PERKIN 1 REVIEW

Saturated and partially unsaturated carbocycles

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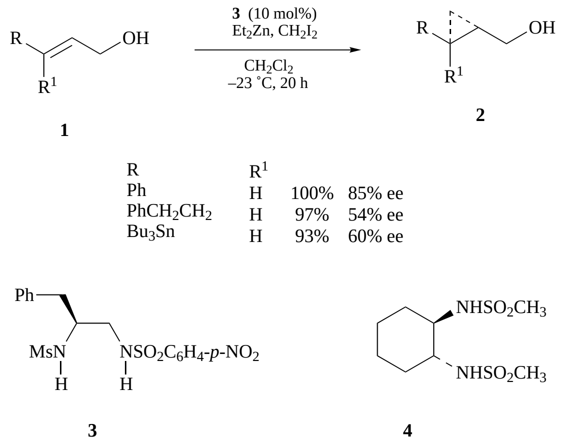
1 Three-membered rings

1.1 Metal carbenoid and carbene based methods

1.1.1 From dihaloalkanes

The most interesting area of research in this field has been to utilise the modified Simmons–Smith cyclopropanation (diethylzinc and diiodomethane) in conjunction with a chiral catalyst or chiral auxiliary to synthesise enantiopure products. Thus Imai *et al.* have subjected allylic alcohols **1** to the usual conditions in the presence of the chiral disulfonamide **3** to produce enantiopure cyclopropanols **2** (**Scheme 1**); similarly Denmark and O'Connor[2] have developed a variant on the modified Simmons–Smith reaction conditions using the chiral catalyst **4** to prepare cyclopropanols in very good yields and enantiomeric excesses.

Luh and co-workers[3] have used a different approach to achieve diastereoselective cyclopropanation of α,β-unsaturated cycloalkenones: with a diol as a chiral auxiliary, they have transformed alkenes **5** into cyclopropanes **6** using classic Simmons–Smith conditions (**Scheme 2**). According to the same principle, Charette *et al.*[4] have used the carbohydrate based chiral auxiliary **9** to achieve chemo- and enantio-selective cyclopropanation (**7**→**8**). To achieve a similar end, the same group has investigated the use of the chiral reagent **10** to convert, for example, allylic alcohol **11** to cyclopropanol **12**. Further to this work, Charette and Lebel[5] have used the same reaction conditions to achieve the enantioselective total synthesis of the pentacyclopropane-containing cholesteryl ester transfer protein inhibitor U-106305. Barrett and co-workers[6,7] have also used chiral reagent **10** in a synthesis of this molecule as well as in the synthesis of the structurally related antifungal agent FR-900848.



R	R^1		
Ph	H	100%	85% ee
$PhCH_2CH_2$	H	97%	54% ee
Bu_3Sn	H	93%	60% ee

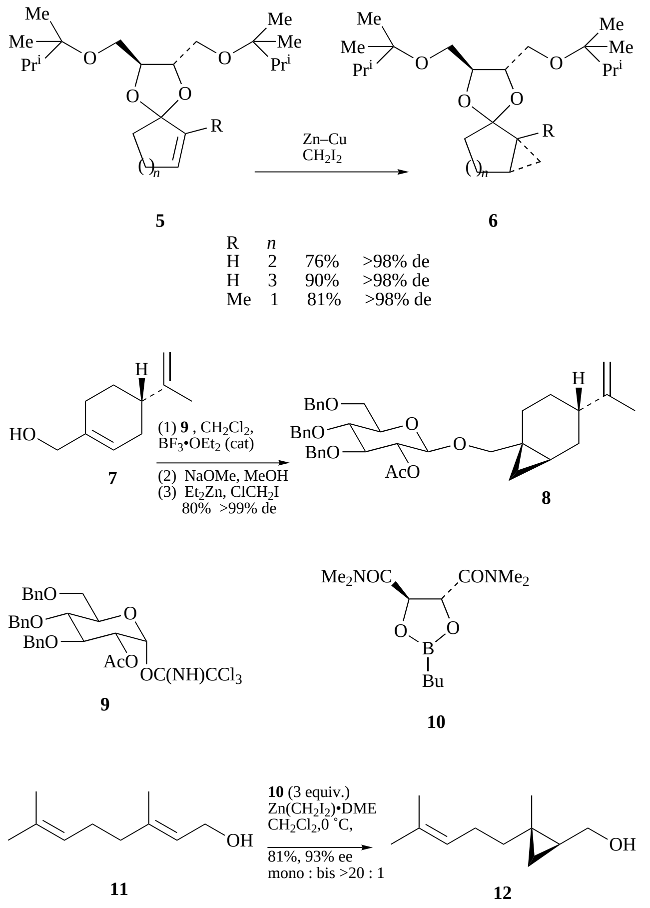
Scheme 1

1.1.2 From diazocarbonyl compounds

As for the dihaloalkane-derived metal carbenoid methods of cyclopropanation, the major area of research in the well-known reaction between alkenes and diazocarbonyl compounds in the presence of a metal catalyst has been the development of enantioselectivity. Not surprisingly the favoured method to achieve this is the use of a chiral catalyst, with rhodium as the metal of choice. Davies and co-workers[8,9] have investigated the use of rhodium(II) (*S*)-*N*-(arylsulfonyl)prolinate catalysts such as **15** in the reaction between alkenes and vinyldiazomethane **13** to give cyclopropanes **14** in very good yields and enantiomeric excesses (**Scheme 3**). Meanwhile Doyle and co-workers have developed the use of dirhodium(II) tetrakis[(4*S*)-alkyl 2-oxoazetidine-4-carboxylates] **16** as catalysts for asymmetric intermolecular cyclopropanations.[10] The same group have also conducted extensive studies on the various catalysts available for these reactions[11] as well as comparing the enantiocontrol obtained in the intramolecular cyclopropanation of diazoacetates using chiral copper(I), rhodium(II) and ruthenium(II) catalysts.[12] Finally Shi and Cai[13] have synthesised bicyclo[2.1.0]pentane ring systems from δ,ε-unsaturated β,β-difluoro-α-diazo esters (**17**→**18**).

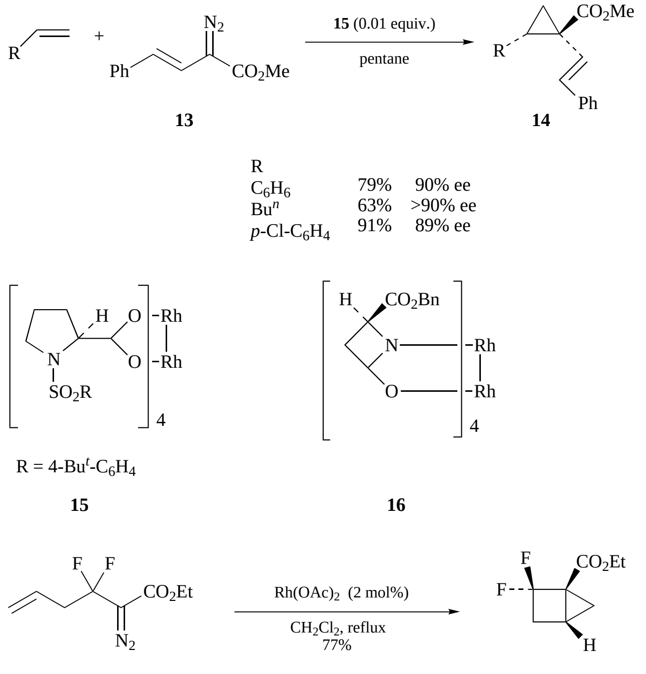
1.1.3 Other carbene/carbenoid derived methods

A number of other interesting methods of carbene/carbenoid derived cyclopropanations have been developed over the review period. For example, Suzuki and co-workers[14] have reported the first synthesis and X-ray analysis of alkenyl(triphenyl)bis-



R	*n*		
H	2	76%	>98% de
H	3	90%	>98% de
Me	1	81%	>98% de

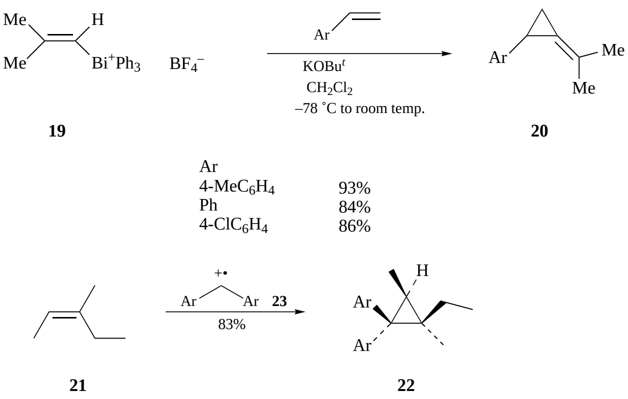
Scheme 2



R		
C_6H_6	79%	90% ee
Bu^n	63%	>90% ee
p-Cl-C_6H_4	91%	89% ee

Scheme 3

muthonium salts such as **19** which on treatment with potassium *tert*-butoxide in the presence of styrenes results in the production of arylcyclopropanes **20** (**Scheme 4**). Goodman and co-workers[15] have shown that the reaction between 4,4′-dimethyldiphenyldiazomethane and tris-(4-methylphenyl)aminium hexafluoroantimonate results in the radical cation **23** which reacts with alkenes to give cyclopropanes (**21**→**22**).



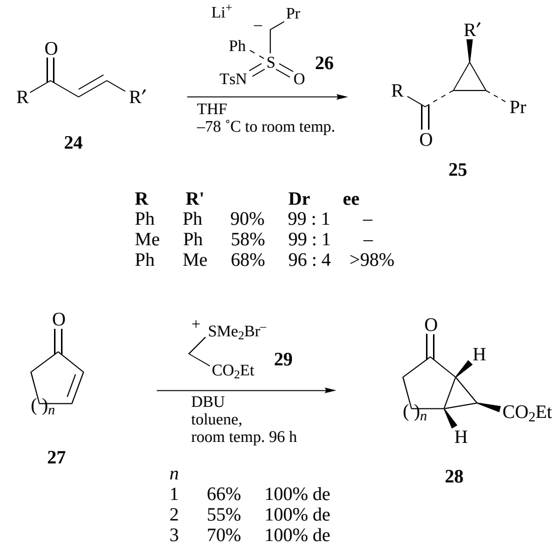
Ar	
4-MeC_6H_4	93%
Ph	84%
4-ClC_6H_4	86%

Scheme 4

1.2 Anion based and related methods

Many of the anion related methods of preparing cyclopropanes that have been reported are based on the familiar reaction between an enone and the trimethylsulfoxonium ylide, and in this area the goal of stereoselective synthesis has predominated. An excellent example of this is the work of Pyne and co-workers[16] who used the chiral, lithiated *N*-tosyl-*S*-butyl-*S*-phenylsulfoximine **26** to synthesise cyclopropanes **25** from enones **24** in good yields, high diastereomeric ratios and excellent enantiomeric excesses (**Scheme 5**). Pedregal and co-workers[17] have produced diastereomerically pure cyclopropanes **28** from enones **27** using the previously reported ethoxycarbonylmethyl(dimethyl)sulfonium bromide **29** in a modified procedure. Along similar lines Huang and co-workers[18,19] have reported the use of the ylides **30** and **31** in the highly selective synthesis of two geometrical isomers of vinyl-substituted cyclopropanes **32** and **33** (**Scheme 6**).

A novel approach to cyclopropane synthesis has been developed by Zwanenburg and co-workers[20] whereby a vinyl aziridine is converted to a cyclopropane (**34**→**35**) *via* a Michael reaction initiated ring closure (**Scheme 7**). Trost *et al.*[21] have used palladium catalysis to prepare the enantiomerically pure bicyclic systems **36**.



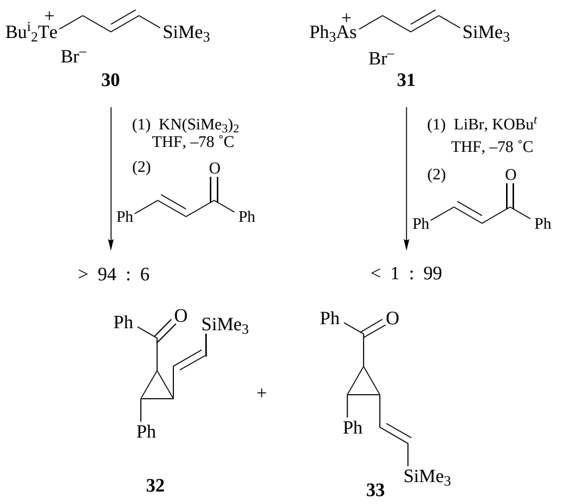
R	R'		Dr	ee
Ph	Ph	90%	99 : 1	–
Me	Ph	58%	99 : 1	–
Ph	Me	68%	96 : 4	>98%

n		
1	66%	100% de
2	55%	100% de
3	70%	100% de

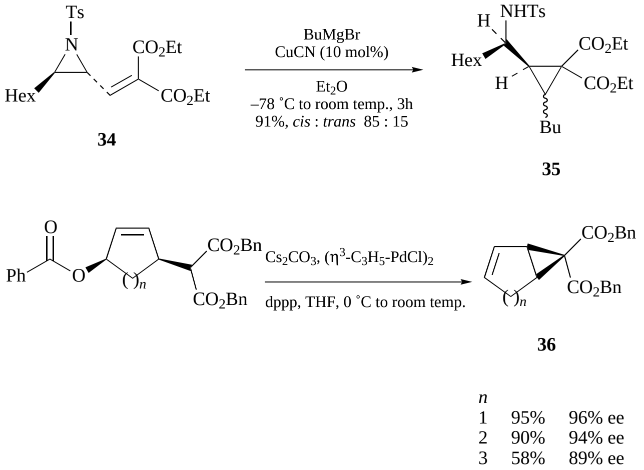
Scheme 5

1.3 Other methods

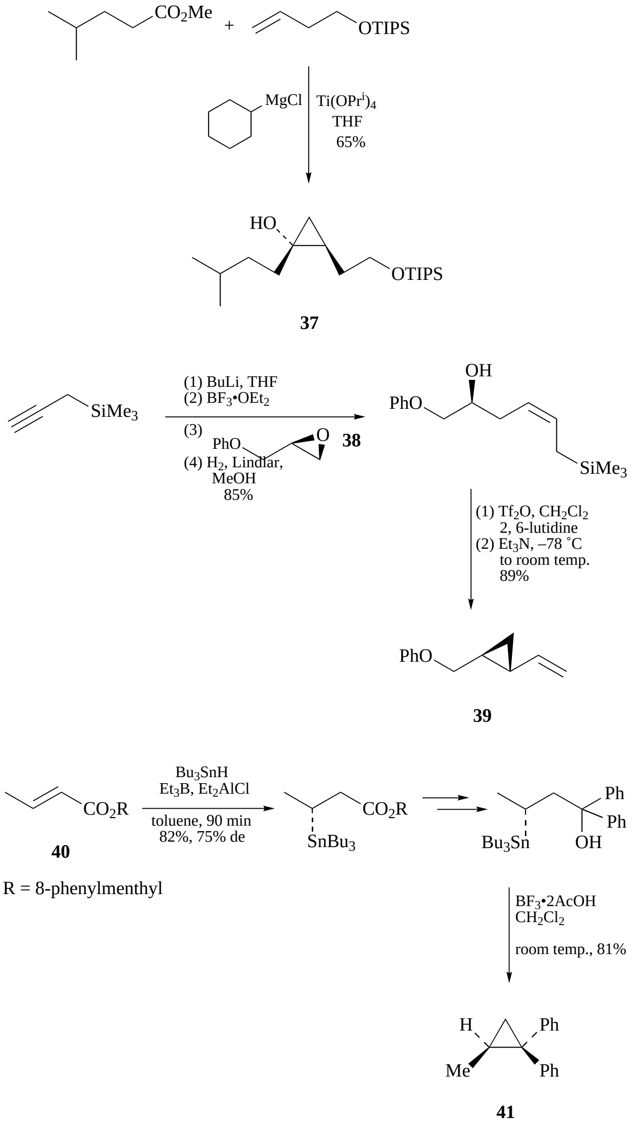
Several synthetically useful methods of preparing substituted cyclopropanes using titanium reagents have been reported. For instance, Cha and co-workers have developed the reductive coupling of terminal alkenes with amides to give cyclopropylamines,[22] with ethylene carbonate to give cyclopropane



Scheme 6



Scheme 7



Scheme 8

hemiketals,[23] and with carboxylic esters to give cyclopropanols[24] (*e.g.* **37**, **Scheme 8**). A related adaptation of the Kulinkovitch procedure has been reported by de Meijere and co-workers.[25] A useful reaction sequence for preparing oligocyclopropane units has been developed by Taylor *et al.*[26] whereby an epoxide, such as **38**, is converted into a vinyl cyclopropane, such as **39**. Optically active cyclopropanes have been synthesised by Nishida and co-workers[27] *via* β-stannyl esters (**40**→**41**) while Mori and Isono[28] have utilised a similar 1,3-elimination of a bis(tributylstannyl)propanol derivative.

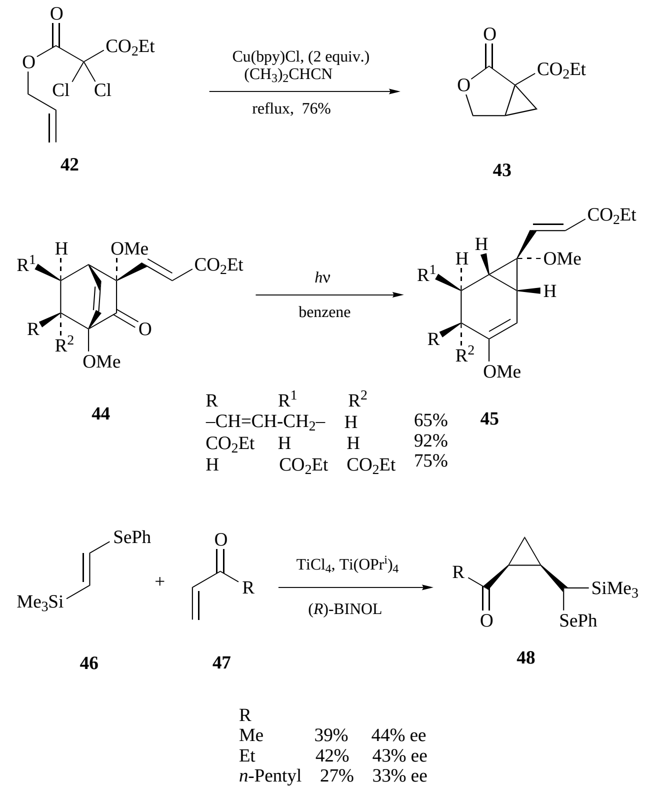
Bertrand and co-workers[29] have prepared 3-oxa- and 3-aza-bicyclo[3.1.0]hexan-2-ones *via* tandem radical cyclisation–intramolecular S_N2 reactions (**42**→**43**, **Scheme 9**). Synthesis of bicyclo[4.1.0]hept-2-enes **45** has been achieved by Yamuchi and co-workers[30] by the photochemical reaction of bicyclo[2.2.2]-oct-5-en-2-ones **44**. And finally asymmetric [2 + 1] cycloaddition reactions of 1-seleno-2-silylethene **46** with enones **47** have been used by Yamazaki *et al.*[31] to produce cyclopropanes **48** in moderate yields and enantiomeric excesses.

2 Four-membered rings

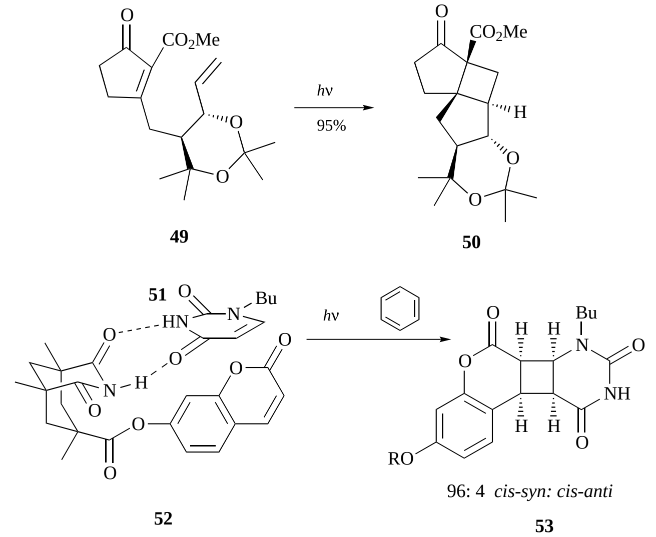
2.1 Photochemical methods

Stereoselectivity has been a strong theme in many of the [2 + 2] photocycloadditions reported over the review period. For example Crimmins *et al.*[32] have found that the chiral acetal **49** provides high levels of diastereocontrol in the cycloaddition to give **50** (**Scheme 10**). Piva, Pete and co-workers[33] have found that enantiomerically pure cycloadducts can be obtained using chiral hydroxy acids as spacers in intramolecular cycloadditions. Molecular recognition between the thymine derivative **51** and the chromone ester **52** has been used to great effect in controlling both the regio- and stereo-selectivity during the intermolecular cycloaddition[34] to **53**. Haddad *et al.*[35] found that a variety of dihydropyrones **54** undergo efficient cycloaddition to the corresponding cyclobutanes **55** (**Scheme 11**). El-Nabi[36] has demonstrated that a number of enol ethers undergo selective cycloaddition to the cyclic keto amide **56** to give predominantly the *exo*-isomers **57**. De Groot and co-workers[37,38] have found that transannular photocycloaddition provides a stereoselective route to tricyclic systems (**58**→**59** and **60**→**61**).

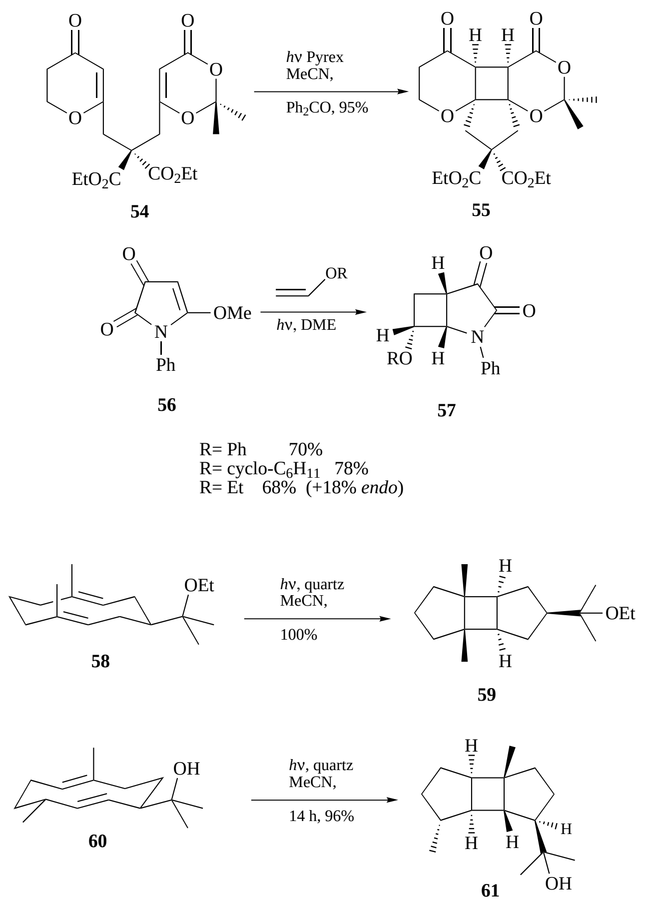
The use of optically active allenylsilanes[39] has proved useful for the formation of functionalised cyclobutanes in high ee (**62**→**63**) (**Scheme 12**). Hegedus and co-workers[40] have utilised the [2 + 2] photocycloaddition chemistry of chromium carbene complexes to good effect as the key step in the synthesis of (+)-cerulenin (**64**→**65**). The power of [2 + 2] photocycloaddition reactions for the synthesis of complex, strained molecular architectures has again been demonstrated by a number of groups.[41,42] For example (**Scheme 13**) Golobish and Dailey[43] were able to prepare the bishomohexaprismanedione ring system by such an approach (**66**→**67** and **66**→**68**). A very interesting enantioselective Norrish type II process has been reported which could lead to a useful synthesis of chiral cyclobutanols from achiral ketones. For example it was found[44] that irradiation of both the ketone **69** and (−)-ephidrine, within a zeolite host, gave the cyclobutanol **70** in 30% ee. Singh and Porinchu[45]



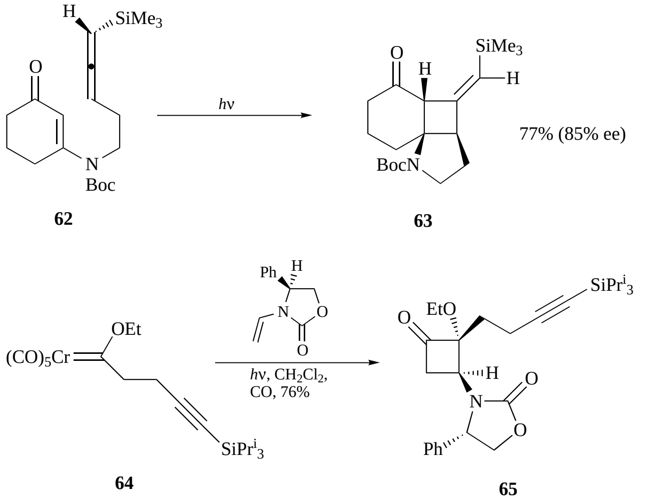
Scheme 9



Scheme 10



Scheme 11



Scheme 12

found that the cyclobutanone **72** could be prepared from the ketone **71** by a photoinduced 1,3-acyl shift.

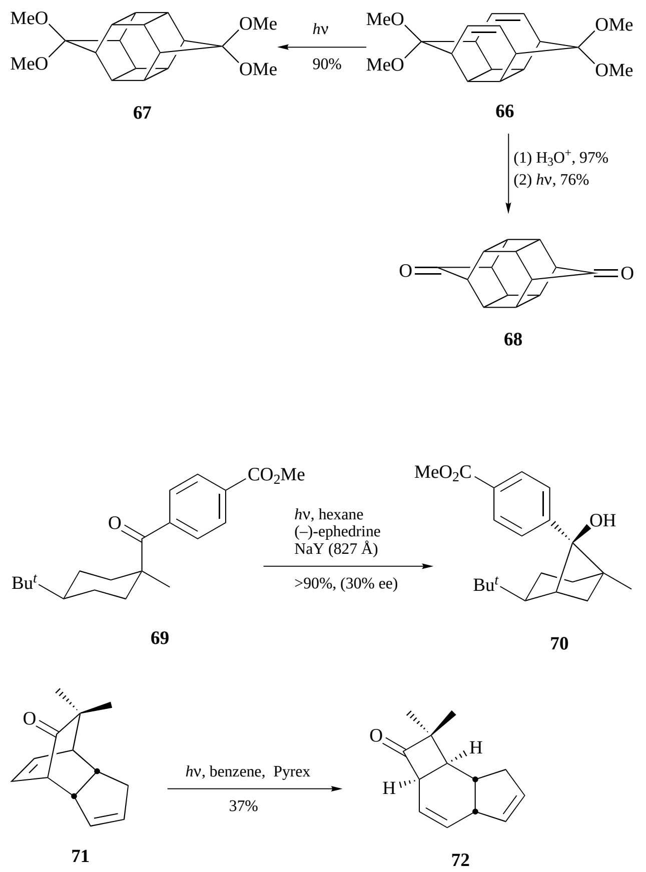
2.2 Other methods

Murphy and Neville[46] have reported the first examples of cyclobutane formation in the Lewis acid catalysed cycloaddition between styrenes and naphthoquinones (**73**→**74**, **Scheme 14**). Rhodium acetate[13] was found to convert diazo esters into the corresponding, highly strained, cyclopropane fused cyclobutanes. Snapper and co-workers[47] have found that alkenyl tethered cyclobutadienes, prepared *in situ*, undergo facile [2 + 2] cycloaddition to give the corresponding bicyclo[2.2.0]-hexenes (**75**→**76**). McNab and co-workers[48] discovered that FVP of the 2-alkenylpyrrole **77** gave the cyclobutane **79** by way of thermal [2 + 2] cycloaddition of the intermediate **78**. Negishi and co-workers[49,50] have found that a number of alkyl alkenyl diiodides gave high yields of cyclobutenes on metal–halogen exchange with *tert*-butyllithium (**80**→**81**, **Scheme 15**). Finally, Hoffmann and co-workers[51,52] reported that a number of diketones undergo facile SmI_2 promoted pinacolisation (**82**→**83**) to the corresponding cyclobutanediols. The process is not just limited to symmetrical diketones, for example treatment of the *cis*-hydrindione **84** with SmI_2 gave a rather neat entry to the norylangane skeleton **85**.

3 Five-membered rings

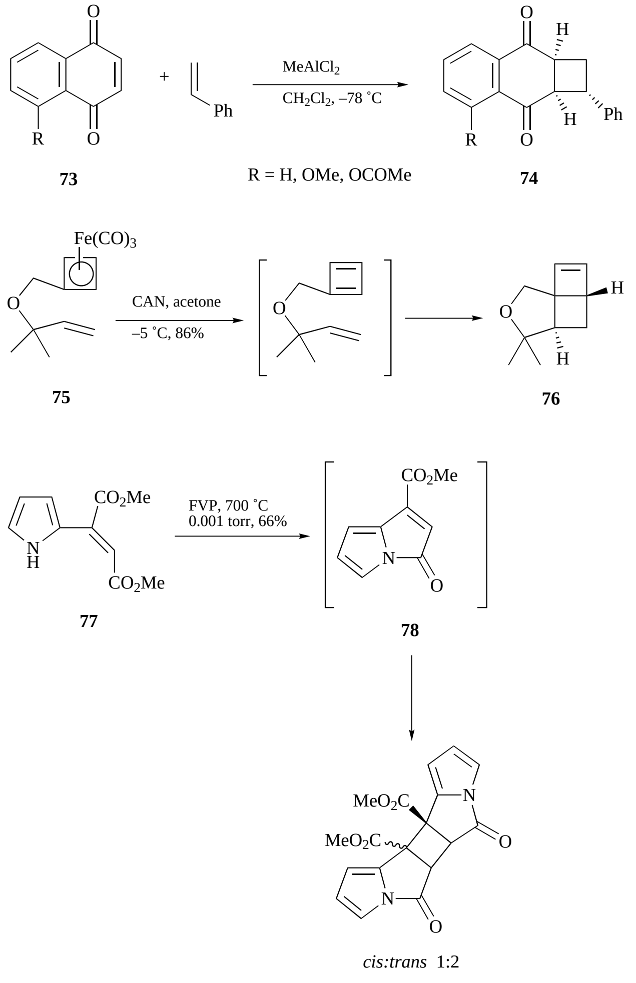
3.1 Free radical methods

Free radical cyclisation remains a very popular method of cyclopentane synthesis and new work in this area comprises studies of alternatives to the standard tributylin hydride method of radical generation, the use of chiral reagents to

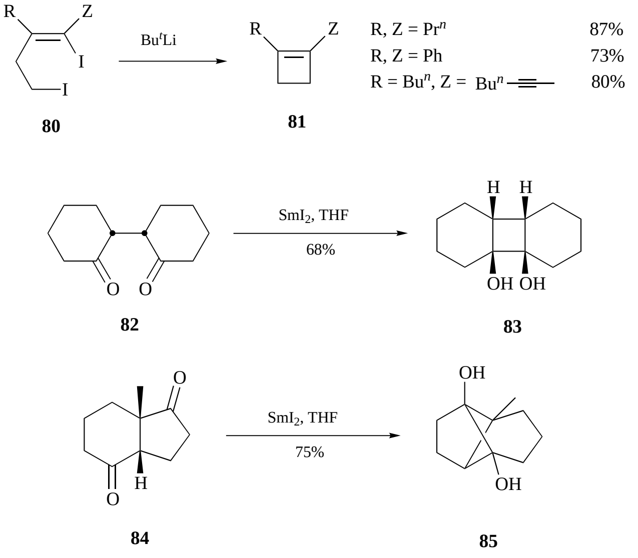


Scheme 13

confer stereoselectivity and the application of existing techniques to natural product synthesis. In the first of these areas, Nishida *et al.*[53] have used the photolysis of a thioacetal group for radical generation (**86**→**87**, **Scheme 16**), while the irradiation of aryl telluride carbohydrates has been developed by Quiclet-Sire and co-workers[54] for the synthesis of carbocycles (**88**→**89**). Photochemical initiation has also been utilised by Pattenden and co-workers[55] in their synthesis of bicyclo[6.3.0]-undec-4,9-dienes **91** from humulene **90**. Little and Lin[56] have extended the field of intermolecular diyl trapping reactions in the synthesis of bicyclic systems to include allene diylophiles. Booker-Milburn *et al.*[57] have developed their iron(III) mediated ring expansion–cyclisations of cyclopropyl ethers to include cyclopropanone acetals (**92**→**93**).

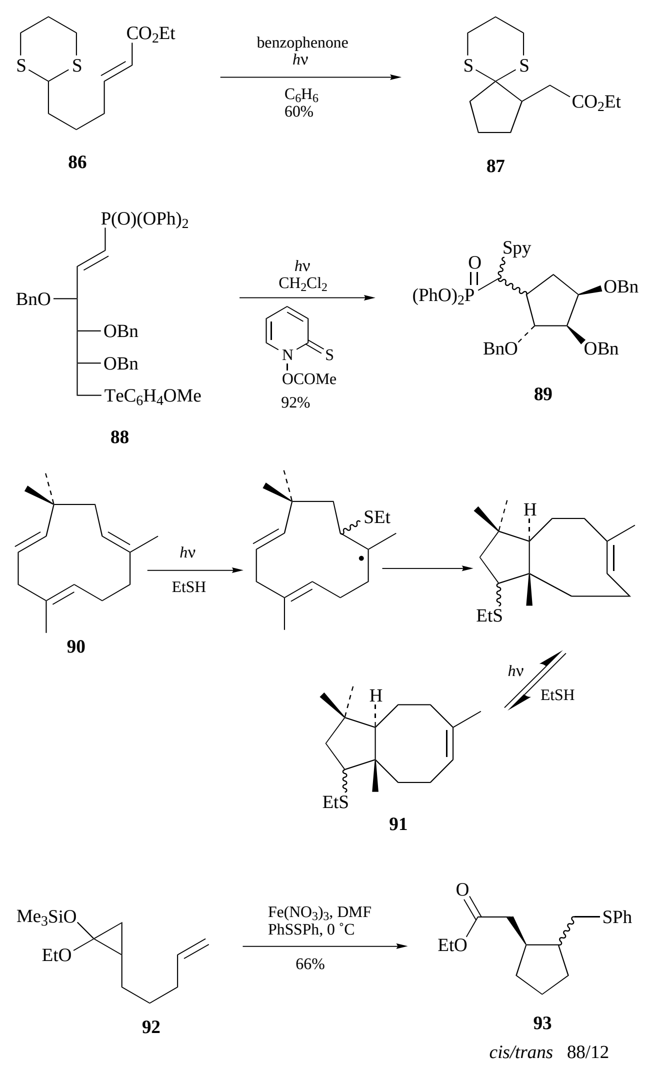


Scheme 14

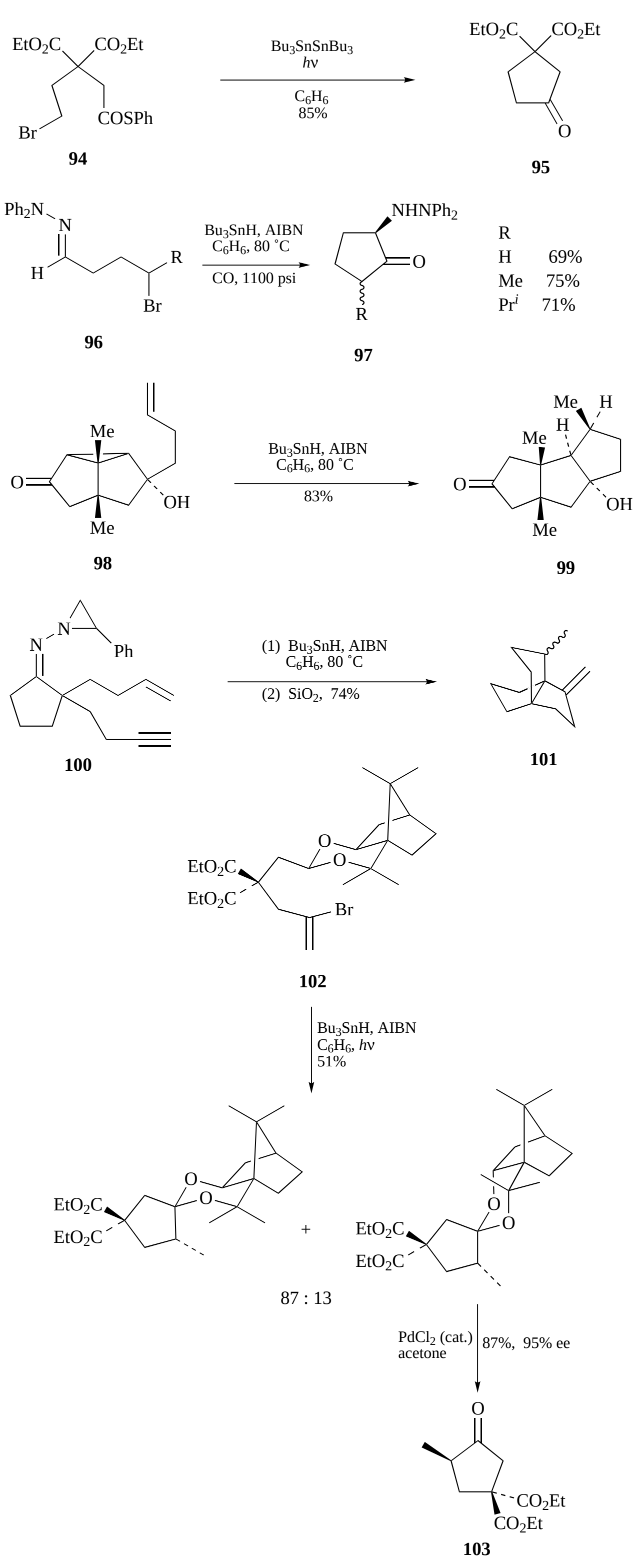


Scheme 15

Two other methods of radical generation have been reported: bromotrichloromethane has been used by Weavers and co-workers[58] in the preparation of haloalkylidene cyclopentanones, and toluene-*p*-sulfonyl bromide has been used by Caddick *et al.*[59] in the synthesis of *p*-tolysulfonyl-substituted dienes. The radical cyclisation of thio- and seleno-esters has been investigated by Kim and Jon,[60] thus cyclopentanone **95** is prepared from thioester **94** in a very good yield (**Scheme 17**). A very neat tandem radical reaction for the preparation of cyclopentanones **97** has been devised by Fallis and Brinza[61] in which the radical generated from bromide **96** reacts first with carbon monoxide before the resulting acyl radical then cyclises on to the *N*,*N*-diphenylhydrazone group. Enholm and Jia[62,63] have made use of the radical ring openings of functionalised cyclopropanes; thus generation of the *O*-stannyl radical from cyclopentanone **98** results in a fragmentation–cyclisation reaction sequence to produce the linear triquinane skeleton **99**. A formal total synthesis of *dl*-modhephene has been reported by Kim and co-workers[64] in which the [3.3.3]propellane **101** is produced from *N*-aziridinyl imine **100** *via* a tandem radical cyclisation. Other syntheses of note involving radical cyclisations include a new approach to the steroid BCD-ring system reported by Takahashi *et al.*[65] and the total synthesis of 15(*RS*)-5,6-dehydro-8-*epi*-PGF$_{2\alpha}$ methyl ester devised by Rossi and co-workers.[66] Finally, in the field of stereocontrol, Crich and co-workers[67,68] have reported the use of the 1,3-dioxan-2-yl compounds, such as **102**, for the generation of chiral acyl radical equivalents which after subsequent radical exchange cyclisation and deprotection give cyclopentanones, such as **103**, in good yields and very high enatiomeric excesses.



Scheme 16



Scheme 17

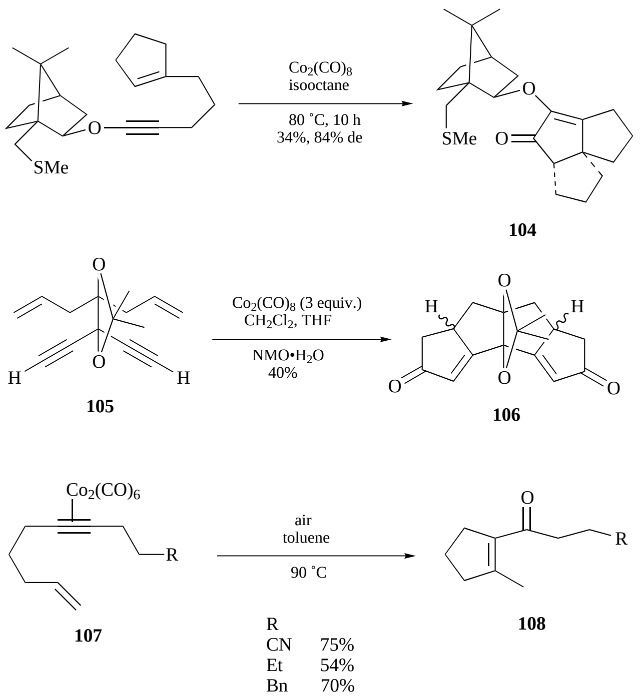
3.2 Metal based methods

3.2.1 Cobalt

As in previous years, the most commonly reported method of forming five-membered rings using cobalt has been the Pauson–Khand reaction (PKR). Of particular interest is the stereocontrolled synthesis of the angular triquinane **104** developed by Moyano and co-workers[69] that uses a chiral auxiliary to obtain a very good diastereomeric excess (**Scheme 18**); the same group[70] has had similar success in the related synthesis of (+)-β-cuparenone. Cook and Van Ornum[71] have used a double PKR to generate four five-membered rings (**105**→**106**) in studies directed to the synthesis of dicyclopenta[*a*,*d*]pentalene, while Kraff *et al.*[72] have reported a novel development in which cobalt complexes **107** are subjected to conditions that result in an interrupted PKR affording cyclopentenes **108** in good yields.

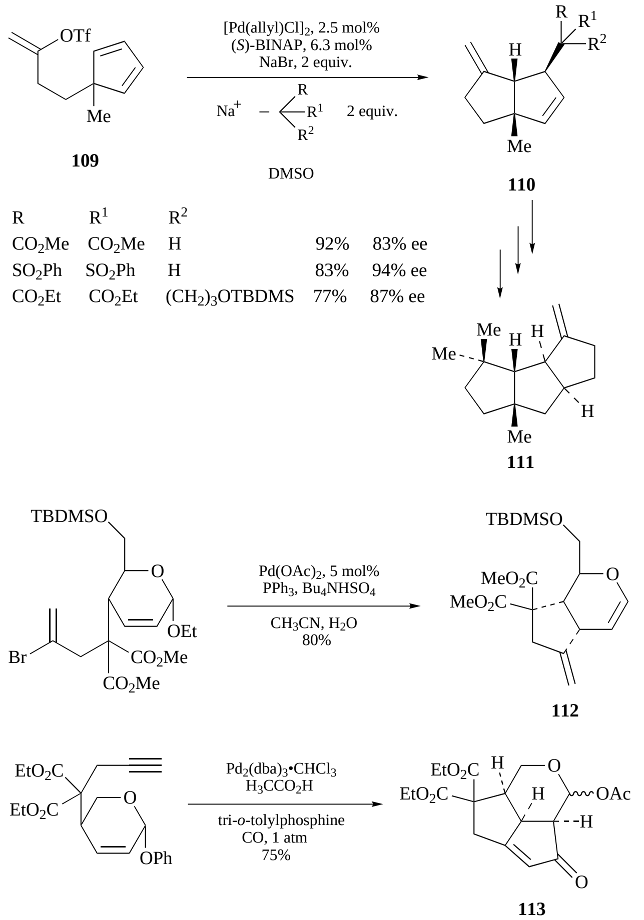
3.2.2 Palladium

Probably the most interesting development in five-membered ring synthesis involving palladium chemistry is that reported by Shibasaki and co-workers[73] in which triflate **109** is subjected to an asymmetric Heck reaction–carbanion capture process resulting in bicycles **110** in very good yields and enantiomeric excesses (**Scheme 19**). This methodology along with a radical cyclisation has resulted in a catalytic asymmetric total synthesis of the linear triquinane (−)-$\Delta^{9(12)}$-capnellene **111**. Two groups have studied stereocontrolled palladium catalysed cyclisations on carbohydrate templates. Sinou and co-workers[74] have prepared enantiopure bicyclic compounds such as **112**, and Holzapfel *et al.*[75] have extended this methodology to include cyclisation–carbonylation reactions as in the synthesis of tricycle **113**. Negishi *et al.*[76] have also developed such cyclisation–carbonylation techniques for the preparation of cyclopentenones as well as conducting in depth studies of the palladium catalysed cyclisation of 1-iodo-substituted dienes. The intramolecular hydrocarbonation of allenes leading to carbocycles has been examined by Yamamoto and co-workers[77] while Kibayashi[78] have looked at the tandem cyclic carbopalladation–vinylation of enynes, and a triple palladium catalysed cyclisation of enediyne **114** has been used by Oh *et al.*[79] to synthesise the linear triquinane **115** (**Scheme 20**). Finally Reissig and co-workers[80] have prepared indane deriv-



R	
CN	75%
Et	54%
Bn	70%

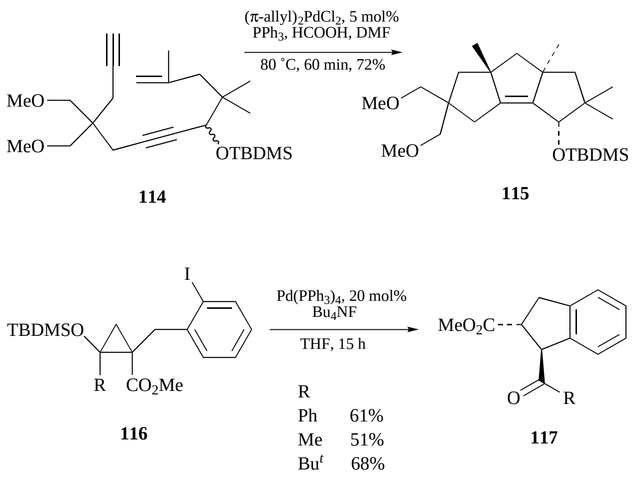
Scheme 18



R	R^1	R^2		
CO_2Me	CO_2Me	H	92%	83% ee
SO_2Ph	SO_2Ph	H	83%	94% ee
CO_2Et	CO_2Et	$(CH_2)_3OTBDMS$	77%	87% ee

Scheme 19

atives **117** from substituted siloxycyclopropanes **116** using palladium chemistry.



R	
Ph	61%
Me	51%
Bu^t	68%

Scheme 20

3.2.3 Titanium

Following on from some of the early developments reported last year, five-membered ring synthesis mediated by titanobicycles has all but replaced that mediated by zirconobicycles and as before the groups of Sato and Buchwald have led the work in this field. Sato and co-workers have used a (μ-propene)-$\mathrm{Ti(OPr^i)_2}$ complex formed *in situ* to effect the tandem cyclisation of 2,7-bis-unsaturated esters to bicyclo[3.3.0.]octane or bicyclo[3.1.0]hexane systems[81] (**118**→**119**, **Scheme 21**) and to synthesise β-alkynyl- and β-ethylidenyl-cyclopentanols.[82] Much of the work of Buchwald and co-workers has been in the area of titanocene mediated or catalysed cyclocarbonylations. Thus the diastereoselective synthesis of γ-butyrolactones **121** from enones **120** has been reported[83] as well as the conversion of enynes to cyclopentenones.[84] Continuing this line of research, the most important development has been the use of the chiral catalyst (*S*,*S*)(EBTHI)Ti(CO)$_2$ **123** [generated *in situ* from (*S*,*S*)(EBTHI)TiMe$_2$ and carbon monoxide, where EBTHI refers to 1,1′-ethylenebis(4,5,6,7-tetrahydroindene)] in the highly enantioselective Pauson–Khand type formation of bicyclic cyclopentenones such as **122**.[85]

3.2.4 Samarium

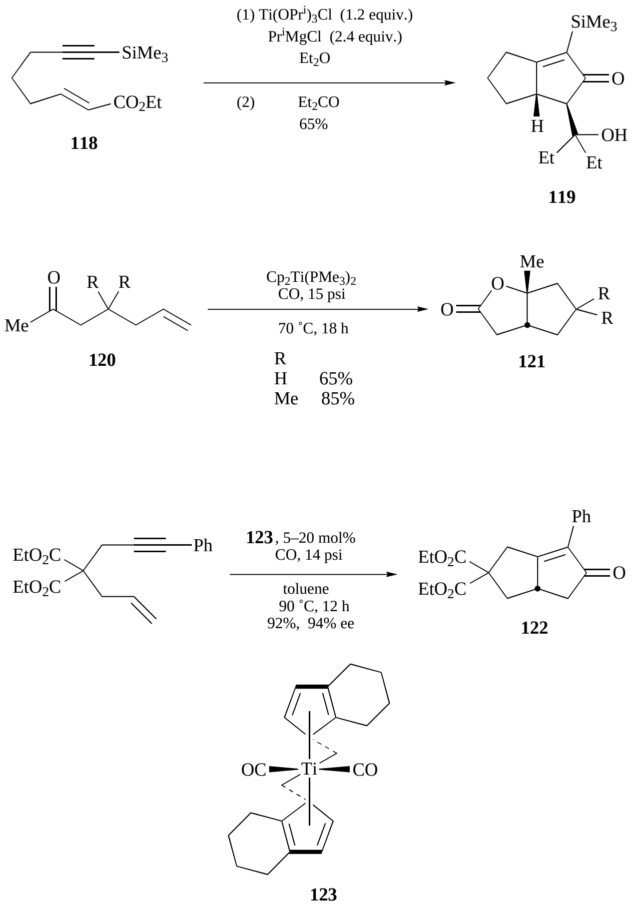
As in previous years the samarium(II) iodide mediated coupling of carbonyl compounds with alkenes has been well represented in the recent literature. Molander and co-workers have been particularly active in this area. For instance bicyclic systems **125** have been prepared from vinyl epoxides **124** in good yields and with very high de values using this reagent (**Scheme 22**).[86] Tandem nucleophilic acyl substitution–ketyl–olefin coupling reactions (**126**→**127**) have also been developed.[87] The same group has also investigated the stereocontrol in ketyl–olefin cyclisations using a chiral auxiliary[88] while Aurrecoechea *et al.*[89] have looked at the diastereoselective synthesis of cycloalkylamines.

3.2.5 Other metals

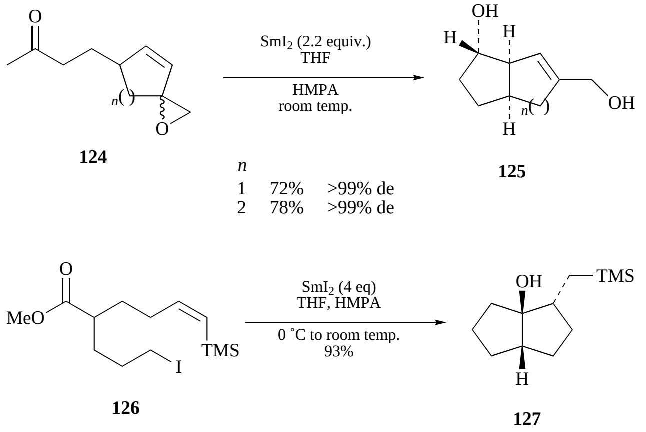
Manganese mediated cyclopentane syntheses have made several appearances in the literature this year. For example Knochel and co-workers[90] have used mixed metal (manganese–copper) catalysis to stereoselectively transform bromoalkenes into cyclopentanes (**128**→**129**, **Scheme 23**), while Alper and Okuro[91] have investigated intramolecular manganese(III) induced carbon monoxide trapping reactions of alkynes with malonate and cyano ester units. Chung and co-workers[92] have synthesised cyclopentanoids using a methylmanganese carbonyl complex. An interesting enantioselective synthesis of β-substituted cyclopentanones **131** from 4-substituted pent-4-enals **130** has been devised by Bosnich and co-workers[93] using the chiral rhodium catalyst **132**. In the field of alkene metathesis, stereoselective syntheses of five-membered rings have been reported using tungsten[94] and ruthenium[95,96] catalysts.

3.3 Anion based methods

The standard anion based methods (aldol, Michael and

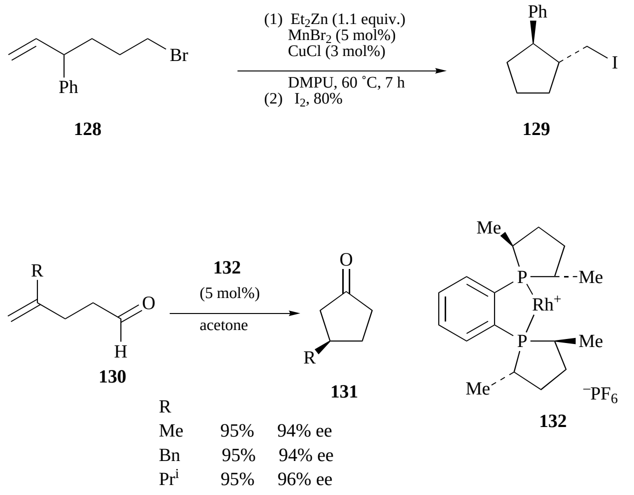


Scheme 21

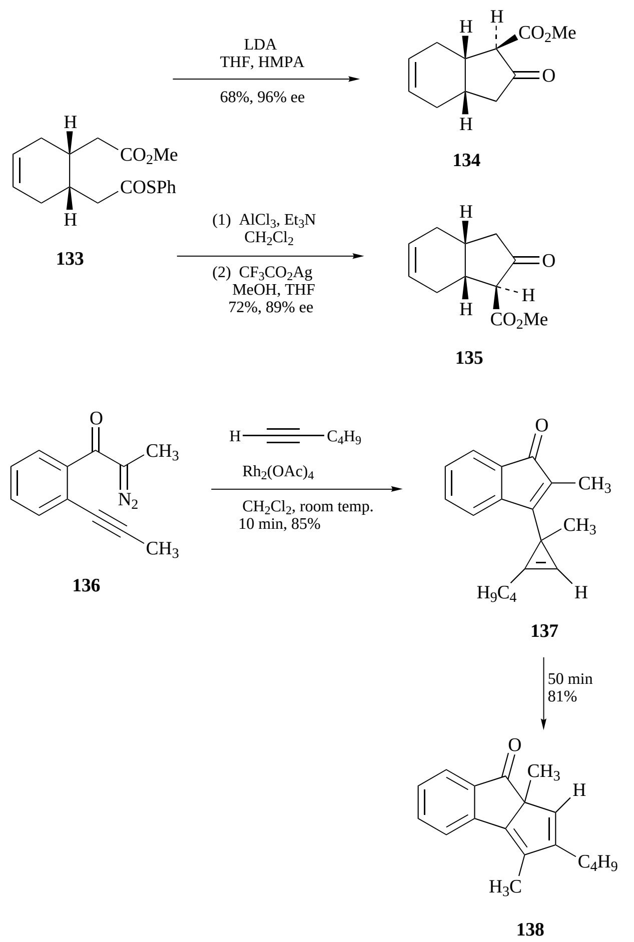


Scheme 22

Dieckmann) of carbon–carbon bond forming have all been much used in five-membered ring synthesis this year particularly in the area of natural product synthesis. Of the more novel examples, many have been extensions of methodology reported in previous years; however there have been a few new developments. A neat enantiodivergent procedure utilising the chemoselective Dieckmann-type cyclisation of chiral monothiol diesters, such as **133**, to give cyclopentenones, such as **134** and **135**, has been devised by Nagoo and co-workers (**Scheme 24**).[97] Yeh *et al.*[98] have diastereoselectively synthesised trisubstituted cyclopentanecarboxylic acid derivatives from an iron tricarbonyl complex while Normant and co-workers[99] have reported a general diastereoselective synthesis of small rings *via* an intramolecular cabolithiation of silylated enynes. As for carbenoid research, Padwa *et al.*[100] have reported many examples of cyclisation reactions involving rhodium complexes, for instance **136**→**137**→**138**.



Scheme 23



Scheme 24

3.4 Ring expansions and rearrangements

The one-carbon ring-expansion of a cyclobutane to a cyclopentane remains a useful synthetic strategy. Trost and Chen[101] have taken this one stage further to include a second ring-forming reaction to prepare several polycyclic systems (**139**→**140** and **141**, **Scheme 25**). Kim and Uh[102] have developed a general thallium ion mediated procedure for the conversion of 1-trimethylsilyloxy-1-alkenylcycloalkanes to α-*exo*-methylenecycloalkanones. Many examples of five-membered ring formation involving a rearrangement reaction (often resulting in a formal ring contraction) have appeared in the literature this year. A very neat enantioselective method for preparing bicyclic systems **143** from *meso*-epoxides **142** has been devised by Hodgson and Lee[103] while Mander and co-workers[104] have used a photochemical-induced one-carbon ring contraction of a diazocyclohexanone in their synthesis of gibberellins. Singh and co-workers[105,106] have also used photochemistry in their efficient routes to tricyclopentanoids and tetraquinanes (**144**→**145**). The arene–alkene *meta*-photocycloaddition reaction has been utilised by Wender *et al.*[107] in their impressive first synthesis of *cis*,*cis*,*cis*,*cis*,*trans*-[5.5.5.5]fenestrane **146** (**Scheme 26**). Chanon and co-workers[108] have also used this photomethodology in a synthesis of ceratopicanol. The oxy-Cope rearrangement has also found use in the preparation of five-membered rings with Paquette and co-workers[109–116] publishing a plethora of papers on their elegant syntheses of linear and angular polyquinanes from squarate esters and Moore and Santora[117] continuing their studies in this area. Finally Uguen and co-workers[118,119] have reported the formation of cyclopentenes **148** *via* the ene reaction of allenic sulfones **147**.

3.5 Cycloadditions and annulations

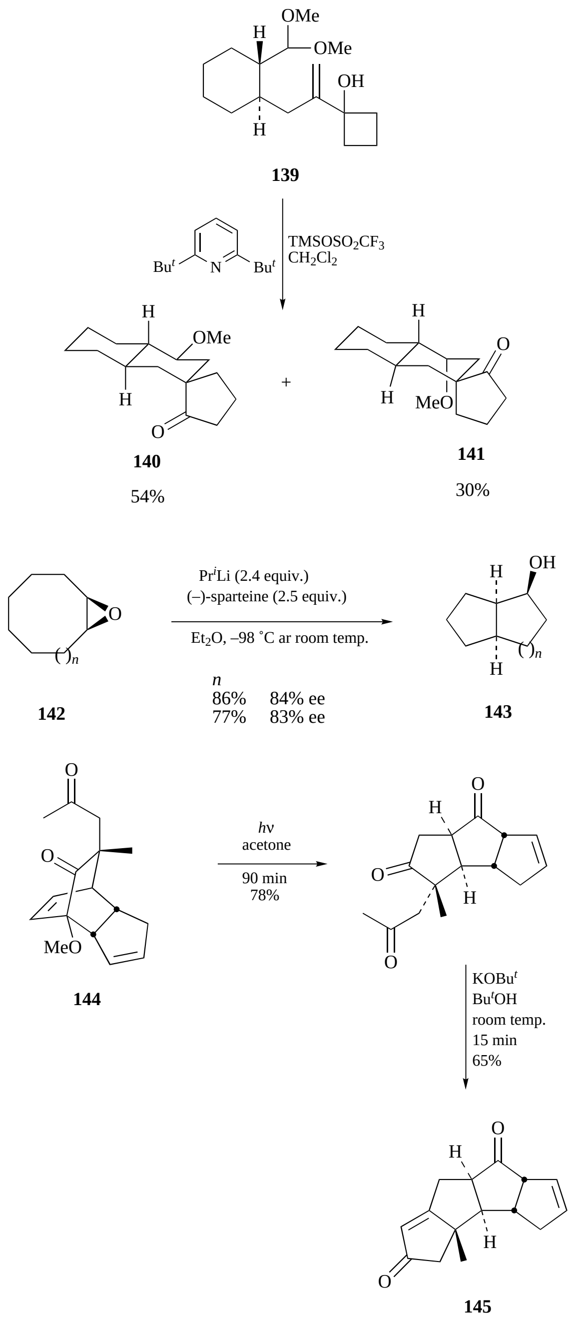
The use of cycloadditions and annulations for the construction of five-membered rings has been particularly popular this year. An outstanding example is the novel asymmetric [3 + 2] cycloaddition reported by Zhang and co-workers[120] in which, for example, the buta-2,3-dienoate **149** reacts with the electron-deficient olefin **150** catalysed by the chiral 2,5-dialkyl-7-phenyl-7-phosphabicyclo[2.2.1]heptane **152** giving cyclopentene **151** in high yield and high enantiomeric excess (**Scheme 27**). Murakami *et al.*[121] have also used allenes **153** in an asymmetric [4 + 1] cycloaddition with carbon monoxide using rhodium catalysis to prepare cyclopentenols **154** in excellent yields and enantiomeric excesses. Palladium catalysed intramolecular [3 + 2] cycloadditions of methylenecyclopropanes have been studied by Lautens and Ren[122,123] while Trost and Higuchi[124] have used an intramolecular palladium catalysed trimethylenemethane [3 + 2] cycloaddition in their synthesis of the perhydroazulene (−)-isoclavukerin A. The rapid and dramatic construction of multi-ring systems *via* photo-induced tandem [6 + 2], homo[6 + 2] cycloadditions of alkynes with chromium(0) tricarbonyl complexes of cycloheptatrienes has been independently reported by two groups; thus Rigby *et al.*[125] have effected the conversion **155**→**156** and Sheridan and co-workers[126] have prepared **158** from **157** using the same methodology (**Scheme 28**).

Moving on to annulations, Ila and co-workers[127] have reported many examples of cyclopentene synthesis using a Lewis acid assisted rearrangement of donor–acceptor substituted cyclopropanes (**159**→**160**, **Scheme 29**). Meyers and Brengel[128] have used tritylallyldimethylsilane–$TiCl_4$ annulations with electron-deficient olefins **161** to prepare diastereomerically pure cyclopentanols **162**. Finally Padwa *et al.*[129] have devised an innovative synthesis of fused cyclopentenes **165** from 2,3-bis(phenylsulfonyl)buta-1,3-diene **164** and alkenes **163** *via* an anion based annulation (**Scheme 30**).

4 Six-membered rings

4.1 Intermolecular Diels–Alder reactions

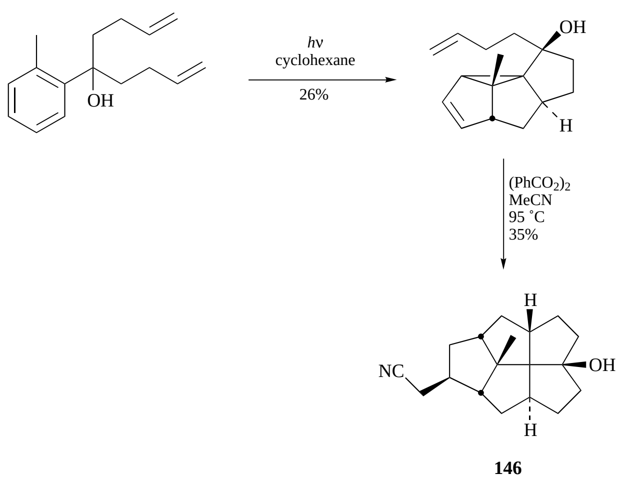
A number of interesting and unusual examples of the Diels–Alder (DA) reaction have been reported over the review period. For example the highly strained bromocyclopropenone **167** underwent rapid cycloaddition[130] with the isobenzofuran **166** to give a dione after hydrolytic work up. Reduction of this dione with tributyltin hydride completed a concise synthesis of (±)-favelonone **168** (**Scheme 31**). In a similar vein the first reported preparation[131] of cycloocta-1,2,3,-triene **169** was proved by trapping as the DA adduct **170**.



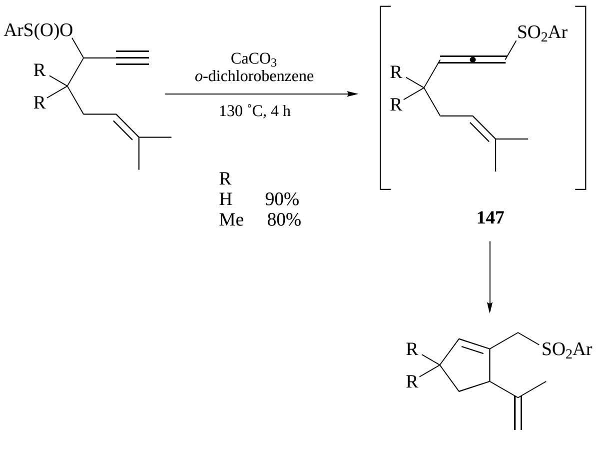
Scheme 25

Muller and Waldmann[132] reported a phenol hydroxylation–oxidation–Diels–Alder cascade using a tyrosinase enzyme (**171**→**172**, **Scheme 32**). The α,β-unsaturated sultone **173** was found to be an efficient dienophile,[133] giving rise to highly functionalised adducts **174** in good yield. Percy and co-workers[134] have shown that a number of substituted difluoromethylenephosophonato alkenes undergo cycloaddition with reactive dienes. The 2-pyridiniobutadiene **175** exhibited both diene and dienophile activity with cyclopentadiene.[135] The adduct **176** formed at lower temperature could be converted into **177** on heating.

The cycloaddition mechanism of ketenes has at last been solved, and has been shown[136] to proceed by initial [4 + 2] cycloaddition followed by facile [3,3] sigmatropic rearrangement to the usually observed [2 + 2] adduct (**178**→**179**→**180**, **Scheme 33**). Good NMR evidence of the intermediate [4 + 2] cycloadduct **179** was provided. Winkler and co-workers[137,138] have utilised an inter-intra–molecular Diels–Alder sequence for the concise synthesis of cyclopropyl taxane analogues (**181**→**182**→**183**). Over the review period there have been a number of catalysts reported[139,140] for the enantioselective Diels–Alder reaction. For example Corey and co-workers[141] reported that **184** was a super-Lewis acidic catalyst giving



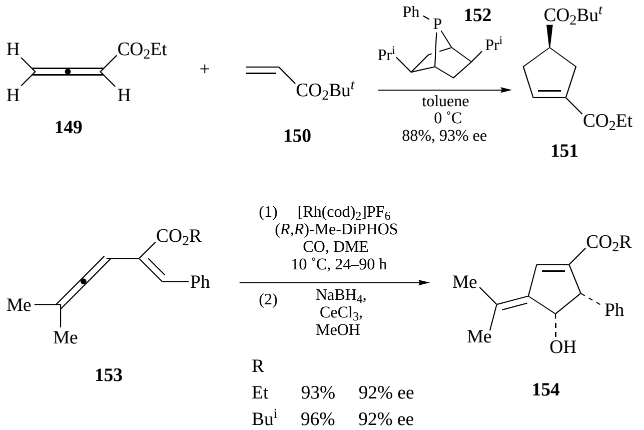
146



R	
H	90%
Me	80%

148

Scheme 26



R		
Et	93%	92% ee
Bu^i	96%	92% ee
Bn	94%	95% ee

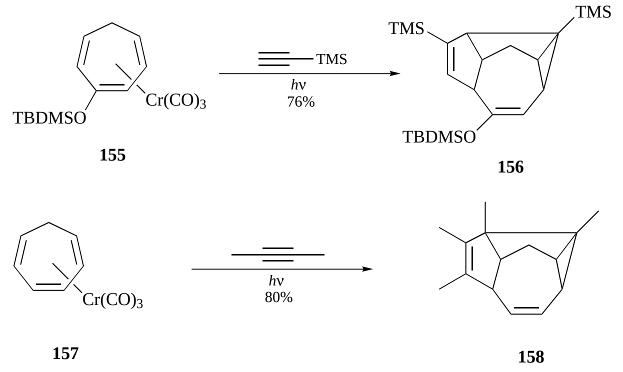
Scheme 27

high ee values in a number of cycloadditions. The resin bound catalyst **185** was found to give high ee values in the cycloaddition between methacrolein and cyclopentadiene.[142]

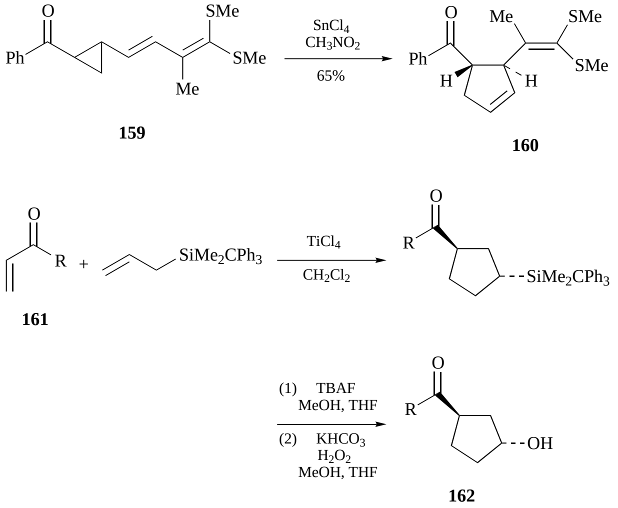
4.2 Intramolecular Diels–Alder reactions

Two different tandem Diels–Alder reactions of bis-furans have been reported from the laboratories of Vogel[143] (**186**→**187**) and Lautens (**188**→**189**, **Scheme 34**).[144] These reactions are noteworthy for both the stereoselectivity of the cycloaddition and complexity of the products obtained.

Ethenesulfonyl chloride **191** has been found to be a useful dienophile[145] with the amino substituted furans **190** (**Scheme 35**). The reaction proceeds *via* formation of the ethene sulfon-

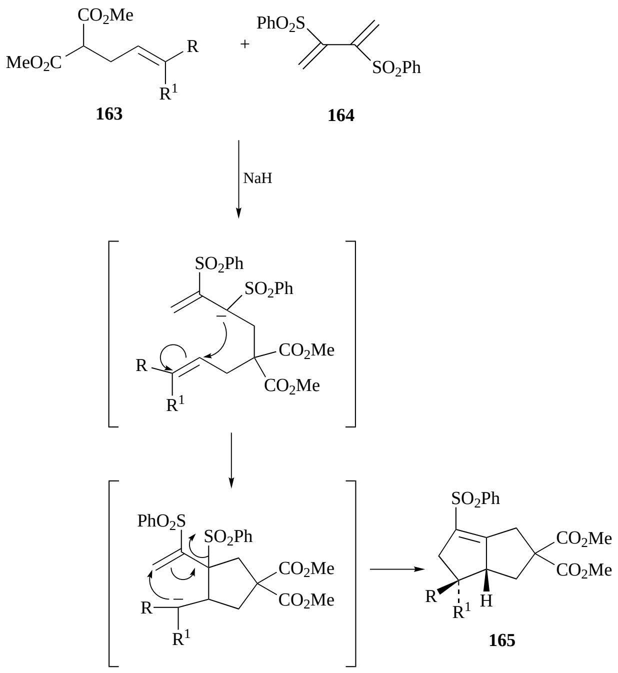


Scheme 28



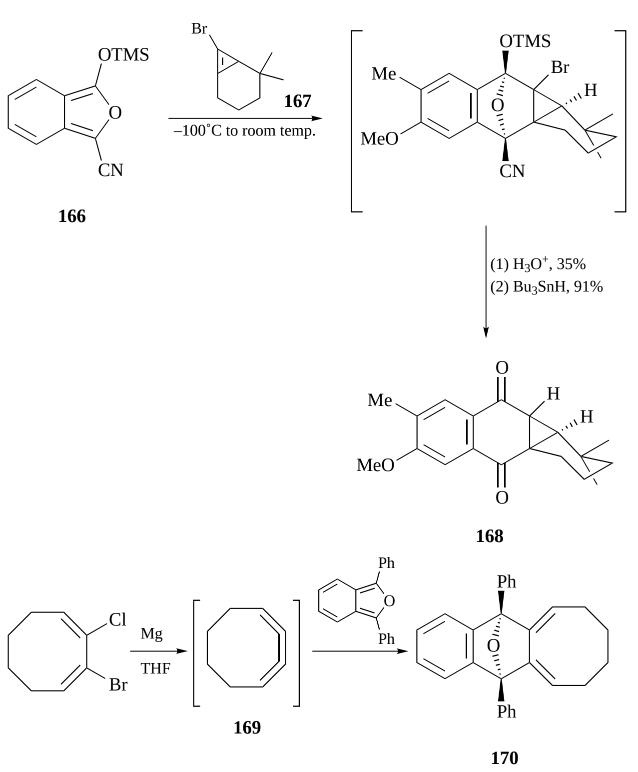
R	
CH$_2$Bu^t	45%
c-C$_5$H$_9$	46%

Scheme 29



R	R^1	
CO$_2$Me	H	74%
H	CN	91%
(*E*)-CHCHCO$_2$Et	H	78%

Scheme 30



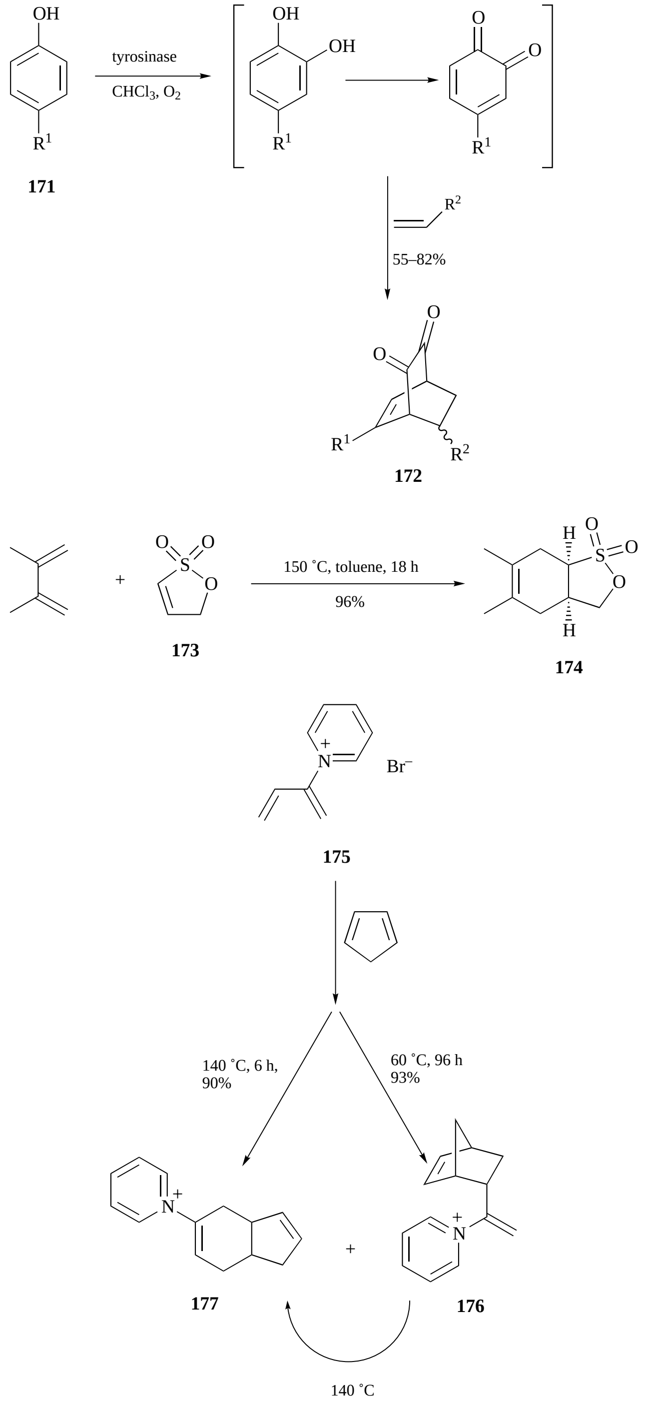
Scheme 31

amide **192** followed by intramolecular cycloaddition. In a related system Sammes and co-workers[146] have made the important observation that the trityl group acts as a steric buttress, greatly accelerating the intramolecular Diels–Alder (IMDA) reaction (**193**→**194**). Where R = H there was no reaction even after several days in boiling toluene.

Stork *et al.*[147] have used the IMDA reaction to good effect in the stereoselective cycloaddition of the thioketene acetal **195** to give the tricyclic steroidal core **196** (**Scheme 36**). The triene **197** underwent spontaneous IMDA cycloaddition to give the ketone **198** on Dess–Martin oxidation.[148] Rodrigo and co-workers[149] found that oxidation of the thioketone **199** with PhI[CF$_3$CO$_2$]$_2$ in the presence of the dienol **200** gave the complex cycloadduct **201**. The reaction is thought to proceed *via* IMDA of the initially formed *ortho*-quinonoid monoketal, and was found to proceed readily with a number of different substrates.

Roush *et al.*[150] reported a highly efficient and stereoselective transannular Diels–Alder reaction in their synthetic studies towards margenicin A$_1$ (**202**→**203**, **Scheme 37**). Roush and Works[151] also reported a very interesting tandem Claisen ring contraction–transannular Diels–Alder sequence (**204**→**205**). Denmark[152] has demonstrated that cyclohexanes can be formed *via* the tandem [4 + 2]–[3 + 2] cycloaddition reactions of nitroalkenes (**206**→**207**).

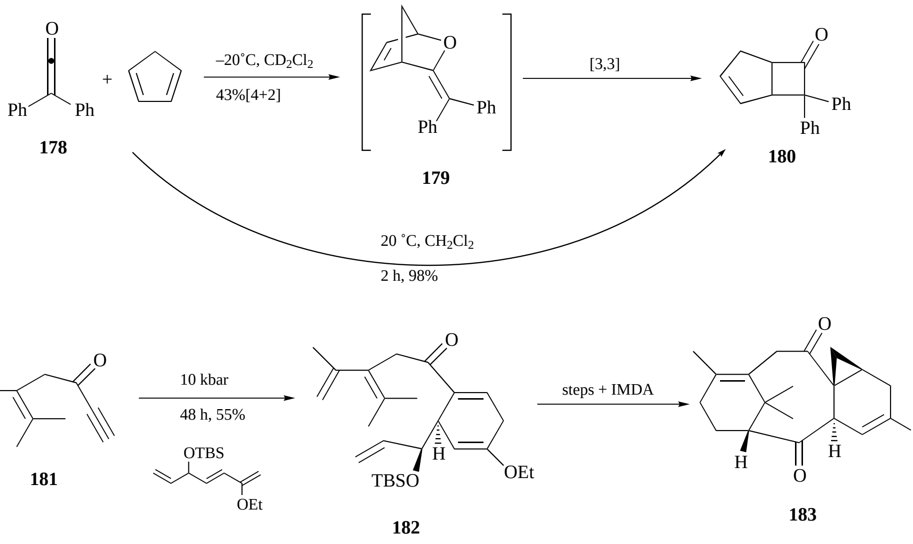
A one pot tandem Pictet–Spengler–Diels–Alder sequence has been used to good effect by Leonard *et al.*[153] for the rapid assembly of apoyohimbines (**208**→**209**, **Scheme 38**). Craig and co-workers[154] have published full details of their investigations into acetal tethered IMDA reactions. Deagostino *et al.*[155] have made the surprising observation that even with a tether of six atoms the IMDA reaction results in a highly stereoselective synthesis of the corresponding 6,8-cycloadduct (**210**→**211**, Scheme 38). Harano and co-workers[156] have described full details of their tandem [3,3] sigmatropic rearrangement–IMDA reactions of unsaturated xanthates (**212**→**213**, **Scheme 39**). Finally, the photosensitised [4 + 2] enone–arene cycloaddition of the α-methylbenzyl substituted ureas **214** has been shown to proceed in a highly stereoselective manner, giving the resulting cycloadducts **215** in reasonable yields.[157]



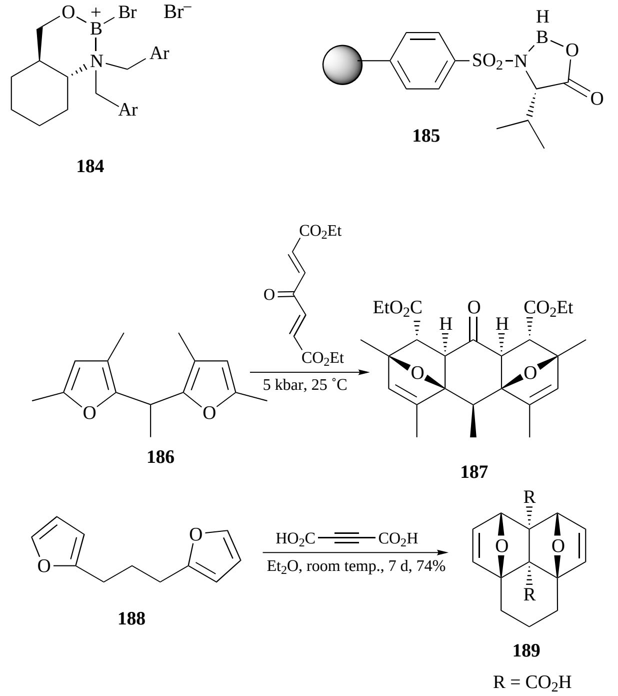
Scheme 32

4.3 Free radical cyclisation

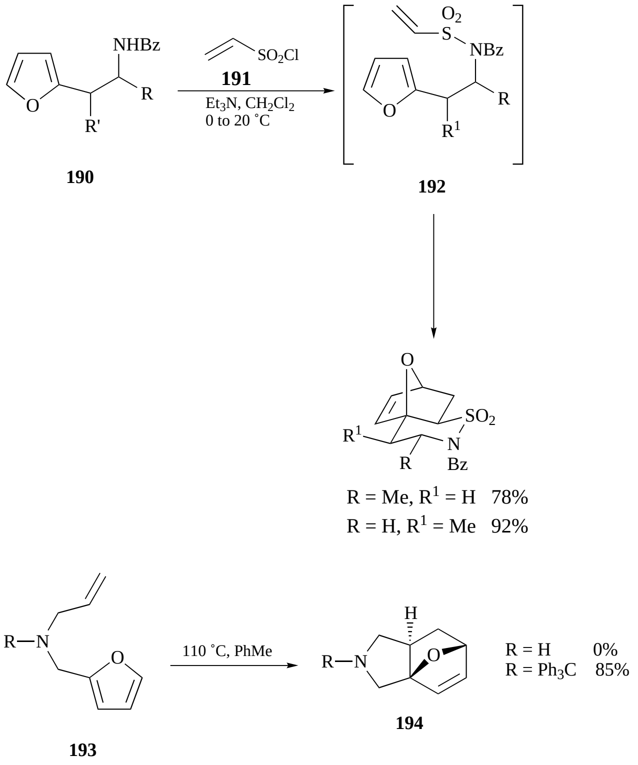
Interest in free radical methods for cyclohexane construction has again been high over the review period. For example, Curran and Martinez-Grau[158] have reported full details of their unimolecular chain transfer cyclisations of silicon hydrides. For example, treatment of the iodide **216** with hexabutylditin gave the (*E*)-alkene **217** exclusively (**Scheme 40**). Tsai *et al.* reported[159] a very elegant sequence involving the radical equivalent of the Brook rearrangement. For example, treatment of the acylsilane **218** with Bu$_3$SnH (TBTH) (Et$_3$B–air initiation) gave the hydrindane system **220** *via* 6-*exo* cyclisation onto the acyl carbon followed by Brook rearrangement to the carbon centred radical **219**. This then undergoes tandem cyclisation to the observed product **220**, which upon oxidative hydrolysis gave the diol **221**.



Scheme 33



Scheme 34

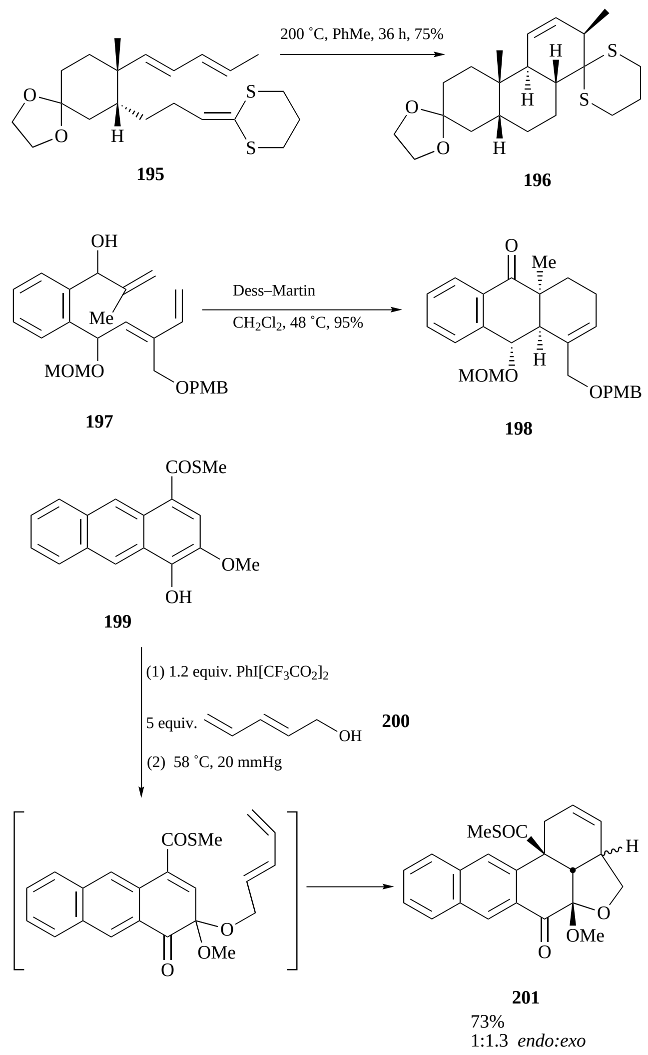


Scheme 35

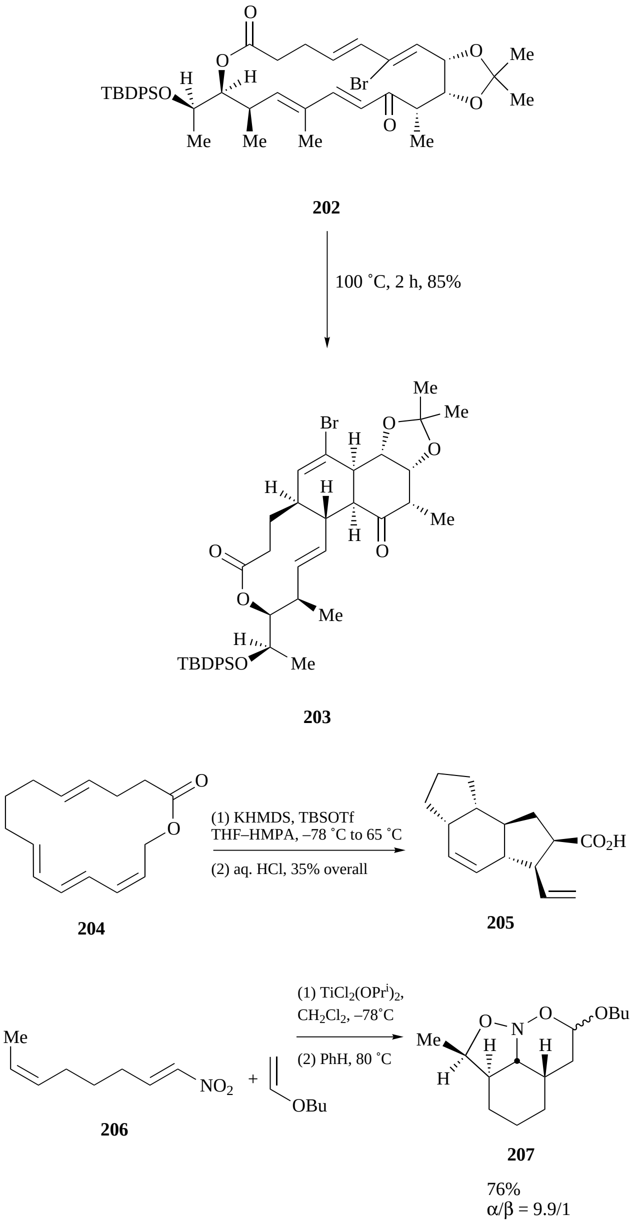
Marco-Contelles[160] also used an $R_3SnH–Et_3B$ mediated triple radical cyclisation sequence for the construction of the tetracycle **223** from the bis-prop-2-ynyl ether **222** (**Scheme 41**). Radical cascades have once again been used in a number of impressively short synthetic approaches to polycyclic systems. For example, Pattenden and Roberts[161] completed a total synthesis of spongian-16-one, utilising three successive 6-*endo* cyclisations in the key conversion of the acyl selenide **224** to the keto lactone **225**. Similarly, Zoretic *et al.*[162] have utilised the chemistry of manganese acetate in a radical cascade involving four successive cyclisations (**226**→**227**).

Samarium diiodide has again proved popular for the formation of cyclohexanes by 6-*exo* reductive cyclisations of ketones. For example reductive cyclisation of the ketone **228** gave the *cis*-decalin system **229** of vinigrol in good yield and with excellent stereoselectivity (**Scheme 42**).[163] In their approach to C10-desmethylarteannium B, Schwaebe and Little[164] reported that the key decalin **231** could be prepared in excellent yield by reductive cyclisation of the keto ester **230** with samarium diiodide. Reductive cyclisation of the keto alkyne was used as the key step in an approach to erigerol (**232**→**233**).[165]

A number of interesting methods for cyclohexane construction involving radical rearrangements or fragmentations have been reported. For example Herbert and Pattenden[166] found that TBTH reduction of the acyl selenide **234** yielded the cyclohexenone **236** *via* fragmentation to the ketene **235** followed by cyclisation (**Scheme 43**). Reductive cleavage of the four-membered ring in **237** followed by rearrangement of the radical **238** to the 6,5-system **239** was used as the key step in the synthesis of (±)-lubiminol by Crimmins *et al.*[167] Similarly, Nemoto *et al.*[168] reported that treatment of the iodide **240** with TBTH gave the decalin **241** *via* a 3-*exo* cyclisation–ring expansion–6-*exo* cyclisation sequence. A useful one-carbon ring expansion procedure[169] was employed in the conversion of the alkynol **242** to the α-methylene cyclohexanones **243** and **244** (**Scheme 44**). Finally, Wang *et al.*[170] reported that good yields of cyclised



Scheme 36



Scheme 37

products **246** could be obtained *via* cascade radical cyclisation on thermolysis of heptatrienynes such as **245**.

4.4 Transition metal mediated cyclisations

Palladium catalysed Heck type cyclisations have been used by a number of different groups for the construction of cyclohexanes. Yokoyama *et al.*[171] were able to construct the A-ring of vitamin D **248** by Heck cyclisation of the vinyl triflate **247** (**Scheme 45**). A similar cyclisation was used by Overman and co-workers[172] to construct the tetracycle **250** from the vinyl triflate **249**.

Grigg[173] has reported a palladium catalysed cyclisation–anion capture cascade where complex tricycles such as **252** can be prepared efficiently from simpler acyclic substrates such as **251** (**Scheme 46**). An interesting tandem Heck–Diels–Alder sequence (**253**→**254**) has been described by de Meijere and co-workers[174] where bicyclo-[4.3.0] and -[4.4.0]ring systems can be prepared in good yield, and in one step from acyclic dienes.

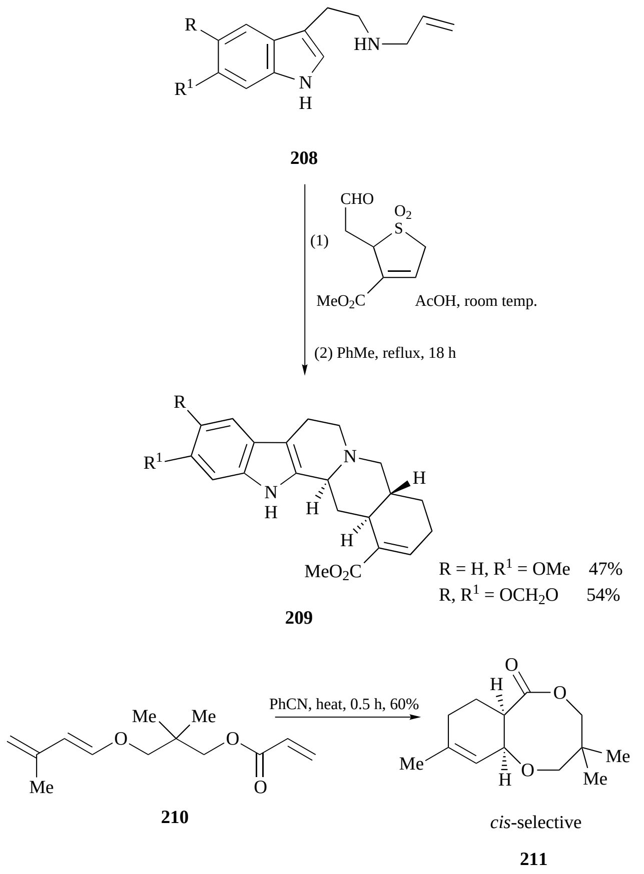
A Pt–Sn catalysed hydroformylation–cyclisation sequence[175] has been used to convert limonene **255** to the bicyclic alcohol **256** in good yield (**Scheme 47**). An interesting tungsten based cyclofunctionalisation procedure has recently been reported[176] which allows the conversion of simple prop-2-ynyl bromides to usefully functionalised carbocyclic systems (**257**→**258**).

4.5 Cationic cyclisations

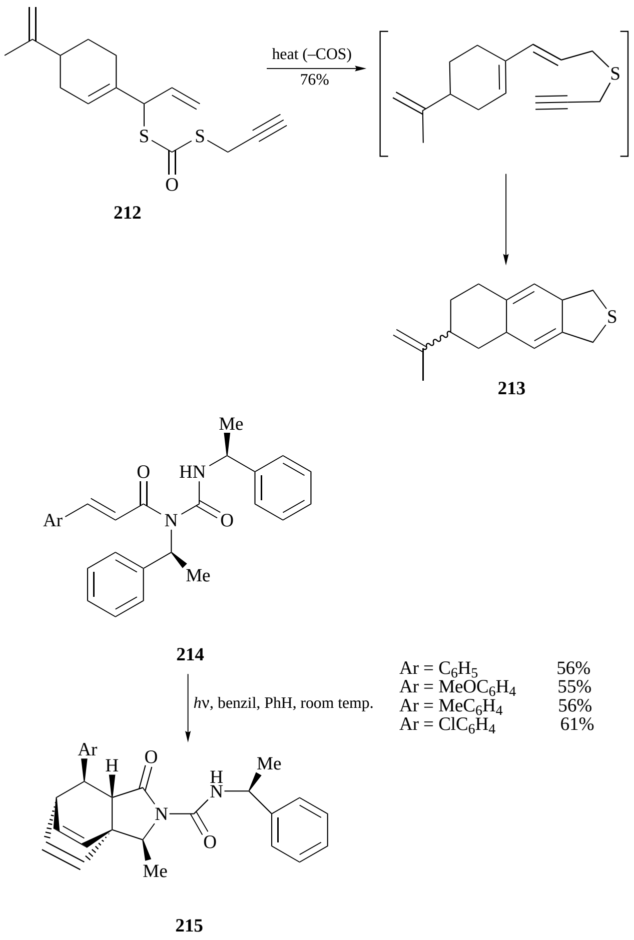
Continued interest in steroid synthesis/biosynthesis has resulted in a number of examples of the cation–olefin cyclisation sequence. For example Corey and Wood[177] reported the efficient formation of the steroidal system **260** by treatment of the epoxy polyene **259** with methylaluminium dichloride (**Scheme 48**). In a biomimetic approach to (+)-peripyropene-E, Smith *et al.*[178] reported that the epoxy polyene **261** underwent tetracyclisation to form the polycyclic fused pyrone **262** with good stereoselectivity. Use of an iminium ion mediated cationic cyclisation allowed the preparation of the azasteroid **264** from the anisole derivative **263** (**Scheme 49**).[179] Sen and Roach[180] reported that the acetal enamine **265** underwent efficient cyclisation to the tricyclic hydroxy enamine **266** upon treatment with hydrated ferric chloride.

An unprecedented acid catalysed transannular cyclisation was reported by Backhaus and Paquette[181] in their synthetic studies towards kaurene (**267**→**268**, **Scheme 50**). Mander and co-workers[182] reported an interesting and complex example of a pinacol rearrangement of a gibberellin aldehyde (**269**→**270**). It is fascinating to note the similarity between **268** and **270**; both routes use different chemistry and are obtained from completely different starting materials.

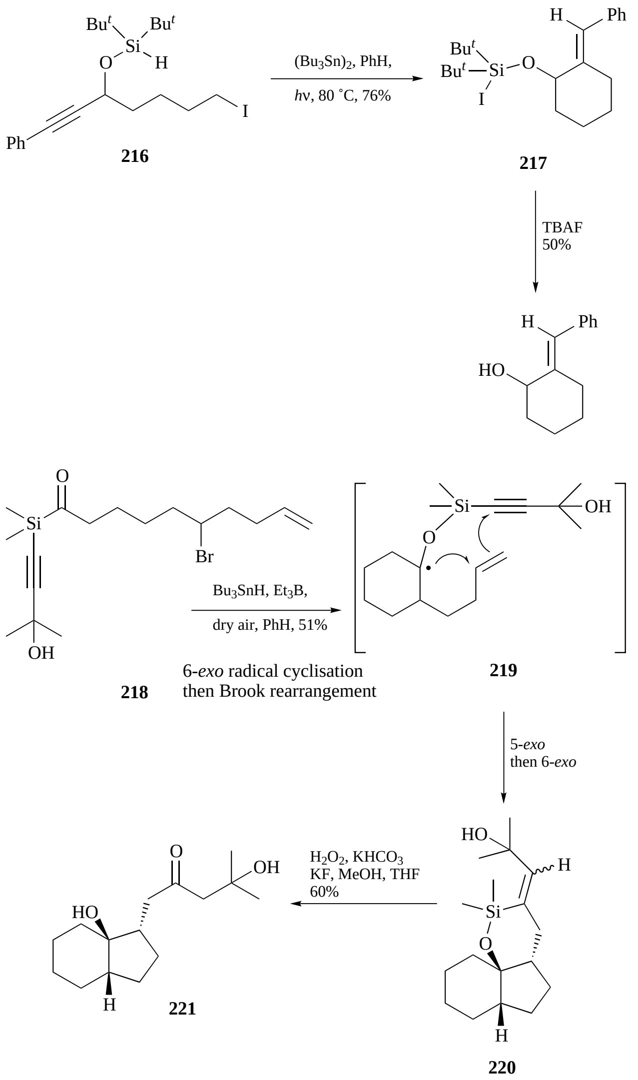
The use of super acid catalysts was reported[183] in the conversion of the allylic alcohol **271** to the complex and unique tricyclic compound **272** (**Scheme 51**). In their synthetic



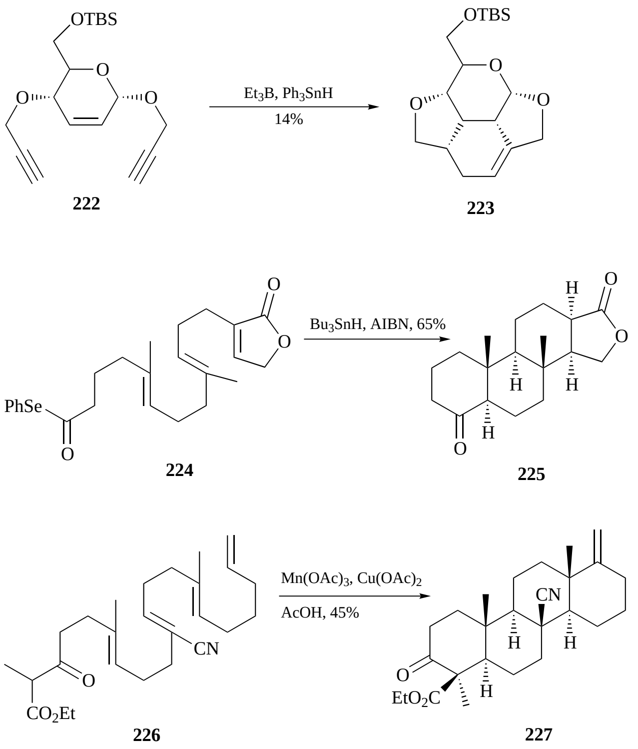
Scheme 38



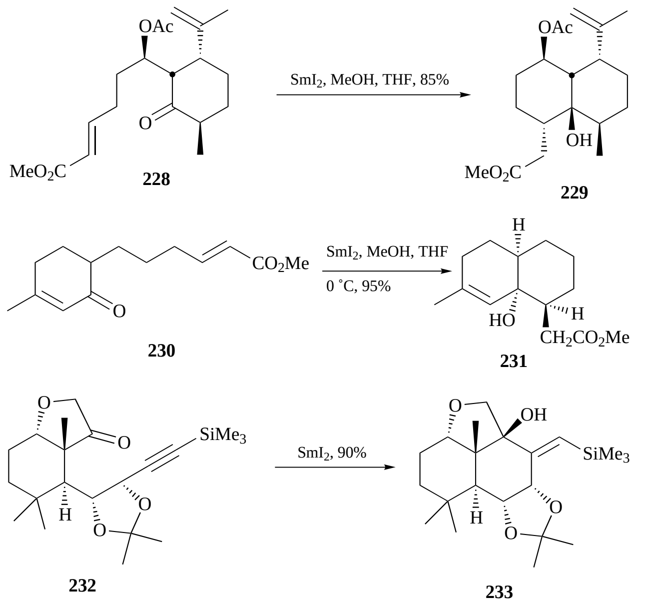
Scheme 39



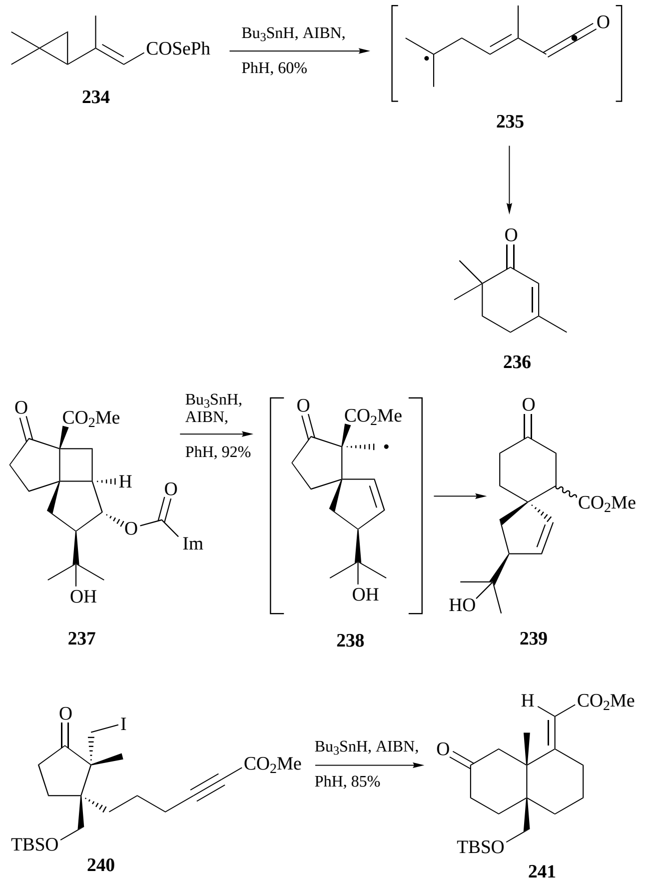
Scheme 40



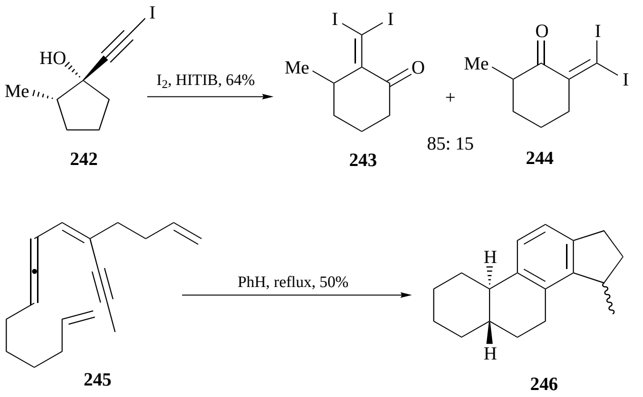
Scheme 41



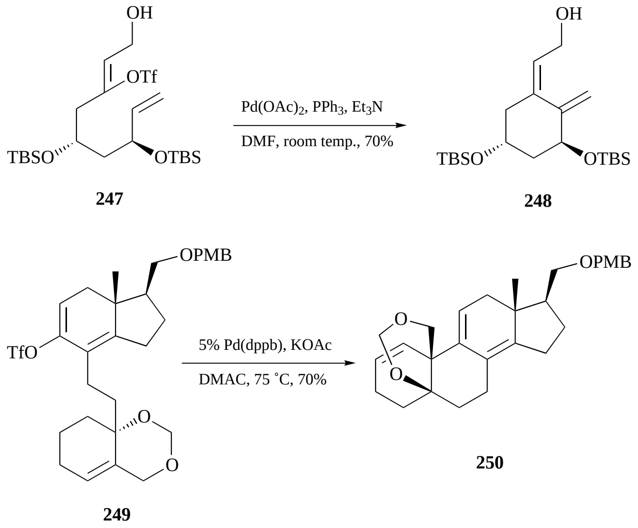
Scheme 42



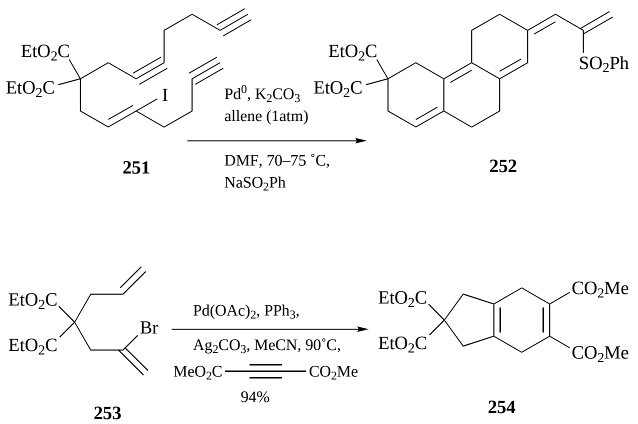
Scheme 43



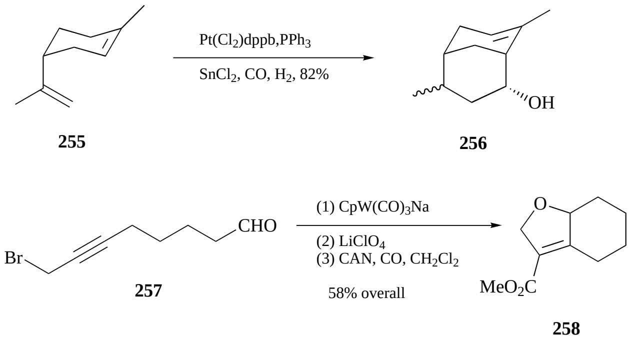
Scheme 44



Scheme 45



Scheme 46

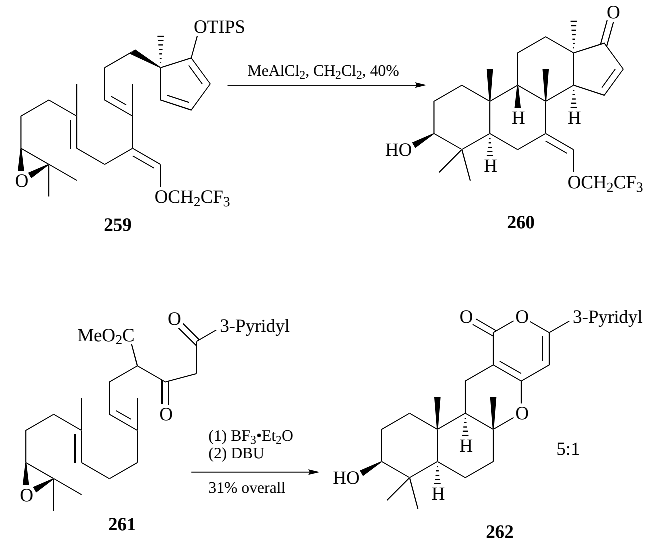


Scheme 47

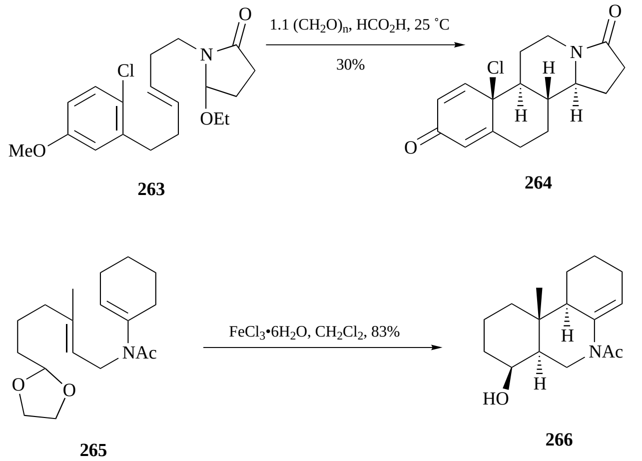
approaches to the pseudopterosins Kocienski and co-workers[184] reported an efficient Lewis acid catalysed cyclisation of the allylic sulfone **273** to the key tricycle **274**. Finally, the allylsilane **275** gave the keto ester **276** with a high degree of *cis*-selectivity on treatment with boron trifluoride–diethyl ether.[185]

4.6 Anionic methods

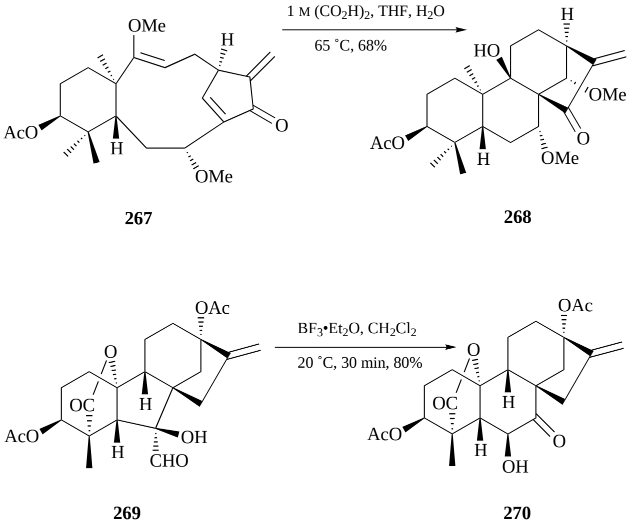
The use of Michael type reactions has continued to prove useful for the facile construction of cyclohexanes.[186] For example Maiti *et al.*[187] described a triple Michael–Dieckmann sequence for the conversion of cyclohexenones into complex fused carbocyclic systems (**277**→**278**, **Scheme 52**). Conjugate addition of



Scheme 48



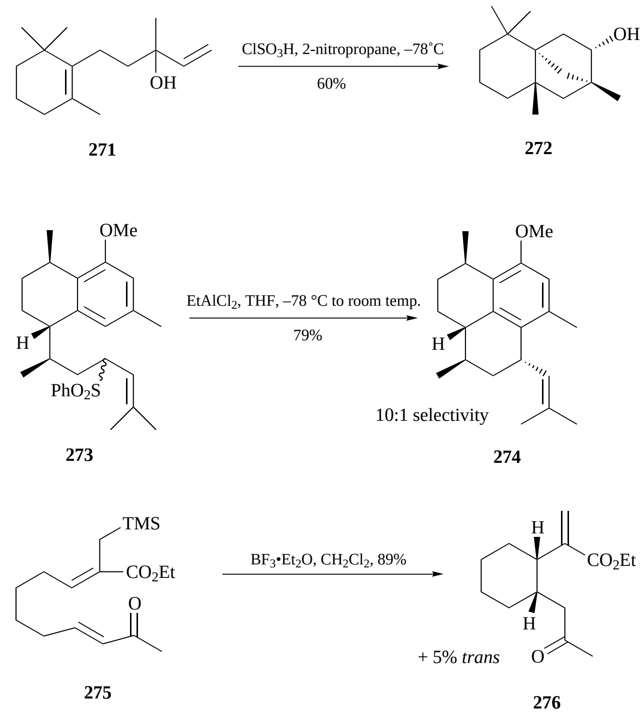
Scheme 49



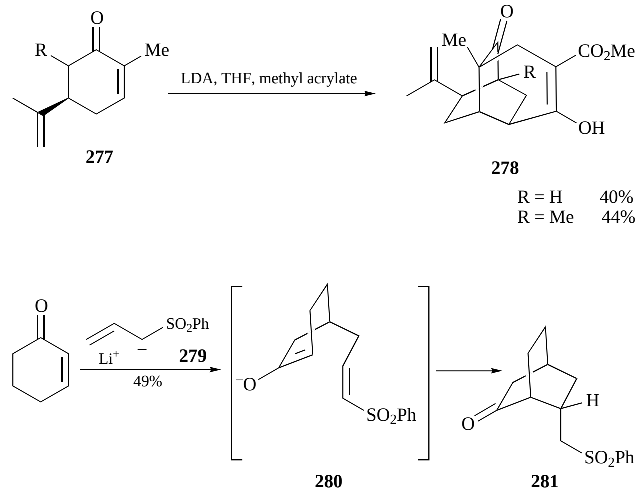
Scheme 50

the sulfone anion **279** to cyclohex-2-enone followed by intramolecular Michael addition of the resulting enolate **280** gave the keto sulfone **281** with excellent stereoselectivity.[188]

An efficient 1,6-conjugate addition sequence (**282**→**283**) was used as a key step in a total synthesis of coronafacic acid (**Scheme 53**).[189] A fascinating one-pot tandem Michael sequence was reported in the reaction of acetophenones with *trans*-dibenzoylethylene whereby resulting cyclohexanes **284** were formed in good yield and as single diastereoisomers.[190]



Scheme 51

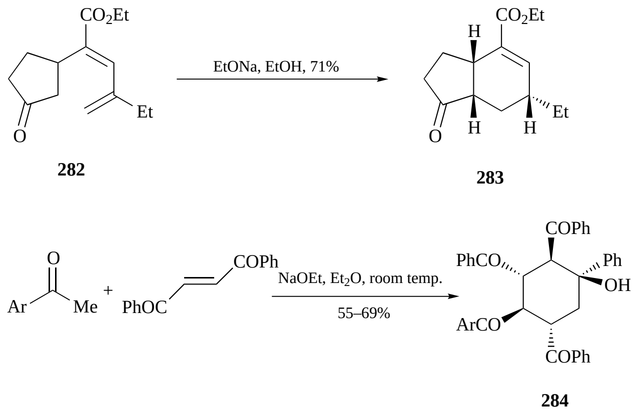


Scheme 52

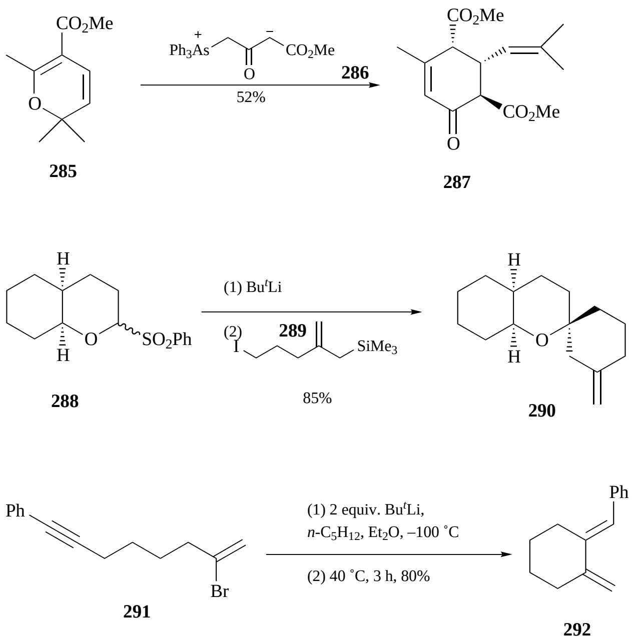
Moorhoff[191] described a very useful synthesis of highly substituted oxocyclohexenedicarboxylates **287** which involved the novel Michael–Wittig condensations of the arsonium ylide **286** with a range of 2*H*-pyrans **285** (**Scheme 54**). Paquette and Tae reported[192] that the tricyclic spiro system **290** could be prepared from the sulfone ether **288** by a sequence involving alkylation with the iodide **289** followed by spontaneous oxonium ion formation and silane mediated cyclisation. Bailey *et al.*[193] have continued their interest in anionic cyclisations and have reported the efficient formation of cyclohexyl fused dienes (*e.g.* **292**) *via* metal–halogen exchange of vinyl halides (*e.g.* **291**) followed by cyclisation on warming.

4.7 Other routes

Paquette has continued to explore the use of the anionic Cope reaction in natural product synthesis. For example in a recently reported[194] approach to spinosyn A it was found that the alcohol **293** underwent smooth rearrangement to the tricyclic ketone **294** (**Scheme 55**). Ho and Chein[195] have utilised a tandem Claisen–ene reaction to construct the decalin ring system of (−)-furodysinin (**295**→**296**). Shanmugam and Rajagopalan have reported[196] a number of useful tandem [3,3]



Scheme 53

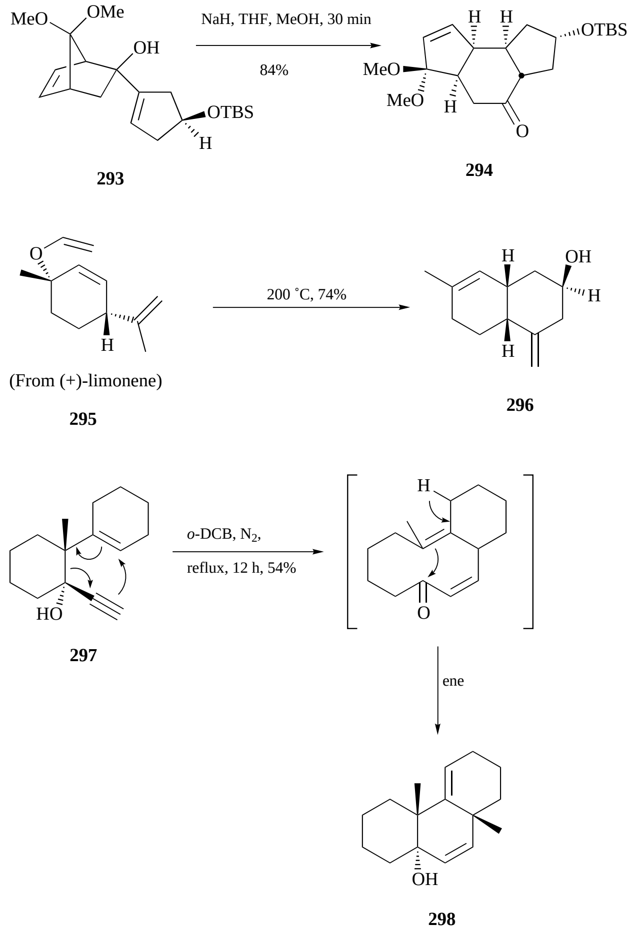


Scheme 54

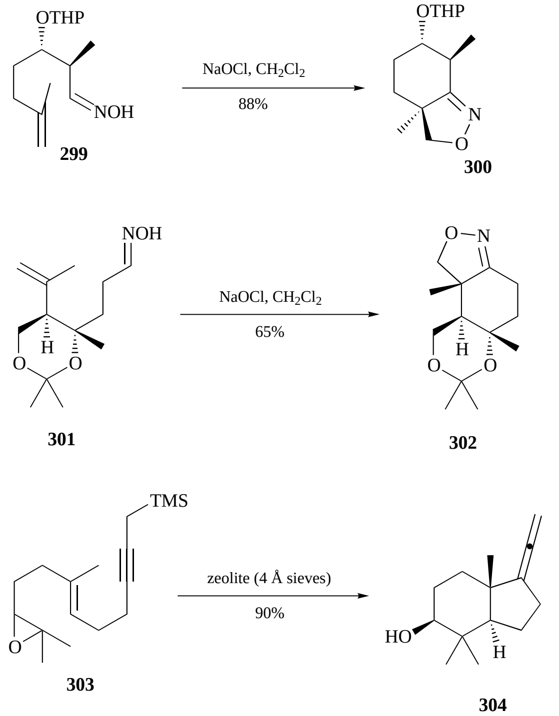
ene cyclisation reactions of prop-2-ynyl and allenyl alcohols for the preparation of polycyclic systems. For example, thermolysis of the prop-2-ynyl alcohol **297** gave the tricyclic alkenol **298** *via* ene cyclisation of the initially formed [3,3] sigmatropic rearrangement product.

Two groups have utilised the intramolecular nitrile oxide cycloaddition for the stereoselective construction of cyclohexanes: (**299**→**300**)[197] and (**301**→**302**, **Scheme 56**).[198] An interesting zeolite induced cyclisation of the epoxyenyne **303** gave a high yield of the 5,6-fused bicyclic allenol **304** with excellent stereocontrol.[199]

Sarkar and Nandy[200] found that samarium diiodide is an excellent catalyst (non-radical) for ene-type cyclisations of dienes to cyclohexenes (**305**→**306**, **Scheme 57**). Bryan and Chan[201] have reported a very useful indium mediated intramolecular carbocyclisation procedure which is carried out in aqueous media. For example treatment of the allylic halide **307** with indium powder in water gave a quantitative yield of the hydroxy ester **308** which under acidic conditions gave the α-methylene lactone **309**. Kuethe and Padwa[202] used a tandem Pummerer–isomünchnone dipolar cycloaddition strategy (**310**→**311**, **Scheme 58**) to form the cyclohexane ring in their synthesis of (±)-pumiliotoxin C. Finally, it has been demonstrated[203] that certain monocyclic silyl enol ethers (**312**) undergo oxidative cyclisation on irradiation in the presence of 9,10-dicyanoanthracene (DCA) to give tricyclic ketones such as **313** with good stereocontrol.



Scheme 55

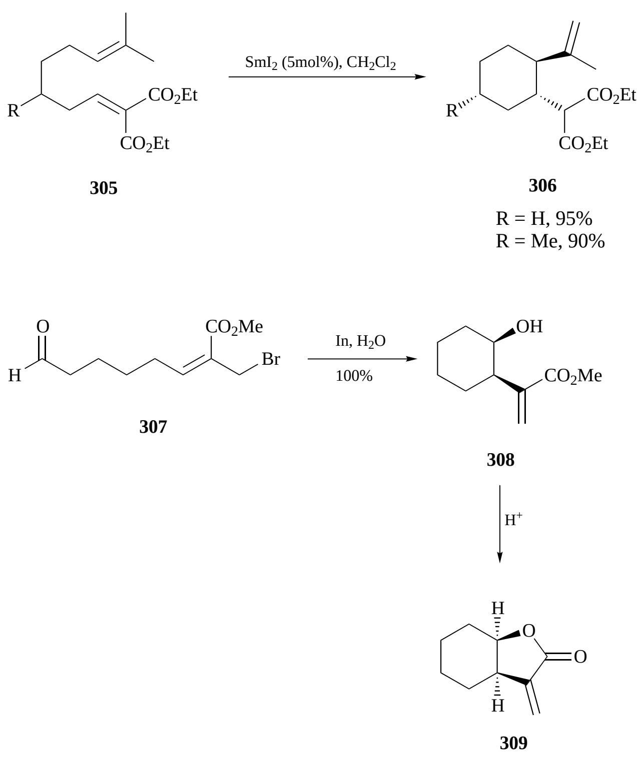


Scheme 56

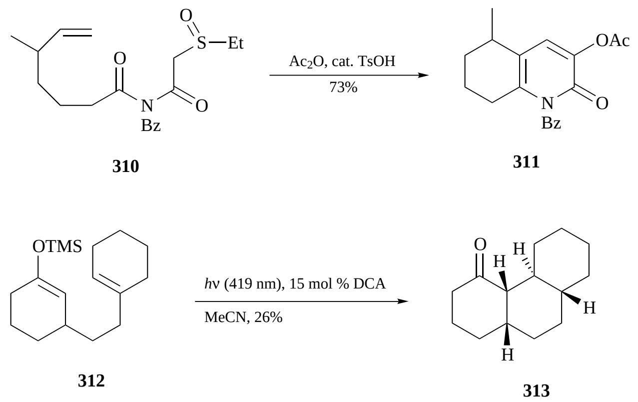
5 Seven-membered rings

5.1 Cycloadditions and annulations

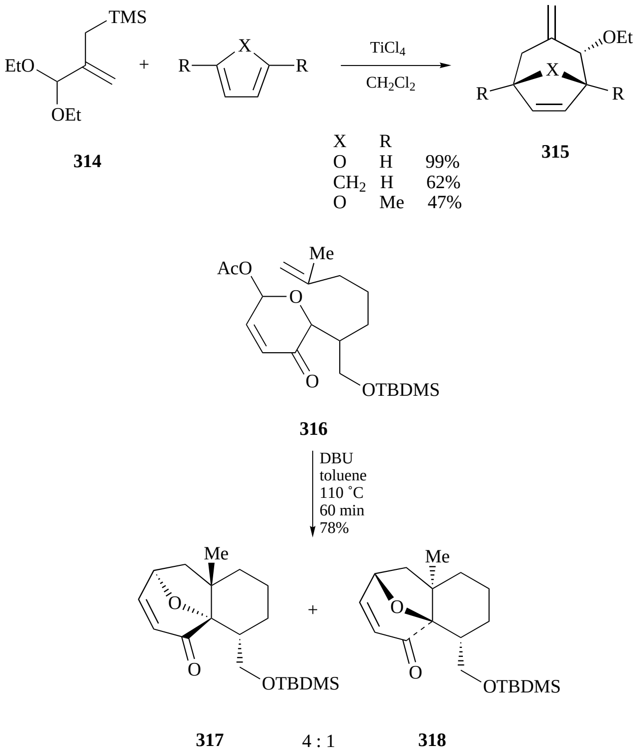
[3 + 4] Cycloadditions have remained a popular method of seven-membered ring synthesis over the review period. A typical example is that reported by Harmata and Jones[204] in which the acetal **314** is treated with $TiCl_4$ and furans or



Scheme 57

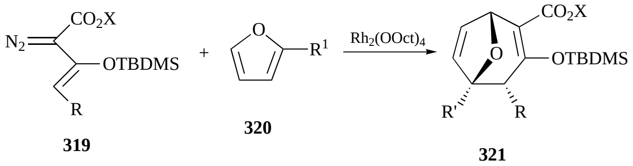


Scheme 58



X	R	
O	H	99%
CH_2	H	62%
O	Me	47%

Scheme 59



X	R	R^1	Yield	de
(*S*)-lactate	H	H	72%	79%
(*R*)-pantolactone	H	H	82%	94%
(*S*)-lactate	H	COMe	74%	79%
(*R*)-pantolactone	Me	Me	69%	94%

Scheme 60

cyclopentadienes to give the bridged systems **315** (**Scheme 59**). An intramolecular example where the α,β-unsaturated ketone **316** is converted into the tricyclic systems **317** and **318** in good yield has been used by Magnus and co-workers[205] for their synthesis of taxane derivatives. Rigby and co-workers[206,207] have continued their studies on cycloadditions of chromium(0) tricarbonyl complexes to produce bicyclo[4.4.1]undecanes while Davies *et al.*[208] have developed an asymmetric synthesis of 8-oxabicyclo[3.2.1]octene derivatives **321** *via* the rhodium induced reaction between furans **320** and diazo compounds **319** (**Scheme 60**).

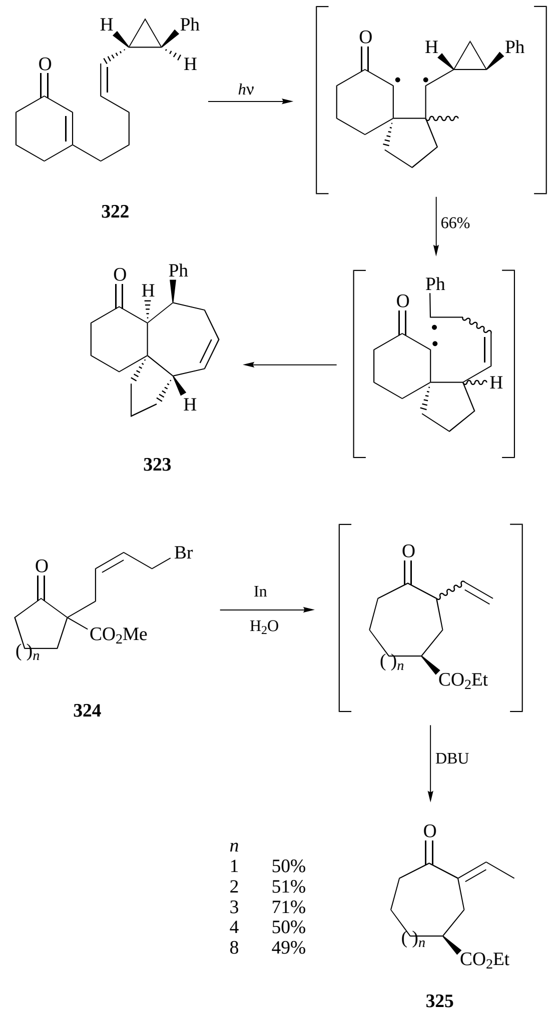
5.2 Other methods

A variety of other methods have been reported for seven-membered ring construction. Shea and co-workers[209] have published full experimental details for their bridged-to-fused ring interchange methodology for the preparation of fused cycloheptanes and cyclooctanes including the total syntheses of ledol, ledene and compressanolide. Davies and Doan[210] have used their well developed tandem cyclopropanation–Cope rearrangement strategy for a rapid and efficient asymmetric synthesis of the tremulane skeleton while Löfström and Bäckvall[211] have studied the BF_3 induced rearrangement of aziridinocyclopropanes to give cycloheptenes. A very clever synthesis of the tricyclic system **323** has been reported by Haddad and co-workers[212] *via* the photocycloaddition of the 3-pent-4-enylcyclohex-2-enone **322** (**Scheme 61**). Li and co-workers[213] have developed a novel indium induced carbocycle enlargement for the general preparation of medium and large rings (**324**→**325**).

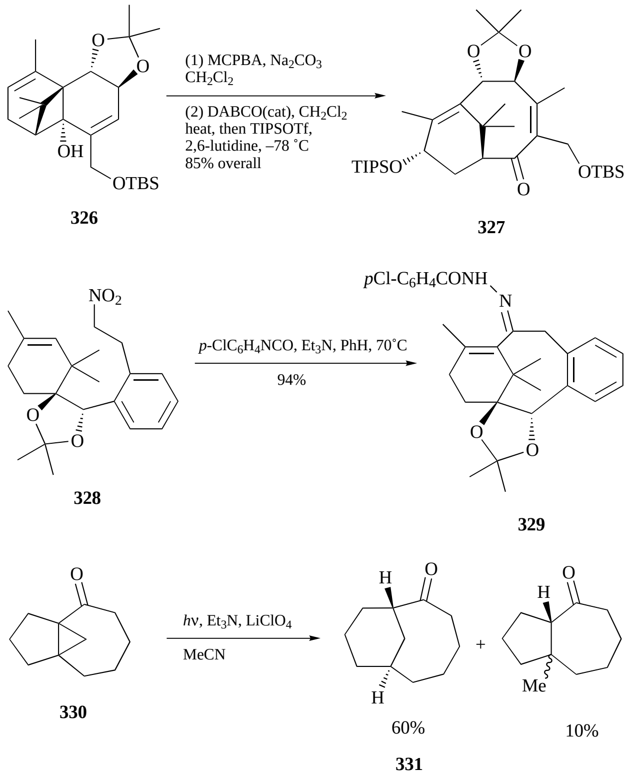
6 Eight-membered rings

Continued interest in Taxol and analogues has resulted in a number of different approaches to cyclooctanes being reported over the review period. In a series of two papers Wender *et al.*[214] described their total synthesis of Taxol which relies upon an elegant base catalysed rearrangement of a verbinone derived epoxide (**326**→**327**, **Scheme 62**). A novel Friedel–Crafts type cyclisation[215] of the nitrile oxide derived from **328** was used to good effect to construct the aromatic taxane system **329**. Cossy and Bouzbouz[216] described a different approach to the taxane skeleton which involved photoselective bond cleavage of the cyclopropane **330** which gave the ketone **331** as the major product.

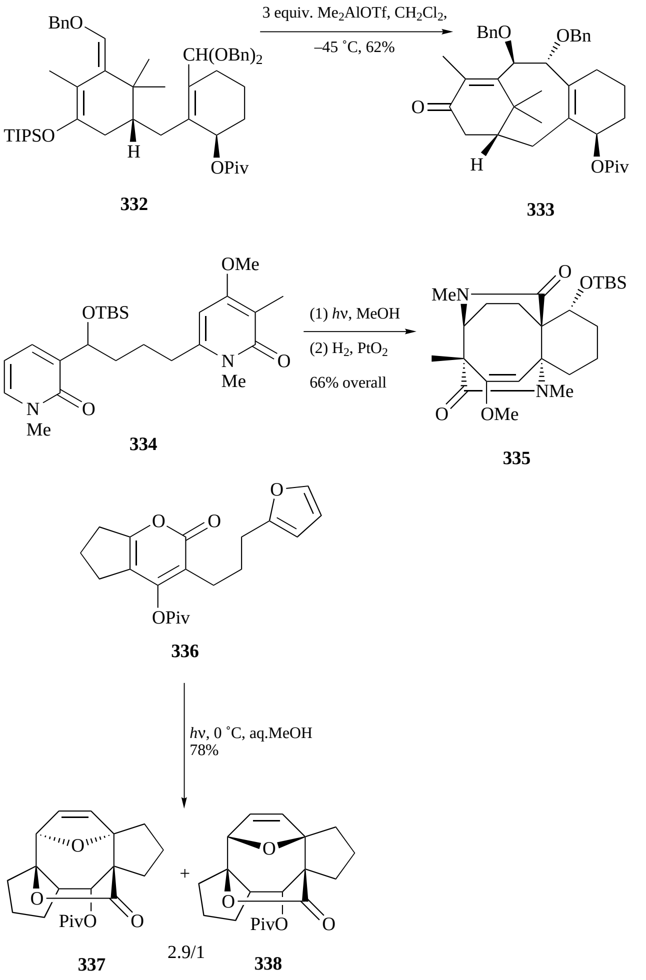
In their total synthesis of taxusin Hara *et al.*[217] described the formation of the taxane framework **333** by Lewis acid catalysed cyclisation of the acetal **332** (**Scheme 63**). Sieburth and co-workers[218,219] used the [4 + 4] photochemical cycloaddition



Scheme 61



Scheme 62



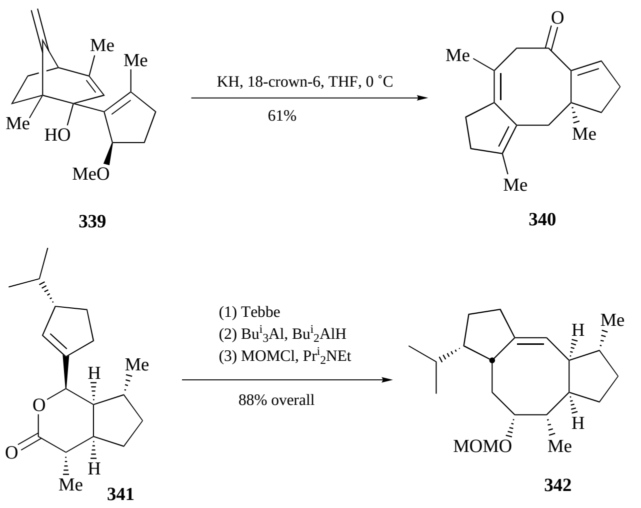
Scheme 63

of pyridones in their approach to the taxanes (**334**→**335**). West and co-workers[220] have also continued their interest in [4 + 4] cycloadditions and found that the furanyl tethered pyrone **336** underwent efficient photocycloaddition to the complex 5,8,5-fused systems **337** and **338**. Such a concise approach to this ring system should prove very useful in the synthesis of natural products such as the ophiobolanes and fusicoccones.

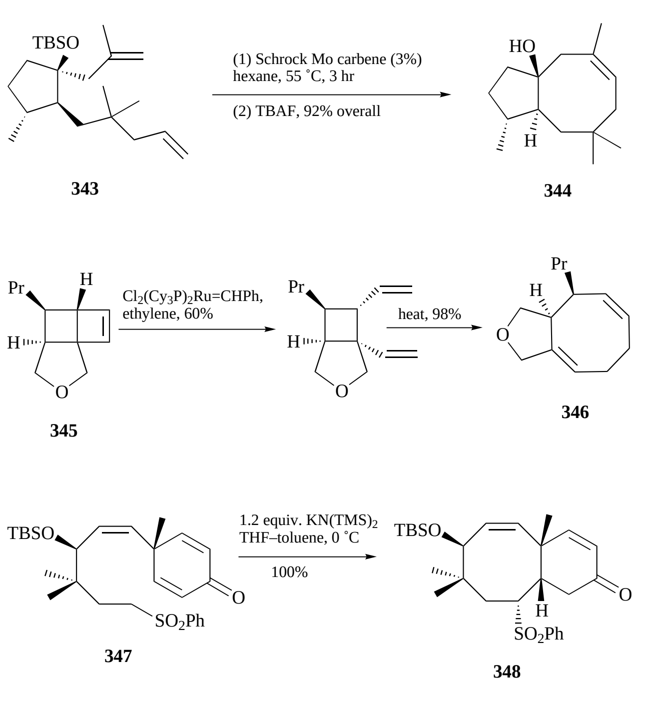
Paquette *et al.*[221] have prepared the core structure of ceroplastin using an anionic oxy-Cope reaction (**339**→**340**, **Scheme 64**). Paquette's group has also reported[222] the use of a Tebbe induced Claisen rearrangement sequence as the key step in an enantioselective total synthesis of epoxydictymine (**341**→**342**).

Metathesis has once again proved popular for the formation of medium sized carbocycles. For example, treatment of the diene **343** with a Schrock molybdenum catalyst followed by desilylation provided the final step in a synthesis of dactylol **344** (**Scheme 65**).[223] Snapper *et al.*[224] reported an interesting metathesis–Cope rearrangement sequence involving the cleavage of the cyclobutane **345** with a Grubbs catalyst, followed by thermolysis to the cyclooctadiene **346**. Direct Michael cyclisation of the sulfone **347** provided a quantitative yield of the [6.4.0] system **348**.[225]

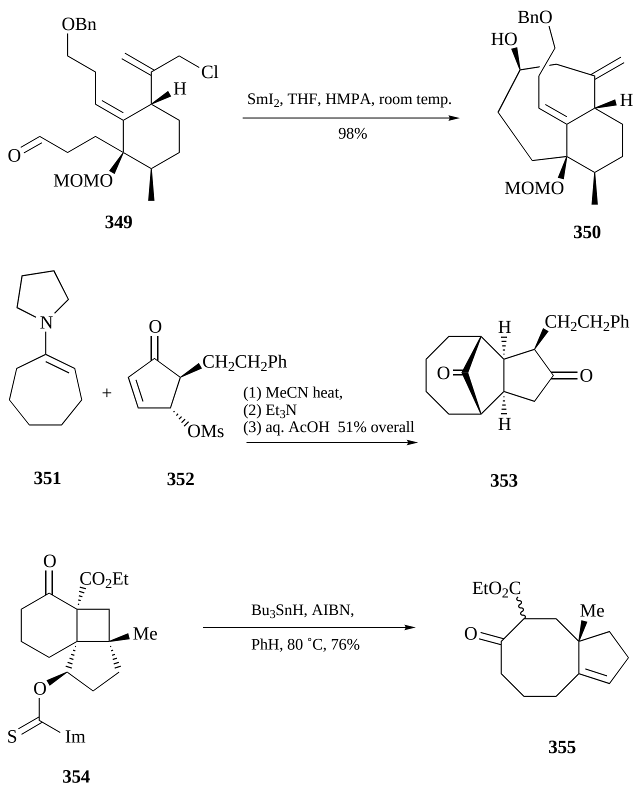
A highly efficient samarium diiodide promoted Barbier coupling was used to construct the eight-membered ring of vinigrol (**349**→**350**, **Scheme 66**).[226] A tandem enamine–Michael addition sequence was described by West and co-workers[227] as a useful method for the formation of keto-bridged cyclooctane systems (**351** + **352**→**353**). Finally, Crimmins *et al.*[228] described the preparation of the 5,8-bicyclic ketone **355** by TBTH mediated radical fragmentation of the cyclobutane **354**.



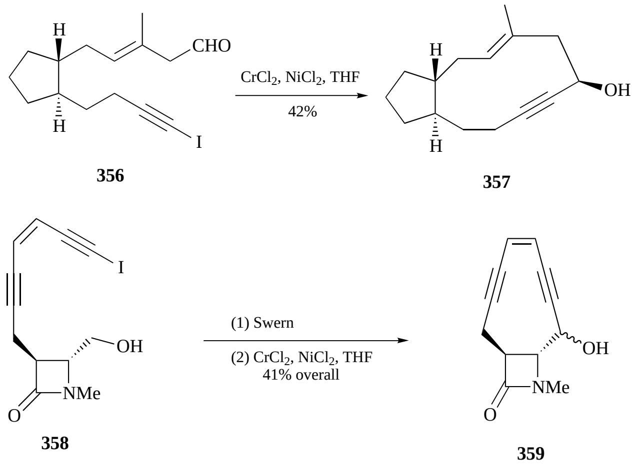
Scheme 64



Scheme 65



Scheme 66



Scheme 67

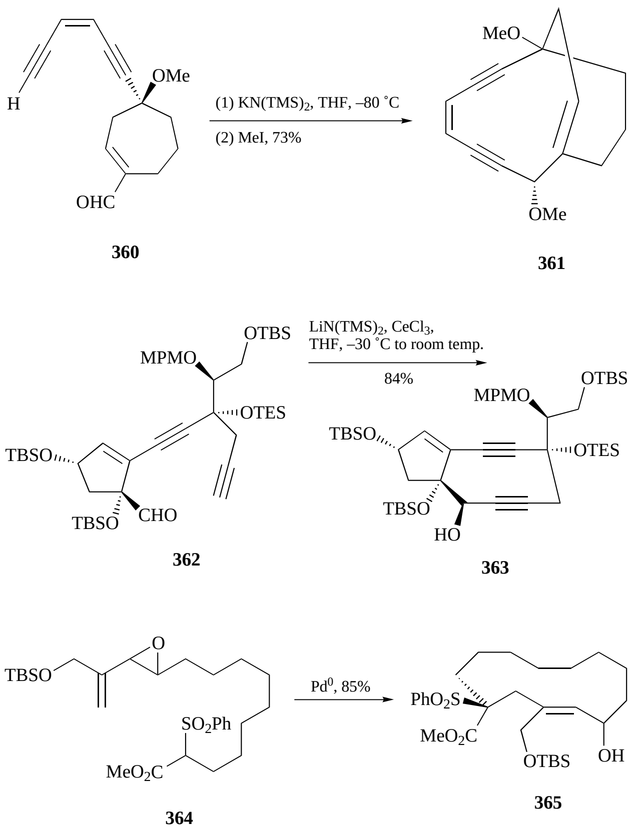
7 Nine-membered and larger rings

The use of chromium mediated cyclisations for the synthesis of nine-membered and larger carbocycles has remained popular over the review period. For example Whitby and Luker[229] found that treatment of the alkynyl iodide **356** under modified Nozaki conditions gave the access to the dolabellane skeleton **357** (**Scheme 67**). Similarly, a sequential Swern oxidation–chromium cyclisation sequence proved useful for the synthesis of novel β-lactam fused enediynes (**358**→**359**).[230]

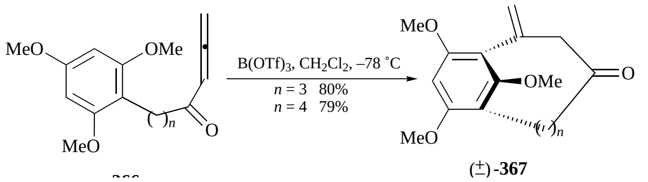
Maier and Langenbacher[231] reported a direct cyclisation to the enediyne **361** by treatment of **360** with potassium bis(trimethylsilyl)amide (**Scheme 68**). Hirama and co-workers[232] also found that a similar cyclisation was useful for construction of the skelton of the C-1027 chromophore (**362**→**363**). In their approach to roseophillin Furstner and Weintritt[233] reported that the palladium catalysed ring closure of the allylic epoxide **364** gave the macrocycle **365** in good yield.

Nagao and co-workers[234] reported a very interesting racemic synthesis of chiral [6]- and [7]-metacyclophanes **367** which involved a Lewis acid catalysed cyclisation of allenones **366** (**Scheme 69**). Paquette and co-workers[235,236] have again used the powerful oxy-Cope fragmentation strategy for the construction of (−)-salsolene oxide (**368**→**369**) and (−)-*o*-methylshikoccin (**370**→**371**, **Scheme 70**). Clark *et al.*[237] have made good use of their oxonium ylide [2,3] sigmatropic rearrangement protocol in the construction of the core of neoliacinic acid (**372**→**373**).

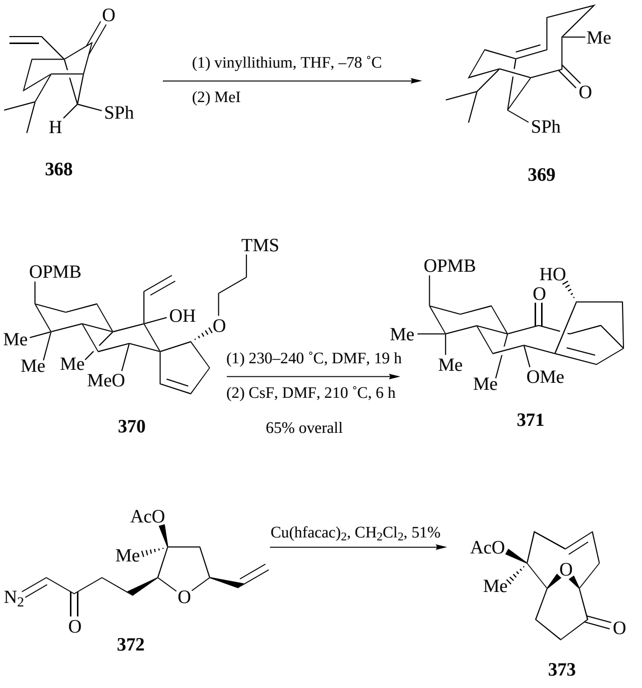
A number of interesting fragmentation procedures to medium sized carbocycles have been reported over the review period. For example, a general two-atom ring expansion procedure based on the indium mediated Barbier–Grignard type reaction has been developed by Li and co-workers (see section 5.2).[213] Saicic reported[238] a high yielding conversion of the alcohol **374** into the 9-membered dione **376** involving β-scission of the alkoxyl radical **375** generated by irradiation of a HgO–I$_2$ mixture (**Scheme 71**). Nowakowski and Hoffmann[239] reported an interesting fragmentation involving the β-scission of alkoxyl radicals generated using CAN. Finally, de Groot and co-workers[37] have utilised a Marshall type fragmentation in their synthesis of allohedycaryol **378** from the decalin **377**.



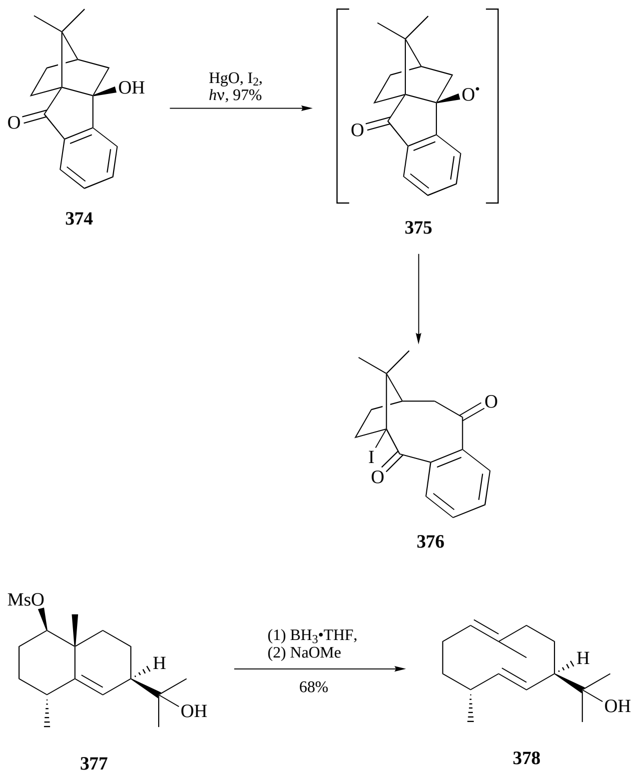
Scheme 68



Scheme 69



Scheme 70



Scheme 71

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