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Account

Synthesis of heterocycles by cyclization of unsaturated organolithiums: a review

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Abstract

The preparation of oxygen and nitrogen containing heterocyclic compounds by cyclization of unsaturated organolithium compounds has been reviewed. The review contains 57 references to relevant literature through early 2001.

Keywords: Intramolecular carbolithiation; Cyclization; Heterocycles; Organolithiums

1. Introduction

The preparation of heterocycles from acyclic precursors constitutes one of the most studied areas of organic chemistry, and over the past two decades, significant advances have been made in the exploitation of main group organometallics for this purpose. This brief review serves to highlight the major achievements in the development of anionic cyclization of olefinic organolithiums as a synthetic tool for the preparation of nitrogen- and oxygen-containing rings.

The 5-*exo*-trig cyclization of 5-hexenyllithiums [1] has received a good deal of attention as a route to cyclopentyl-containing systems and the preparation of carbocycles via cyclization of unsaturated organolithiums has been reviewed [2–4]. Surprisingly, the development of this methodology for the efficient preparation of heterocyclic systems has received less attention. It is hoped that this survey will provide sufficient insight into the nature of these species to stimulate further growth in this area of main group chemistry.

2. Oxygen heterocycles

The first reports of carbolithiation of an unactivated carbon–carbon double bond for the preparation of a heterocycle appear to be two brief accounts in the early 1970s describing the intramolecular addition of α-oxyallyllithiums [5] and α-oxybenzyllithiums [5,6] to the carbon–carbon π-bond of a norbornene. In 1980, Baldwin and co-workers observed the novel rearrangement of **1** to benzofuran **2** upon *ortho*-lithiation and, as illustrated in Scheme 1, proposed a mechanism for the transformation that involved intramolecular addition of the aryllithium to the tethered π-bond [7]. It should be noted that the parent system, 2-(2-propenoxy)-phenyllithium (**3**), rearranges upon warming in the presence of TMEDA via 5-*exo*-cyclization to give **4**, however, as shown in Scheme 1, subsequent γ-elimination affords the lithium salt of 2-cyclopropylphenol [8].

The first systematic studies of intramolecular carbolithiation for the preparation of a heterocycle were the seminal reports by Broka and co-workers detailing the preparation of substituted tetrahydrofurans by anionic cyclization of olefinic α-alkoxyorganolithiums [9,10]. These cyclizations were found to be highly stereoselective as a consequence of the progression of the isomerization through a rigid, chair-like transition state analogous to that observed in the cyclization of the parent 5-hexenyllithium [11,12].

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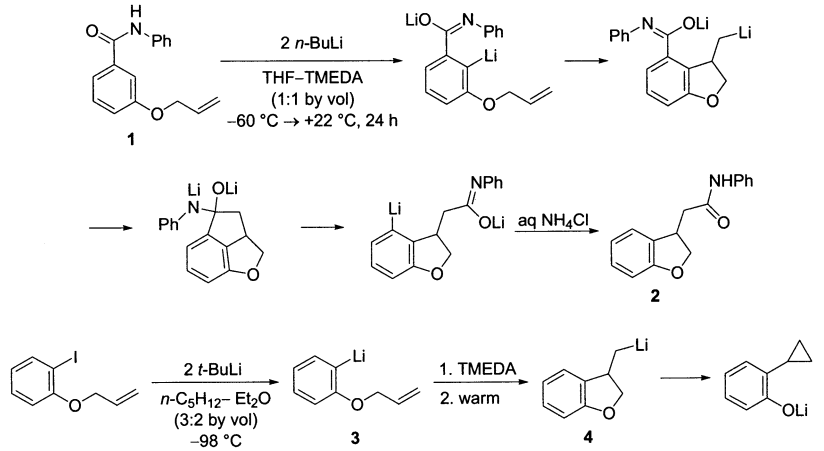
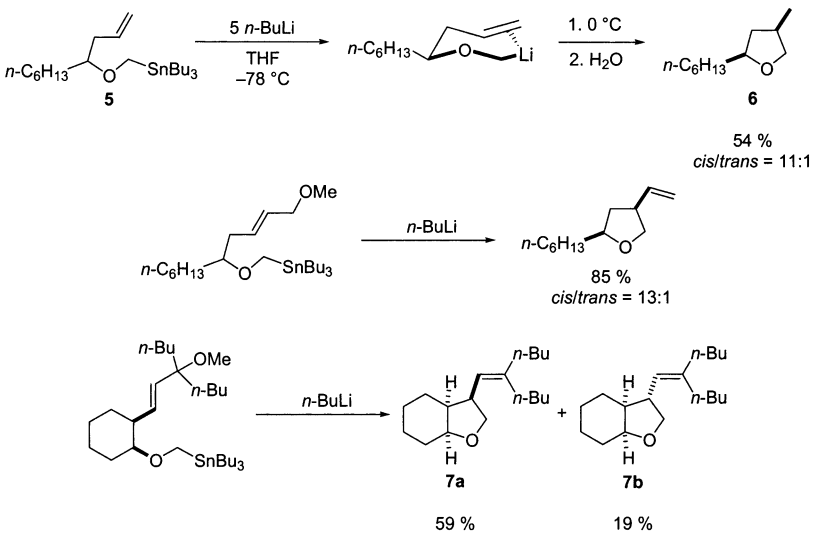
In seeking a general route to 2,4-disubstituted tetrahydrofurans [9], Broka and co-workers examined the cyclization of α-alkoxyorganolithiums derived from homoallylic (tri-*n*-butylstannyl)methyl ethers. After generation of the organolithium by low temperature lithium–tin exchange in THF, the organolithiums were observed to cyclize in a highly *cis*-selective fashion upon warming. For example as shown in Scheme 2, the cyclization of **5** to **6** proceeded in 54% chemical yield with a 11:1 preference for the *cis*-stereoisomer [9]. Moreover, as also illustrated in Scheme 2, the incorporation of a leaving group at the distal allylic position enhances not only the yield of cyclic product but the stereoselectivity as well. The 6,5-ring system of **7a**–**b** was constructed as a 3:1 mixture of isomers using this technique (Scheme 2).

Using reductive lithiation of *O*,*S*-thioacetals as a route to secondary alkoxy organolithiums, Broka and Shen further demonstrated that *trans*-2,3-disubstituted tetrahydrofurans could be prepared with good stereocontrol via intramolecular carbolithiation [10]. As illustrated in Scheme 3, treatment of α-(phenylthio)ether **8** with lithium napthalenide (LN) in THF at 0 °C gave **9** in 52% yield as a 7:1 mixture of *trans* and *cis* isomers. Here again, incorporation of a distal allylic leaving group improved both the chemical yield and the stereoselectivity of the cyclization (c.f. **10**→**11**).

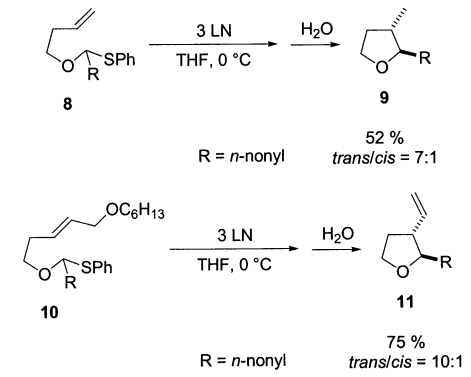
More recently, Lautens and Kumanovic have extended the pioneering work of Broka to the synthesis of bicyclo[5.3.0] systems [13]. Treatment of oxabicyclo[3.2.1] substrates such as **12** (Scheme 4) with five equivalents of MeLi in THF at −78 °C, followed by warming to 0 °C, delivered products such as **13** in 70–85% yield. This elegant methodology leads to structures containing up to five contiguous stereocenters, a bridgehead tertiary alcohol, and the newly constructed ring. Moreover, the intramolecular ring opening reaction is also applicable to the preparation of *N*-



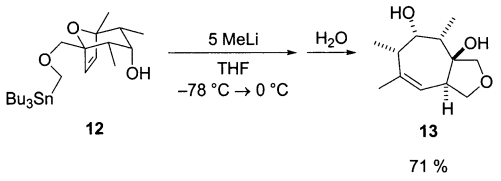
Scheme 1.



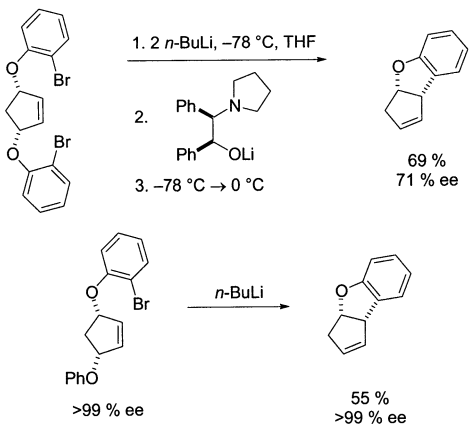
Scheme 2.



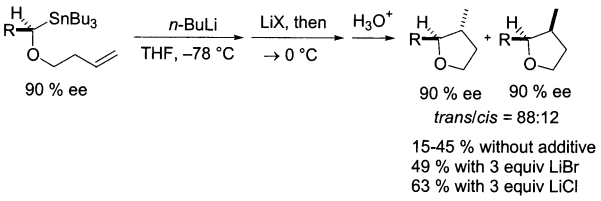
Scheme 3.



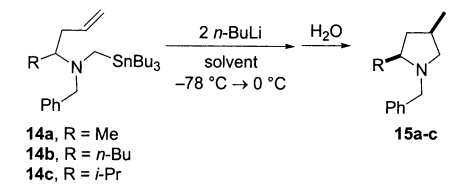
Scheme 4.



Scheme 5.



Scheme 6.



Scheme 7.

methylpyrrolidine and thiophene moieties in the bicyclo[5.3.0] systems [13].

As shown in Scheme 5, a related intramolecular S_N2' cyclization has been conducted in the presence of chiral lithium alkoxides [14]. In several instances, good chemical yields and modest enantioselectivities were observed. Nishiyama and co-workers have also reported the preparation of cyclopenta[*b*]benzofurans from chiral precursors, as also illustrated in Scheme 5 [15].

In the preparation of carbocycles, Hoppe and co-workers observed that the cyclization of 6-phenyl-5-hexenyl-(α-carbamoyloxy)alkyllithiums proceeded with complete retention of configuration at the carbanionic center [16]. Nakai's group has demonstrated that cyclization of enantio-enriched α-(homoallyloxy)alkyllithiums, prepared from the corresponding organostannanes, also proceeds with complete retention of configuration at the carbanionic center [17,18]. Thus, as illustrated in Scheme 6, a 9:1 mixture of *trans*- and *cis*-2,3-disubstituted tetrahydrofurans may be prepared with no loss of enantiomeric purity. To provide useful chemical yields, the β-elimination of lithium methoxide, pioneered by Broka, was employed to facilitate the cyclization [17,18]. The addition of lithium halide also served to improve the yield of product, presumably through disruption of intramolecular chelation of the lithium atom with the ether oxygen of the substrate which inhibits the lithium–alkene coordination essential for cyclization [18].

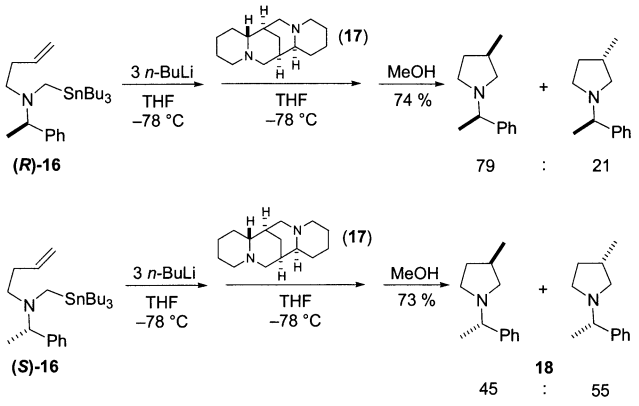
3. Nitrogen heterocycles

Not surprisingly, efforts to extend anionic cyclization to the preparation of nitrogen-containing heterocycles have been fruitful. In fact, much recent work has demonstrated that this technique is an efficient method for diastereoselective and enantioselective synthesis of this class of compounds.

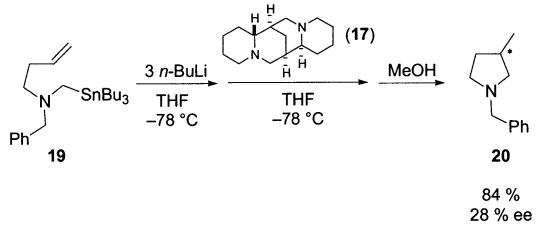
The preparation of pyrrolidines has been the focus of an intensive investigation by the Coldham group in Exeter [19–22]. For example, as depicted in Scheme 7, treatment of **14a** with two equivalents of *n*-BuLi in THF leads to **15a** in 46% yield with $>25{:}1$ *cis–trans* selectivity [21]. Conducting the cyclization in a system composed of a 10:1 (by volume) hexane–diethyl ether solvent resulted in higher chemical yields of **15a–c**, but the increase in yield was purchased at the expense of stereoselectivity (*cis–trans* $\sim 6{:}1$) [21]: the erosion of the stereoselectivity was attributed to the fact that tin–lithium exchange is not complete at -78 °C in hexane–ether solution and cyclization perforce occurs at a higher temperature in this case [21].

The presence of a chiral auxiliary on nitrogen in the anionic cyclization of the α-aminoorganolithium, such as that derived from (*R*)-**16** (Scheme 8), was found to

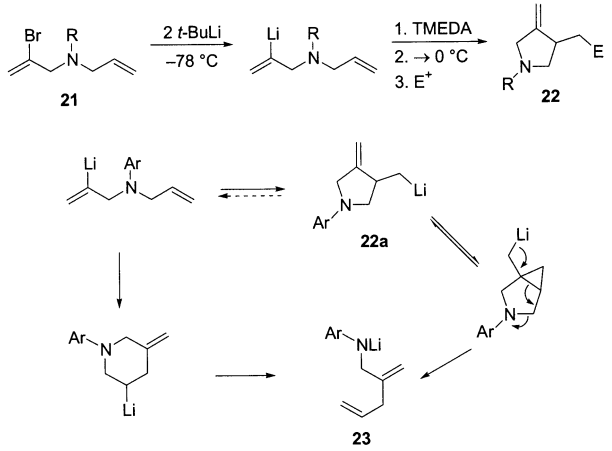
induce moderate enantioselectivity [21]. A variety of solvent variations failed to boost the ratio of the resulting diastereomers above 74:26 [21]. Optimal conditions for the cyclization involved transmetallation at −78 °C in THF, followed by addition of (−)-sparteine (**17**) dissolved in THF, which increased the diastereomeric ratios (d.r.) to 79:21 [21]. The use of the mismatched pair, (*S*)-**16** and (−)-sparteine, resulted in only 45:55 d.r. of product **18** [21]. As illustrated in Scheme 9, disappointingly low levels of enantioselectivity were observed in the (−)-sparteine-mediated cyclization of achiral **19**; *N*-benzyl-3-methylpyrrolidine (**20**) was generated in only 28% ee [21].



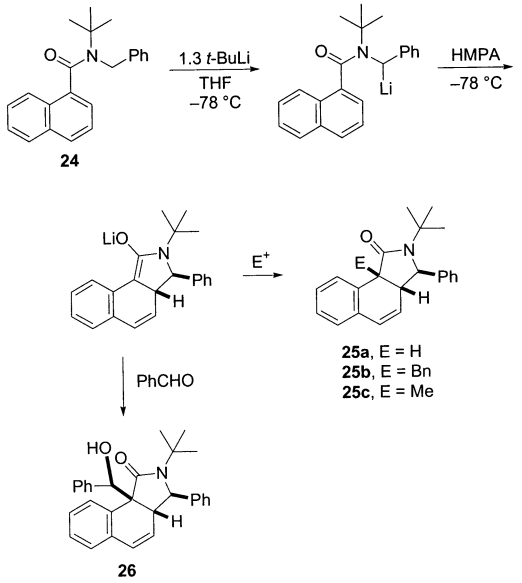
Scheme 8.



Scheme 9.



Scheme 10.

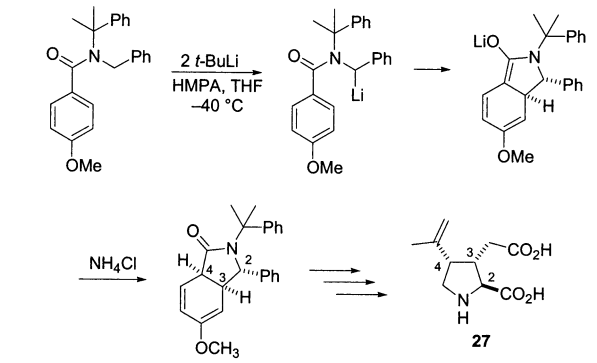


Scheme 11.

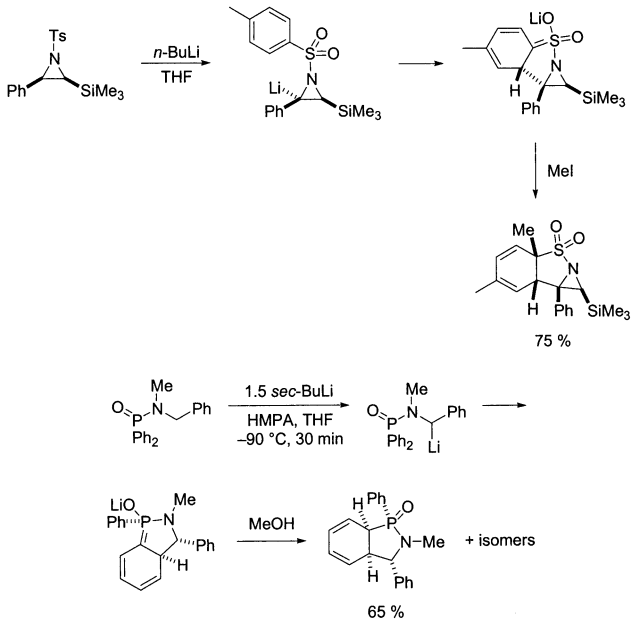
As depicted in Scheme 10, Barluenga and co-workers have reported that cyclization of the vinyllithium derived from N-protected substrates such as **21** deliver 3-methylene-4-methylpyrrolidines (**22**) in good yield [23]. The cyclic organolithium undergoes reaction with a variety of electrophiles to provide a highly functionalized pyrrolidine nucleus. However, as shown in Scheme 10, the protective group on nitrogen plays a crucial role in the reaction topology, as rearranged products such as **23** were observed when the vinyllithium was generated from an arylamine substrate [23]. Barluenga and co-workers posit that, when the nitrogen bears an aryl group, the kinetically favored 5-*exo* product (**22a**) may revert to the acyclic vinyllithium which then undergoes a slower 6-*endo* cyclization followed by irreversible cleavage to the lithium amide **23** [23,24]. There appear to be no other examples of reversible 5-*exo* cyclizations of unsaturated organolithiums in the literature [2–4], and an alternative explanation of the mechanistic course of these reactions may be found in the work of Chamberlin and co-workers [25]. Thus, as depicted in Scheme 10, a reversible 3-*exo* cyclization of **22a** [25], followed by rapid and irreversible fragmentation of the strained intermediate, would also account for the rearrangement favored by aromatic amines.

The cyclization of an organolithium into an aromatic system has been reported by Clayden and co-workers [26–34]. For example, as shown in Scheme 11, lithiation of *N*-*tert*-butyl-*N*-benzyl-1-napthalamide (**24**) with *t*-BuLi in THF at −78 °C, followed by addition of an excess of hexamethylphosphoramide (HMPA) and standing at −78 °C for 16 h, affords tricyclic product, **25**, after quench with an electrophile [26]. With the exception of protonolysis, which produces a single

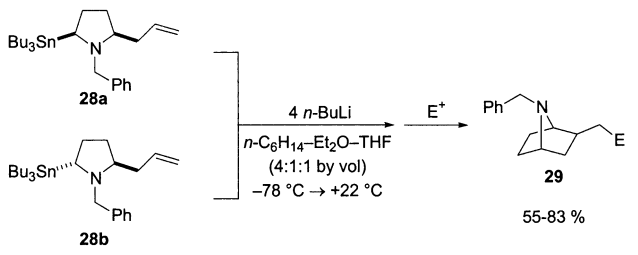
diastereomer (**25a**), the diastereoselectivity of the reaction appears to depend on the steric bulk of the electrophile; only **25b** was formed upon capture with benzyl bromide, while a 3:1 mixture of diastereomers, **25c** and epi-**25c**, were formed upon treatment with iodomethane [26]. Aldol condensation of the cyclic enolate proceeded with high stereoselectivity with benzaldehyde, leading to **26** in 81% yield, however, the diastereoselectivity



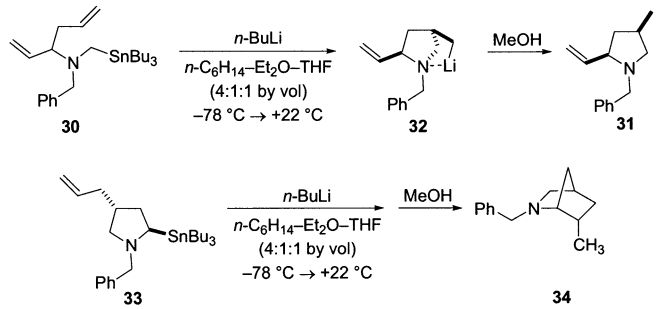
Scheme 12.



Scheme 13.



Scheme 14.



Scheme 15.

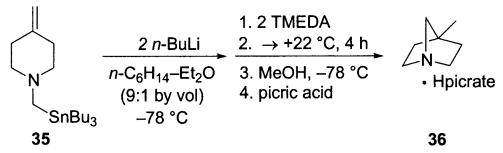
diminished appreciably when other aldehydes such as *n*-butanal were used [26]. Nonetheless, as depicted in Scheme 12, this chemistry has recently been exploited for setting the relative stereochemistry of three contiguous stereocenters in the synthesis of (±)-kainic acid (**27**) [31] and related systems [34].

Similar examples of dearomatizing anionic cyclizations using sulfonamide [35] and phosphinamide [36] groups as activators have been documented recently, and a pair of examples are illustrated in Scheme 13.

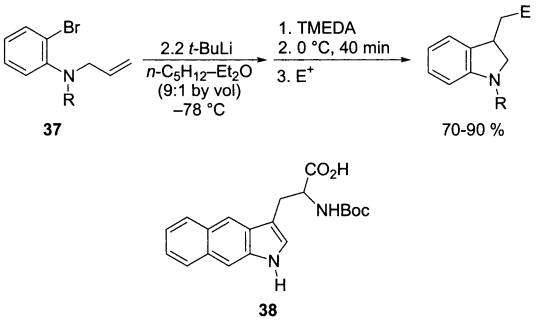
Coldham's group has demonstrated that bicyclic systems, such as the 7-azabicyclo[2.2.1]heptanes illustrated in Scheme 14, may be prepared by cyclization of pyrrolidinyllithiums tethered to a remote alkene [37,38]. Thus, treatment of either stannane **28a** or **28b** with an excess of *n*-butyllithium in a 4:1:1 mixture of hexane–diethyl ether–tetrahydrofuran at −78 °C followed by warming to room temperature for 6 h afforded *exo*-2-substituted-7-azabicyclo[2.2.1]heptanes (**29**) [37,38]. The bicyclic organolithium was trapped with a variety of electrophiles in a wide range of chemical yields; quench of the organolithium with DMF produced the aldehyde in only 34% yield, while allylation proceeded in 60% yield [37,38].

Attempts to prepare the 2-azabicyclo[2.2.1]heptane system by tandem cyclization of an α-aminoorganolithium derived from a dienyl organostannane (**30**), as shown in Scheme 15, resulted in monocyclic 1-benzyl-4-methyl-2-vinylpyrrolidine (**31**) [38]. The authors reasonably concluded that the second cyclization step failed due to intramolecular chelation of the alkyllithium by the nitrogen lone pair (**32**). However, judicious choice of the substrate can circumvent such problems; as illustrated in Scheme 15, cyclization of pyrrolidine **33** proceeded smoothly to give **34** in 60% yield [38].

The 1-azabicyclo[2.2.1]heptane system may be conveniently accessed, as portrayed in Scheme 16, by transmetallation and cyclization of piperidinyl stannane **35**, the picrate salt **36** was isolated in 60% yield [38]. It should be noted that TMEDA was needed to facilitate the cyclization of the organolithium derived from **35** [38]; the analogous carbocyclic cyclization is known to



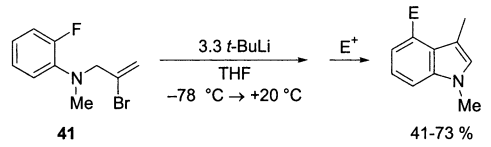
Scheme 16.



Scheme 17.

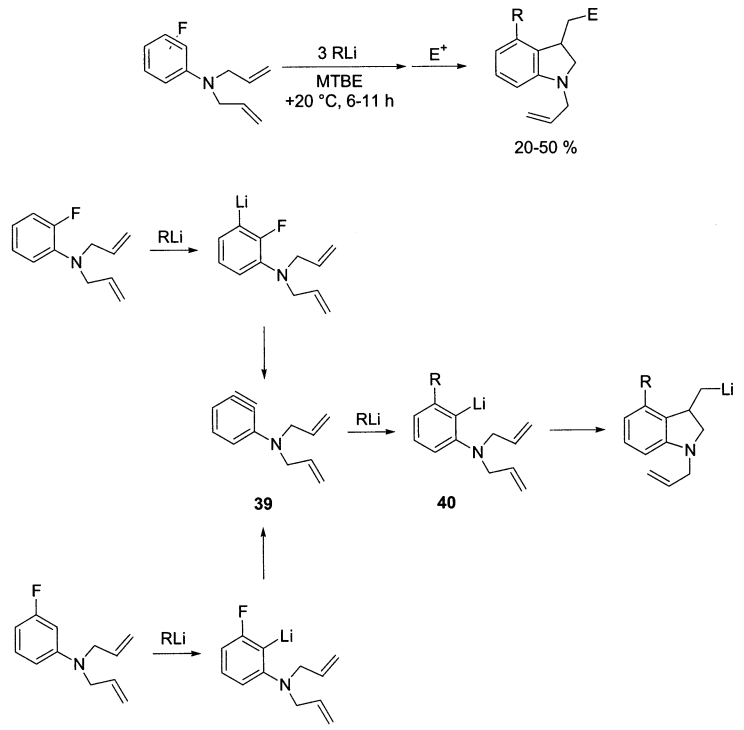
be relatively rapid, but it too experienced a significant rate acceleration upon the addition of TMEDA [39].

Indolines and indoles may be conveniently synthesized by a variety of anionic cyclization techniques. Thus, as illustrated in Scheme 17, the aryllithium derived from an *N*-allyl-2-bromoaniline (**37**) by lithium–bromine exchange [40], cyclizes on warming to 0 °C in the presence of TMEDA and quench with a variety of electrophiles affords three-substituted indolines in 70–90% isolated yield [41,42]. Oxidation of the indolines to the corresponding indoles was accomplished by treatment with *o*-chloranil [41,42]. A similar approach was employed in the synthesis of *N*-α-*t*-Boc-benz[*f*]tryptophan (**38**), a fluorescent amino acid probe by Yokum and co-workers [43].



Scheme 19.

Metal–halogen interchange is not the sole means by which cyclization to give indolines may be initiated. Bailey and Carson have described a novel cascade reaction that delivers 3,4-disubstituted indolines in modest yield [44]. As illustrated in Scheme 18, regioselective *ortho*-lithiation of either 2-fluoro- or 3-fluoro-*N*,*N*-diallylaniline initiates the anionic cascade leading to an *N*-allyl-3,4-disubstituted indoline. The transformation apparently involves loss of LiF from the *ortho*-lithiated species, regioselective intermolecular addition of the organolithium to the benzyne intermediate (**39**), and cyclization of the resulting aryllithium (**40**) [44]. In a similar fashion, Barluenga's group has shown, as portrayed in Scheme 19, that 3,4-disubstituted indoles may be prepared in one-pot (thus avoiding the oxidation of the indoline) via an analogous cascade involving cyclization of a vinyllithium-tethered benzyne [45].

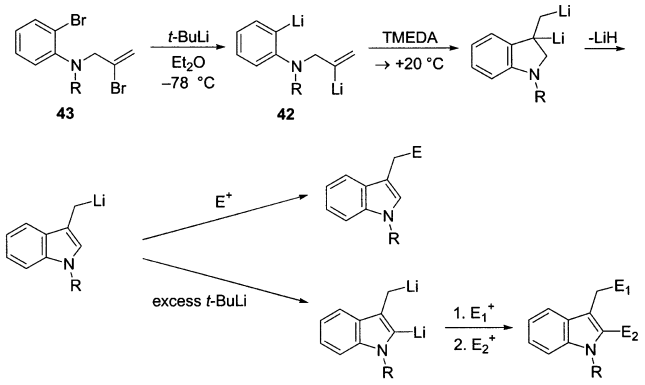


Scheme 18.

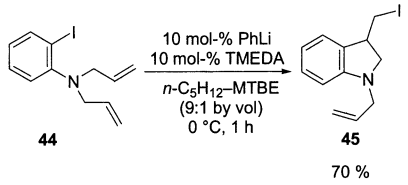
Thus, treatment of a THF solution of **41** with 3.3 equivalents of *t*-BuLi, followed by electrophilic quench gave functionalized indoles in 41–73% yield [45].

Indoles and pyrroles have also been synthesized, as shown in Scheme 20, by cyclization of dilithio-species such as **42**, prepared by low temperature lithium–bromine exchange of **43** in diethyl ether with four equivalents of *t*-BuLi, followed by the addition of four equivalents of TMEDA and warming to room temperature [46,47]. The proposed mechanism for the transformation is portrayed in Scheme 20 [46]. Careful control of the organolithium stoichiometry allowed the authors to selectively prepare either 3-functionalized (four equivalents *t*-BuLi) or 2,3-bifunctional (five equivalents *t*-BuLi) indoles [46].

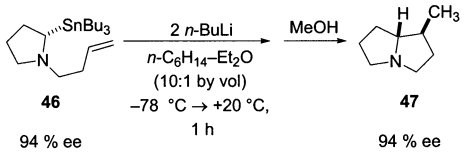
In certain cases, a catalytic quantity of organolithium may be employed to effect a lithium–iodine exchange-



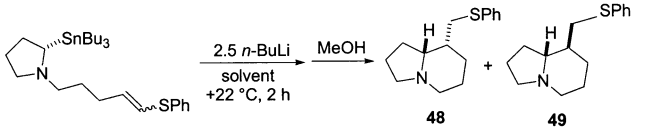
Scheme 20.



Scheme 21.



Scheme 22.



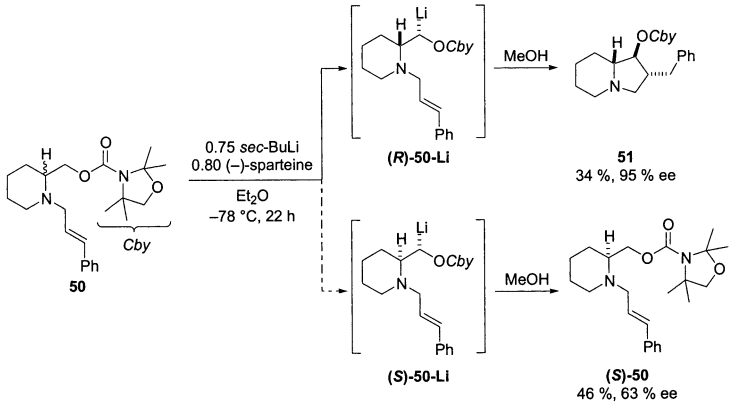
Scheme 23.

mediated cycloisomerization of an unsaturated iodide substrate [48–50]. As illustrated in Scheme 21, this technique has been applied to the conversion of *N*,*N*-diallyl-2-iodoaniline (**44**) to 1-allyl-3-iodomethylindoline (**45**) in 70% isolated yield [49].

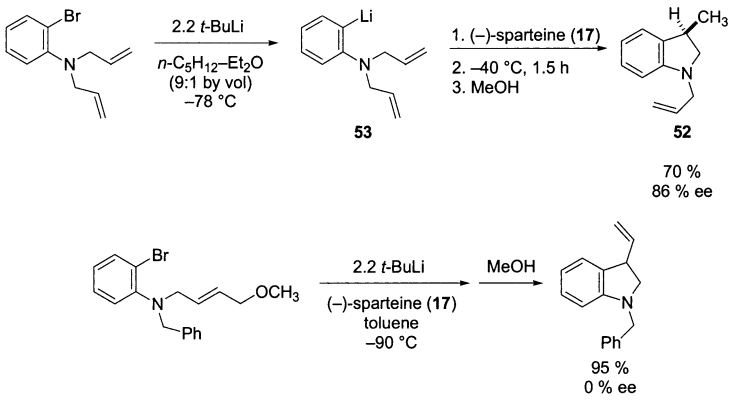
Anionic cyclization of chiral organolithiums has been exploited for the synthesis of a variety of nitrogen heterocycles. As depicted in Scheme 22, Coldham and co-workers found that transmetallation of optically active stannane **46** in 10:1 hexane–diethyl ether proceeded with retention of configuration at the carbanionic center to give an organolithium that cyclized on warming to deliver (+)-pseudoheliotridane (**47**) with complete stereocontrol [51]. The corresponding cyclization to prepare the indolizidine ring system was complicated by competitive racemization of the chiral organolithium species prior to the sluggish cyclization step [52]. The racemization issue was resolved, as illustrated in Scheme 23, by employing a thiophenyl stabilizing group at the alkene terminus to enhance the rate of cyclization [52]. Unfortunately, this move compromised of the diastereoselectivity of the cyclization and afforded octahydroindolizidines **48** and **49** as a 70:30 isomeric mixture [52].

Hoppe and co-workers have demonstrated that the powerful technique of asymmetric deprotonation may be coupled with the cyclization of an unsaturated organolithium to afford functionalized indolizidines in high enantiomeric excess [53]. As shown in Scheme 24, asymmetric deprotonation of **50** by *sec*-BuLi in the presence of (−)-sparteine (**17**) results in a matched [(*R*)-**50**-Li] and mismatched [(*S*)-**50**-Li] pair of organolithiums that are kinetically resolved into indolizidine **51** by cyclization and recovered (*S*)-**50** following methanolysis [53]. Similar chemistry has been described for asymmetric deprotonation and cyclization into allylic chlorides to prepare 3,4-divinylpyrrolidines in 85% yield and 95% ee [54].

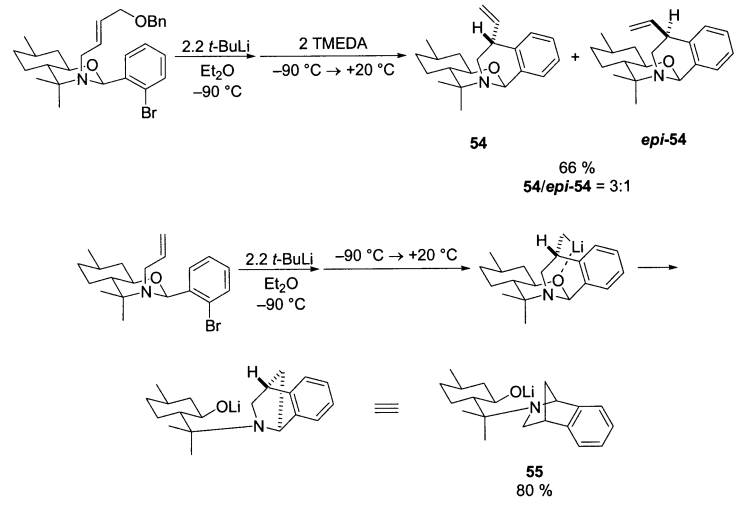
Recently, the ring closure of achiral olefinic organolithiums has been found to proceed enantioselectively in the presence of (−)-sparteine (**17**) [55,56]. Upon complexation with a chiral bidentate ligand, the lithium atom of the olefinic organolithium is rendered stereogenic and may transmit this information in the cyclization step [55,56]. As illustrated in Scheme 25, (*R*)-1-allyl-3-methylindoline (**52**) has been prepared in 86% ee by cyclization of achiral aryllithium **53** in the presence of an equivalent of (−)-sparteine [55]. It was noted that conducting the reaction in the presence of THF afforded virtually racemic products [55,56]. Similar results have been reported by Gil and Groth who also found, as depicted in Scheme 25, that the presence of a leaving group at the distal allylic position of substrates analogous to **53** leads to racemic product albeit in high overall yield [56].



Scheme 24.



Scheme 25.



Scheme 26.

Pedrosa and co-workers have examined the 6-*exo* cyclization of the aryllithiums derived from chiral 2-(*o*-bromophenyl)-substituted perhydro-1,3-benzoxazines [57]. As shown in Scheme 26, the incorporation of a distal allylic leaving group facilitated the selective formation of perhydrobenzoxazines **54** and epi-**54** [57]. Attempted 6-*exo* cyclization of the aryllithium derived from substrates lacking the allylic leaving group, or an anion stabilizing substituent, was unsuccessful in the presence of TMEDA. Surprisingly, products such as **55** were observed in the absence of an additive [57].

In summary, intramolecular carbolithiation, which has been widely used for the construction of carbocyclic rings, also provides a convenient and selective route to a variety of five- and six-membered heterocyclic systems.

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