**Synthesis of six-membered azaheterocycles by means of the
β-lactam synthon method**

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**ABSTRACT**

Azaheterocycles comprise relevant target structures within organic chemistry due to the broad diversity of biological activities associated with these scaffolds. In this review, the most important recent procedures for the preparation of functionalized six-membered azaheterocyclic compounds by ring transformation of β-lactams are summarized and discussed.

# KEYWORDS

β-lactams – ring transformation – azaheterocyclic chemistry – morpholines – piperidines – oxazinanes – piperazines

# Introduction

The β-lactam nucleus comprises the key structural motif in β-lactam antibiotics and has been identified as crucial for bioactivity.[[[3]](#endnote-1)] Next to their antibacterial properties, β-lactams also exhibit other pharmacological activities enabling their use in different therapeutic areas.[[[4]](#endnote-2)] Examples in that respect include inhibition of HIV-1 protease,[[[5]](#endnote-3)] antitumor activity,[[[6]](#endnote-4)] antimalarial activity,[[[7]](#endnote-5)] cholesterol absorption inhibition,[[[8]](#endnote-6)] and antiviral activity.[[[9]](#endnote-7)]

In addition to their indisputable importance as bioactive agents, β-lactams have also acquired a prominent position in organic chemistry as synthons for further elaboration by exploiting the strain energy associated with the four-membered ring system. Selective bond cleavage of the β-lactam nucleus followed by further intriguing transformations renders these compounds powerful synthetic building blocks. In this way, β-lactams act as important intermediates toward a wide variety of nitrogen-containing acyclic and heterocyclic target compounds (β-lactam synthon method).[[[10]](#endnote-8)]

The present mini-review provides a survey of the recent salient synthetic achievements dealing with the transformation of functionalized β-lactams into six-membered azaheterocyclic systems exploiting selective bond cleavage of the β-lactam nucleus through any of the three possibilities, i.e., N1-C2, C3-C4 or C4-N1 bond cleavage. Six-membered azaheterocycles are recognized as essential substructures in a wide range of bioactive compounds, hence the current broad interest in the development of new synthetic approaches for their selective construction. The emphasis in this overview lies on new literature data published during the period 2005-2012.

# Ring transformation through N1-C2 bond cleavage

Two concise, complementary stereocontrolled routes to optically pure orthogonally protected *anti*,*anti*-4-amino-3,5-piperidinediols **4** have been described. Key features of the first approach (method A) include a chemoselective reductive ring opening of the β-lactam nucleus with LiBH4 to 3-amino-5-hydroxypentanenitriles **2**, followed by reductive cyclization of conveniently functionalized δ-mesyloxynitriles **3** with NaBH4/NiCl2 (Scheme 1). The second approach (method B) involves a LiAlH4-induced reduction of protected *anti*,*anti*-4-amino-3,5-dihydroxypiperidin-2-ones, which were easily obtained by chemoselective reduction of the cyano group in γ-cyano-β-amino esters **5** and subsequent intramolecular ring closure of the resulting diamino esters (Scheme 1).

[[[11]](#endnote-9)]



Scheme 1

According to an analogous reaction sequence, β-lactams have been shown to play a key role in the synthesis of cisapride, a drug used for the treatment of various gastrointestinal disorders.[[[12]](#endnote-10)] The synthetic strategy consists of methanolysis of nitro-β-lactams **6** followed by a Pd-catalyzed reductive cyclization by means of ammonium formate and reduction of the carbonyl moiety upon treatment with borane (Scheme 2).[[[13]](#endnote-11)] The construction of the [gastroprokinetic agent](http://en.wikipedia.org/wiki/Gastroprokinetic_agent) cisapride **10** was achieved in an additional three-step synthesis.[[[14]](#endnote-12)]



Scheme 2

An alternative (diastereoselective) approach for the synthesis of piperidine derivatives from β-lactams comprises the ring transformation of 3-(3-chloropropyl)-β-lactams **11**, synthesized by treatment of *N*-(arylmethylidene)amines with 5-chloropentanoyl chloride in benzene in the presence of 2,6-lutidine.[[[15]](#endnote-13)] The synthetic strategy involves a two-step synthesis of *trans*-2-arylpiperidine-3-carboxylates **14**, compounds of significant interest due to their potential use in the treatment of Alzheimer’s disease,[[[16]](#endnote-14)] upon subsequent treatment of halogenated β-lactams **11** with hydrogen chloride in methanol and triethylamine in dichloromethane (Scheme 3). This reaction has been proposed to proceed through initial nucleophilic ring opening of the protonated β-lactam **12** by methanol, followed by intramolecular displacement of chloride by the *in situ* formed free amine **13** upon addition of the base (Scheme 3).[13] Interestingly, *cis*-piperidines would be expected, suggesting that epimerization has occurred during this transformation. Furthermore, these *trans*-2-arylpiperidine-3-carboxylates **14** were easily converted into their corresponding *cis*-isomers **15** by means of hydrazine monohydrate in methanol (Scheme 3).[13]



Scheme 3

Several examples are known in which aryl-substituted β-lactams are rearranged into functionalized quinolone derivatives, a family of compounds with *inter alia* broad-spectrum antibiotic,[[[17]](#endnote-15)] antidiabetic,[[[18]](#endnote-16)] antidepressant, sedative and antiparkinson[[[19]](#endnote-17)] properties. For example, 1-arylazetidin-2-ones **16**, synthesized by a Goldberg-Buchwald-type copper-catalyzed coupling of *N*-unsubstituted azetidin-2-ones with the appropriate aryl halides or using Mitsunobu cyclization processes,[[[20]](#endnote-18)] have been treated with triflic acid under mild reaction conditions in CH2Cl2, which ensued a smooth Fries rearrangement delivering 2,3-dihydro-4(1*H*)-quinolinones **22** in good to high yields (71-98%) (Scheme 4).[18] This intramolecular Friedel-Crafts acylation is the result of an acid-mediated amide bond cleavage in β-lactams **16**, generating a highly reactive free acylium ion in intermediates **21**, which subsequently undergo an intramolecular electrophilic aromatic substitution (Scheme 4). Recently, this transformation has been used in the synthesis of TRPV1 antagonists as analgesic agents.[[[21]](#endnote-19)]



Scheme 4

Another method for the construction of dihydroquinolinones from β-lactams starts with the microwave-assisted transfer hydrogenation of the *ortho*-nitro group in azetidinones **23**, synthesized *via* the Staudinger reaction, to afford intermediates **24** by using ammonium formate in ethylene glycol. Subsequent *in situ* intramolecular β-lactam ring opening provided 4-amino-3,4-dihydroquinolin-2-ones **25** in 74-90% yield (Scheme 5).[[[22]](#endnote-20)]



Scheme 5

Isoquinoline-based scaffolds represent an important group of biologically active compounds and are attracting increasing attention in contemporary biomedical research and drug discovery programs. Several members of this group exhibit various pharmacological and biological activities, including potential anticancer properties.[[[23]](#endnote-21)] In that respect, recently, the single-step diastereoselective synthesis of functionalized hexahydroisoquinolinones **30** and tetrahydroisoquinoline-1,3-diones **28** has been realized by intermolecular NaOMe-induced amidolysis of 1-aryl-β-lactams **26** (Z = OMe) and subsequent intramolecular cyclization upon reflux in xylene, and by intramolecular base-induced amidolysis of 1-aryl-β-lactams **26** (Z = NH2) with concomitant two-carbon ring enlargement by stirring in MeOH at room temperature, respectively (Scheme 6).[[[24]](#endnote-22)]



Scheme 6

Furthermore, it has been observed that treatment of β-lactams **26** (Z = NH2) with NaOMe and I2 in methanol at room temperature gave rise to iodinated octahydropyrroloisoquinoline-1,3-diones **32** and 4-[(4-iodophenylamino)arylmethyl]tetrahydroisoquinoline-1,3-diones **34** depending upon the *N*-substituent of the β-lactam ring.[22] In the case *para*-substituted 1-arylazetidin-2-ones **26** (Z = NH2, R1 = Me, Cl) were deployed as synthetic precursors, electrophilic addition of molecular iodine across the double bond in the initially formed tetrahydroisoquinoline-1,3-dione derivatives **28** yielded intermediate iodonium ions **31**, which upon intramolecular cyclization afforded the corresponding functionalized tricyclic tetrahydropyrrole derivatives **32** in 49-55 % yield (Scheme 7).[22] Interestingly, *N*-phenyl-β-lactams **26** (Z = NH2, R1 = H) underwent electrophilic aromatic substitution instead of iodocyclization upon addition of I2, which has been explained considering the initial formation of diketones **33** having a negative charge on the nitrogen atom. In this way, the electron density at the *para*-position of the phenyl substituent increases, thus favouring aromatic electrophilic substitution with molecular iodine, resulting in the selective preparation of iodinated tetrahydroisoquinoline-1,3-diones **34** in 68-74% yield after re-aromatization (Scheme 7).[22]



Scheme 7

The synthetic usefulness of β-lactam to piperidinone transformations has also been demonstrated through the synthesis of dihydroindolizinones. Enynyl β-lactams **35** have been rearranged into 5,6-dihydro-8*H*-indolizin-7-ones **42** through a regiospecific Au-catalyzed β-lactam ring opening and recyclization sequence. The reaction mechanism of this ring expansion has been rationalized by considering an initial 5-*exo*-*dig* cyclizationof the lactam nitrogen to the metal-activated alkyne moiety, followed by a heterocyclic fragmentation of the amide bond to generate acyl cations **39**, which subsequently undergo cyclization to the enamine moiety to afford bicyclic zwitterions **40**. Finally, recuperation of the Au-catalyst and subsequent 1,5-hydride migration gives bicyclic pyrroles **42** (Scheme 8).[[[25]](#endnote-23)] This synthetic strategy was further extended by the development of naturally occurring indolizidine alkaloids, as demonstrated by the synthesis of racemic indolizidine 167B **43**, an alkaloid isolated from neotropical poison dart frogs (Scheme 8).[23]



Scheme 8

A one-step approach has been reported for the conversion of 4-acyloxy-β-lactams **44** into 1,3-oxazin-6-ones **48** by using acyl chlorides in the presence of DBU (Scheme 9).[[[26]](#endnote-24)] After initial acylation of the β-lactam nitrogen, the acidity of the H-3 proton of the β-lactam nucleus is enhanced by the electron-withdrawing *N*-acyl group, thus making the β-lactam carbonyl group more “ketone-like”. As a result, the organic base DBU promotes the elimination of the carboxylic acid (R1CO2H) across the β-lactam C3-C4 bond generating highly strained azetinones **46**, which rapidly experience a four-centered electrocyclic ring opening to *N*-acylimidoylketenes **47**, which in turn provide 1,3-oxazin-6-ones **48** in 40-76% yield through a six-centered electrocyclic ring closure (Scheme 9).[24]



Scheme 9

Another example of a ring transformation of β-lactams into nitrogen- and oxygen-containing six-membered heterocycles comprises the synthesis of 1,3-oxazinanes **51** *via* LiAlH4-promoted reductive ring opening of *cis*-β-lactams **49** towards γ-aminoalcohols **50**, followed by recyclization using formaldehyde in THF (Scheme 10).[[[27]](#endnote-25)] The biological importance of these classes of compounds has been demonstrated by evaluation of their *in vitro* antiplasmodial activity and cytotoxicity, pointing to their promising potential as a novel type of antimalarial agents.[25]



Scheme 10

In a single example, racemic 3-allyl-4-formyl-β-lactam **52** was treated with *N*-methylhydroxylamine hydrochloride in the presence of triethylamine, which, upon intramolecular protonation of the olefin moiety toward the corresponding zwitterionic bicyclic hemiaminal **54** followed by imination of the latent aldehyde, gave rise to the selective formation of nitrone **56** in 50% yield (Scheme 11). This nitrone **56** proved to be unstable in chloroform and after one week 1,2-oxazinane-6-one **58** was obtained in quantitative yield through intramolecular ring opening of the β-lactam nucleus *via* the N1-C2 bond (Scheme 11).[[[28]](#endnote-26)]



Scheme 11

Indane-fused dihydropyrimidinones **61** and **63** have been obtained by ring enlargement of 3,4-benzo-6-azabicyclo[3.2.0]heptan-7-one **59**, prepared from indene by chlorosulfonyl isocyanate addition, upon melting with imidates or lactim ethers at 150-160 °C for 8 hours (Scheme 12). The first step in the reaction is the formation of amidine intermediates **60** and **62**, which, after intramolecular transamidation with simultaneous N1-C2 bond fission, rearrange into tri- and tetracycles **61** and **63**, respectively (Scheme 12).[[[29]](#endnote-27)]



Scheme 12

In addition, 1,3-diamine **64**, synthesized *via* N1-C2 bond cleavage of tricyclic β-lactam **59**, has been treated with 2-formylbenzoic acid or levulinic acid in boiling toluene, which ensued, after initial imination, a smooth two ring-closure sequence delivering indane-fused hexahydropyrimidines **67** and **70** with complete diastereoselectivity in 65% and 63% yield, respectively (Scheme 13).[27] The stereochemical outcome of this overall ring rearrangement has been rationalized assuming the formation of a tautomeric equilibrium between the intermediates **65** and **68**, respectively, in combination with a kinetic control governing the second cyclization step (Scheme 13).[27]



Scheme 13

# Ring transformation through C3-C4 bond cleavage

The tandem cycloetherification/β-lactam ring cleavage of racemic γ-olefinic α-allenols **71**, prepared from the appropriate 4-oxoazetidine-2-carboxaldehydes *via* a regiocontrolled indium-mediated Barbier-type carbonyl-allenylation in aqueous medium,[[[30]](#endnote-28)],[[[31]](#endnote-29)] in the presence of catalytic iron(III) trichloride in dichloroethane at 80 °C in a sealed tube has been described to selectively afford allenic morpholinones **75** in good yields (78-85%) (Scheme 14).[29] Probably, the hydroxyl-iron complex **72**, formed initially through coordination of FeCl3 to the oxygen atom of olefinic allenols **71**, considerably increases the acidity of the hydroxyl protons, thus inducing a chemo- and regioselective intramolecular protonation of the alkene moiety with concomitant 4-*exo* oxycyclization to yield bicycles **73**, which, driven by relief of the strain associated with the four-membered ring, rapidly evolve to intermediates **74** through selective β-lactam ring cleavage. Finally, demetalation regenerates the iron catalyst and affords morpholinones **75** (Scheme 14).[29] Alternatively, initial activation by coordination of FeCl3 to the olefinic double bond cannot be excluded.



Scheme 14

As described above, β-lactams are excellent substrates for the synthesis of functionalized piperidinone derivatives through selective fragmentation of the N1-C2 amide bond of the β-lactam nucleus followed by ring expansion. Also, β-lactams have been proven to be suitable building blocks for the ring enlargement towards dihydropyridones, as demonstrated by the thermally induced [1,3]-sigmatropic rearrangement with concomitant C3-C4 bond cleavage of 4,4-dienyl-β-lactams **77**, which have been obtained through [2+2]-cyclocondensation of azatrienes **76** with the appropriate ketenes, upon heating in toluene or xylene (Scheme 15).[[[32]](#endnote-30)] When the starting β-lactams **77** have two different vinyl substituents (R1 = Ph; R2 = CO2Et; R3 = H or R1 = Ph, CO2Et; R2 = R3 = Me), the regioselectivity of the rearrangement reaction depends on steric factors and on the electronic demands of the substituents. Whereas in the former case (R1 = Ph; R2 = CO2Et; R3 = H) the predominant formation of dihydropyridones **78** can be attributed to the benzylic stabilization of the developing carbenium ion, in the latter case (R1 = Ph, CO2Et; R2 = R3 = Me) steric factors play a predominant role rather than electronic factors, inducing reaction at the monosubstituted diene C-terminus even if the substituent is an electron-withdrawing ethoxycarbonyl group (Scheme 15).[30]



Scheme 15

A β-lactam to piperazinone rearrangement has been reported in the synthesis of 1,4-diazabicyclo[4.3.0]nonanones **85** from 4-formyl-spiro-β-lactams **80** by means of a Pd-catalyzed hydrogenation. This ring transformation involves, after initial hydrogenolytic removal of the benzyloxycarbonyl protecting group, a retro-Mannich process, which induces β-lactam ring opening through selective C3-C4 bond fission, affording intermediate enols **82** (Scheme 16).[[[33]](#endnote-31)] Further hydrogenation, nucleophilic addition of the *in situ* liberated secondary amine to the aldehyde group and elimination finalizes the reaction pathway, generating pyrrolidine-fused pyrazinones **85** in good yields (70-90%) (Scheme 16).[31] 1,4-Diazabicyclo[4.3.0]nonanes comprise remarkable structural units encountered in several biologically active products, as demonstrated by their potential use in the treatment of *inter alia* schizophrenia, depression, memory dysfunction,[[[34]](#endnote-32)] filariasis[[[35]](#endnote-33)] and angina pectoris.[[[36]](#endnote-34)] In that respect, further derivatization of bicyclic piperazinone **85** [R = CH2CH2(3,4-Cl2)C6H3], i. e., monochloroalane-mediated reduction of the carbonyl functionality, enabled the synthesis of 1,4-diazabicyclo[4.3.0]nonane **86** (Scheme 16),[31] a compound claimed for the treatment of central nervous system disorders.[[[37]](#endnote-35)]



Scheme 16

# Ring transformation through C4-N1 bond cleavage

The first two-carbon ring expansion of a β-lactam through cleavage of the C4-N1 bond has been described in the synthesis of 1,3,4,5-tetrasubstituted glutarimides. The presence of a 4-hydroxyphenyl substituent at the 4-position in the starting 3-alkylazetidin-2-ones **87** enabled a base-mediated C4-N1 bond fission upon treatment with potassium *tert*-butoxide in DMF, which induced the formation of the corresponding phenolate anions followed by rearrangement to intermediate quinone methides **88** with simultaneous C4-N1 bond cleavage (Scheme 17).[[[38]](#endnote-36)] The latter reactive quinone methides **88** are subsequently quenched by the *tert*-butyl methyl malonate anion in a Michael-type 1,6-conjugate addition at the benzylic carbon atom and are transformed into glutarimides **91** upon cyclization and removal of the *tert*-butyl group with trifluoroacetic acid (Scheme 17). The stereochemistry of the ring expansion proved to be dependent on the specific C3-substituent of the starting β-lactams **87**.[36]



Scheme 17

Next to the base-catalyzed ring opening of 4-(4-hydroxyphenyl)-β-lactams, the latter azetidinones are also cleaved under acidic conditions. It has been observed that treatment of β-lactams **92** with neat trifluoroacetic acid gave rise to the formation of intermediates **93**, which upon intramolecular Friedel-Crafts alkylation ensued to recyclize towards 3,4-dihydroquinolin-2-ones **94** in quantitative yields (Scheme 18).[[[39]](#endnote-37)] It has to be noted that the 4-(4-hydroxyphenyl) substituent in the starting β-lactams **92** induces C4-N1 bond cleavage, whereas in the absence of a C4-substituent cleavage of the amide bond occurs upon treatment with trifluoroacetic acid (Scheme 4).



Scheme 18

# Conclusion

This short review demonstrates that the β-lactam skeleton is a very useful and versatile building block exhibiting an extremely rich organic chemistry. The selective bond cleavage of the β-lactam nucleus has proven to have many applications in stereocontrolled synthesis, including the synthesis of azaheterocyclic six-membered ring systems such as morpholinones, piperidines, oxazinanes, oxazinones, piperidinones, piperazines, piperazinones, glutarimides, dihydropyrimidines and dihydroquinolinones (Figure 1).



Figure 1

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