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Oxidative Lactamization of Amino Alcohols: An Overview

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ABSTRACT

Lactams are essential functional groups in a number of pharmacologically and biologically active compounds. They are widely found in many natural products, marketed drugs, as well as in the base of polymeric structures (e.g., polyamides/Nylons). In this context, it is quite important to develop novel and efficient methods for the synthesis of these compounds. Recently, intramolecular dehydrogenative coupling reactions of amino alcohols, which generate only hydrogen as a side product, have emerged as one of the most versatile and powerful synthetic strategies to construct lactam rings. In the present review we will discuss recent advances on this chemistry with the emphasis on the mechanistic aspects of the reactions.

1. Introduction

Lactams are important heterocyclic motifs found in a significant number of natural and non-natural biologically active molecules (Figure 1) [1, 2] and have therefore been widely used as privileged structures and substructures in the drug design processes. They are also monomers of polyamides (Nylons), which are frequently used polymer materials in our everyday life and industry [3]. Although numerous synthetic approaches have been described for the construction of these heterocycles [4], they are often suffering from poor selectivity, intolerance to various functional groups, undesired by-products, and/or low atom economy. Therefore, development of new, efficient, and convenient approaches for the synthesis of titled compounds from simple and easily available starting materials is of considerable attention.

Cross-dehydrogenative coupling, which generally refers to the formation of C(X)–C(X) (X= heteroatom) directly from two C(X)–H bonds with liberation of H_2, has emerged as a very powerful and valuable tool for increasing the structural complexity in organic molecules [5-7].

Along this line, dehydrogenative lactam synthesis from amino alcohols has received much attention, because readily available starting materials are used, and H_2, a clean source of energy, is generated as the sole by-product. To the best of our knowledge, a comprehensive review has not appeared on this novel and atom-economical synthetic route of lactams in literature so far. In continuation of our recent works on the synthesis of lactam cores [8, 9] and cross-coupling reactions [10] herein, we will highlight the most important advances and developments on intramolecular cross-dehydrogenative coupling of amino alcohols (Figure 2) which will be helpful in the development of improved methods for the construction of natural and biologically important compounds.

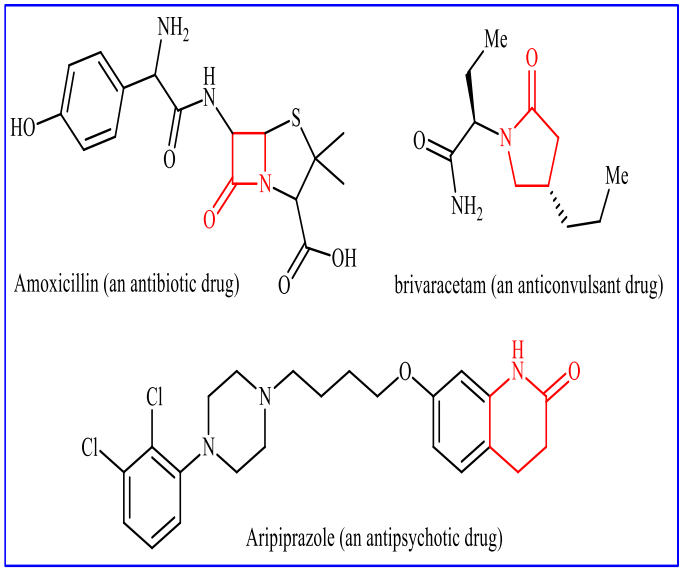

Figure 1. Chemical structure of some of the marketed drugs containing lactam rings

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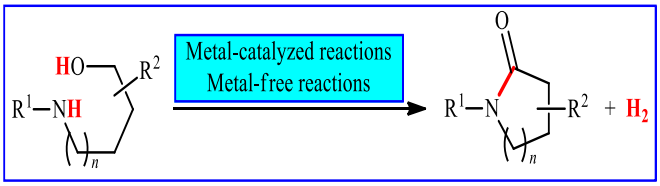


Figure 1. Oxidative lactamization of amino alcohols.

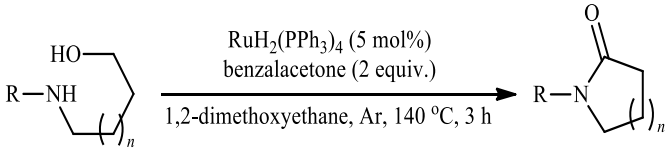
2. Metal-Catalyzed Reactions

In this section, we describe the current literature on the synthesis of lactams through the metal-catalyzed intramolecular cross-dehydrogenative coupling of the respective amino alcohols. For clarity, the section was organized based on the type of catalytic metal cores (Ru, Rh, Au, Fe).

2.1. Ruthenium

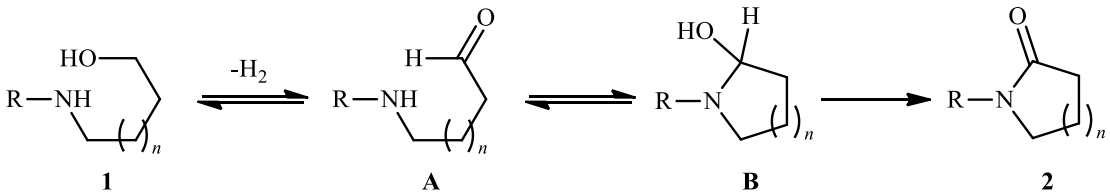
The last few years have witnessed the extremely rapid development of ruthenium-based catalysts for the oxidative lactamization of amino alcohols. These catalysts are indeed at the heart of intramolecular cross-dehydrogenative coupling reactions between amines with alcohols: up to 90 % of all reported examples of the synthesis of lactams from the corresponding amino alcohols are used Ru-based catalysts. This part will focus exclusively on this chemistry with the emphasis on the mechanistic aspects of the reactions.

Naota and Murahashi were the first to report the synthesis of lactams through the Ru-catalyzed oxidative lactamization of the corresponding amino alcohols [11]. They showed that treatment of 1,4- and 1,5-amino alcohols **1** with a catalytic amount of $RuH_2(PPh_3)_4$ and 2 equiv. of benzalacetone as a hydrogen acceptor in 1,2-dimethoxyethane under an inert atmosphere afforded the expected lactams **2** in good to high yields within 3 h (Scheme 1). They found that the presence of a hydrogen acceptor was crucial for the reaction, since it prevent the formation of cyclic amines. When the reaction was carried out in the absence of benzalacetone, the reductive condensation took placed to afford the corresponding cyclic amines exclusively. It should be mentioned that $Ru_3(CO)_{12}$ was also found to promote this coupling reaction. However, the complexes bearing chloride, such as $RuCl_3•nH_2O$, $RuCl_2(PPh_3)_3$, as well as $RhCl(PPh_3)_3$ were not able to effectively catalyze this transformation. Of note, the optimal catalytic system was also successfully applied for the fabrication of arylamides through the condensation of benzaldehydes with amines. The mechanism shown in Scheme 2 was proposed for this oxidative lactamization reaction. It consists of the following key steps: (i) partial oxidation of amino alcohol **1** with catalyst to form the amino aldehyde **A**, (ii) intramolecular condensation of intermediate **A** produces hemiaminal intermediate **B**, and (iii) dehydrogenation of intermediate **B** in the presence of a hydrogen acceptor gives the desired lactone **2**. Inspired by this work, Madsen and co-workers reported the synthesis of 2-pyrrolidinone **2a** in good yield (65%) *via* intramolecular oxidative lactamization of 4-aminobutan-1-ol **1a** using $Ru(COD)Cl_2$ as a catalyst, KO^tBu as a base, and $^iPr_2ImCl$ and $PCyp_3•HBF_4$ as the ligands [12]. Noteworthy, the authors also studied the scope of intermolecular fashion of this reaction and successfully synthesized a variety of secondary and tertiary amides from the corresponding alcohols and amines. Similarly, Hong's [13] an Bera's [14] research teams synthesized 2-pyrrolidinone **2a** in moderate yields, 51% and 45%, respectively, by performing the process in the presence of $RuCl_3/^iPr_2ImBr$/NaH and Ru(py-NHC)$(CO)_2Br_2$ /NaH combinations, respectively, as the catalytic systems.



1a, R= H, n= 1 — **2a**, (59%)
1b, R= H, n= 2 — **2b**, (65%)
1c, R= Me, n= 2 — **2c**, (81%)
1d, R= Et, n= 2 — **2d**, (68%)

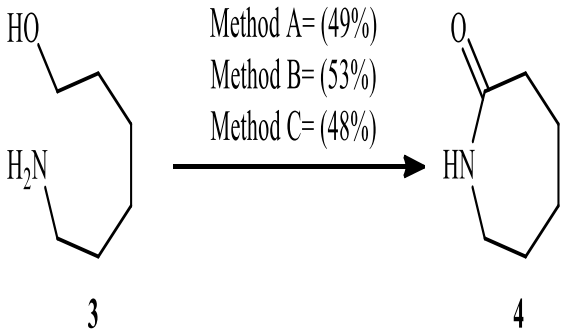
Scheme 1. Synthesis of lactams **2** developed by Murahashi.



Scheme 2. Proposed mechanism for the formation of lactams **2**

Table 1. Oxidative lactamization of 5-aminopentan-1-ol derivative catalyzed by Ru-complexes.

Entry	Catalyst	Conditions	Yield (%)	Ref.
1	$[Ru(benzene)Cl_2]_2$	$^{i}Pr_2ImBr$, MeCN, NaH, toluene, reflux, 36 h	94	[15]
2	$[Ru(p\text{-cymene})Cl_2]_2$	$^{i}Pr_2ImBr$, pyridine, NaH, toluene, reflux, 36 h	92	[15]
3	$[RuH_2(PPh_3)_4]$	$^{i}Pr_2ImBr$, NaH, MeCN, reflux, 24 h	92	[16]
4	[Ru(benzene)($^{i}Pr_2Im$)Cl_2]	KO^{t}Bu, reflux, 24 h	90	[17]



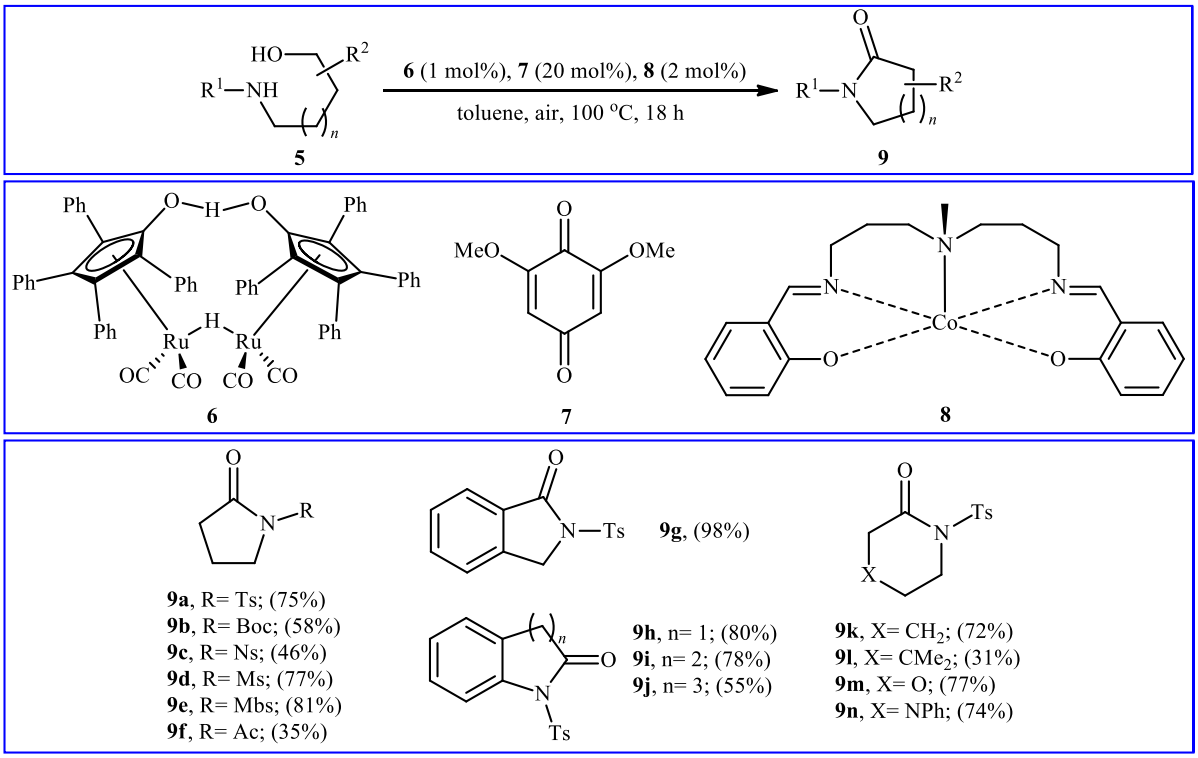
Method A= [$RucCl_2(cod)$] (2 mol%), $^{i}Pr_2ImBr$ (2 mol%), PCy_3.HBF_4 (2 mol%), KO^{t}Bu (8 mol%), toluene, reflux, 24 h
Method B= [$Ru(p\text{-cymene})Cl_2(^{i}Pr_2Im)$] (2 mol%), PCy_3 (2 mol%), KO^{t}Bu (8 mol%), toluene, reflux, 24 h
Method C= Hoveyda-Grubbs I catalyst (2 mol%), PCy_3 (2 mol%), KO^{t}Bu (8 mol%), toluene, reflux, 24 h

Scheme 3. Madsen's synthesis of caprolactam **4**.

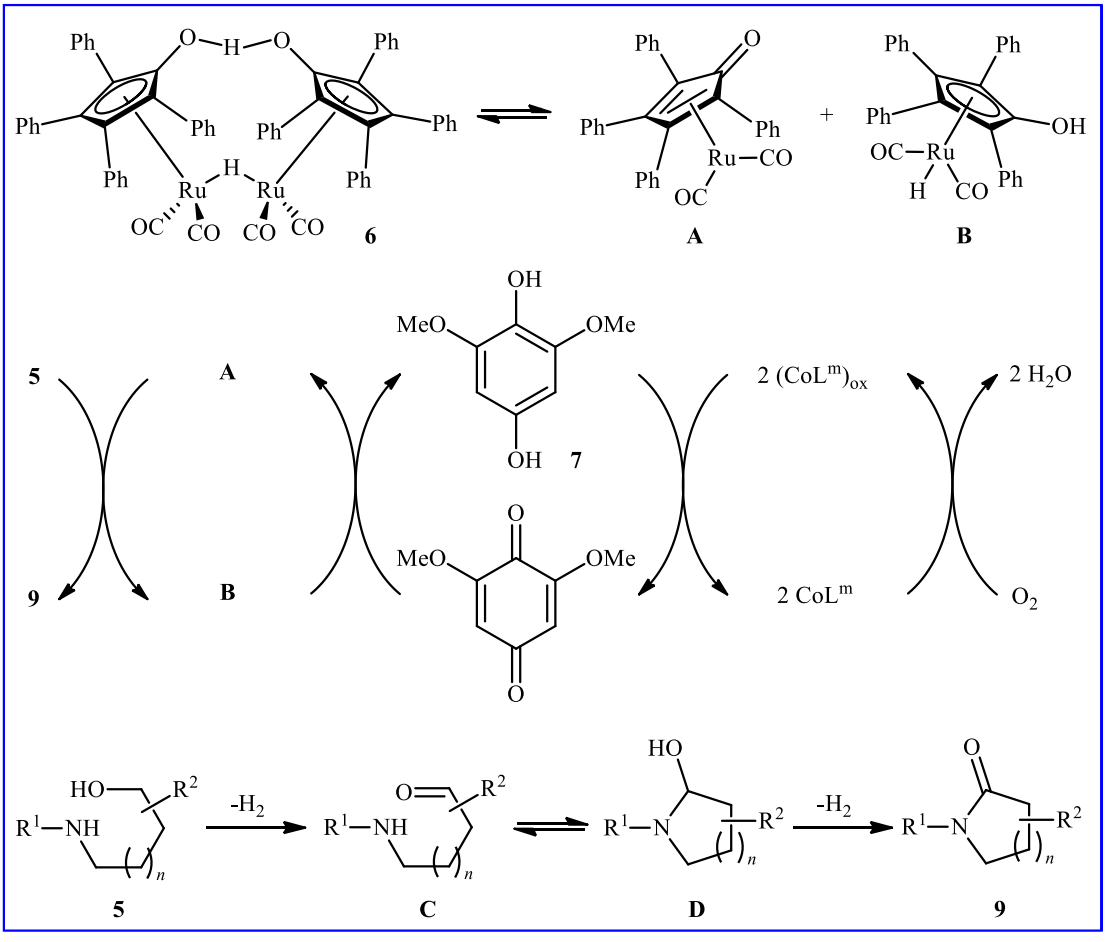
In this context, various Ru-based catalytic systems have been developed which effectively catalyzed the oxidative lactamization of 5-aminopentan-1-ol **1b** to produce γ-lactam **2b** in excellent yields (Table 1). These include: $[Ru(benzene)Cl_2]_2$, [15] $[Ru(p\text{-cymene})Cl_2]_2$ [15], $[RuH_2(PPh_3)_4]$ [16], and [Ru(benzene)($^{i}Pr_2Im$)Cl_2] [17].

In 2010, Madsen and co-workers disclosed the first synthesis of seven-membered lactam **4** through the Ru-catalyzed oxidative cyclization of respective amino alcohol **3** using three alternative sets of reaction conditions [18]: (i) $[RucCl_2(cod)]$ in the presence of $^{i}Pr_2ImBr$, PCy_3•HBF_4, and KO^{t}Bu (ii) $[Ru(p\text{-cymene})Cl_2(^{i}Pr_2Im)]$ in the presence of PCy_3 and KO^{t}Bu; and (iii) Hoveyda–Grubbs I catalyst in the presence of PCy_3 and KO^{t}Bu in refluxing toluene. As shown in Scheme 3, these catalytic systems showed similar reactivity in the titled reaction. Although caprolactam was prepared in moderate yield, only one example was provided, without any substrate scope exploration. A related system ($[Ru(p\text{-cymene})Cl_2(^{i}Pr_2Im)]$, KO^{t}Bu) was also utilized by the same research team for the high yielding synthesis of five- and six-membered lactams [19].

In 2012, with the objective of designing a comprehensive procedure to lactams through Ru-catalyzed intramolecular dehydrogenative coupling of amino alcohols, Bäckvall and colleagues were able to



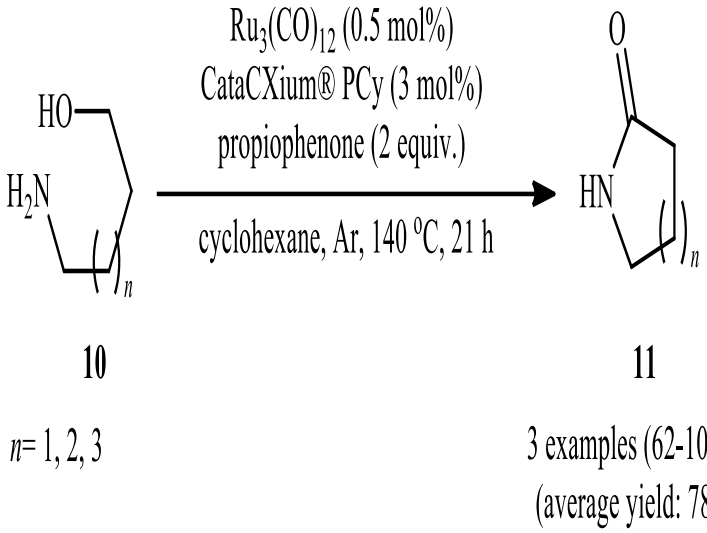
Scheme 4. Aerobic lactamization of N-protected amino alcohols **5**.



Scheme 5. Proposed mechanism for synthesis of lactams **9**.

demonstrated that a variety of five-, six-, and seven-membered lactams **9** could be obtained in fair to excellent yields from the corresponding N-protected amino alcohols **5** employing Shvo's catalyst **6** and a combination of 2,6-dimethoxy-1,4-benzoquinone **7** (DMBQ) and a cobalt complex **8** as the electron-transfer mediators (ETMs) [20]. A wide variety of protecting groups are compatible with the reaction conditions, including Ts, Ns, Ms, Ac, and Boc groups (Scheme 4). However, N-benzoyl protected amino alcohols afforded intermolecular oxidative-esterification products and Fmoc-protected substrates decomposed under the reaction conditions. Moreover, attempts to expand this methodology to include the synthesis of four-membered lactams were unsuccessful because intermolecular oxidative esterification was preferred over the intramolecular lactam formation. According to the authors proposed mechanism (Scheme 5), this oxidative coupling reaction proceeds through the formation of active 16-electron complex **A** *via* the thermal dissociation of Shvo's catalyst **6** which dehydrogenates amino alcohol **5** to amino aldehyde **C** whilst itself undergoes reduction to hydride **B**. Subsequently, ETMs (**7** and **8**) mediated transfer of hydrogen from **B** to molecular oxygen regenerates the active complex **A**. Next, amino aldehyde **C** cyclizes to hemiaminal intermediate **D**. Finally, dehydrogenation of hemiaminal **D** by complex **A** leads to the final product **9**.

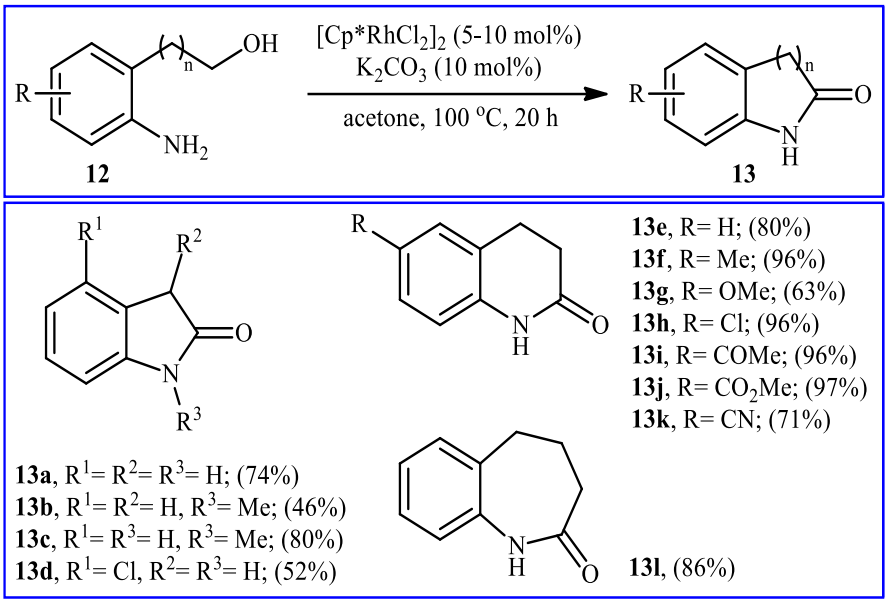
In a related investigation, Pingen and Vogt also found that NH-free amino alcohols **10** were converted to the corresponding lactams **11** *via* Ru-catalyzed oxidative cyclization using $Ru_3(CO)_{12}$/CataCXium® PCy combination as the catalytic system and propiophenone as the H-acceptor in cyclohexane at 140 °C (Scheme 6) [21]. However, 3-amino-1-propanol failed to give the desired β-lactam under the present reaction conditions. Moreover, N-protected substrates were incompatible in this reaction. The authors observed that, by the addition of water instead of propiophenone under the standard reaction conditions, cyclic amines were selectively obtained and without an additive a mixture of both the amine and the amide was observed.



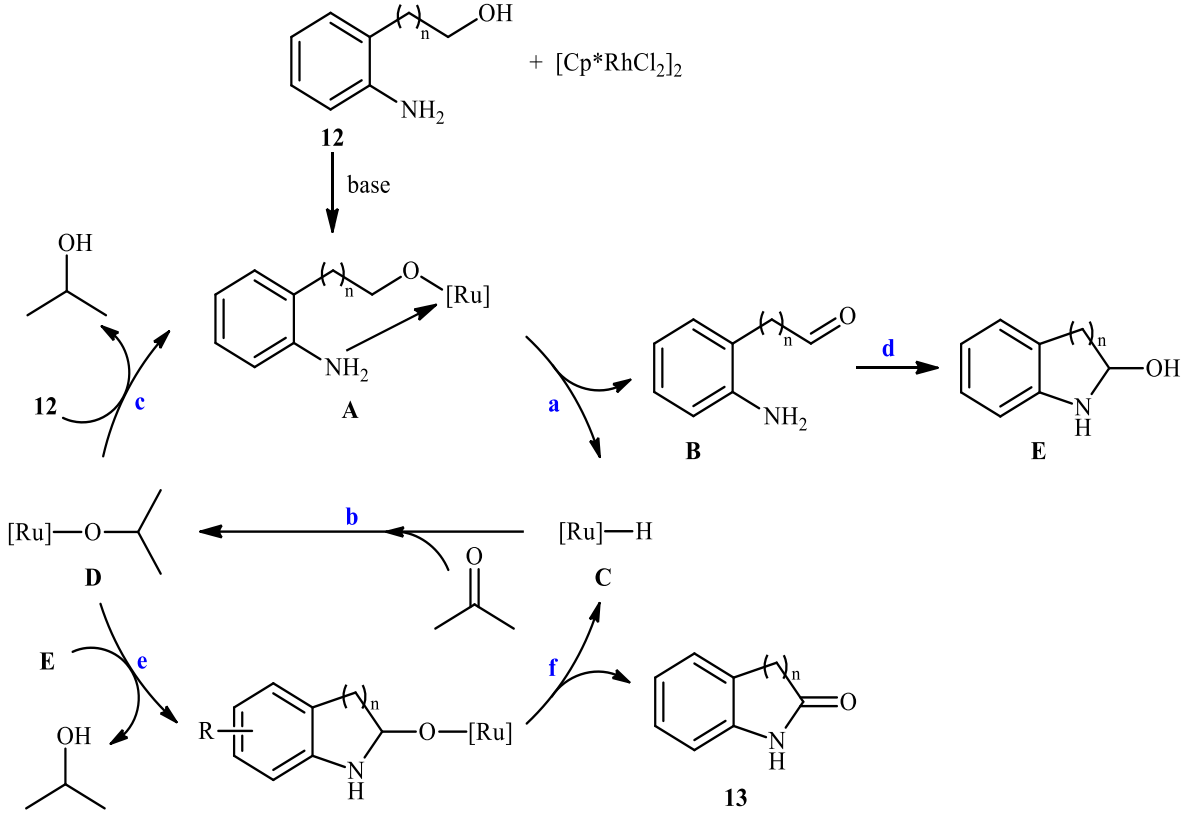
Scheme 6. $Ru_3(CO)_{12}$-catalyzed oxidative lactamization of amino alcohols **10**.

2.2. Rhodium

In 2004, Fujita and Yamaguchi along with their co-workers described the first and only Rh-catalyzed oxidative lactamization of amino alcohols [22]. Thus, the treatment of aromatic amino alcohols **12** with 5-10 mol% of $[Cp^*RuCl_2]_2$ and 10 mol% of K_2CO_3 in acetone at 100 °C afforded benzene-fused γ-lactams **13** in moderate to almost quantitative yields (Scheme 7). The cyclization showed good functional group tolerance, including methoxy, chloro, cyano, ester and ketone functionalities. Thus, this procedure offers scope for further manipulation of products. Of note, the process could also be conducted successfully on a gram scale without sacrificing the yield or outcome of the protocol. The mechanistic course of this reaction sequence is shown in Scheme 8, and involves the initial formation of intermediate **A** through the coordination of amino alcohol **12** to the rhodium center of the catalyst. Then, β-hydrogen elimination from intermediate **A** affords an amino aldehyde **B** and a rhodium hydride species **C** (step a). Next, insertion of acetone into the rhodium-hydride bond in **C** leads to a rhodium isopropoxide species **D** (step b), which after an alkoxy exchange reaction with amino alcohol **12** regenerates intermediate **A** (step c). Concurrently, the intermediate **B** undergoes intramolecular condensation to produce a cyclic hemiaminal intermediate **E** (step d). Finally, Oxidation of **E** by the rhodium catalyst *via* β-hydrogen elimination affords the target lactam product **13** (steps e and f).



Scheme 7. Rh-catalyzed synthesis of benzene-fused γ-lactams **13** from amino alcohols **12**.

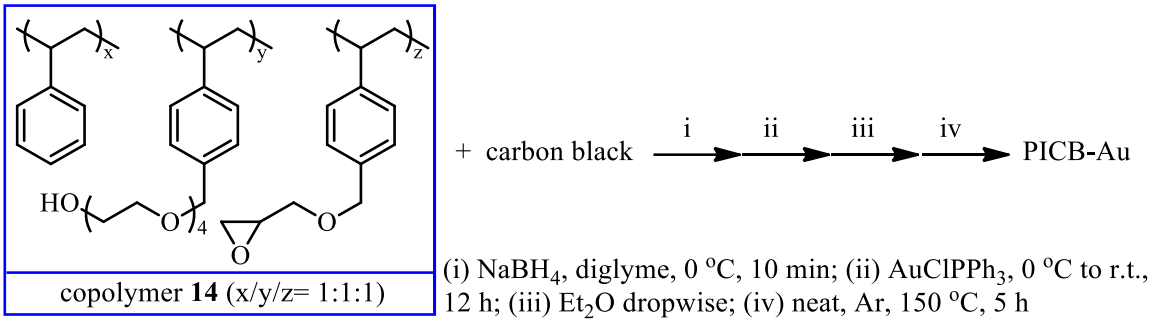


Scheme 8. Proposed mechanistic pathways for the formation of benzene-fused γ-lactams **13**.

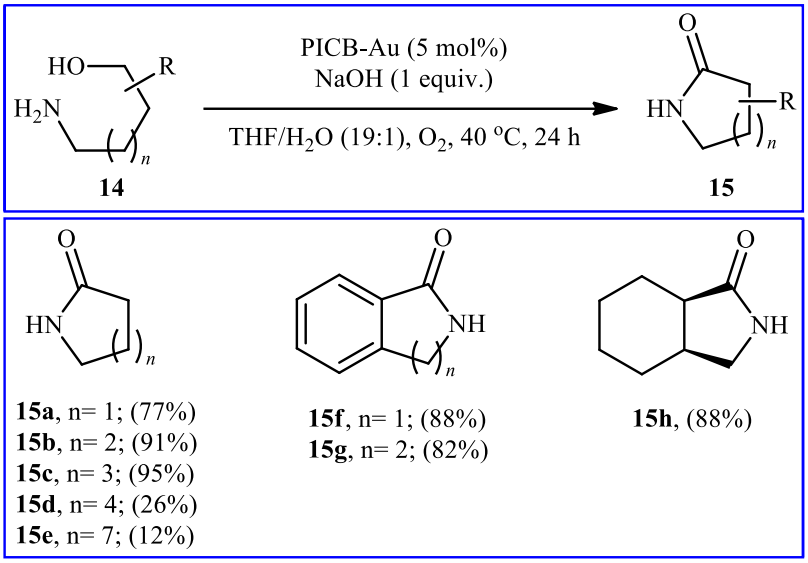
2.3. Gold

Although gold-catalyzed intermolecular dehydrogenative coupling reactions of alcohols and amines have been well established, reports on intramolecular version of these reactions are scarce [23-26]. Indeed, only one example to date was reported on this chemistry.

In 2012, Kobayashi and co-workers designed and synthesized a novel carbon-stabilized polymer-incarcerated gold nanocatalyst (PICB-Au) *via* a four-step procedure through the reaction of copolymer **14** with carbon black and PPh_3AuCl (Scheme 9) [27]. The hybrid system was applied as an efficient catalyst for the synthesis of NH-free lactams **16** *via* intramolecular oxidative coupling of the corresponding amino alcohols **15** in the presence of NaOH in binary solvent THF/H_2O with ratio 19:1 (Scheme 10). Various five-, six-, seven-, eight-, and twelve-membered lactams were successfully synthesized by this catalytic system. However, like



Scheme 9. Schematic diagram showing the formation of PICB-Au.

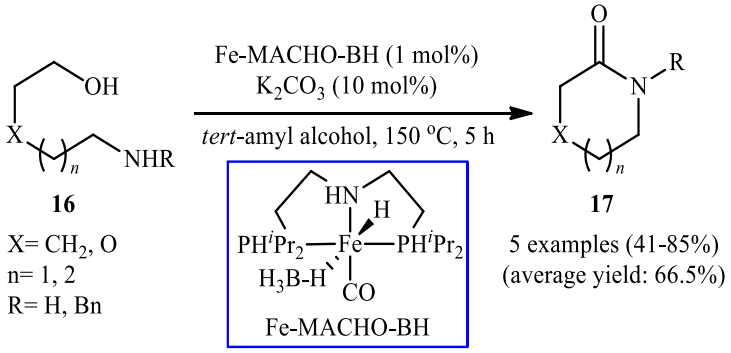


Scheme 10. PICB-Au-catalyzed intramolecular dehydrogenative coupling of amine

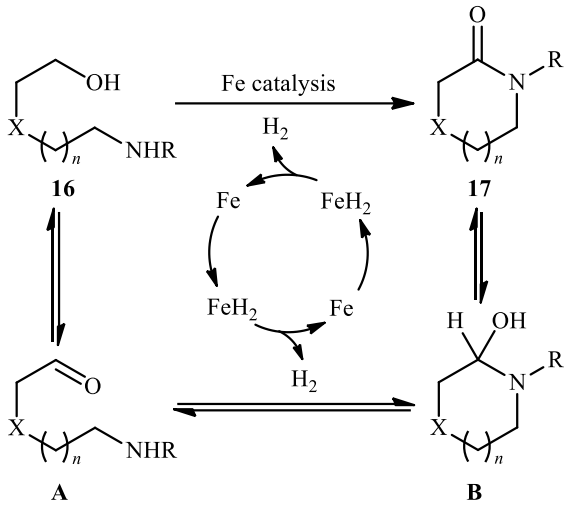
previous works, β-Lactam was incompatible in this reaction. It should be mentioned that the lactam products were obtained as almost pure compounds (>95 % purity) after simple filtration.

2.4. Iron

In 2015, Beller and co-workers reported for the first time the use of iron catalysts in the synthesis of cyclic amides from the corresponding amino alcohols [28]. Thus, in *tert*-amyl alcohol, an iron(II) pincer-catalyzed intramolecular dehydrogenative coupling of amino alcohols **16** furnished six- and seven-membered lactam products **17** in moderate to high yields, ranging from 41% to 85% (Scheme 11). However, the methodology was ineffective in the formation of four- and five-membered cyclic amides. Other iron catalysts such as $FeBr_3$ and $Fe(acac)_2$ completely failed to promote this reaction. Noteworthy, the optimal condition was also successfully used for the synthesis of lactones from simple diols. The mechanism shown in Scheme 12 is proposed for this transformation. To the best of our knowledge, this is the only example of Fe-catalyzed intramolecular amidation of amino alcohols reported thus far.

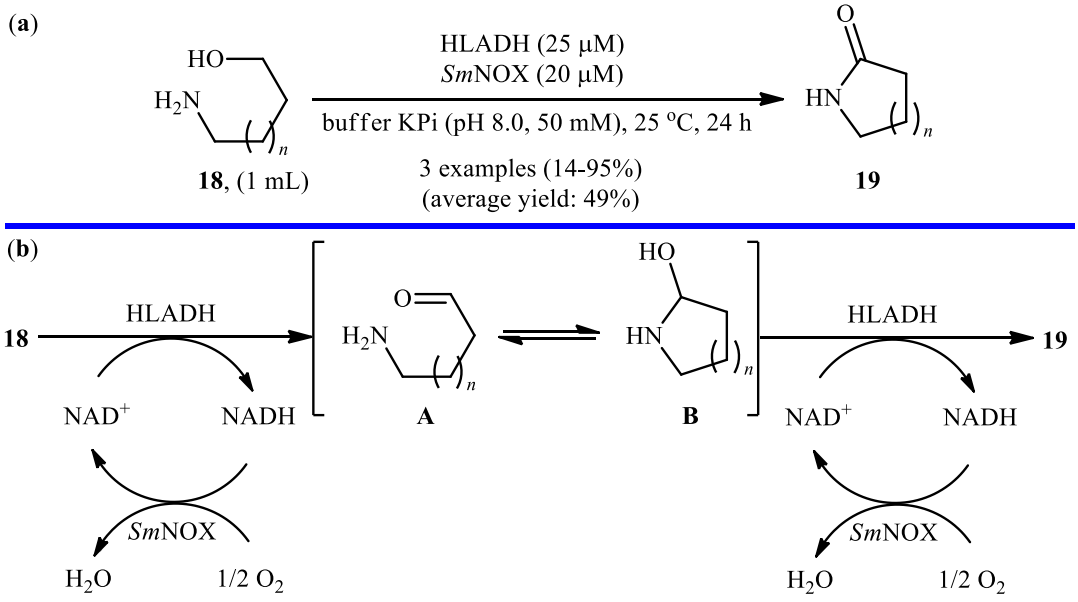


Scheme 11. Fe-catalyzed synthesis of lactams **17** from amino alcohols **16**.



Scheme 12. Mechanistic proposal for the formation of lactams **17**.

By using horse liver alcohol dehydrogenase (HLADH) together with the NADH-oxidase from *Streptococcus mutans* (*Sm*NOX) as a biocatalytic system in KPi buffer (pH 8.0), a small series of aliphatic amino alcohols **18** underwent oxidative cyclization to afford the corresponding five-, six-, and seven-membered lactams **19** in poor to excellent yields (Scheme 13). It was elucidated that the pH of the reaction mixture plays an important role in the performance of the enzymatic oxidative lactamization reaction, as the conversion increased with increasing pH values from 6.0 to 9.0 and then decreased at more alkaline conditions.



Scheme 13. Bienzymatic oxidative lactamization of aliphatic amino alcohols **18** developed by Turner.

3. Conclusion

Lactams are at the heart of a wide number of natural and non-natural biologically active molecules and commercialized drugs that display an impressive variety of biological activities including antibacterial, antimalarial, anticonvulsant, antipsychotic, antidepressant, anti-inflammatory, antitumor, and antidiabetic properties. These pharmacological and biological activities have made the synthesis of these heterocycles quite attractive. The direct synthesis of lactam cores through the oxidative cyclization of amino alcohols has recently been reported. This environmentally friendly approach does not require the use of pre-functionalized starting materials, and hydrogen is produced as the sole by-product. Although, considerable successes have been achieved using this novel and robust synthetic protocols, the reported examples are narrow and there is still further need to study the scope and limitations of this page of lactam synthesis.

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