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Review Article

MEDIUM RING BENZOFUSED HETEROCYCLIC COMPOUNDS: RELEVANCE AND RECENT SYNTHETIC APPROACHES

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ABSTRACT

Benzofused medium ring heterocycles find a wide range of application in pharmaceuticals and medicinal chemistry due to their binding capability with multiple receptors. Synthesis and exploration of these types of compounds will help researchers to discover biologically active compounds with a broad range of medicinal values.

Keywords: Heterocycles, Synthesis, Pd-catalyzed reactions, ring closing metathesis, radical cyclization

1. INTRODUCTION

The term 'medium size ring' introduced by Prelog and Brown [1] is usually applied to alicyclic compounds having a ring size in the range of 8 to 11. However, 7-membered and 12-membered rings are often included for comparison purpose. Benzofused medium ring heterocyclic compounds are worth our attention for many reasons; chief among them are their biological activities. More than 80 % of approved drug molecules contain a nitrogen heterocycle within their structure [2]. Almost all of these are easy-to-make 5-7 membered rings. Although medium-ring nitrogen heterocycles (containing 8–11 members) are present in biologically active natural products, they are absent from medicinally important structures. This is principally due to the difficulties associated with their synthesis. Efficient access to medium-sized rings remains a challenging goal in synthetic organic chemistry. The unfavorable entropy effect and transannular interactions are among the difficulties that have to be overcome in order to achieve such transformations. Therefore, organic chemists have been making extensive efforts to produce these heterocyclic compounds by developing new and efficient synthetic transformations.

Benzofused cyclic molecules incorporating at least one nitrogen atom in the structure are often referred to as "privileged structures" owing to their capability of binding to multiple receptors with high affinity [3]. Benzofused seven- or eight membered cyclic amines, named as benzazepines or benzazocines respectively, exhibit important pharmacological properties and are currently under intense scrutiny for their physiological activity. For example, 1-benzazepine systems [4] have shown significant antimicrobial and analgesic activity. 2-Benzazepines have been reported [5] to be used as non-peptide mimics for the well-known tri-peptide sequence Arg-Gly-Asp (RGD), which interacts with αvβ3 integrin, a pivotal protein that plays a key role in cell-cell signaling and acts as its antagonist. 3-Benzazepines have inhibitory effect on reverse transcriptase [6]. 1-Benzazocine derivatives are described as CCR-5 antagonists and used against HIV infections and some other diseases also [7]. These cyclic amine moieties are present in many pharmaceutically active naturally occurring molecules, for example: i) Galanthamine (**1**) [8] isolated from *Galanthus woronowii* or *Galanthus nivalis* is one of the effective drugs for Alzheimer disease, the most common case of elderly dementia; ii) (+) –FR900482 (**2**) [9] isolated from *Streptomyces sandaensis* and acts as an anti-tumor antibiotic;. iii) (-) Pancracine (**3**) [10] isolated from *Rhodophiala bifida* of USA shows hypotensive and anticonvulsive activities. Lorcaserin (**4**), [11] a 3-benzapepine derivative, is a selective serotonin 5-HT2C receptor agonist for the treatment of obesity is recently synthesised. Besides cephalotaxine (**5**), [12] buflavine (**6**), [13] lycoramine (**7**), [14] chilenine (**8**), [15] and montanine (**9**) [16] are notable for their unique and synthetically challenging structures (Fig 1). Similarly a number of natural products endowed with diverse biological activities are found to incorporate oxygen heterocycles of varying ring sizes, linearly fused with aromatic moiety (Fig 1). Among them heliannuol A (**10**) belongs to a new group of phenolic allelochemicals.

These were isolated from the cultivated sunflowers (*Helianthus annuus*) which exhibit activity against dicotyledonous plant species [17]. The analogous eight-membered benzannulated ether helianane (**11**) has been isolated from a marine sponge [18]. Bauhinoxepin J (**12**), containing seven membered benzofused oxacycle shows Cytotoxic and antimycobacterial activity [19]. There are many more benzofused medium ring heterocyclic compounds having pharmaceutical importance. It is therefore not surprising that the development of simple and convenient procedures for the synthesis of medium ring heterocycles continue to attract organic chemists.

There are several methodologies for the synthesis of medium ring heterocyclic compounds. Previously these were prepared mainly by utilizing Beckmann rearrangement, Friedel Crafts reaction, Schmidt reaction or simply by acid catalyzed cyclization [20]. These methods have lot of limitations in terms of yield and reaction conditions. In recent years, using transition-metal catalysts, significant success for the formation of benzofused medium-sized rings has been achieved. Recent reports based on intramolecular cyclization offer attractive routes for the synthesis of these ring systems under milder conditions [21]. The intramolecular cyclization processes mainly involved palladium catalyzed reactions, free radical cyclization and ring closing metathesis. There are also a number of reports for the synthesis of medium ring benzofused systems using greener approaches.

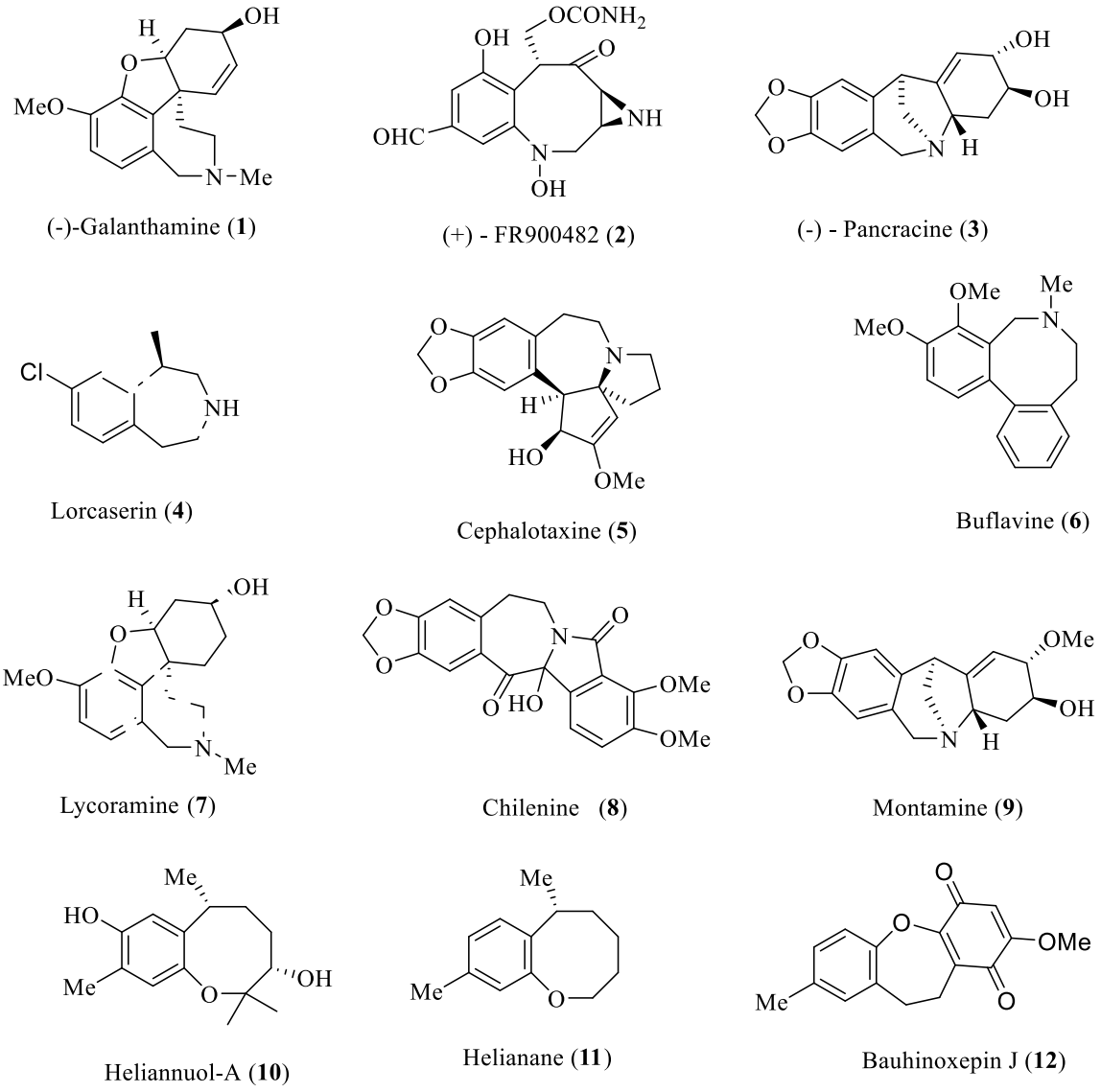
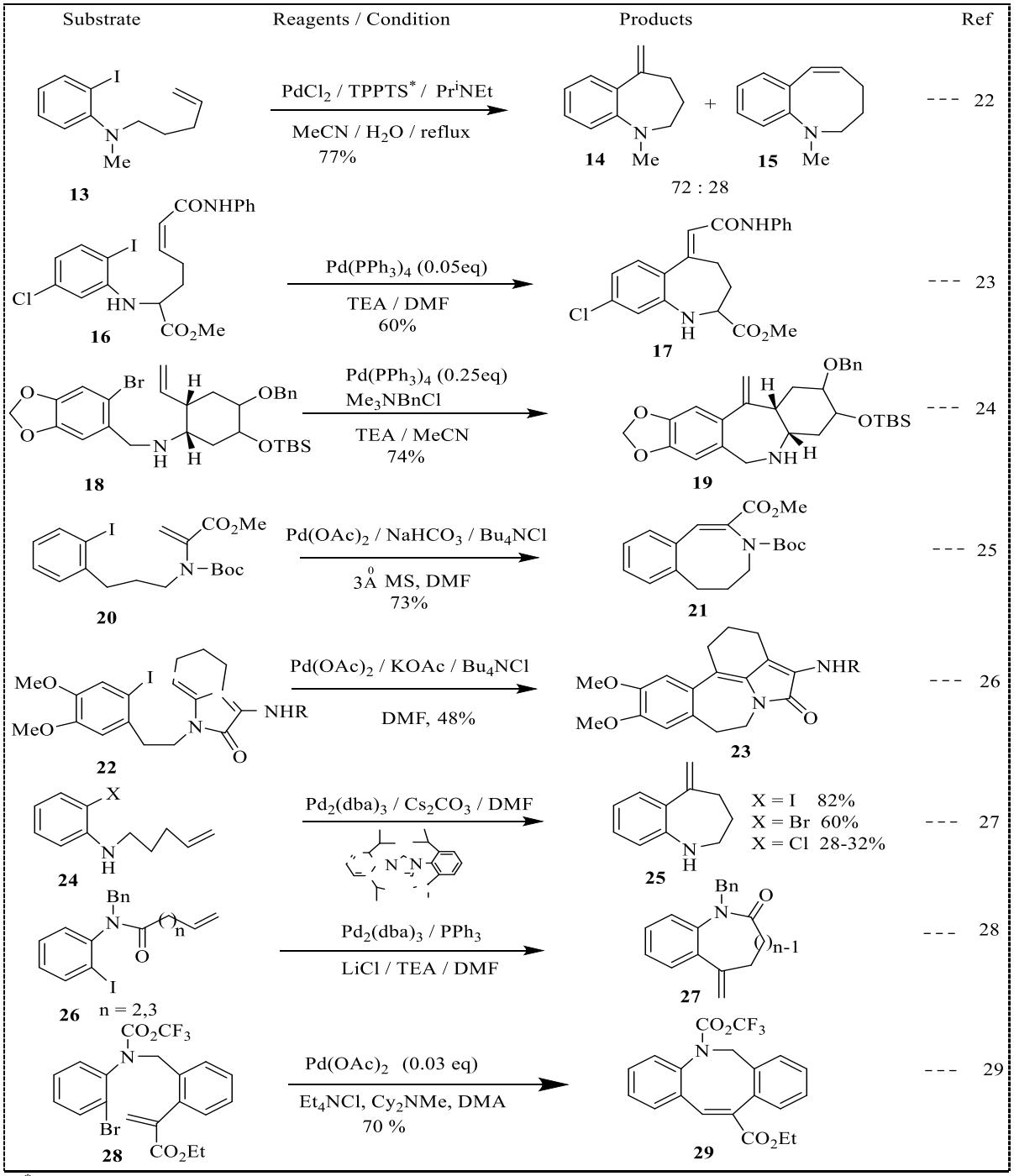


Fig.1: Structures of various medicinally active heterocycles

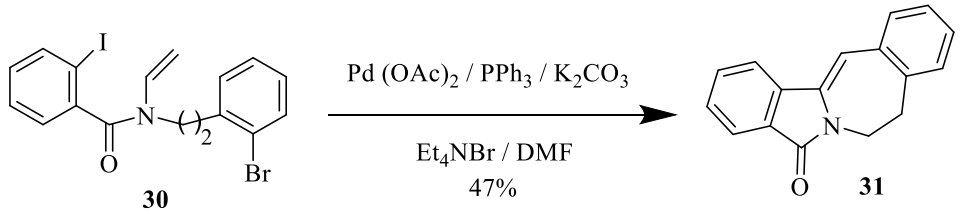
2. Syntheses Using Pd-Catalyzed Reactions

Palladium catalysis has achieved the status of an indispensable tool for organic synthesis. Among basic types of palladium-catalyzed transformations, Heck reaction and related chemistry occupy a special place. Heck reaction, the palladium catalyzed arylation of olefins, is widely used for C-C bond formation in organic syntheses. The intramolecular Heck cyclization of alkenes, with or without ligands, has also emerged as a powerful tool for the construction of benzannulated cyclic amines and analogues. An overwhelming preference for the *exo*- mode of cyclization has been noted in most of the cases. There are, however, few examples where *endo*- mode is preferred depending upon the nature of the catalyst and the electronic effect of the substituent group in the substrate molecule as presented below (Chart-1).

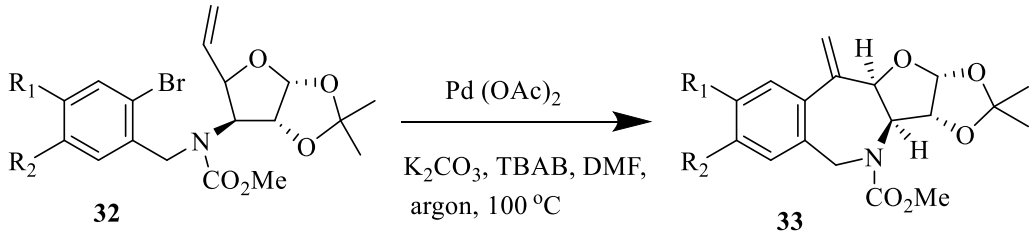


*TPPTS = triphenylphosphine trisulfonate sodium salt

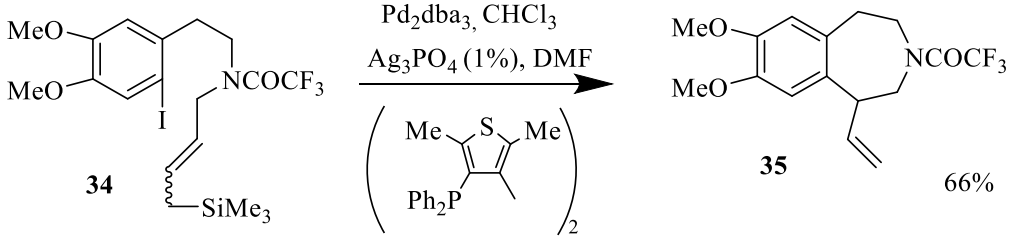
Chart-1



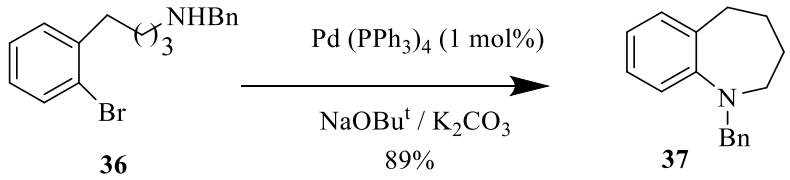
Scheme:1



Scheme: 2



Scheme: 3



Scheme: 4

Domínguez *et al*. [30] have reported a synthesis of the benzofused seven membered nitrogen heterocycle **31** using a tandem Heck reaction of n-vinyl-2-iodobenzamides **30** (Scheme-1).

A novel syntheses of chiral functionalized 2-benzazepines derivatives **33** using D-glucose derivatives **32** as precursors have been reported by Majhi *et. al.* (Scheme 2) [31]. Facile annulation with good yield of benzazepines onto furano-sugars has been achieved through Pd-catalysed intramolecular Heck reaction.

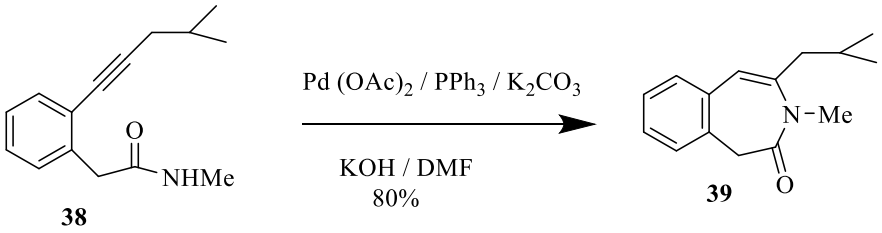
The intramolecular Heck reaction of the iodoaryl compound **34** having a *N*-allylsilane moiety was carried out in presence of the chiral ligand (+)-4,4′-bis(diphenylphosphino)-2,2′,5,5′-tetramethyl-3,3′-bithiophene leading to the chiral synthesis of 3-benzazepine **35** with 92% (Scheme 3) [32].

Other palladium catalyzed reactions:

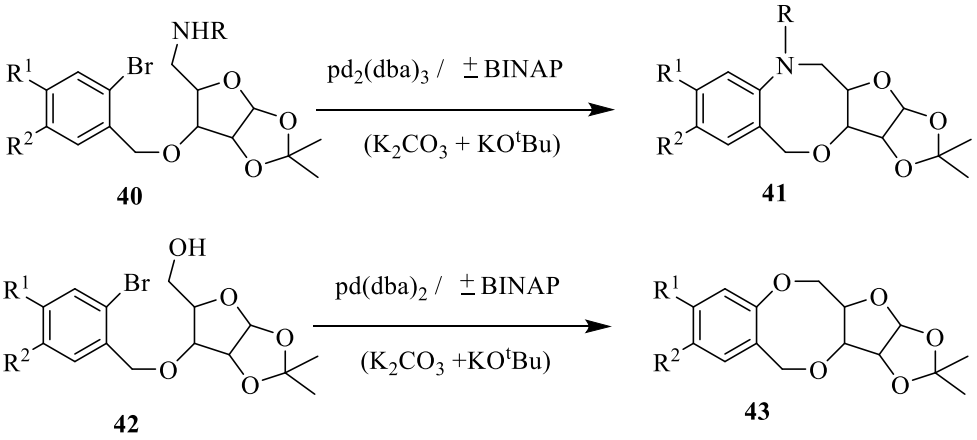
i) A series of intramolecular palladium catalyzed arylaminations were reported by Buchwald and co-workers leading to the synthesis of 1-benzazepine derivatives **37** from secondary amine **36** (Scheme-4) [33].

ii) Mitchell and co-workers [34] reported the synthesis of 3-benzazepines **39** in excellent yield from **38** using Pd-catalyzed intramolecular cyclization of amides to alkynes (Scheme-5).

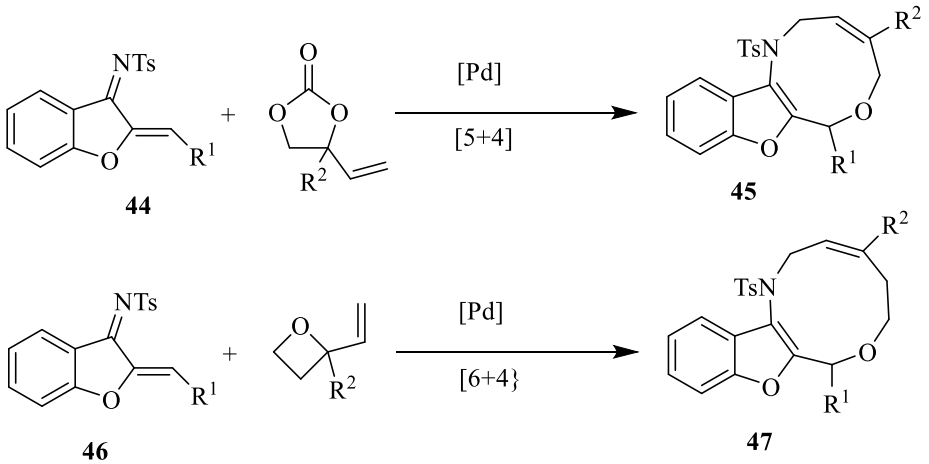
iii) Chattopadhyay *et. al*. [35] have reported syntheses of a series of chiral 8-membered benzofused heterocycles using Buchwald-Hartwig methodology. Palladium catalyzed intramolecular amination and etherification reactions were successfully used to synthesis 8-membered chiral benzofused rings **41**, **43** starting from carbohydrate as chiral precursors (Scheme-6).



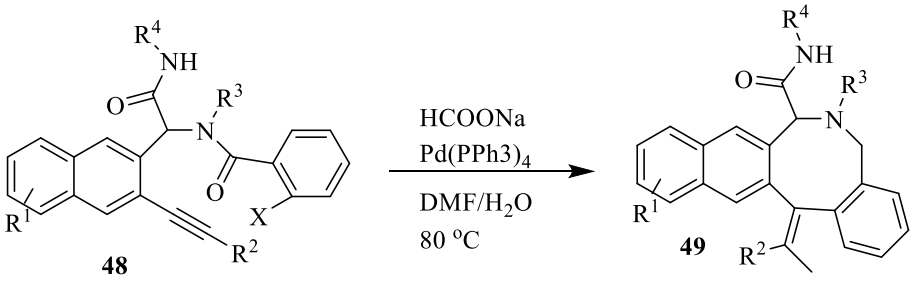
Scheme: 5



Scheme: 6



Scheme: 7



Scheme: 8

iv) As a direct strategy for the formation of medium-sized rings from readily available building blocks, Very recently Yu Zhao and co-workers [36] have successfully synthesized nine- and ten- membered ring using palladium catalyzed [5+4] and [6+4] cycloaddition reactions respectively (Scheme-7).

Very recently, Balalaie and co-workers [37] have disclosed a concise diastereo- and regioselective cyclization approach to highly functionalized azocinoquinolines **49** based upon a cyclizing reductive carbo-palladation (Scheme-8).

3. Syntheses using ring closing metathesis

Ruthenium catalyzed ring closing metathesis has emerged as a powerful methodology for the construction of a great variety of nitrogen heterocycles. During the past few years the RCM reaction has been extensively used for the synthesis of benzofused nitrogen heterocyclic systems. The versatility of Grubbs 1st and 2nd generation catalysts **50** and **51** respectively (figure-2) for the cyclization of different ring sized olefins is well documented [38]. Some selective examples of synthesis of benzofused heterocycles are given below.

Using RCM van Otterlo and co-workers [39] have reported an expedient approach to the synthesis of 2-benzazepines and azocines derivatives **53** and **55** from **52** and **54** respectively (Scheme-9).

Construction of 1-benzazepine (**57**) and 2-benzazepine (**59**) derivative from diolefins **56** and **58** using RCM has been reported by Snieckus and Lane (Scheme-10) [40].

Bennasar *et al*. [41] have reported the synthesis of a variety of enamide-containing benzofused heterocycles **61** by RCM reaction of appropriate enamides **60** (Scheme-11).

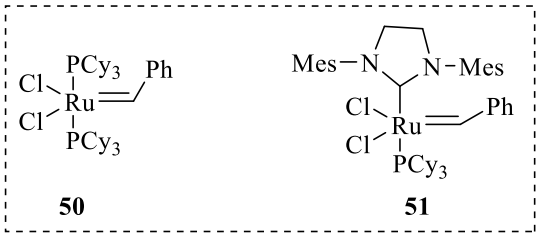
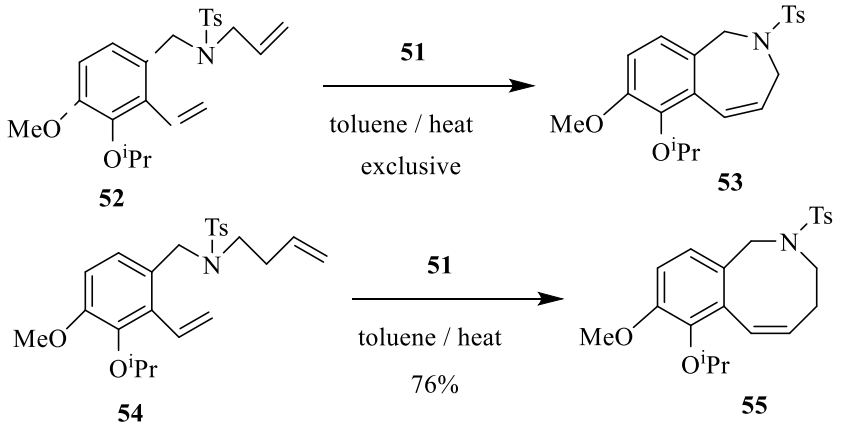
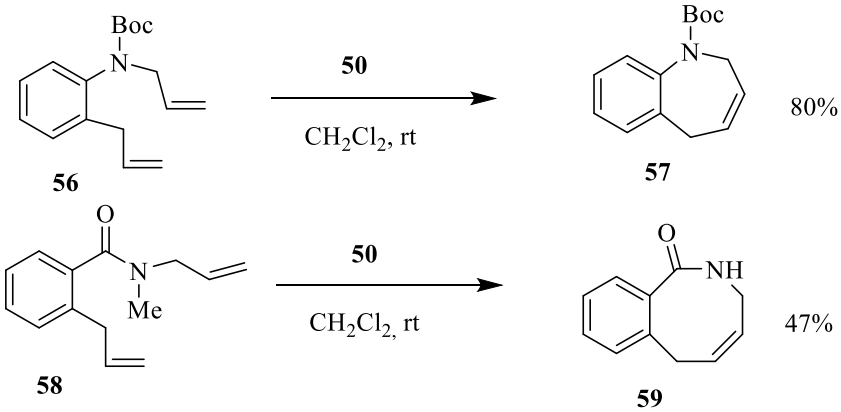


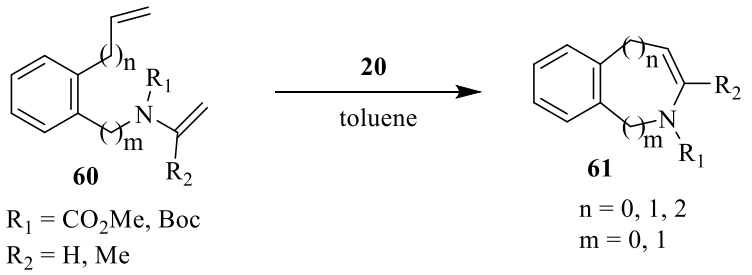
Fig. 2: Grubbs 1st and 2nd generation catalysts



Scheme: 9



Scheme: 10

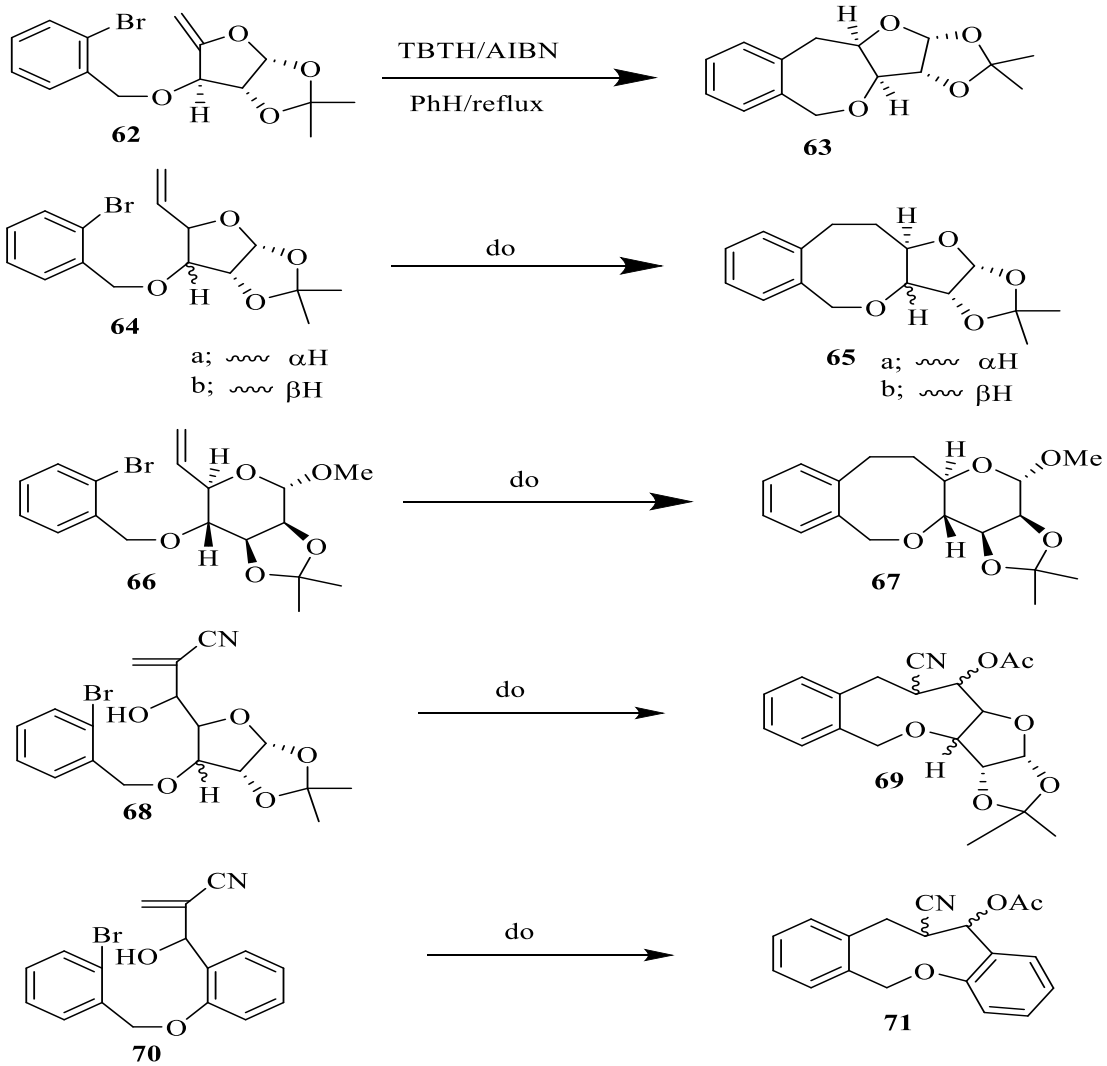


Scheme: 11

4. Syntheses using radical cyclization method

Free radical cyclization reactions are important tools for the construction of various types of cyclic compounds including biologically active natural products and pharmaceuticals. The advantages these reactions offer to the synthetic organic chemist include mild reaction conditions with high levels of regio- and stereo-control along with significant functional group tolerance. Radical cyclization is widely used for the synthesis of seven and eight membered oxygen and nitrogen heterocycles.

Chattopadhyay and co-workers [42] have reported the syntheses of seven, eight and nine member benzofused chiral heterocycles from carbohydrates as precursors (Scheme-12). Using tributyltinhydride a highly regioselective *endo-trig*-aryl radical cyclization has been achieved. In another example an efficient synthesis of dibenzofused nine-membered ring with very high yield using sequential Baylis-Hillman and radical cyclisation has been reported (Scheme-12) [42b].



Scheme: 12

Using similar methodology Kamimura *et al*. [43] have synthesized 2-benzazepine analogues **73** from the easily accessible bromobenzyl amide **72** (Scheme-13).

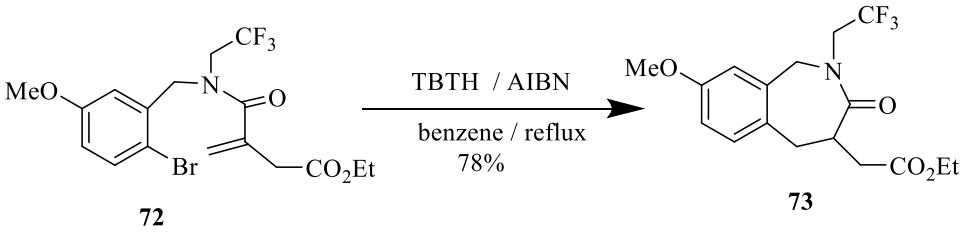
Castedo and co-workers [44] developed an exclusive stereoselective 7-endo cyclization method towards the synthesis of the *trans*-3-benzazepine system **75** from the biologically active alkaloid. They synthesized the 3-benzazepine derivative **77** from **76** by intramolecular radical cyclization. In this case also only compound **77**, arising from a 7-*endo-trig* cyclization route, was obtained from the reaction mixture in 65% yield; no *exo*-product was detected in the reaction (Scheme 15).

There are few other reports in the literature regarding TBTH mediated radical cyclization for the preparation of nitrogen heterocycles and analogues [46]. In all the cases the general guidelines for the radical cyclization 7- and 8-bromo enamide **74** (Scheme-14). In this case the intramolecular radical cyclization was too fast to get any reaction product arising from hydrogen transfer to aryl radical even under very high TBTH concentration.

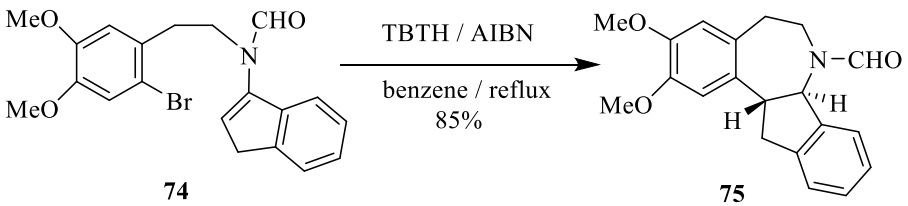
Rigby *et al*. [45] demonstrated an elegant approach towards the synthesis of a structurally complex and membered *N*- heterocycles has been found to similar to be that of carbocycles favoring the *endo* process [46b].

Ishibashi and co-workers reported synthesis of lennoxamine **79** from iodo-olefin **78** using radical cascade reaction involving 7-*endo* cyclization and homolytic aromatic substitution of the resulting radical onto phenyl ring (Scheme-16) [47].

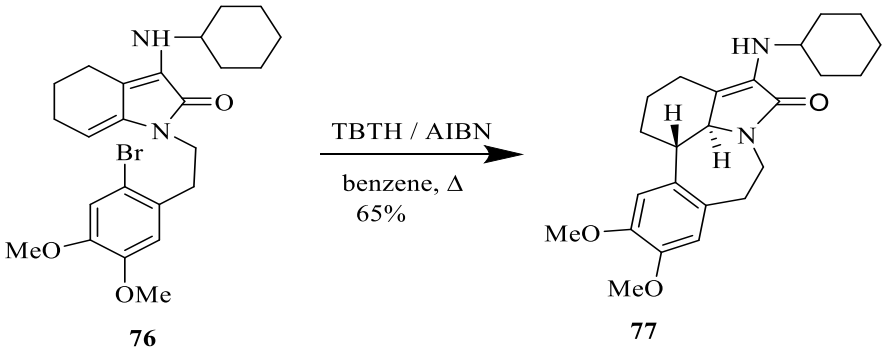
Similar methodology was applied (Scheme-17) for a concise construction of the ring skeleton of cephalotaxine (**5**) [48] from **80**. Bu_6Sn_2 is used to facilitate the homolytic aromatic substitution.



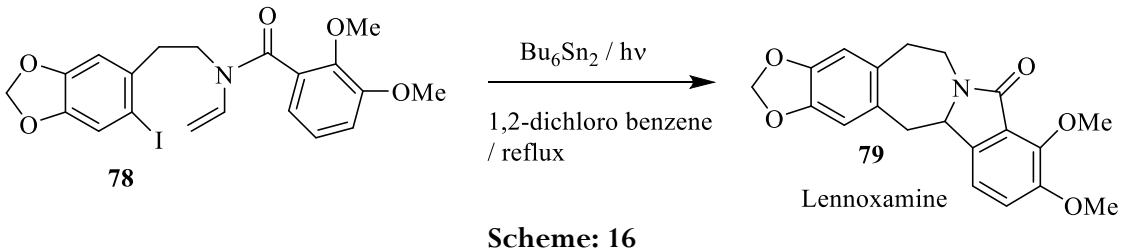
Scheme: 13



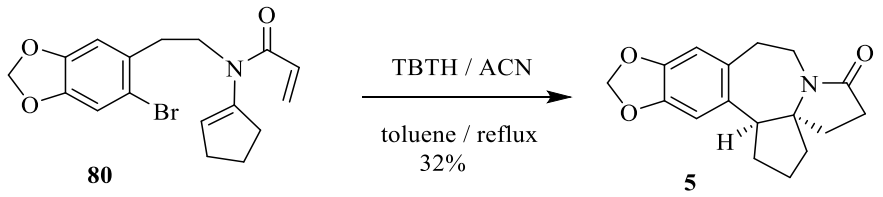
Scheme: 14



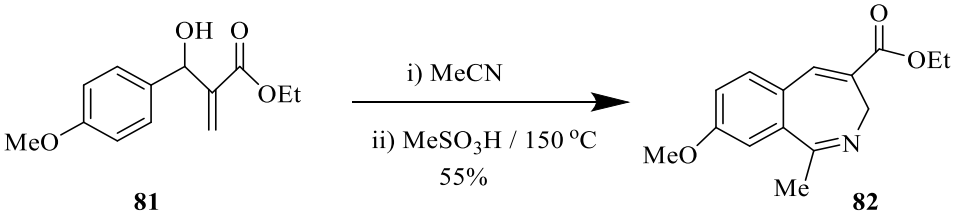
Scheme: 15



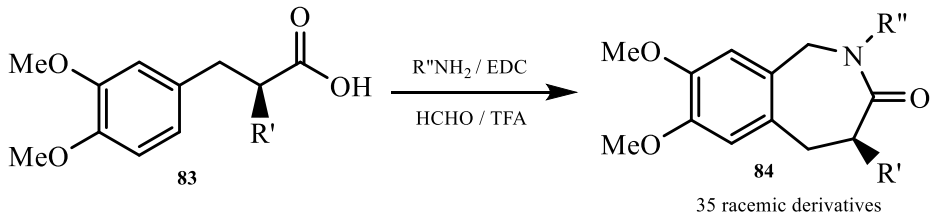
Scheme: 16



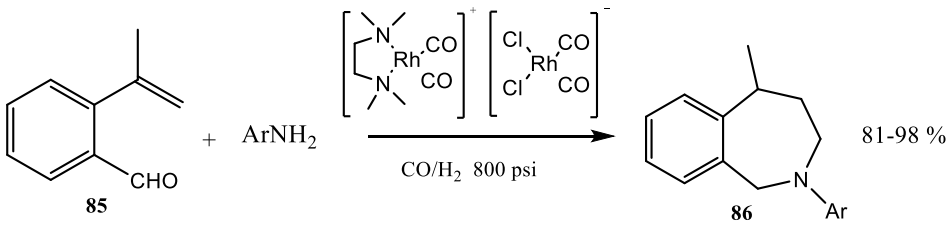
Scheme: 17



Scheme: 18



Scheme: 19



Scheme: 20

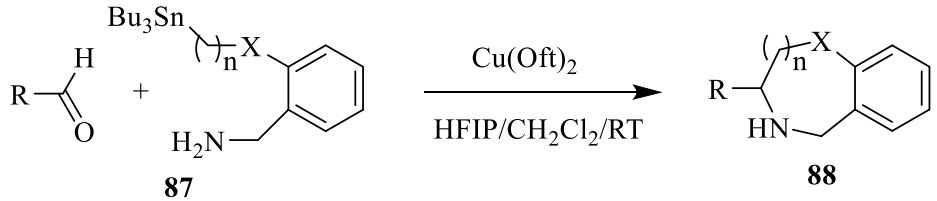
5. Syntheses by other methods

i) Basavaiah and co-workers [49] have developed a facile one pot synthesis of the benzazepine derivative **82** using tandem construction of C-N and C-C bonds via the simultaneous Ritter and Houben-Hoesch reactions on Baylis-Hillman adduct **81** (Scheme-18).

ii) Synthesis of 2-benzazepines, which are potentially good candidates for new drug therapies to treat skin wounds have been prepared by Kakimura *et. al*. [50]. They synthesized the molecules 84 from substituted cinnamaldehyde 83 via an intramoecular Friedel-Crafts reaction (Scheme-19).

iii) A novel and efficient three-component, one-pot synthesis of 2-benzzepine derivatives using rhodium catalysed hydroaminomethylation (Scheme-20) have been reported by Alper *et. al*. [51] using different aniline derivatives they got excellent yield of compound **86**.

iv) Bode and co-workers [52] have recently developed new SnAP reagents for the synthesis of saturated N-heterocycles with seven-, eight- and nine-membered rings, including oxazepanes, tetrahydrobenzoxazepines, diazepanes, tetrahydrobenzodiazepines, oxazocanes and others. They reported a mild copper catalyzed cyclisation where they used SnAP reagents for cross-coupling with aldehydes to afford N-unprotected, substituted and saturated medium-sized heterocycles.

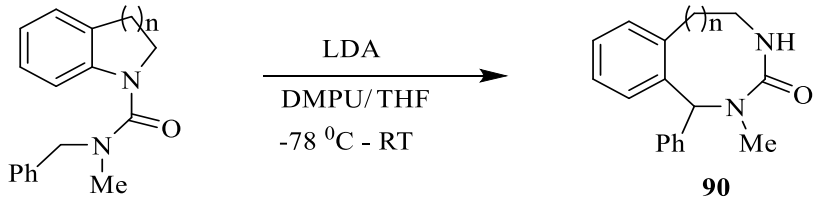


Scheme: 21

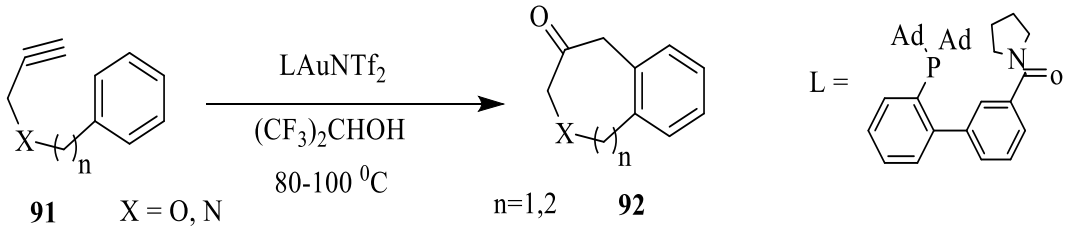
v) Clayden and co-workers [53] have report a method for the synthesis of medium ring (8- to 12-membered) benzo-fused nitrogen-containing heterocycles through n to (n+3) ring expansion of readily available heterocyclic precursors using LDA under cold condition (Scheme-22).

vi) Recently, Zhang and coworkers have reported [54] an efficient gold catalyzed one-pot expedient preparation of benzo-fused 7- and 8-membered cyclic ketones, including O-/N-heterocycles **92** from easily accessible arylsubstituted linear alkyne substrates **91** (Scheme-23).

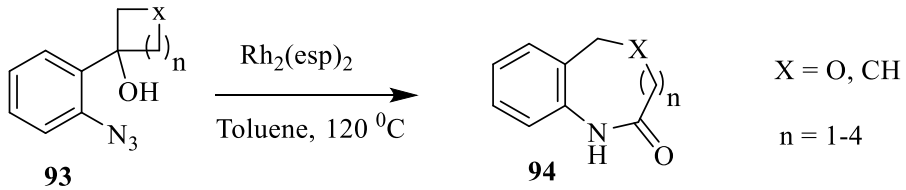
vii) A Rh_2(II)-catalyzed reaction (Scheme-24) of o-cyclobutanol-substituted aryl azides was discovered to afford medium-ring N-heterocycles **94** by Mazumder *et. al*. [55] In this method domino reaction was triggered by formation of the rhodium N-aryl nitrene, which unravels the o-cyclobutanol substituent.



Scheme: 22



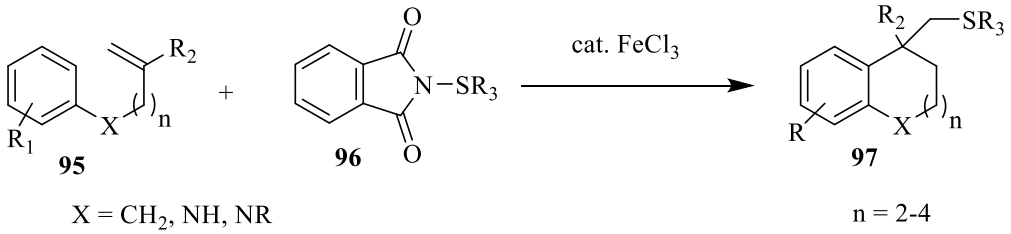
Scheme: 23



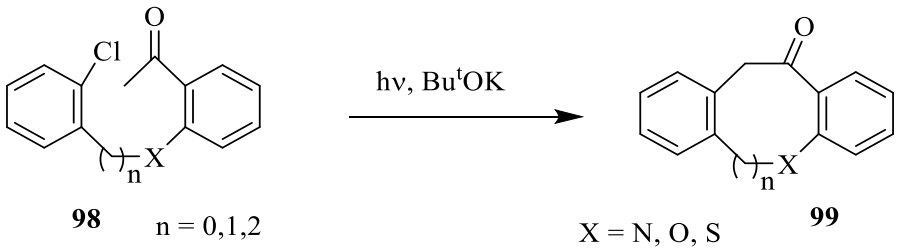
Scheme: 24

viii) Organosulfur plays a vital role in many significant biological activities, such as regulating hormone release, improving immune resistance, and maintaining a human's healthy metabolism. Very recently, Zhiping and Leiyang [56] have demonstrated synthesis of a variety of medium-sized rings, especially strained 7- and 8-membered heterocyclic compounds **97** using Friedel Crafts' reaction from unactivated alkenes **95** tethered with a sulfur atom (Scheme-25).

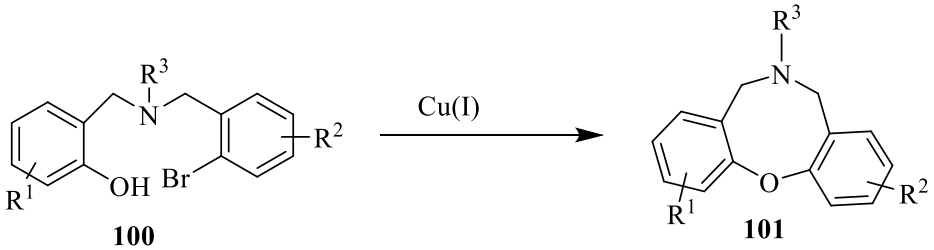
ix) The α-arylated carbonyl compounds are versatile synthetic building blocks and the structural unit of a variety of bioactive natural products and therapeutic agents. Rossi and co-workers have reported [57] a general synthesis of benzofused 7- to 9- membered heterocycles **99** by photostimulated intramolecular $S_{RN}1$ reactions using acetyl enolate anions **98** as nucleophiles.



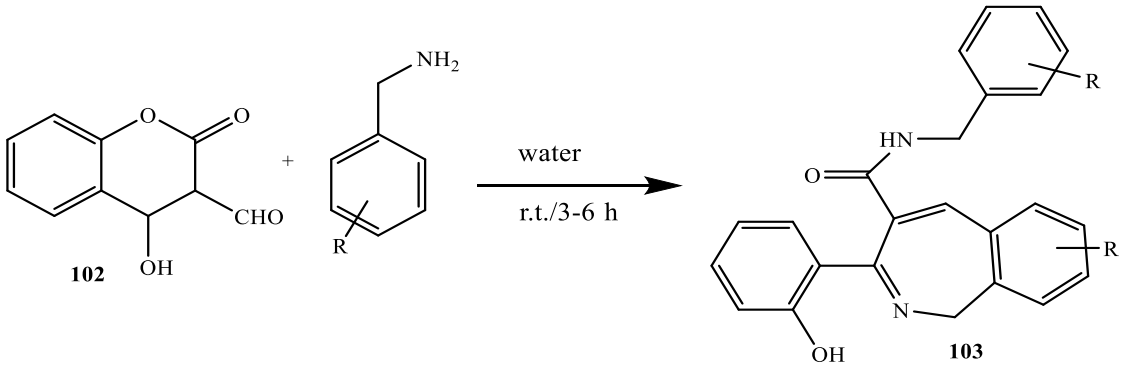
Scheme: 25



Scheme: 26



Scheme: 27



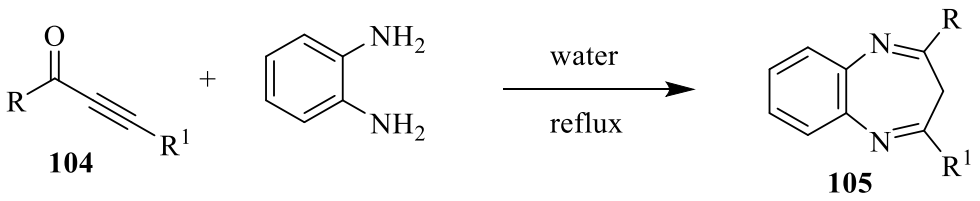
Scheme: 28

x) Tricyclic aza-hetereocyclic ring systems incorporating a biaryl ether motif are present in a number of biologically active and pharmaceutically relevant compounds. Spring and coworkers [58] have developed a new, concise, and general protocol for the synthesis of compounds based around tricyclic biaryl ether-linked scaffolds in corporating seven-, eight-, and nine-membered ring amines, using copper(I) catalyzed intramolecular carbon-oxygen bond-forming reaction from readily accessible acyclic precursors.

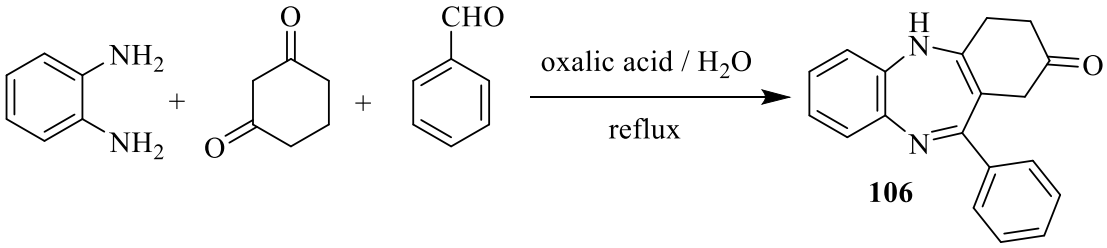
xi) An efficient one-pot catalyst-free domino approach for the synthesis of 2-benzazepine **103** derivatives in water has been reported by Kumar and co-workers (Scheme-28) [59].

xii) A facile, green approach for the synthesis of benzofused 1,4-diazepines **105** was reported by Srinivasan and co-worker [60] by cyclo condensation of ynone **104** with ortho-phenylenediamine using water as solvent.

xiii) A very high yielding one pot three component reaction in water medium was developed by Sangshetti *et. al.* [61] for the synthesis of some new benzofused-1,4-diazepine-1-one **106** using Diamine, 1,3 diketone and aromatic aldehyde and oxalic acid as catalyst (Scheme-30).



Scheme: 29



Scheme: 30

6. Conclusion

Benzofused medium ring heterocycles are very important scaffolds in medicinal chemistry. A number of publications in last few years bears ample testimony to the enormous interest in this field. We hope that the use of these compounds for the development of drugs and search for different routes to their syntheses will continue. In this context, this review will help the researchers interested in the field of benzofused medium ring heterocycles.

7. References

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