

Microwave-Assisted Claisen and Aza-Claisen Rearrangements

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Abstract: This review covers the key advances in the study of Claisen rearrangement under microwave irradiation conditions. It surveys [3,3] sigmatropic rearrangements of vinyl allyl ethers, aryl allyl ethers, ortho-ester and aza Claisen rearrangements. Applications of the method for the synthesis of heterocyclic and natural compounds are surveyed.

Key Words: Claisen rearrangement, microwave, irradiation, natural compounds.

INTRODUCTION

Over two decades ago Gedeye *et al.* [1] described the use of microwave irradiation for organic synthesis, and since then microwave assisted chemistry became a useful practical tool [2-6] including areas of medicinal [7-9], combinatorial [10-15], heterocyclic [16-20], carbohydrate [21-22], amino acid [23] and polymer [24-25] chemistry. Many types of reactions, *i.e.* cycloadditions [26-27], metal-catalyzed coupling reactions [28], and fluoros synthesis [29], require extended heating periods under traditional conditions and proceed considerably faster when subjected to microwave irradiation.

Claisen rearrangement (CR) gains the continuous interest as a fundamental synthetic tool for the preparation of natural and pharmaceutically valuable compounds and its latest developments and photochemical [30], catalysed [31-33] and asymmetric [34-36] variations have been extensively reviewed. Special topics of aza-CR [37-38], tandem Diels-Alder/Claisen sequences [39], applications in the synthesis of fragrance compounds [40] and synthesis of coumarins, quinolones and thiocoumarins [41] have been surveyed. The CRs have been traditionally carried out under thermal conditions and, thus, are an attractive class of reactions for microwave assisted synthesis [42-44]. The influence of the physical parameters on the course of the process was the part of the recent general review on CR [45]. Furthermore, this transformation was studied to exemplify the microwave instrumentation [46-47], and microwave high-temperature aqueous [46, 48] and ionic [49] media applications.

In this first specialized review we present the key advances in the study of CR under microwave irradiation conditions. It covers the literature from early 2000 till 2007 and surveys applications for the synthesis of natural, biologically and industrially important compounds.

REARRANGEMENTS OF VINYL ALLYL ETHERS

Several recent reports have demonstrated the rate accelerating effect of microwaves on the [3,3] sigmatropic rearrangement of allyl vinyl ethers. These reactive species can be prepared by a variety of methods to generate vinyl or allyl

moieties, and synthetic sequences can be performed stepwise or by synthetic cascades.

Thus, starting from allyl alcohols and vinyl iodides a domino copper-catalyzed *C-O* coupling-CR process has been reported [50]. Reaction can be carried out under both conventional heating and microwave irradiation. The later conditions significantly reduce reaction time, although they result in decreased diastereoselectivity and yields.

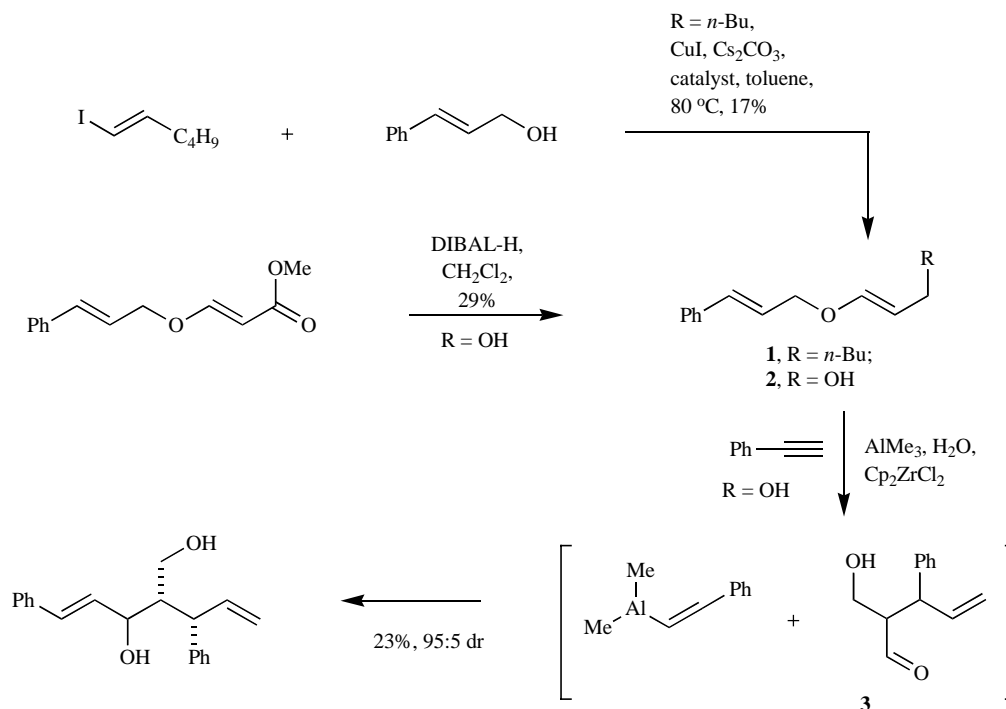
The above Buchwald's coupling method and ester reductive strategy (Scheme (1)) were used for the preparation of the intermediate allyl vinyl ethers **1** and **2**, correspondingly. A three-step cascade reaction involving a water-accelerated catalytic carboalumination, a CR, and a nucleophilic carbonyl addition further converts terminal alkynes and allyl vinyl ethers into allylic alcohols containing up to three contiguous asymmetric carbon centers. Formation of the intermediate aldehyde **3** can be performed both under conventional heating and microwave conditions. Stoichiometric quantities of water as an additive increase the rate of the [3,3] sigmatropic rearrangement as well as the diastereoselectivity of the carbonyl addition process [51].

The use of microwave irradiation accelerates a one-pot synthesis of decahydrobenzo[*e*]azulen-5(1*H*)-one **6** which includes base-catalyzed 5-*exo* cyclization of substituted 4-alkyn-1-ol **4** and subsequent CR of the intermediate 2-methylene tetrahydrofuran derivative **5** (Scheme (2)). The resulting cycloheptanoid ring systems were produced under microwave irradiation in the presence of catalytic amounts of MeLi or potassium *tert*-butoxide in DMF or ethoxybenzene. Solvent-free conditions are also compatible with the reaction sequence [52].

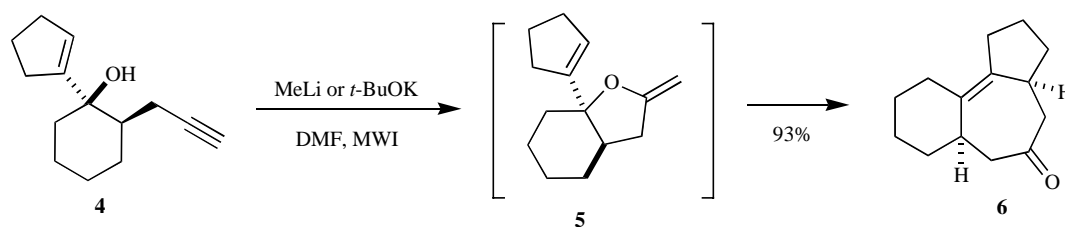
Craig *et al.* demonstrated the utility of the method in the synthesis of homoallylic sulfones **10a** through a decarboxylative CR reaction (Scheme (3)) [53-54]. The transformations could be carried out in the presence of *N,O*-bis(trimethylsilyl)acetamide (BSA) (1.0 equiv) and potassium acetate (0.1 equiv) by 15 h reflux in toluene, while microwave irradiation requires only 3 min exposure in the presence or absence of solvent. When the same reaction was performed in the absence of base the acid **9** was isolated as the hydrolysis product of the intermediate silyl ester **8**.

Similarly, allylic esters containing *N*-arylsulfonyl sulfoximines **7** ($X = \text{NSO}_2\text{Ar}^2$) can be submitted to the decarboxylative CR reaction. Rearranged products **10b** were isolated in generally good yields and diastereoselectivity [55]. Similar methodology was further extended to heteroaromatic sys-

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Scheme 1.



Scheme 2.

tems (Ar¹ = heteroaryl) to produce the corresponding furan, thiophene and pyrrole or indole sulfone derivatives [56].

A tandem three-step one-pot approach for the conversion of aldehydes into β -substituted-2-oxohex-5-enoic acids **12** is depicted in Scheme (4). The optimized sequence is carried out in water with microwave irradiation and involves sequential Horner-Wadsworth-Emmons olefination, CR and ester hydrolysis [57]. The sequential three-step process gives an overall yield of 69% with a combined reaction time of 12 hours, compared to the tandem process which gives 88% yield in a single operation lasting ten minutes. The aqueous medium employed for the tandem sequence presumably assists the sigmatropic rearrangement *via* the hydrophobic acceleration effect, as well as effects the *in situ* hydrolysis of the intermediate α -ketoester **11**.

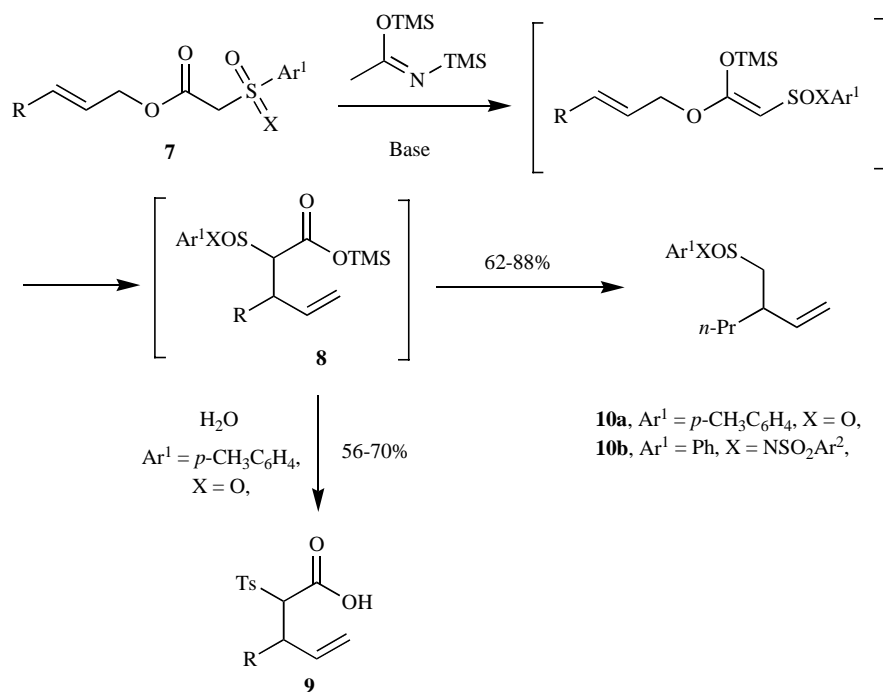
Enantiospecific aliphatic CR can be realized with high chirality transfer (Scheme (5), [58]). Synthetic pathway includes Pd-catalyzed asymmetric allylic alkylation, sequential protection, olefin isomerization ($[(C_8H_{14})_2IrCl]_2$, PCy₃, NaBPh₄) of the resultant bisallyl ethers **13** into vinyl allyl derivatives **14** and *in situ* CR to afford chiral aldehydes **15**. The latter tend to racemize, and a balance between time and temperature for the rearrangement is crucial for the chirality transfer. Conventional heating of the substrate leads to inefficient conversion and selectivity. Reaction under microwave

conditions affords full conversion at 150 °C within 10 min and using a microwave at 140 °C for 15 min gives the least chirality loss. Adding 1 equiv of BSA could further inhibit the racemization. A Lewis acid catalyzed CR has been also attempted, although it did not decrease the required temperature with acceptable conversion or chirality transfer.

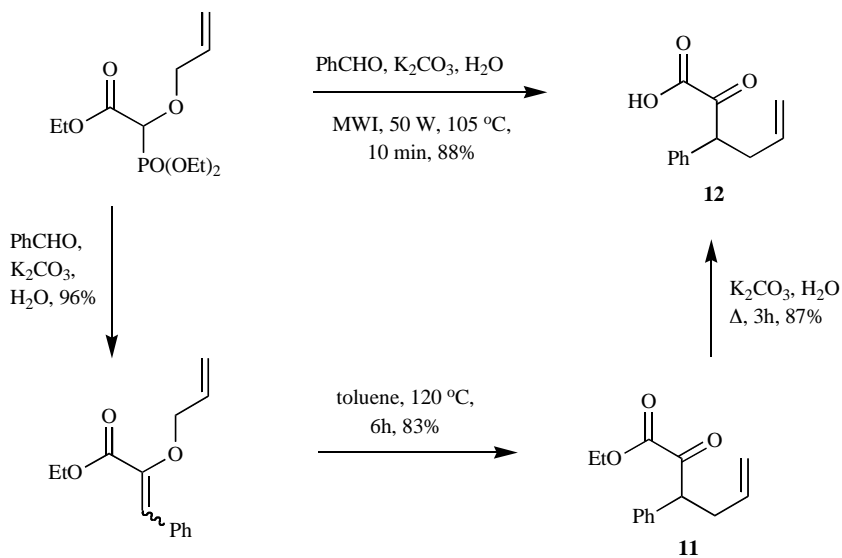
Similar olefin isomerization-CR reactions have been used for a synthesis of other chiral unsaturated aldehydes suitable for a further tandem intramolecular Sakurai-aldol reaction affording substituted cyclohexenone derivatives with high diastereoselectivity [59].

Cascade of three highly stereoselective pericyclic reactions leading to the synthesis of skeletons possessing two contiguous quaternary centers has been reported by Barriault group (Scheme (6), [60-61]). The reaction of allylic ether **17** is triggered under microwave conditions by an oxy-Cope rearrangement to create *in situ* a 10-membered ring enol ether macrocycle **18**, which immediately rearranges *via* a Claisen [3,3] shift to the corresponding *E*-cyclodec-6-en-1-one **19**. The latter spontaneously cyclizes *via* a transannular ene reaction to produce Decalin **20**.

α -Allyl ketones **21** (Fig. (1)) can be obtained from tetralones (X = CH₂) or chromanones (X = O) in moderate to good yields with only minor quantities of diallylation by-



Scheme 3.



Scheme 4.

products. The reaction proceeds through the initial formation of enol allyl ethers and is also applicable to substituted acetophenones, five-membered 2,3-dihydro-1*H*-inden-1-ones and seven-membered 6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-ones [62].

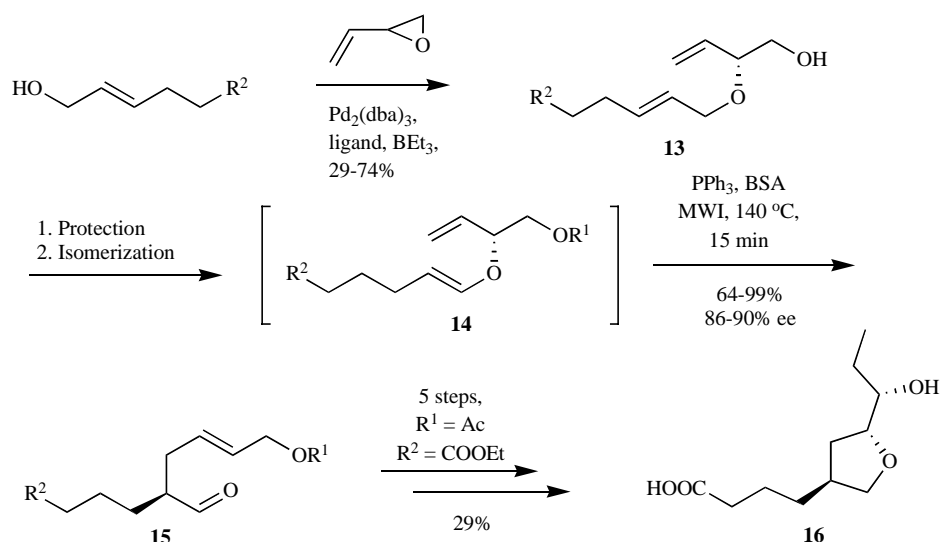
REARRANGEMENTS OF ARYL ALLYL ETHERS

Examples of microwave-assisted CR of aryl allyl ethers include solid-phase preparation of salicyl acids **22** [63], solvent-free synthesis of bis(3-allyl-4-hydroxyphenyl) sulfone **23** [64] and 1,4-benzoquinone derivative **24** [65] (Fig. (1)). The later transformation was performed on the silica gel and included spontaneous oxidation of the intermediate 1,4-bis-hydroxybenzene.

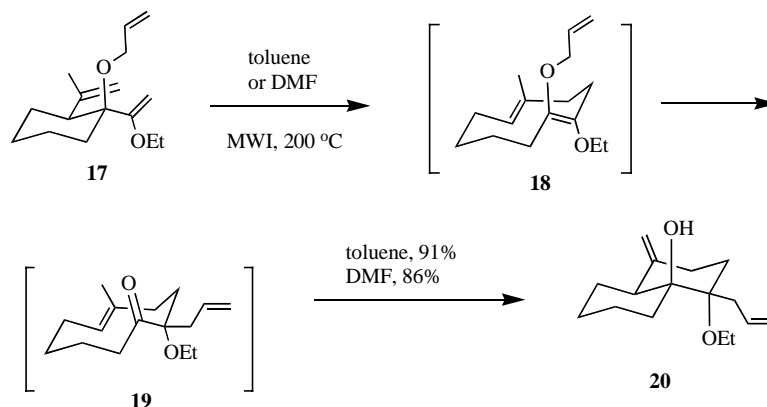
CLAISEN ORTHO-ESTER REARRANGEMENT

Several examples of direct reaction of allyl alcohols with triethyl ortho acetate under microwave conditions have been reported. This variation of the thermal Johnson's method [66], significantly accelerates the process and gives access to γ,δ -unsaturated esters **25** in moderate to good yields (Scheme (7), [67-70]).

In the similar manner reaction of the propargyl alcohol leads to ethyl penta-3,4-dienoate **26** with allen moiety (Fig. (2), [67]). Other examples of this type of transformation include synthesis of ethyl 6-oxo-11-vinyl-hexahydro-1*H*-pyrido[1,2-*b*]isoquinolin-11-yl)acetate **27** [71] and γ,δ -unsaturated esters **28** and **29**, which are valuable precursors of



Scheme 5.



Scheme 6.

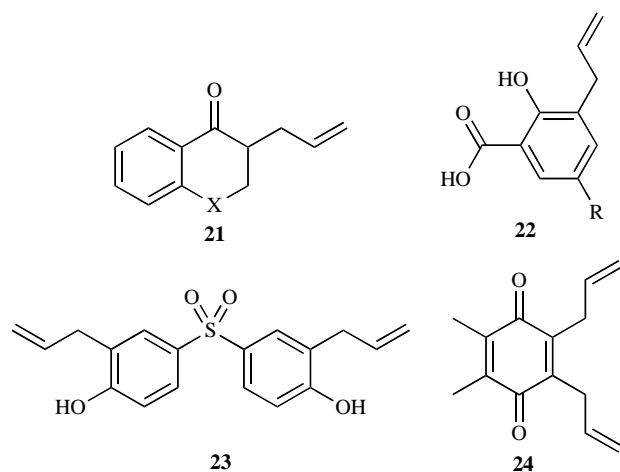


Fig. (1).

bicyclic lactones [68-69] and 2,2-divinyladamantane [72], correspondingly.

HETEROCYCLIC CHEMISTRY

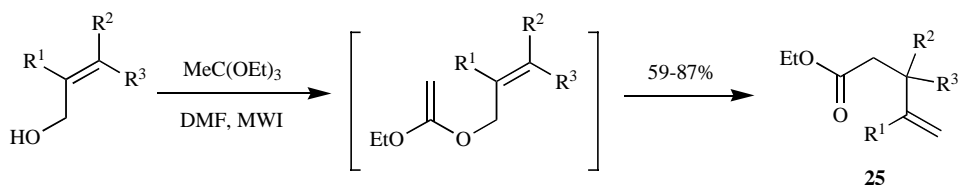
[3,3]-Sigmatropic rearrangement of allyl tetronates **30** ($\text{R}^1 = \text{H}$, $\text{R}^2 = \text{Ar}$) and the corresponding allyl tetramates (not depicted) gives 3-allyltetronic acids **31** and proceeds within

20–60 min under microwave irradiation (Scheme (8)). Consecutive sigmatropic [1,5] H shifts such as oxa-ene Conia reaction, which are common for the reactions under conventional heating, are less sensitive to microwave irradiation. Thus, the successful isolation of Claisen intermediates **31** of sigmatropic domino sequences with minimal contamination with Conia products **32** has been reported [73].

Similarly, allyl methylall] tetronic acid derivative **34**, useful for RCM synthesis of annulated butanolides, was obtained in quantitative yield starting from ether **33** [74].

Microwave induced rearrangement of 4-methyl-7-hydroxycoumarin propargyl ethers **35** proceeds regioselectively onto position 8 and the nature of the product depends on the substitution on propargyl C2 carbon (Scheme (9)). Thus, tertiary propargyl ether ($\text{R} = \text{Me}$) affords pyranocoumarin **37** in 71% yield as the result of 1,5-sigmatropic shift/cyclization sequence. Primary ethers ($\text{R} = \text{H}$) undergo intramolecular nucleophilic attack on allen carbon of **36** to give furocoumarins **38** [75-76]. 4-Unsubstituted 7-hydroxycoumarin is of similar reactivity, while transformations of 4-propargyloxy derivatives occur regioselectively and directed onto position 3 [75].

CR of 5-*O*-prenyl flavones is regioselective and depends on a nature of solvent and source of heating. Irradiation of 7-MEM-5-prenyl chrysin **39** at 750 W in *N,N*-diethylaniline



Scheme 7.

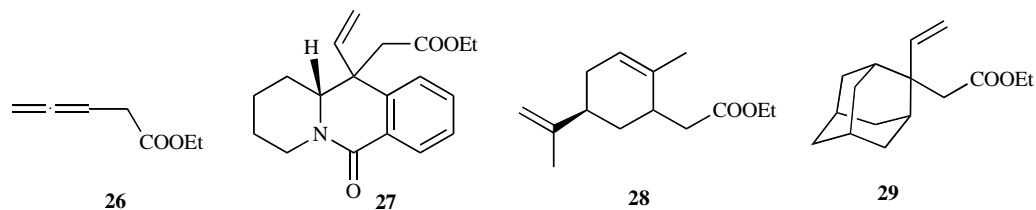
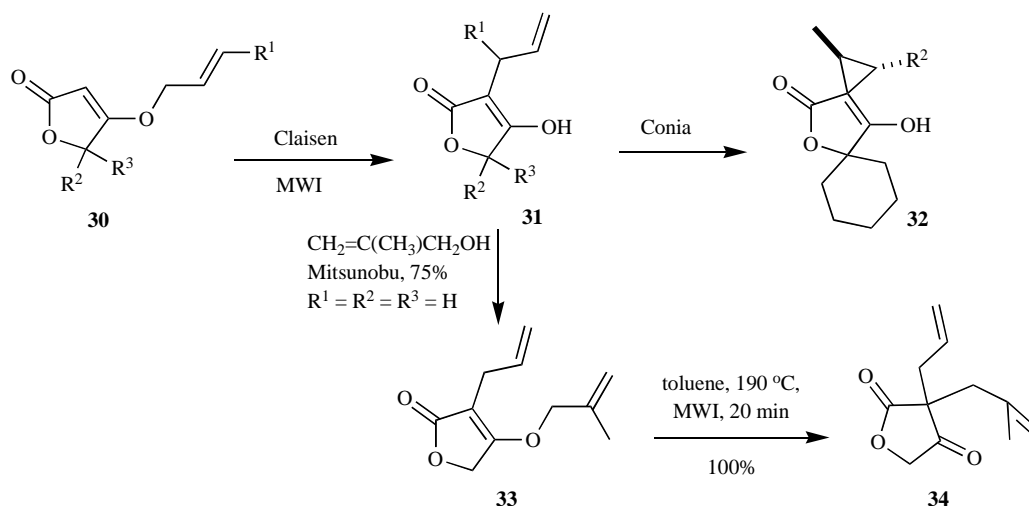
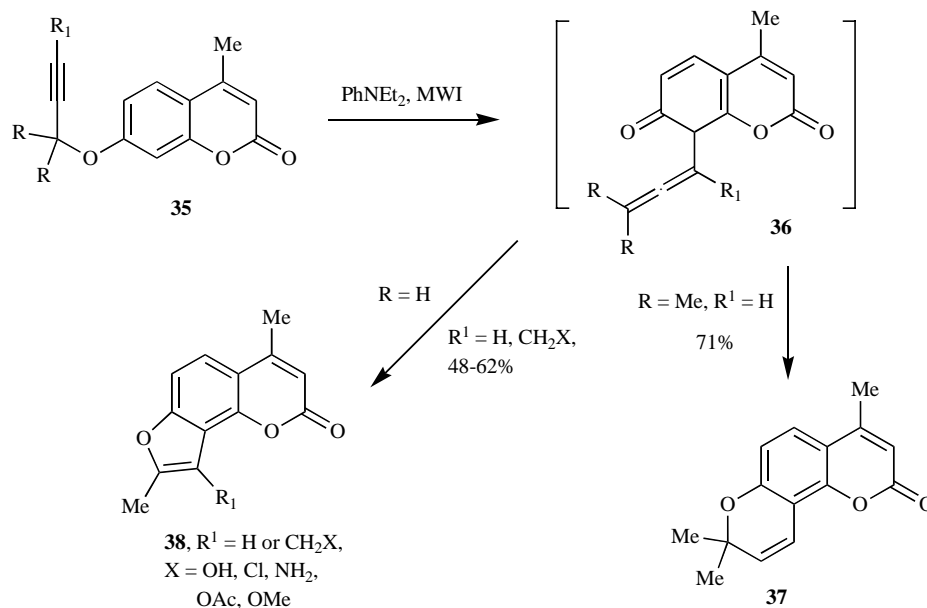


Fig. (2).



Scheme 8.

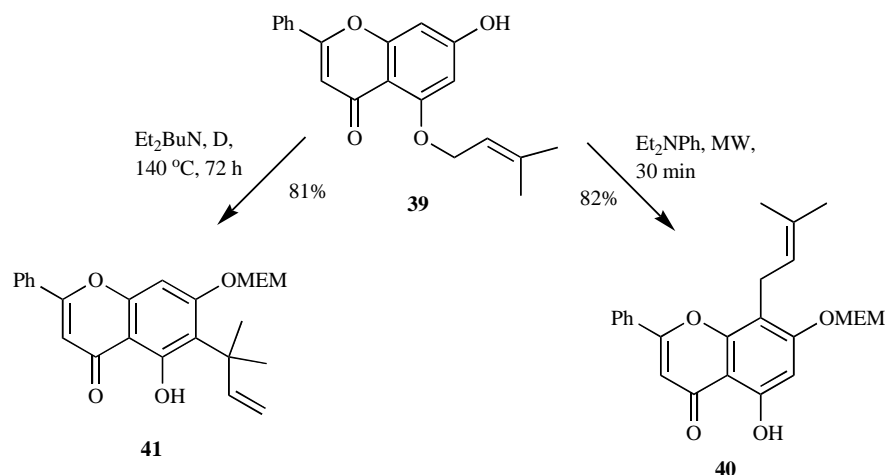


Scheme 9.

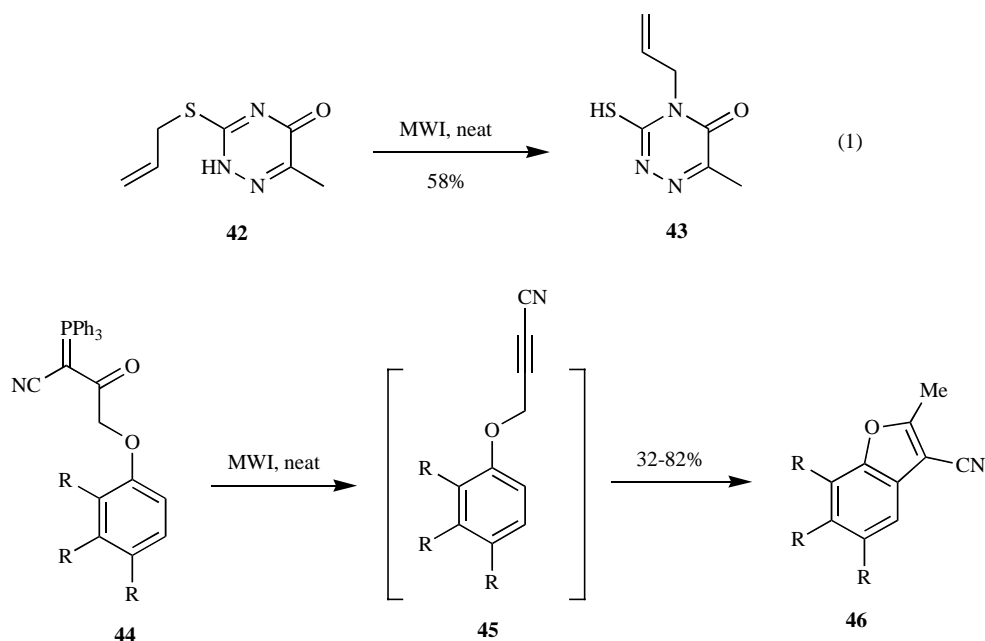
specifically yields the 8-C-(3,3-dimethylallyl)flavone **40** as a major *para*-rearranged product, while refluxing in *N,N*-diethylbutylamine gives selective access to the *ortho*-rearranged 6-C-(1,1-dimethylallyl)flavone **41** (Scheme (10), [77]).

Similarly, a rapid microwave assisted CR of 4-allyloxy-2-methylquinolines gives 65-91% 3-allyl-4-hydroxy-2-methylquinolines [78].

3-Allylsulfanyl-6-methyl-2*H*-[1,2,4]-triazin-5-one **42** undergoes [3,3] sigmatropic shift under solvent-free microwave



Scheme 10.



Scheme 11.

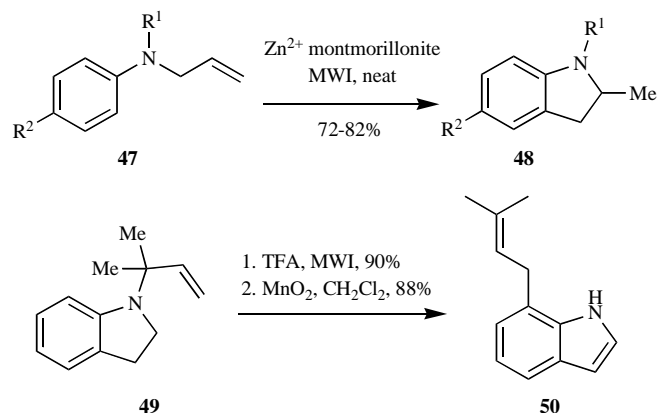
irradiation conditions to afford 4-*N*-allyl derivative **43** as a sole product in good yield (Equation (1), [79]).

One-pot synthesis of 3-cyano-2-methyl-benzo[*b*]furans **46** [80-81] starts from Wittig type reagents **44**, which under microwave irradiation undergo intramolecular olefination and spontaneous CR of the intermediate cyano derivatives **45** (Scheme (11)).

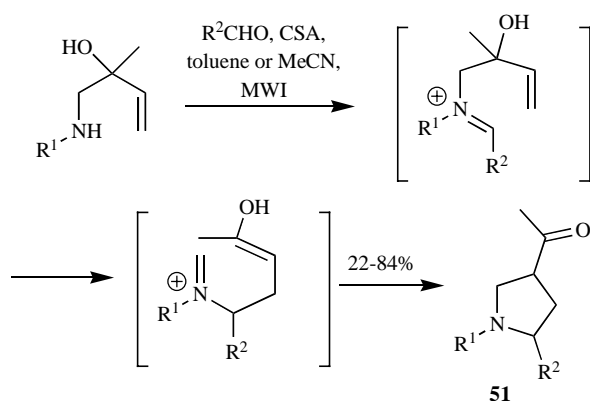
AZA CLAISEN REARRANGEMENT

Contrary to the CR, the nitrogen analog of this reaction, i.e. aza-CR has received less attention, due to several limitations. Uncatalyzed aromatic aza-CR requires long reaction times at high temperature of $250\text{--}260^\circ\text{C}$ resulting in considerable thermal decomposition of *N*-allylanilines [82]. Even though this temperature threshold could be considerably reduced by using Lewis or protic acids as catalysts, the products were isolated only in low yields. Nevertheless, catalytic approach combined with microwave irradiation proved to be advantageous. Two reported examples are depicted in

Scheme (12). *N*-Allylanilines **47** undergo 3-aza-Claisen rearrangement in the presence of Zn^{2+} montmorillonite under microwave irradiation in the absence of solvent to afford exclusively 2-methyl indoline derivatives **48** in high yields



Scheme 12.



Scheme 13.

as the product of intramolecular hydroamination [82]. Using a TFA-catalyzed aza-CR as the key step followed by MnO_2 oxidation, 7-prenylindole **50** has been prepared from indoline **49** in 62% overall yield [83].

The microwave-assisted version of the tandem aza-Cope rearrangement–Mannich cyclization has been developed (Scheme (13)). This sequence provides acylpyrrolidines **51** in a single synthetic step, which diastereoselectivity depends on the substituent R^1 and can be improved by increasing its size [84].

NATURAL PRODUCTS

Microwave assisted synthesis has been widely used for the preparation of various natural products and their syn-

thetic analogs. Thus, an asymmetric synthesis of communiol **A 16**, a tetrahydrofuran derivative from the coprophilous fungus *Podospora communis*, was accomplished applying on the key steps Pd-catalyzed asymmetric allylic alkylation/olefin isomerization/CR sequence (Scheme (5), [58]).

The naturally occurring benzoquinones primin **52a**, pal-lasone **B 52b** and isoarnebifuranone **53** (Fig. (3)) were synthesized by a simple protocol involving microwave accelerated CR of the aryl allyl ethers, followed by hydrogenation of the side chain alkene, and oxidation to the quinone [85-87]. Similar methodology with an additional ring-closing metathesis step, was used for the synthesis of the bridged benzoquinone **54**, related to the ansamycin antibiotics [85].

Synthesis of 6'-prenylisoflavone **56** ($\text{R}^1 = \text{OMEM}$, $\text{R}^2 = \text{OH}$) is a key step in the preparation of kwakhurin, a characteristic component of *Pueraria mirifica* (Leguminosae) with estrogenic activity, which can be obtained in high yield by CR of ether **55** in *N,N*-diethylaniline (Equation (2)) [88]. Successive methylation and deprotection yielded the target kwakhurin **56** ($\text{R}^1 = \text{OH}$, $\text{R}^2 = \text{OMe}$) in 12% overall yield from 2,4-dihydroxybenzaldehyde.

Fronodosin **C 57** is a novel sesquiterpene hydroquinone derivative isolated from the Micronesian marine sponge *Dysidea frondosa*, found to be antagonists of interleukin-8. Construction of the tetracyclic frondosin C framework may be achieved in straightforward manner using a tandem 5-exo-cyclization/ CR strategy (Equation (3)) [89-91].

The enantiospecific total synthesis of the cytotoxic guaipyridine sesquiterpene alkaloid (+)-cananodine **60** has been described [92]. Preparation of the intermediate **59**

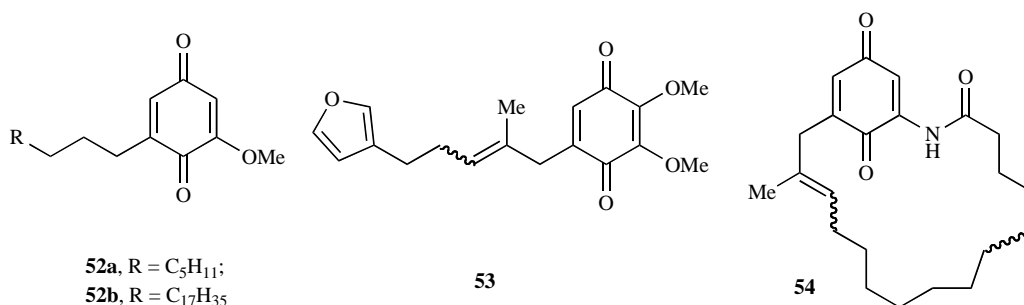
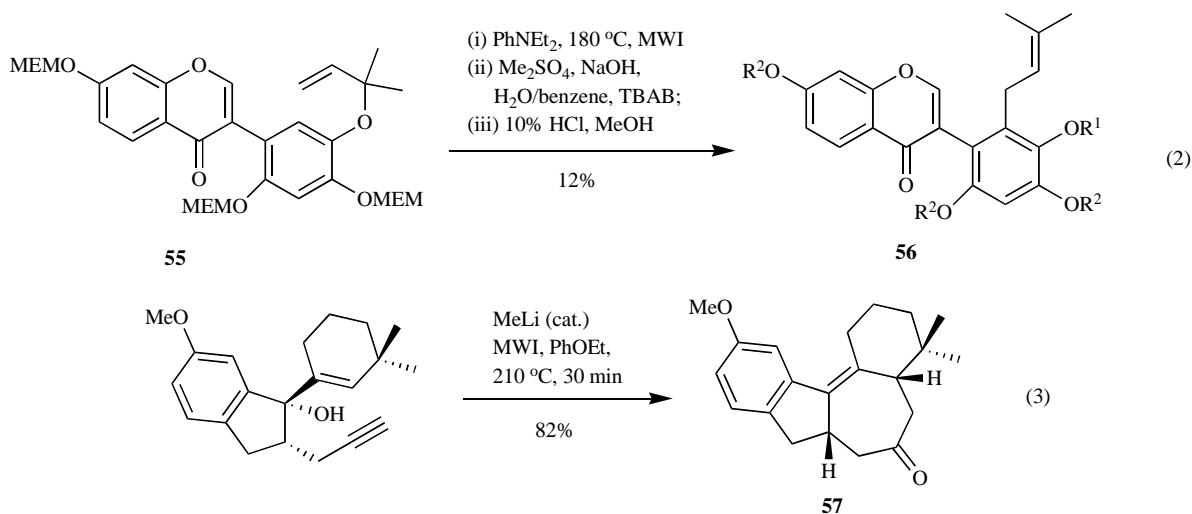
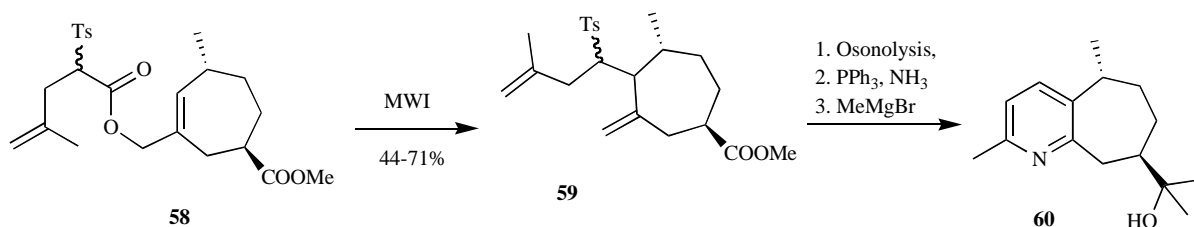
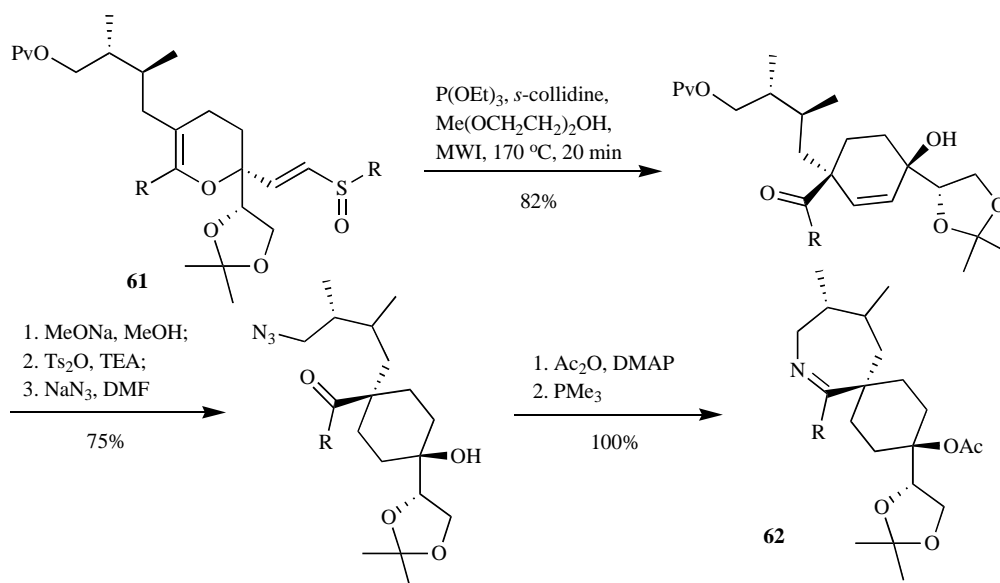


Fig. (3).

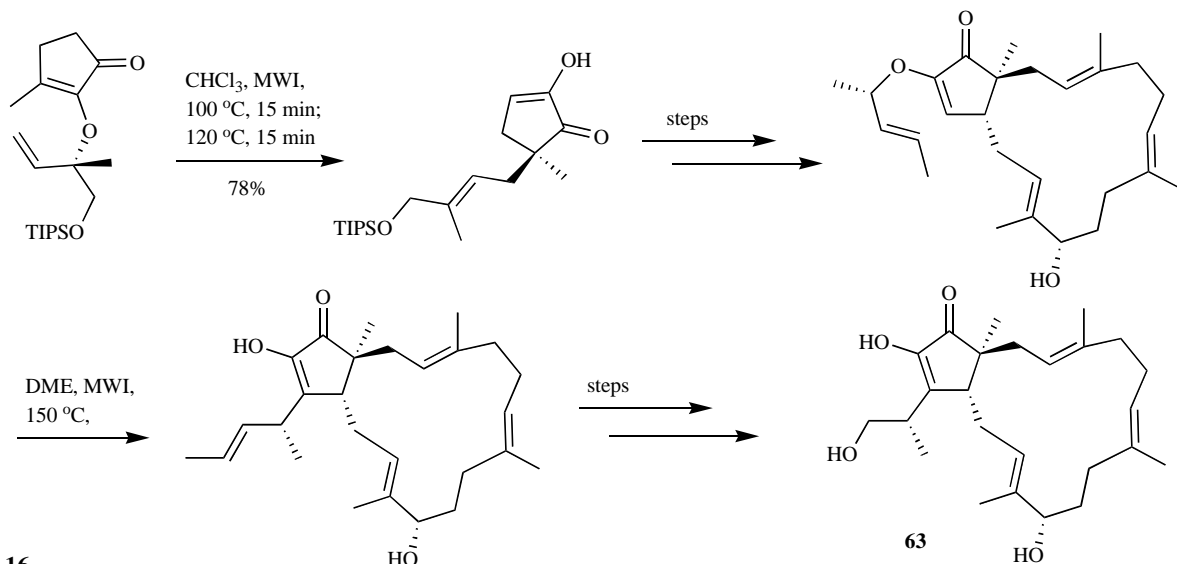




Scheme 14.



Scheme 15.

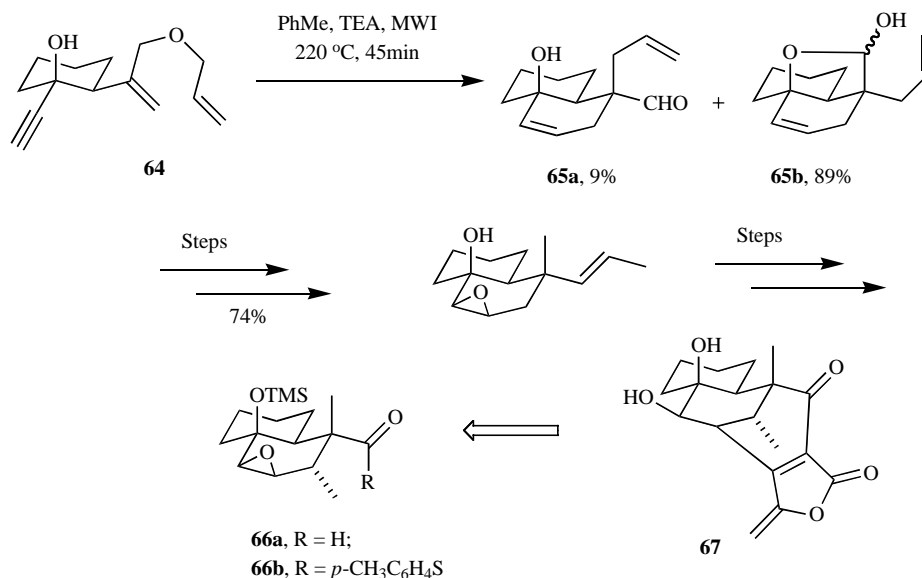


Scheme 16.

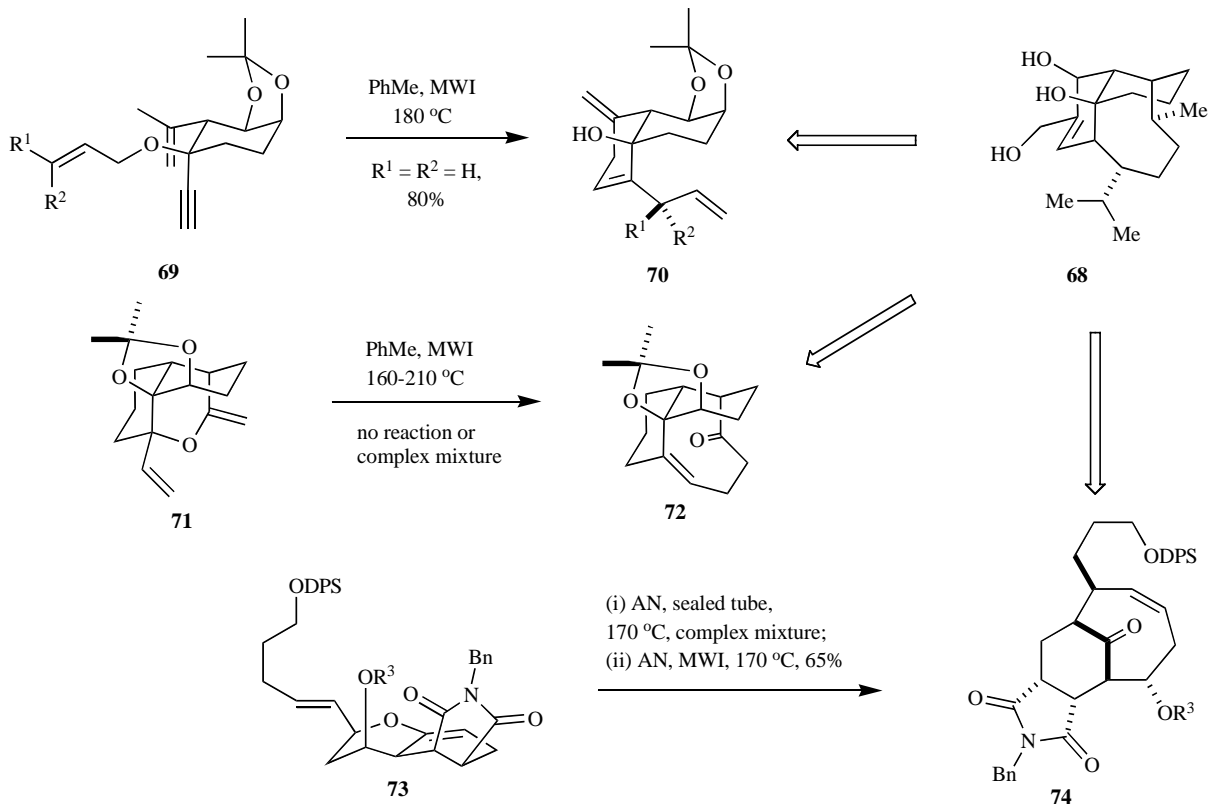
(Scheme (14)) involved microwave-assisted decarboxylative CR reaction, similar to the depicted in Scheme (3). Interestingly, treatment of **58** with BSA and potassium acetate in toluene under conventional thermal conditions yields only 52% of 3-methyl-1-(4-tolylsulfonyl)-3-butene due to the hydrolysis and decarboxylation of the 4-tolylsulfonyl-substituted ester. Contrary, reaction under microwave irradiation conditions is highly stereoselective and gives the 1,6-diene **59** in 44–71% yield as a mixture of two of the four possible diastereoisomers.

The A,G-spiroimine motif **62** of pinnatoxins, an expanding group of marine natural products characterized by the presence of unusual spirocyclic imines in their structure, can be synthesized by the multistep sequence (Scheme (15)). It includes Claisen sigmatropic process followed Mislow-Evans rearrangement [93]. The efficiency of the CR for **61** is significantly higher comparing to that of the thermal process [94].

Terpestacin **63** (Scheme (16)), a potential drug lead for anticancer chemotherapeutics, was originally isolated from



Scheme 17.



Scheme 18.

Arthrinium sp. FA1744, inhibits angiogenesis and the formation of syncytia by HIV-infected T cells. It contains the cyclic 1,2-diketone moiety which serves, due its unique reactivity as a critical core onto which can be installed all of the carbon chains. The sequence of *O*-allylation-CRs results in chemo- and regioselective enolate allylations, which can be performed asymmetrically with respect to the enolate or/and allyl fragment [95].

Synthesis of C7-C15 *trans* Decalin units **66a** and **66b** of the natural antibiotic Tetrodecamycin **67** has been accom-

plished using tandem oxy-Cope/ene/CR in 16 and 18 steps, respectively [96] (Scheme (17)). The mechanism of the process is similar to the cascade depicted on Scheme (6), although in the case of alcohol **64** the diastereoselectivity of the process was diminished by the planarization of the ring imposed by unsaturated bond, which leads to the formation of the side product aldehyde **65a**.

Two synthetic studies towards Vinigrol **68** (Scheme (18)) have been reported by Barriault group [97-98]. This natural diterpene is known as antihypertensive agent, platelet aggre-

gation-inhibiting, and tumor necrosis factor antagonist; and it bears a *cis*-decalin subunit surmounted by an eight-membered ring that constitutes an unprecedented tricyclo[4.4.4.0.4a,8a] tetradecane skeleton. Stereoselective synthesis of the *cis* decalin subunit of **68** includes microwave irradiation of allyl ethers **69** [97]. Intermediate **70** ($R^1 = R^2 = H$) has been produced in 80% yield as a product of tandem oxy-Cope/Claisen/ene reaction. Surprisingly, both *cis* ($R^1 = H, R^2 = i\text{-Pr}$) and *trans* ($R^1 = i\text{-Pr}, R^2 = H$) bulky substituents in the allyl terminal position are unfavorable for the rearrangement sequence.

The assembly of the eight membered unit of vinigrol is more challenging [98]. Attempts to generate the eight-membered ring in **72** through a Claisen-type ring expansion of the intermediate **71** were not successful. Nevertheless, cyclooctane ring can be created by CR of hydroxy Diels-Alder product **73**. Interestingly, the choice of the heating source and protecting groups is crucial for the process and the best results have been achieved for trimethylsilyl derivative **74** ($R^3 = \text{TMS}$) under microwave irradiation.

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