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Recent Advances in Decarboxylative Nitration of Carboxylic Acids

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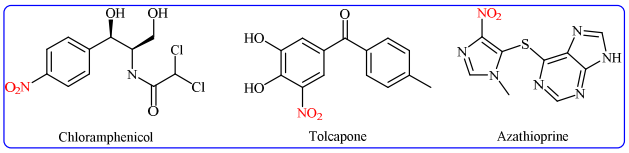
ABSTRACT

In this review, we will summarize the available literature on the preparation of nitro compounds from the corresponding carboxylic acids through nitrodecarboxylation. The review is divided into three major sub-sections. The first focuses exclusively on nitrodecarboxylation of aliphatic carboxylic acids. The second will discuss decarboxylative nitration of aromatic carboxylic acids. The third section will cover the synthesis of nitroolefins *via* decarboxylative nitration of α,β-unsaturated carboxylic acids. Particular emphasis is paid to the mechanistic aspect of reactions.

1. Introduction

Nitro compounds are organic compounds that contain one or more nitro functional groups (–NO_2) in their structure. This versatile functional group is part of the chemical structure of veterinary medications called nitro-drugs [1]. These drugs are used in the treatment of many diseases, including heart failure, hypertension, cancer, trypanosomiasis, and chagas disease (Scheme 1) [2]. The nitro group is also one of the most important and versatile functional group in organic synthesis [3]. It can readily be reduced to amines and oximes, common functional groups required in many pharmaceutical compounds. Recently, nitro compounds have been frequently utilized as versatile and eco-friendly alternatives to traditional organohalide coupling partners in various C-C cross-coupling reactions. These reactions known as denitrative cross-coupling reactions and recently have been highlighted by Cai and co-workers in their interesting review article entitled "denitrative coupling reaction: a powerful synthetic tool in functional transformation" [4]. Due to the widespread synthetic applications and biological activities of nitro compounds, development of novel, straightforward, and efficient synthetic routes to their preparation is always interesting.



Scheme 1. Selected examples of drugs containing nitro group

Carboxylic acids are conveniently available, easy to store, and simple to handle compounds which extensively utilized as building blocks in organic and polymer synthesis [5]. Recently, the decarboxylative cross-coupling reactions carboxylic acids has attracted considerable attention, since it opens a new avenue for fabrication of various carbon–carbon and carbon–heteroatom bonds [6]. In this context, the preparation of nitro compounds through decarboxylative cross-coupling reactions has experienced an explosive growth in recent years. To the best of our knowledge, a comprehensive review has not appeared of the nitrodecarboxylation of carboxylic acids in literature. In connection with our review articles [7], herein, we will summarize the data available from the literature for the synthesis of nitro compounds from the corresponding carboxylic acids (Figure 1). The review is divided into three major sub-sections. The first focuses exclusively on nitrodecarboxylation of aliphatic carboxylic acids. The second will discuss decarboxylative nitration of aromatic carboxylic acids. The third section will cover

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the synthesis of nitroolefins *via* decarboxylative nitration of α,β-unsaturated carboxylic acids.

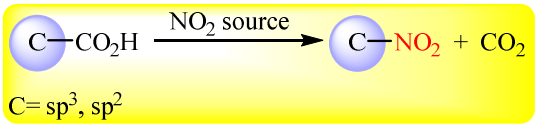
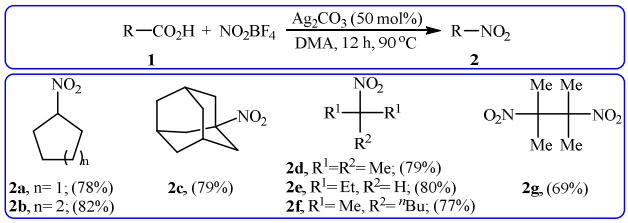


Figure 1. Nitrodecarboxylation of carboxylic acids

2. Aliphatic carboxylic acids

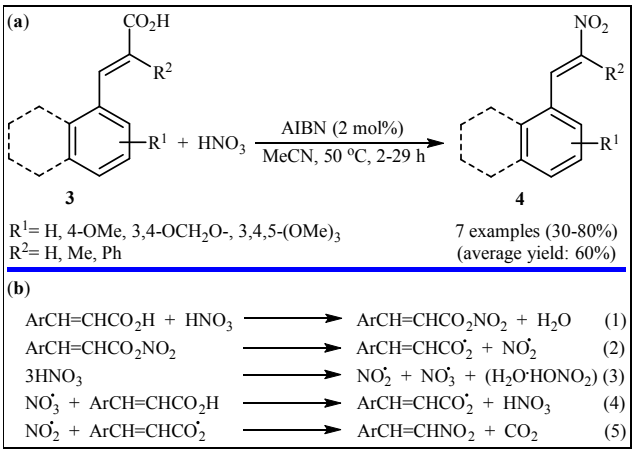
In 2015, the first metal-catalyzed nitrodecarboxylation of aliphatic carboxylic acids was published by Natarajan, Chaudhary, and Venugopalan [8], who showed that the reaction of secondary and tertiary aliphatic carboxylic acids **1** with nitronium tetrafluoroborate (NO_2BF_4) in the presence of 50 mol% Ag_2CO_3 as a decarboxylation reagent in dimethylacetamide (DMA) afforded the corresponding nitro compounds **2** in good to high yields (Scheme 2). Notably, using of DMA as the solvent was crucial to the success of this reaction. Replacing DMA with some other solvents (e.g., MeCN, $CHCl_3$, DCM, DCE, tetrahydrofuran, and tetrachloroethane) led to very poor yields or even no target product at all. Of note, this reagent system was also successfully applied in the *ipso*-nitration of aromatic carboxylic acids to nitroaryls. To the best of our awareness, this is the only example on the decarboxylative nitration of aliphatic carboxylic acids reported so far.



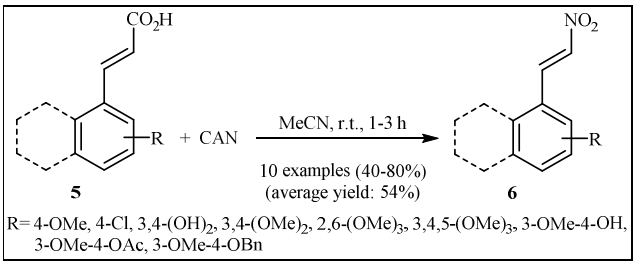
Scheme 2. Natarajan's synthesis of aliphatic nitro compounds **2**

3. Olefinic carboxylic acids

The possibility of decarboxylative nitration of olefinic carboxylic acids was first realized by Roy and colleagues, in 2002, who synthesized a series of nitroolefins **4** from the corresponding α,β-unsaturated carboxylic acids **3** using nitric acid as the NO_2 source and azobisisobutyronitrile (AIBN) as an additive in MeCN (Scheme 3a) [9]. Mechanistically, a free radical process was likely involved in this nitrodecarboxylation (Scheme 3b). In a related study, Rao and co-workers found that electron-rich cinnamic acids **5** were converted to the corresponding nitroolefins **6** through a decarboxylative nitration process using ceric ammonium nitrate (CAN) as the nitrating agent in MeCN at room temperature (Scheme 4) [10]. The system was also amenable to the nitrodecarboxylation of heteroaryl-substituted α,β-unsaturated carboxylic acids, providing synthetically important β-heteroaryl nitroethylene products in good yields. However, when an electron-poor cinnamic acid was employed under the optimal reaction conditions, poor yield of the target product was obtained.



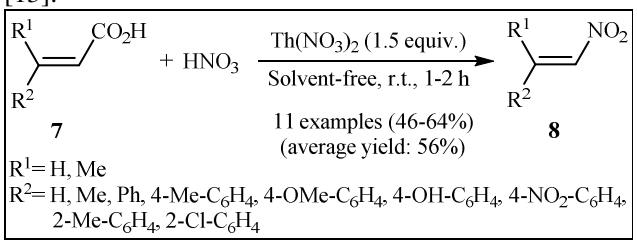
Scheme 3. (a) AIBN-mediated decarboxylative nitration of olefinic carboxylic acids **3** with HNO_3; (b) Plausible mechanism for the formation of nitroolefins **4**



Scheme 4. Nitrodecarboxylation of cinnamic acids **5** with CAN

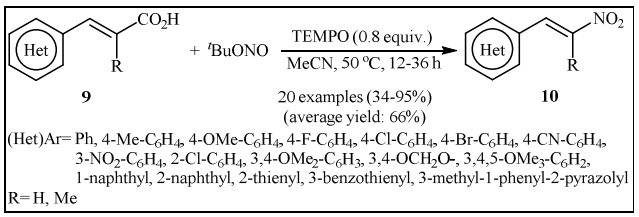
In 2007, Rajanna's research team was able to demonstrate that a range of nitroolefins **8** could be obtained from the metal nitrates mediated nitrodecarboxylation of the respective α,β-unsaturated carboxylic acids **7** under solvent-free conditions employing HNO_3 as the nitro source at ambient temperature [11]. Various metal nitrates such as $Mg(NO_3)_2$, $Sr(NO_3)_2$, $Al(NO_3)_3$, $Ca(NO_3)_2$, $Ni(NO_3)_2$, $Cd(NO_3)_2$, $Zn(NO_3)_2$, $Hg(NO_3)_2$, $AgNO_3$, $ZrO(NO_3)_2$, $UO_2(NO_3)_2$, $Th(NO_3)_2$ have been found to be effective in this transformation. Overall the relative effectivity of the titled metal nitrates in the promoting of nitration of the same set of olefinic carboxylic acids followed the order: $Th(NO_3)_2 \geq Mg(NO_3)_2 \approx Sr(NO_3)_2 \approx Al(NO_3)_3 \approx UO_2(NO_3)_2 \geq ZrO(NO_3)_2 \approx Hg(NO_3)_2 \approx Zn(NO_3)_2 \approx Ni(NO_3)_2 > AgNO_3$. As shown in the Scheme 5, performing the process in the presence of 1.5 equiv. of $Th(NO_3)_2$ afforded the expected products with yield ranging from 46% to 64% within 1-2 h. Later the same research team found that addition of a small amount of PEG gradually decreased the reaction times gradually with an increase in the molecular weight of PEG. Among the several PEGs, PEG-300 has been found to be much more effective than other PEGs [12]. The catalytic activity was found to be in the increasing order: PEG-300 > PEG-400 > PEG-600 > PEG-200. Subsequently, they improved the efficiency of their reaction in terms of reaction time and yields by

performing the process under microwave irradiation conditions employing Zeolite Y as a reusable catalyst [13].



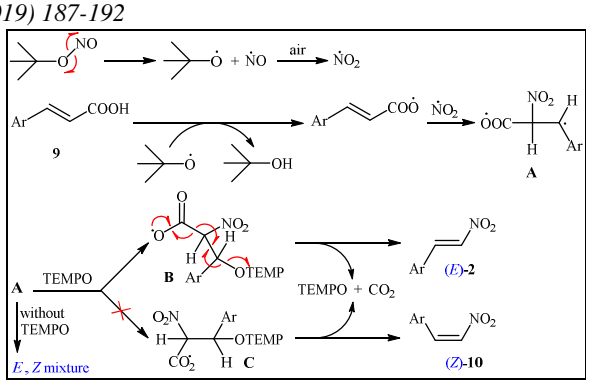
Scheme 5. Rajanna's synthesis of nitroolefins **8**

In 2013, Li and co-workers reported an efficient protocol for the stereoselective synthesis of (*E*)-nitroolefins from the reaction between α,β-unsaturated carboxylic acids and *t*-butylnitrite (*t*-BuONO) through a 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)-mediated nitrodecarboxylation strategy [14]. They discovered that the reaction of 1 equiv. of cinnamic acids **9** with 2-3 equiv. of *t*-BuONO in MeCN at 50 °C in the presence of 0.8 equiv. of TEMPO under aerobic conditions produced the desired (*E*)-nitroolefins **10** in moderate to excellent yields (Scheme 6). This methodology can also be scaled up to provide multi-gram quantities of the target products without sacrificing the yield or outcome of the methodology. Unfortunately, aliphatic carboxylic acids did not form the expected decarboxylative nitration products under the reaction conditions. The author proposed mechanism for this C-NO_2 bond formation reaction is depicted in Scheme 7. The reaction starts with the formation of nitro free radical ($NO_2^{\cdot}$) *via* hemolytic cleavage of O–NO bond and subsequent oxidation under air. Next, $NO_2^{\cdot}$ attacks olefin **9** to form a benzylic radical **A**, which after reaction with TEMPO produces intermediate **B**. Finally, anti-elimination from **B** selectively affords the observed products (*E*)-**10**. Noteworthy, in the absence of TEMPO, a mixture of (*E*) and (*Z*) isomers was generated.



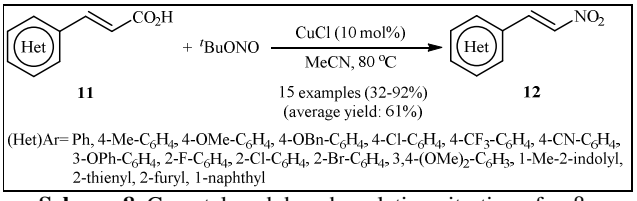
Scheme 6. Synthesis of (*E*)-nitroolefins **10** *via* decarboxylative nitration using *t*-BuONO and TEMPO

Concurrently, Rokade and Prabhu reported an efficient methodology for the synthesis of functionalized nitroolefins **12** *via* Cu(I)-catalyzed nitrodecarboxylation of various aromatic and heteroaromatic α, β-unsaturated acids **11** in the presence of air [15].



Scheme 7. Possible mechanism for the reaction in Scheme 6

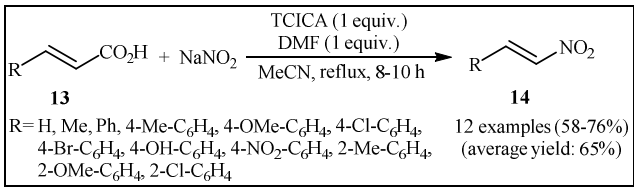
The optimum conditions for this reaction utilize CuCl as the catalyst, *t*-BuONO as the nitrating agent and MeCN as the solvent. Under optimized conditions, the reaction tolerated both electron-donating and electron-withdrawing substituents and gave the desired C-NO_2 bond formation products in moderate to excellent yields (Scheme 8). The authors were also able to demonstrate that a range of β-alkyl nitroolefins could be obtained in high yields from the corresponding β-alkyl cinnamic acids under the standard condition. It is noted that other Cu-salts such as CuI, $CuCl_2{\cdot}2H_2O$, $Cu(ClO_4){\cdot}6H_2O$, and $Cu(OTf)_2$ could also promote this C-N bond formation reaction; albeit in lower yields.



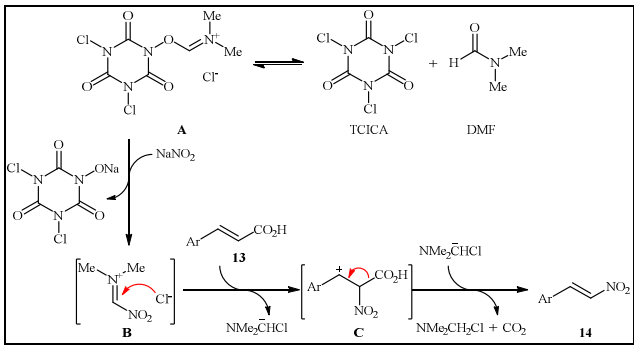
Scheme 8. Cu-catalyzed decarboxylative nitration of α, β-unsaturated carboxylic acids **11** developed by Rokade

Later, Kumar and co-workers reported the preparation of a library of nitroolefins shown in Scheme 9 [16]. These nitroolefins were formed by the treatment of α,β-unsaturated carboxylic acids **13** and NaNO in the presence of trichloroisocyanuric acid (TCICA)/DMF system. The reaction was carried out in DMF at reflux temperature resulted in relatively slow (8–10) decarboxylative nitration to give moderate to good isolated yields of nitroolefins **14**. Interestingly, when the reactions were performed under sonication, the reaction times reduced from 8–10 h to 1.0–1.5 h, even though the yields are comparable under both the conditions. The authors also showed that nitrodecarboxylation of aromatic carboxylic acids under the standard reaction conditions provided a useful route to nitroarenes. According to the author proposed mechanism this reaction proceeds via the initial formation of [TCICADMF] adduct **A** from the interaction of TCICA with DMF. Next, reaction of this intermediate with $NaNO_2$, leading to the formation of nitro iminium ion **B** (nitro methyleniminum ion), which then interacts with

the substrate **13** to afford the corresponding nitroolefin **14** through the intermediate **C** as shown in Scheme 10.

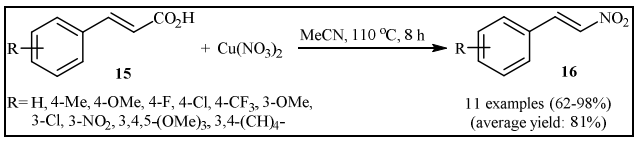


Scheme 9. Synthesis of nitroolefins **2** through the reaction of α,β-unsaturated carboxylic acids **13** and NaNO



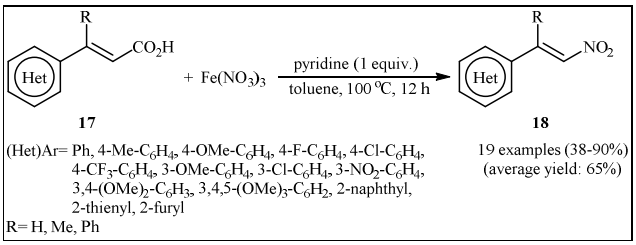
Scheme 10. Plausible mechanistic pathway for the formation of nitroolefins **14**

Alongside this, Luo and co-workers presented an elegant additive-free nitrodecarboxylation of α,β-unsaturated carboxylic acids **15** for the stereoselective synthesis of (*E*)-nitroolefins **16** using inexpensive commercially available $Cu(NO_3)_2$ as both nitrating agent and catalyst in refluxing MeCN (Scheme 11) [17]. Of note, other metal nitrates including $Fe(NO_3)_3$, $Ag(NO_3)_2$, $Ni(NO_3)_2$ and $Na(NO_3)_2$ were proved to be completely ineffective in this transformation. The reaction is noteworthy in that both aryl- and heteroaryl-substituted olefinic carboxylic acids were tolerated. The results demonstrated that the electronic character of the substituents in the phenyl ring periphery of cinnamic acids had little effect on the rate of the reaction.

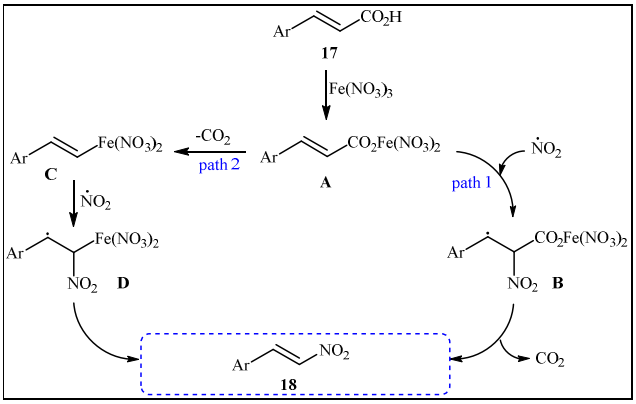


Scheme 11. Luo's synthesis of (*E*)-nitroolefins **16**

Shortly afterwards, Yang and Zhou along with their co-workers re-investigated this reaction using $Fe(NO_3)_3{\cdot}9H_2O$ as the catalyst and nitro source, pyridine as an additive, and toluene as the solvent [18]. Under optimized conditions, the reaction tolerated various aryl and heteroaryl α,β-unsaturated acids **17** and gave the corresponding (*E*)-nitroolefins **18** in moderate to excellent yields (Scheme 12). On the basis of a series of control experimental results and literature precedent, the radical mechanism shown in Scheme 13 was proposed by the authors. The reaction starts with the formation of intermediate **A** by reaction between the $Fe(NO_3)_3{\cdot}9H_2O$ and cinnamic acid **17**. Its reaction with *in situ* generated nitro radical (from $Fe(NO_3)_3{\cdot}9H_2O$ under standard conditions) leads to a radical intermediate **B** that, after decarboxylation affords the target product **18** (Scheme 13, path a). In another possibility, decarboxylation of intermediate **A** yields intermediate **C** which after reaction with $^{\bullet}NO_2$ and demetalation furnishes the final product **18** (Scheme 13, path b).

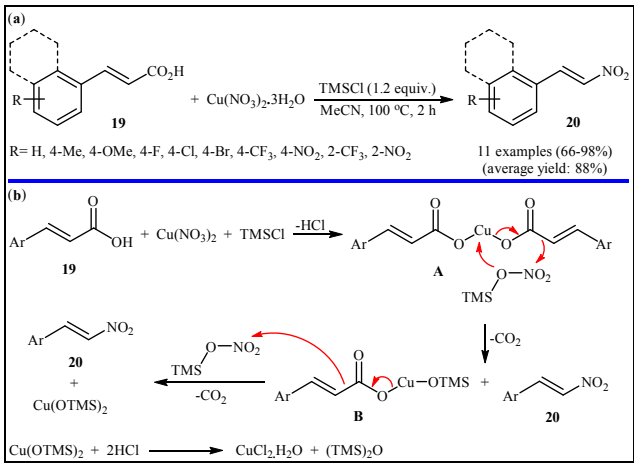


Scheme 12. Fe(III)/pyridine-mediated decarboxylative nitration of α,β-unsaturated acids **17** with $Fe(NO_3)_3$



Scheme 13. Proposed mechanism for the decarboxylative nitration process reported by Yang and Zhou

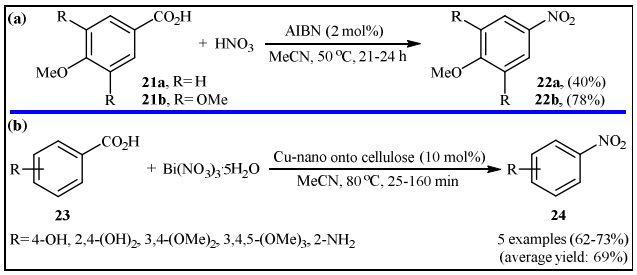
In 2017, Mathew and Prakash reported a beautiful chlorotrimethylsilane (TMSCl)-mediated decarboxylative *ipso*-nitration of cinnamic acids to prepare β-nitrostyrenes [19]. It was found that upon treatment with $Cu(NO_3)_2{\cdot}3H_2O$ as a NO_2 source and TMSCl as an additive, various electron-rich and electron-poor cinnamic acids **19** could be converted into the corresponding β-nitrostyrenes **20** in good to almost quantitative yields (Scheme 14a). However, aliphatic conjugated carboxylic acids such as acrylic acid and 1-cyclopentene carboxylic acid failed to participate in this reaction and reaction of 4-*N*,*N*-dimethylaminocinnammic acid resulted in a mixture of products that could not be separated. Interestingly, when the reaction was carried out in the presence of bromotrimethylsilane (TMSBr) and much lower amount of the nitrate salt, the corresponding *anti*-2,3-dibromo-3-phenylpropanoic acids were obtained in good to excellent yields ranging 77% to 96% instead the desired β-nitrostyrenes. The authors proposed mechanistic pathway for the formation of β-nitrostyrenes **20** is depicted in Scheme 14b.



Scheme 14. (a) TMSCl-mediated decarboxylative *ipso*-nitration of cinnamic acids **19** with $Cu(NO_3)_2$; (b) possible mechanism for the formation of β-nitrostyrenes **20**

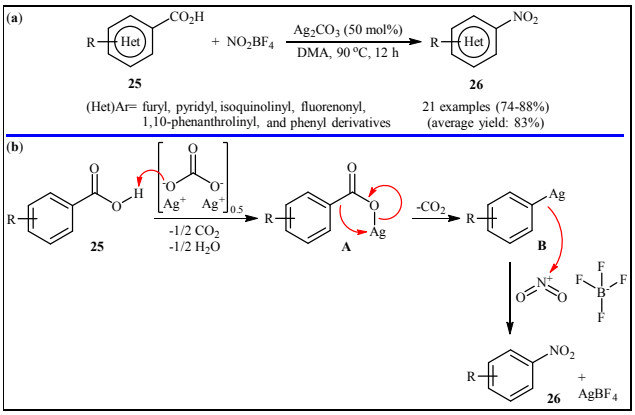
4. Aromatic carboxylic acids

The first mention of the fabrication of nitroaryls through decarboxylative nitration of aromatic carboxylic acids can be found in a 2002 paper by Roy and co-workers, although only two examples were reported (Scheme 15a) [9]. With the objective of designing a comprehensive protocol to nitrobenzenes *via* nitrodecarboxylation of the respective benzoic acids, Baruah and colleagues presented a cellulose supported copper nanoparticle catalyzed decarboxylative nitration of a library of electron-rich aromatic carboxylic acids **23** with $Bi(NO_3)_3{\cdot}5H_2O$ into the corresponding nitroaryls **24** (Scheme 15b) [20]. The reactions were run in MeCN at 80 °C, tolerated various electron-donating substituents (e.g., OH, NH_2, OMe), and generally provided the target products in good yields. Unfortunately, electron withdrawing benzoic acids did not form desired nitrobenzenes under this reaction conditions at all. It should be mentioned that this catalytic system was also successfully applied in decarboxylative nitration of cinnamic acids.



Scheme 15. (a) Roy's synthesis of nitrobenzenes **22**; (b) CuNPs-catalyzed decarboxylative nitration of benzoic acids **23** with $Bi(NO_3)_3{\cdot}5H_2O$

Concurrently, the group of Natarajan developed an analogous one-pot access to nitroaryls **26** by the reaction of various aromatic and heteroaromatic carboxylic acids **25** with NO_2BF_4 in the presence of 50 mol% of Ag_2CO_3 in DMA at 90 °C (Scheme 16a) [21]. Under optimized conditions, the reaction tolerated various important functional groups such as CHO, CO_2Me, OMe, CF_3, F, Cl, Br, , and CN and gave the expected C-NO_2 bond forming products in good to high yields. Interestingly, the authors nicely applied their methodology in the synthesis of two nitro drugs, namely, fexinidazole and nitazoxanide. The mechanism proposed by the authors to explain this C-NO_2 bond formation reaction starts with the formation of metal carboxylate **A** *via* an anion exchange at the silver center, and then extrusion of carbon dioxide from this intermediate to give an arylmetal species **B**. Subsequently, the reaction of intermediate **B** with nitronium ion affords the observed product **26** (Scheme 16b).



Scheme 16. (a) Ag_2CO_3-mediated nitration of aromatic carboxylic acids **25** with NO_2BF_4; (b) Mechanism that accounts for the formation of nitroaryls **26**

5. Conclusion

Nitro compounds are organic compounds that contain at least one nitro group in their structure. These compounds display an impressive variety of biological properties including anti-cancer, anti-Parkinson, anti-infective, anti-inflammatory, anti-leukemic, and anti-bacterial activities. Several commercially available drugs, including tolcapone, nitrofural, chloramphenicol, furazolidone, nitrofurantoin, nitroglycerin, tinidazole, metronidazole, oxamniquine, and nitroxoline are nitro compounds. Due to wide biological activity, synthesis of compounds bearing nitro group are quite attractive, and a large number of straightforward and robust methods for the installation of this versatile group on organic compounds was established. Recently, the decarboxylative cross-coupling reactions carboxylic acids has attracted considerable attention, since it opens a new avenue for fabrication of various carbon–carbon and carbon–heteroatom bonds. As illustrated, nitrodecarboxylation of carboxylic acids has gained a great deal of interest in recent years as useful procedures for the construction of nitro compounds. Easily available starting materials, simplicity of operation, and good yields are the salient features of this synthetic procedure. Hopefully, this methodology will be

employed in the synthesis of biologically important and complex nitro compounds in future studies.

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