e.g.* **1119**) for total synthesis of cruentaren A [1132–1134]; (4) construction of a macrocyclic lactone (*e.g.* **1122**) for total synthesis of 16-epi-lantruculin B [1135] and structural analogs [1136]; (5) construction of the macrocyclic ring of myxovirescin A [1137]; and (6) construction of a macrocyclic lactone (**1124**) and subsequent use of a cyclic alkyne in enyne metathesis as key steps in a total synthesis of amphidinolide V [1138].

Nitrile–alkyne metathesis was reported (see Scheme 99) [1139]. The reaction of tungsten nitrides (*e.g.* **1125**) with 3-hexyne led to the tungsten carbyne complex (**1126**), the metallacyclobutadiene (**1127**), and propionitrile. The nitride complex serves as a catalyst for the co-metathesis of nitriles and alkynes. In the presence of a "sacrificial" alkyne (*e.g.* 3-hexyne) the nitride complexes can also serve as an alkyne metathesis catalyst.

3.3.3. Other processes involving metal–carbyne complexes

Isomerization reactions were reported for several tungsten carbyne complexes in 2007 (see Scheme 100). Aryl isomerization of mesitylcarbyne–tungsten complex **1133** to arylcarbyne complex **1134** was reported [1140]. A mechanism involving reversible formation of a carbene complex (**1135**), followed by C–H oxidative addition to afford a cyclic carbene complex (**1136**), followed by



Scheme 103.

hydrogen migration and reformation of the carbyne complex functionality at what was originally a methyl group was proposed. The mechanism was supported through deuterium labeling studies and DFT calculations. The reaction of tungsten carbyne complex **1140** [in equilibrium with isomeric bis(alkylidene) complex **1141**] with trimethylphosphine was reported [1141]. The reaction led to a stable nonfluxional carbene–carbyne complex, **1142**.

The one-electron oxidation of tungsten methylidyne complex **1143** (Scheme 101) was reported [1142]. Treatment of the tungsten methylidyne complex **1143** with trityl tetraarylborate led to the cationic tungsten(V) complex **1144** and the trityl dimer **1145**. Attempts to deprotonate the paramagnetic complex led to the reduction product, original carbyne complex **1143**, accompanied by a minor amount of bridged structure **1146**. Complex **1146** results from deprotonation of the phosphine ligand in the original complex **1143**.

A DFT study of the formation of molybdenum carbide complexes (*e.g.* **1147**, Scheme 102) from tris(amido)molybdenum(III) complexes (*e.g.* **1121**) and CO was reported [1143]. The mechanistic steps evaluated are depicted in Scheme 102. All steps of the reaction become energetically more favorable when the simple NH_2 ligand is replaced by the (*t*-Bu)(3,5-dimethylphenyl)amido ligand. The cleavage of CO and cleavage N_2 cleavage were also compared for various molybdenum complexes. Although the overall conversion of CO to molybdenum carbide and molybdenum oxide is exothermic, the final step was endothermic. The analogous step for nitrogen cleavage was exothermic. Similar studies were reported for the generation of rhenium carbides from the reaction of rhenium carbonyls with tris(amido)tantalum(III) complexes [1144].

Several hypothetical reaction processes involving metal–carbyne complexes were reported (Scheme 103). The energetics for the hypothetical metalla-Bergman cyclization were studied computationally [1145]. Formation of the metallabenzene diradical (**1156**) from the enynyl(carbyne) osmium complex **1155** was an exergonic process requiring low activation energy. Isomerization of the alkynylcarbene complex (**1157**) to the enynylcarbyne complex (**1159**) was also an exergonic process, where both formation of the metallabenzene diradical (**1158**) and its ring opening occur at relatively low activation energy. The hypothetical N–H activation of NH_3 by titanium–carbyne complexes was also studied computationally [1146].

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