

Cross-dehydrogenative coupling of aldehydes with *N*-hydroxyimides: An efficient and straightforward route to *N*-hydroxyimides esters

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ABSTRACT

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Esterification of *N*-hydroxyimides to corresponding active esters (*N*-hydroxyimide esters) is one of the most important organic transformations not only for their importance as versatile intermediates for amides and esters but also their use as coupling partners in various C-S, C-C and C-N coupling reactions. Therefore, there is continuing interest in the development of efficient, practical, and straightforward methodologies for their construction. Nowadays, cross-dehydrogenative coupling reactions, which combine two unmodified C(X)-H (X = heteroatom) bonds for the fabrication of new C(X)-C(X) bonds, are recognized as a fundamental synthetic tool for highly atom-economical synthesis of a wide variety of organic compounds. Along this line, recently, several procedures have been reported for the synthesis of *N*-hydroxyimide esters through the oxidative C-O coupling of with aldehydes with *N*-hydroxyimides. This review highlights recent progresses in this interesting research field.

1. Introduction

N-Hydroxyimide esters are one the most specific class of active esters and extremely promising building blocks in organic synthesis due to their diverse reaction patterns.

These compounds are widely used as versatile raw materials in various carbon-carbon and carbon-heteroatom bond forming reactions. For example (Figure 1), they were successfully applied in the synthesis of sulfides [1], boronate esters [2], hydrazones [3], aryloxyamines [4] and many more [5-10].

Traditionally, the titled compounds were synthesized by the direct coupling of carboxylic acids with *N*-hydroxyimides in the presence of *N,N'*-dicyclohexylcarbodiimide [11].

However, poor atom efficiency and difficult purification have limited the range of applications this approach. Thus, new and efficient methodologies for *N*-hydroxyimide esters are very desirable.

Cross-dehydrogenative-coupling reactions, which combine two C(X)-H (X= heteroatom) bonds to form new C-C and C-X bonds, are valuable and powerful tools in modern organic synthesis that can avoid the use of pre-functionalized starting materials and thus make synthetic schemes shorter, simpler and cleaner [12].

Recently, several groups have reported the synthesis of *N*-hydroxyamide esters from aldehydes and *N*-hydroxyimides *via* oxidative cross-dehydrogenative coupling, either in the presence of a metal catalyst or under metal-free conditions.

In connection with our series of review papers on cross-coupling reactions [13-19] and green chemistry [20-25], we summarize here a variety of methods for the synthesis of *N*-hydroxyamide esters from aldehydes and *N*-hydroxyimides (Figure 2), with special emphasis on the mechanistic features of the reactions.

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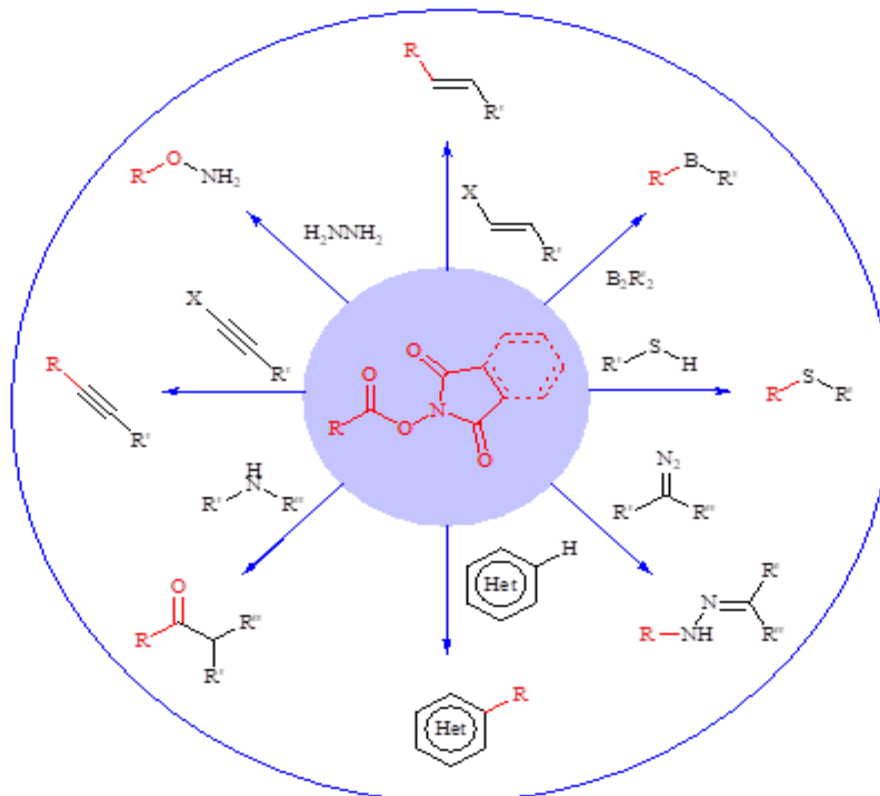


Figure 1. Selected examples on synthetic applications of *N*-hydroxyimide esters.

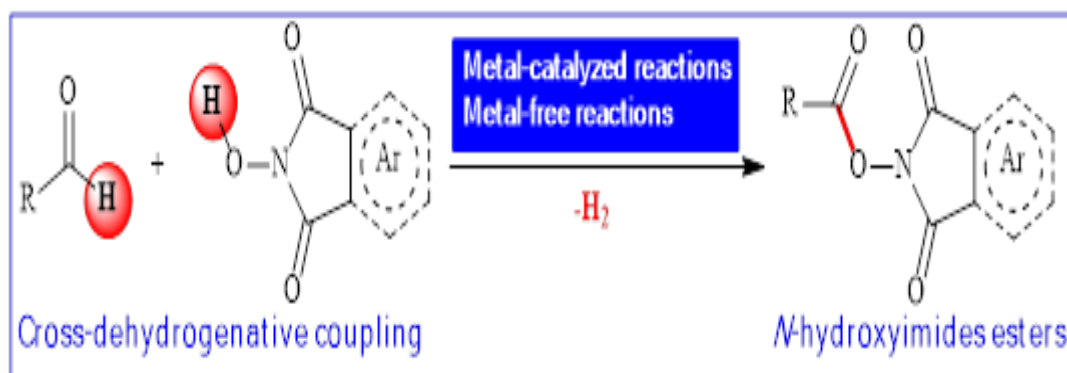


Figure 2. Cross-dehydrogenative coupling of aldehydes with *N*-hydroxyimides.

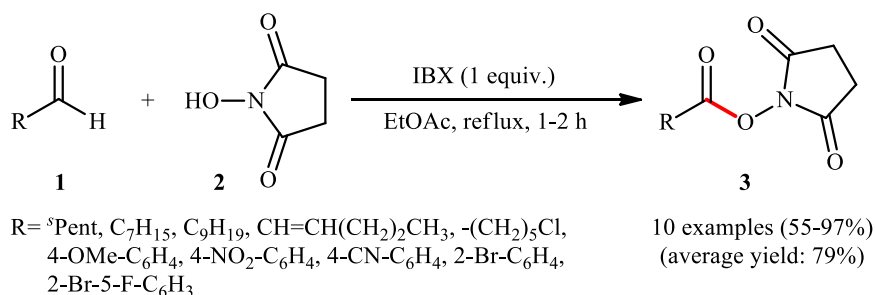
2. Metal-free reactions

In 2004, Schulze and Giannis published the first example of the synthesis of *N*-hydroxyimides esters through the direct coupling reaction between aldehydes and *N*-hydroxyimides under metal-free conditions [26]. They showed that the treatment of various aromatic and aliphatic aldehydes **1** with *N*-hydroxysuccinimide (NHSI; **2**) in the presence of a stoichiometric amount of 1-hydroxy-1,2-benziodoxol-3(1H)-one 1-oxide (IBX) as hypervalent iodine oxidant in refluxing EtOAc rapidly produced the corresponding *N*-hydroxysuccinimide esters **3** in moderate to excellent yields (Scheme 1). The results demonstrated that aliphatic aldehydes were more reactive than aromatic aldehydes and the electronic character of the substituents on the aryl rings of

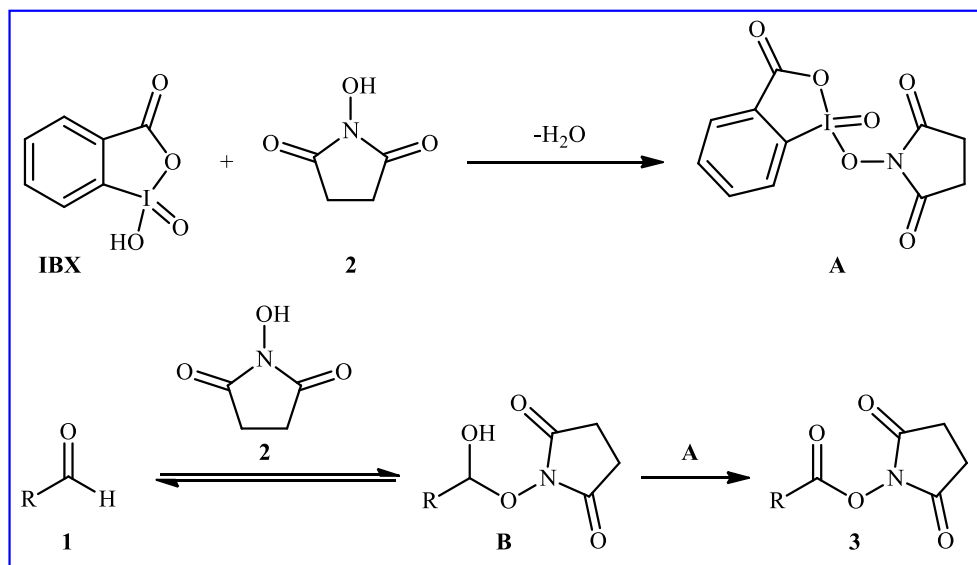
benzaldehydes had no significant impact on the facility of the reaction. However, this protocol was not successful in the case of amino-substituted benzaldehydes. Interestingly, the optimized conditions were also found to be highly efficient for the direct oxidation of alcohols to the corresponding active esters, albeit in this case two equivalents of IBX are required. The author proposed mechanism to explain the formation of *N*-hydroxysuccinimide esters **3** is based on the generation of an IBX-NHSI adduct **A** via the reaction of IBX with NHSI, followed by oxidation of *in situ* generated aldehyde adduct of type **B** (through the reaction of aldehyde **1** with NHS) by this oxidizing agent **A** (Scheme 2). The kinetic isotope effect experiments ($k_H/k_D \approx 9.3$) indicated that the abstraction of the aldehyde hydrogen is

the rate-limiting step of this transformation [27]. In a related study, Liu, Liang, and co-workers demonstrated that $\text{PhI}(\text{OAc})_2$ could also promote this cross-

dehydrogenative coupling reaction [28]. However, only three aldehydes were exemplified.



Scheme 1. IBX-mediated cross-dehydrogenative coupling of aldehydes **1** and *N*-hydroxysuccinimide **2** developed by Giannis.



Scheme 2. Proposed mechanistic pathway for the formation of *N*-hydroxysuccinimide esters **3**.

from the corresponding (hetero)aryl aldehydes **7** (Scheme 3b). Mechanistically, a

Drawing inspiration from these works, Barbas's research team successfully synthesized a series of aryl *N*-hydroxyphthalimide esters **6** from the reaction of respective aromatic aldehydes **4** with *N*-hydroxyphthalimide (NHPI; **5**) in good to excellent yields, using a catalytic amount of tetrabutylammonium iodide tBu_4NI as a catalyst and 2 equiv. of *tert*-butyl hydrogen peroxide (TBHP) as the precatalyst under the near reflux temperature of EtOAc (Scheme 3a) [29].

The reaction showed excellent good group tolerance, including trimethylfluoro, chloro, nitro, and ether functionalities that would allow further elaboration of the products. Moreover, heteroaryl and vinyl aldehydes were also compatible with this methodology. However, nitrogen-containing heteroaromatic and linear aliphatic aldehydes failed to participate in this C-O heterocoupling reaction. Interestingly, the process could be easily scaled up to the gram quantities without sacrificing the yield or outcome of the methodology.

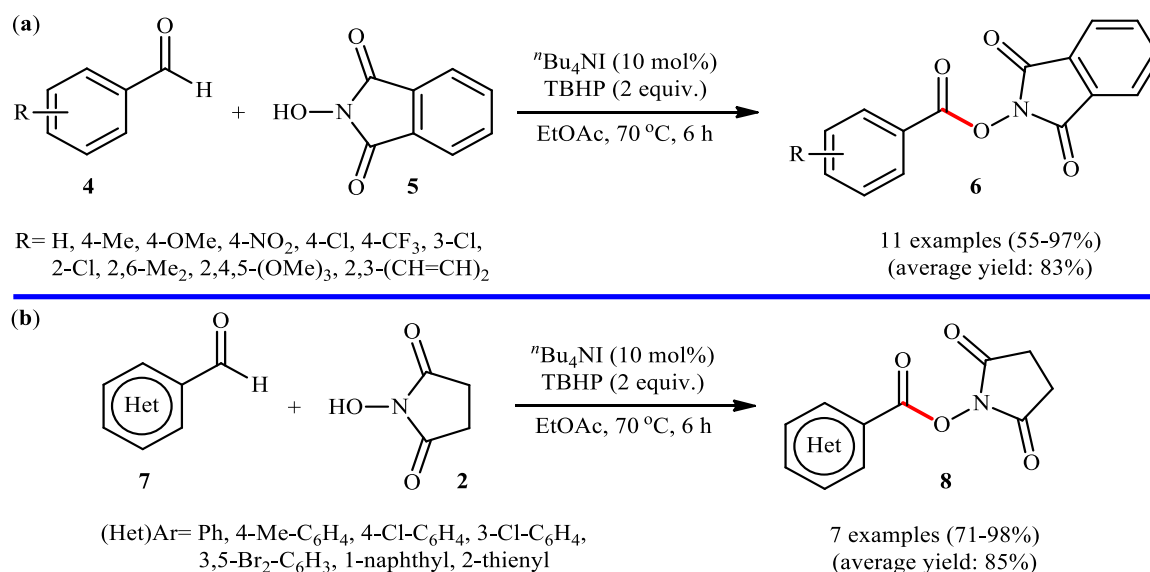
It should be mentioned that the optimized catalytic system was also found to be highly efficient for the synthesis of (hetero)aryl *N*-hydroxysuccinimide esters **8**

free radical process was likely involved in this transformation (Scheme 4). With the objective of designing a general and practical procedure to *N*-hydroxyimides esters through the direct esterification of aldehydes, in 2016, the group of Lv was able to demonstrate that a wide variety of aryl, alkyl, and vinyl *N*-hydroxyphthalimide esters **10** could be obtained in fair to excellent yields from the treatment of corresponding aldehydes **9** with *N*-hydroxyphthalimide **5** in the presence of 1.2 equiv. of selectfluor as an oxidant in MeCN at 90 °C (Scheme 5) [30]. *N*-Hydroxysuccinimide was also tested in the coupling reaction with a library of aromatic aldehydes and gave the corresponding aryl *N*-hydroxysuccinimide ester products in good to excellent yields. Of note, like Barbas's work, this protocol was also allowed large-scale experiments without any difficulty. It is worth mentioning that the presence of selectfluor was crucial for the success of this transformation. No desired product was obtained in the absence of the oxidant.

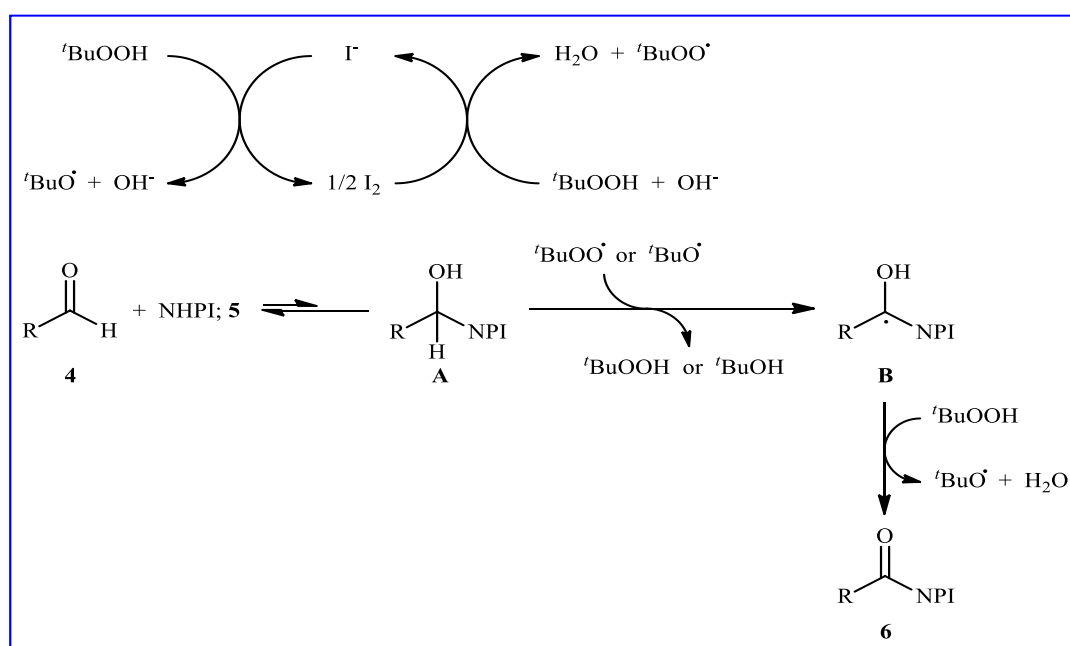
Recently, Xie and colleagues disclosed a beautiful protocol for the construction of (hetero)aryl *N*-

hydroxyphthalimide esters **12** by simple heating of (hetero)aromatic aldehydes **11** and *N*-hydroxyphthalimide **5** in MeCN under catalyst- and additive-free conditions (Scheme 6) [31]. The results indicated that the electronic character of benzaldehydes had little effect on the rate of the reaction. Generally, both electron-donating (e.g., Me, OMe) and electron-withdrawing groups (e.g., CF₃, CO₂Me) were well tolerated, providing the target *N*-hydroxyphthalimide esters **12** in good to excellent yields, ranging from 75% to 95%. The authors successfully applied the prepared active esters as starting materials in the synthesis of various amide products via the treatment of primary amines under catalyst-free conditions. Moreover, they

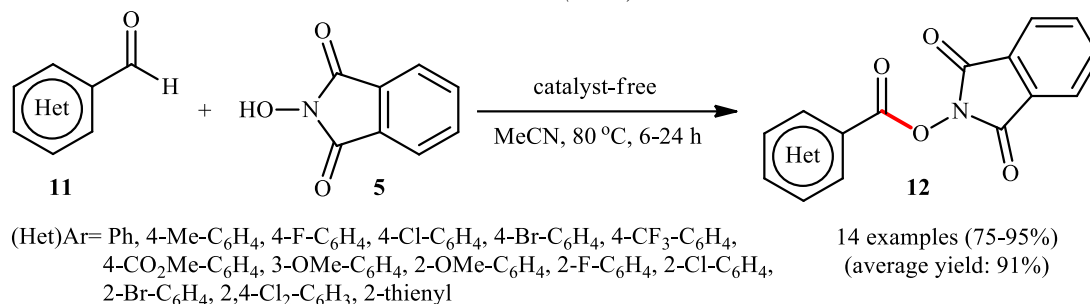
disclosed one-pot synthesis of amides directly from aldehydes, NHPI and amines. According to the authors proposed mechanism, this C-O bond formation reaction may proceed *via* the initial formation of *N*-oxyphthalimide radical **A** through the oxidation of starting *N*-hydroxyphthalimide **5** in the presence of molecular oxygen and under heating. Next, abstraction of a hydrogen from *in situ* generated aldehyde adduct **B** by radical **A** leads to the formation of radical **C**, which then undergoes further oxidation to produce the expected product (Scheme 7).



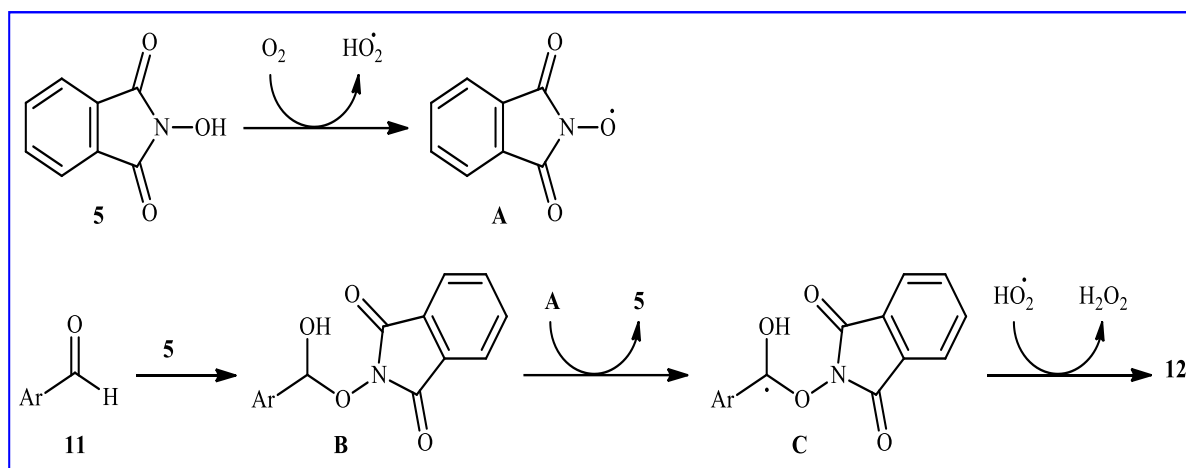
Scheme 3. (a) *t*Bu₄NI-mediated reaction between aromatic aldehydes **4** and *N*-hydroxyphthalimide **5**; (b) C-O heterocoupling between (hetero)aryl aldehydes **7** and *N*-hydroxysuccinimide **2** mediated by *t*Bu₄NI.



Scheme 4. Plausible mechanism for the formation of aryl *N*-hydroxyphthalimide esters **6**.



Scheme 6. Catalyst-free synthesis of *N*-hydroxyphthalimide esters **12**.



Scheme 7. Proposed mechanism for the reaction in Scheme 6.

3. Metal-catalyzed reactions

In 2013, Luca and co-workers reported the first example of the synthesis of *N*-hydroxyimides esters through the metal-catalyzed cross-dehydrogenative coupling of aldehydes with *N*-hydroxyimides [32]. To evaluate the activity of different catalytic systems, 4-methoxybenzaldehyde and *N*-hydroxysuccinimide were chosen as the model reactants. The combination of easily available Cu(OAc)₂•H₂O with TBHP was found to be more effective, which gave a better yield of the target ester. After the optimization of the reaction conditions, they investigated the possibility of performing a one-pot transformation of aldehydes into amides. Thus, by using Cu(OAc)₂•H₂O/TBHP as the catalytic system in refluxing MeCN under an inert atmosphere, the coupling of various aliphatic and aromatic aldehydes **13** with *N*-hydroxysuccinimide quickly afforded the corresponding *N*-hydroxysuccinimide esters **A**, which after treatment with amines **14** at room temperature, converted to the amides **15** in good to almost quantitative yields (Scheme 8).

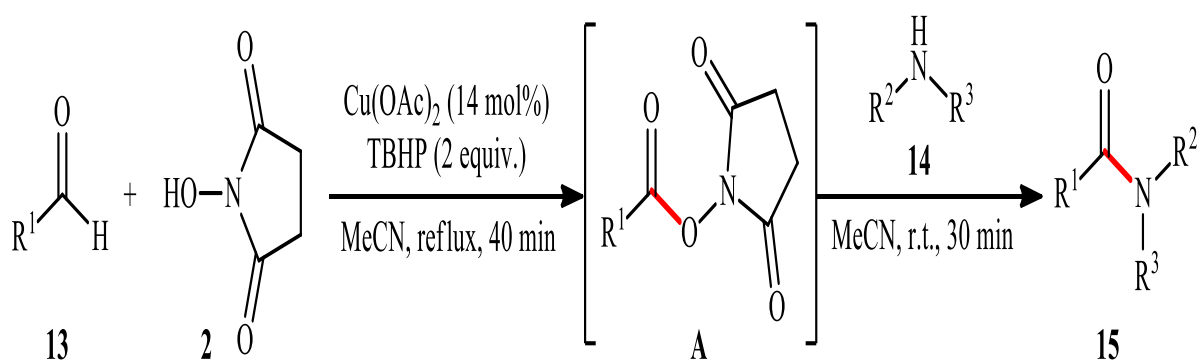
Shortly afterwards, Maity's research team disclosed an interesting visible-light-mediated photoredox-catalyzed cross-dehydrogenative C-O coupling of aldehydes **16** with *N*-hydroxyimides **2** and **5** using only 2 mol% of [Ru(bpy)₃]Cl₂ as a commercially available photoredox catalyst under additive-free conditions [33].

The reactions were carried out in MeCN at room temperature and under irradiation with a LED light bulb, completed within 6-24 h, and afforded the corresponding *N*-hydroxyimide ester products **17** in poor to excellent yields (Scheme 9). The method showed a broad substrate scope including various aryl, heteroaryl, and vinyl aldehydes. However, Aliphatic aldehydes failed to produce any product under the optimized conditions. The mechanistic pathway depicted in Scheme 10 was suggested for this C-O bond formation reaction. It consists of the following key steps: (i) photoexcitation of the ground state Ru^{II} complex by visible light to form the excited state [Ru^{II}]*; (ii) electron transfer from excited [Ru^{II}]* to *N*-hydroxyimide **2** (or **5**) to produce a radical cation and a Ru^I complex; (iii) deprotonation of intermediate **A** to generate imide *N*-oxyl radical **B**; (iv) reaction of radical **B** with aldehyde **16** to produce hemiaminal radical **D** through the intermediate **C**; and (v) oxidation of intermediate **D** to give the final product **17**.

In 2017, with the aim of designing a greener procedure to *N*-hydroxyimides esters through metal-catalyzed cross-dehydrogenative coupling reactions, Jin and Su along with their co-workers were able to demonstrate that a diverse range of *N*-hydroxyphthalimide esters **19** could be synthesized from the reaction of corresponding aldehydes **18** with *N*-hydroxyphthalimide **5** employing Cu(0)/selectfluor/PhI(OAc)₂ combination as a catalytic

system in the most environmentally-friendly solvent, water, at room temperature (Scheme 11a) [34]. Various alky, benzyl, vinyl, aryl, heteroaryl aldehydes were successfully utilized to establish the general applicability of this synthetic process. Beside the use of environmentally benign solvent and mild reaction conditions, broad substrate scope, short reaction time, as well as the use of the inexpensive elemental copper catalyst were the advantages, mentioned for this methodology. Concurrently, the group of Xie showed that aldehydes **20** can be effectively coupled with *N*-hydroxyimides **2** and **5** in the presence of 20 mol% of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ as a magnetically separable catalyst in

MeCN under an atmosphere of air (Scheme 11b) [35]. The results showed that *N*-hydroxyphthalimide **5** compare to the *N*-hydroxysuccinimide **2** gave higher yield of the desired products. picolinamide derivatives bearing electron-donating groups were high yielding compared to electron-withdrawing ones. The relative reaction rates of aldehydes followed the order: aryl aldehydes \geq alkyl aldehydes $>$ vinyl aldehydes $>$ heteroaryl aldehydes. It is noteworthy that other metal catalysts such as AgNO_3 , $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ were also found to promote this reaction, albeit at lower efficiencies.



$\text{R}^1 = \text{C}_6\text{H}_{13}, \text{C}_8\text{H}_{17}, \text{CH}_2\text{C}(\text{Me})_3, \text{CH}=\text{CHPh}, \text{Ph}, 4\text{-Me-C}_6\text{H}_4, 4\text{-OMe-C}_6\text{H}_4, 4\text{-COMe-C}_6\text{H}_4,$
 $4\text{-NO}_2\text{-C}_6\text{H}_4, 4\text{-Cl-C}_6\text{H}_4, 3\text{-Cl-C}_6\text{H}_4, 2\text{-Me-C}_6\text{H}_4, 2\text{-Cl-C}_6\text{H}_4, 2\text{-thienyl}$

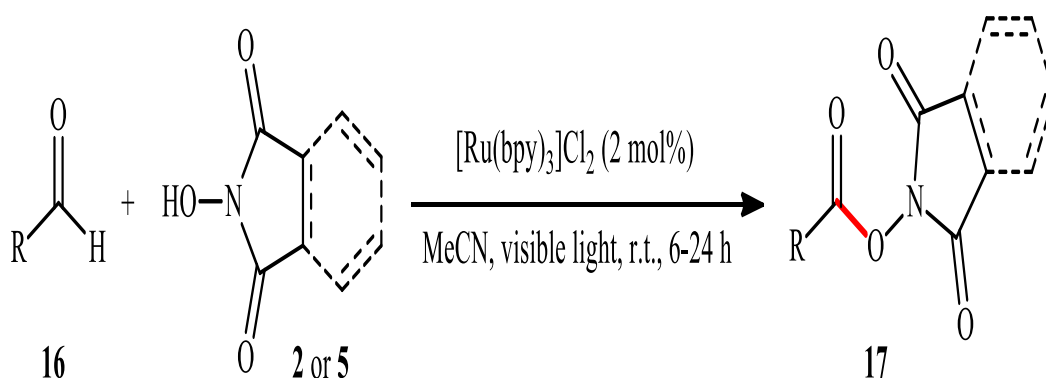
19 examples (65-98%)
 (average yield: 86%)

$\text{R}^2 = \text{H}, \text{Me}, i\text{Pr}$

$\text{R}^3 = \text{C}_4\text{H}_9, \text{C}_7\text{H}_{15}, \text{CH}_2\text{CH}_2\text{OH}, \text{CH}_2\text{CH}_2\text{OMe}, \text{CH}_2\text{CH}=\text{CH}_2, \text{OMe}, \text{CO}_2\text{Me}, \text{Bn}, \text{Ph}, 4\text{-Me-C}_6\text{H}_4$

$\text{R}^2 + \text{R}^3 = \text{-(CH}_2\text{)}_5\text{-}, \text{-(CH}_2\text{)}_2\text{O(CH}_2\text{)}_2\text{-}$

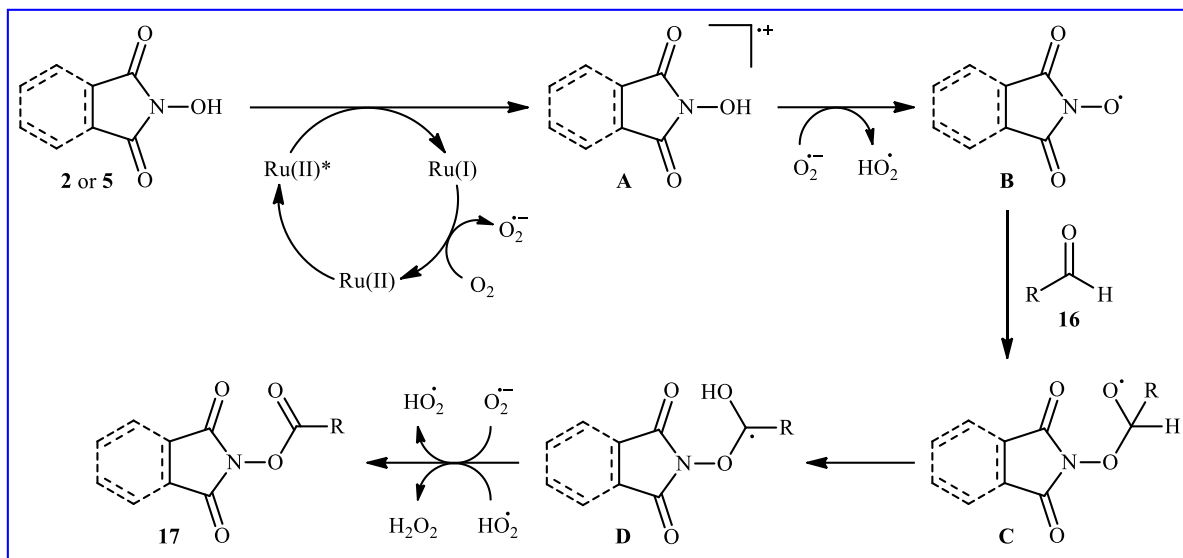
Scheme 8. Luca's synthesis of amides **15**.



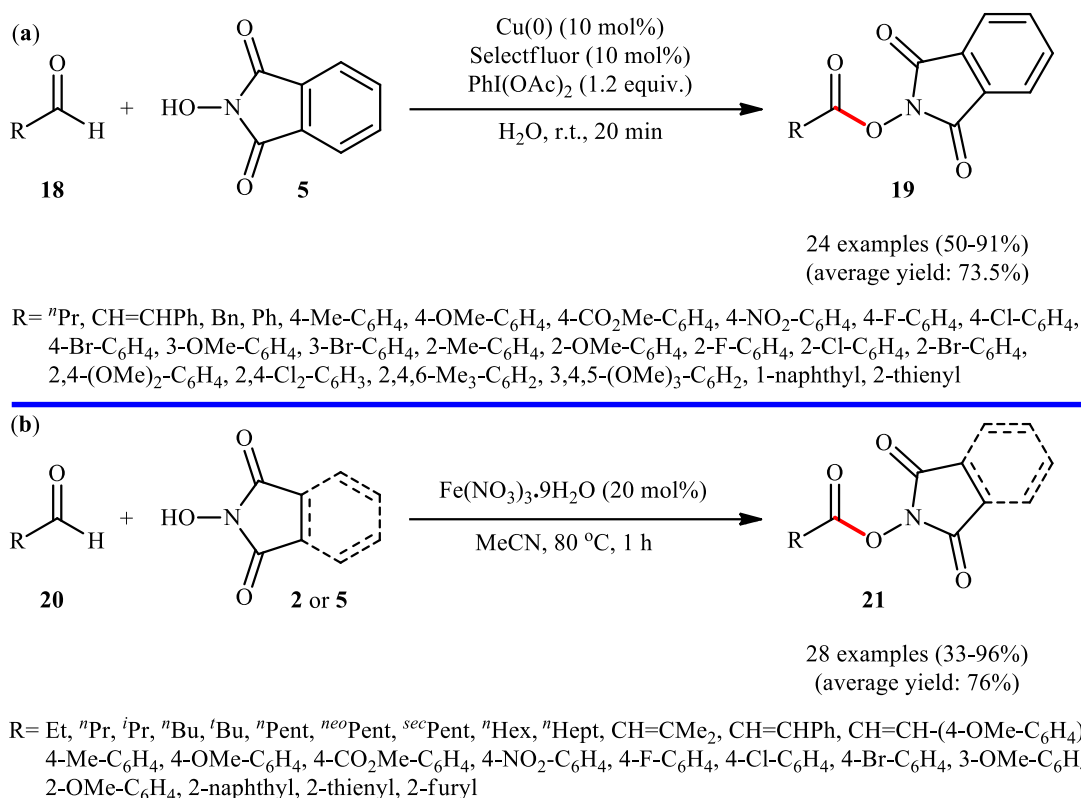
$\text{R} = \text{CH}=\text{CHPh}, \text{Ph}, 4\text{-Me-C}_6\text{H}_4, 4\text{-OMe-C}_6\text{H}_4, 4\text{-NO}_2\text{-C}_6\text{H}_4, 4\text{-F-C}_6\text{H}_4,$
 $4\text{-Cl-C}_6\text{H}_4, 4\text{-Br-C}_6\text{H}_4, 3\text{-F-C}_6\text{H}_4, 3\text{-Br-C}_6\text{H}_4, 2\text{-F-C}_6\text{H}_4, 3\text{-pyridyl},$
 $2\text{-furyl}, 2\text{-thienyl}$

21 examples (21-92%)
 (average yield: 54%)

Scheme 9. Visible-light-mediated photoredox-catalyzed coupling of aldehydes **16** with *N*-hydroxyimides **2** and **5**.



Scheme 10. Proposed reaction mechanism for the formation of N-hydroxyimides esters **17**.



Scheme 11. (a) Cu-catalyzed cross-dehydrogenative coupling reaction between aldehydes **18** and N-hydroxyimides **2** and **5** in water; (b) Fe-catalyzed synthesis of N-hydroxyphthalimide esters **21** from aldehydes **20** and N-hydroxyimides **2** (or **5**).

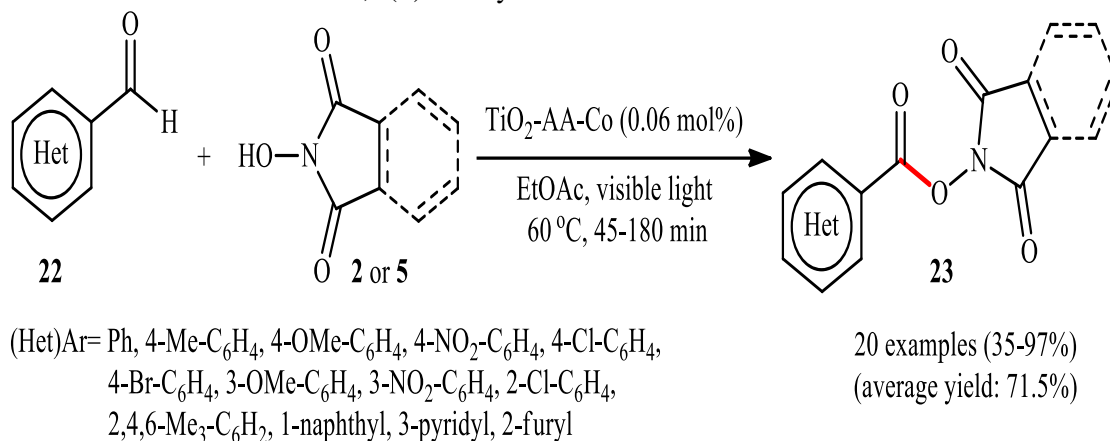
In 2018, Feizpour, Jafarpour, and Rezaeifard developed an aerobic photocatalytic cross-dehydrogenative coupling reaction between aldehydes **22** and N-hydroxyimides (**2** and **5**) using titanium dioxide–cobalt–ascorbic acid nanohybrid (TiO₂–AA–Co) as a photocatalyst for the high yielding synthesis of N-hydroxyimides esters **23** without using any additive or oxidant [36]. The reactions were performed in EtOAc under irradiation of a fluorescent lamp (CFL: 40 W, 1.1 W m⁻²) at 60 °C, tolerated various important functional groups, and generally afforded the target C–O bond

formation products in moderate to excellent yields (Scheme 12). Importantly, the reaction can also be scaled up without loss of the yield. Of note, the catalyst can be readily separated by centrifugation and reused over five consequent runs without a significant decrease in activity. The authors proposed mechanistic course for this heterocoupling reaction is illustrated in Scheme 13.

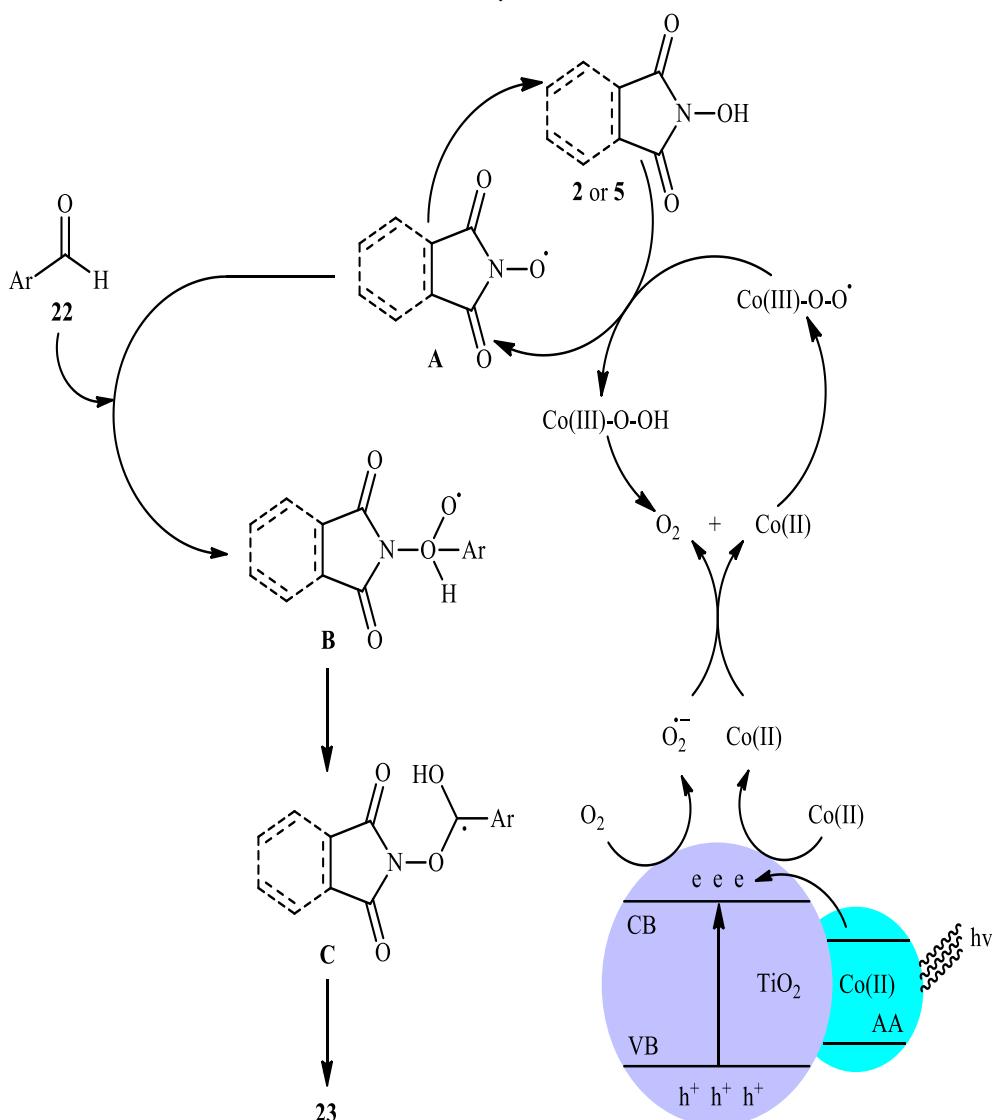
In a related investigation, Panda and co-workers synthesized nineteen N-hydroxyimides esters **25** in poor to excellent yields (20-92%) through simultaneous

selective oxidation of alcohols **24** to the corresponding aldehydes followed by cross-dehydrogenative coupling with *N*-hydroxyimides (**2** and **5**) using N-doped yellow TiO₂ (N-TiO₂) hollow sphere as a visible light active photocatalyst and TBHP as an oxidant (Scheme 14) [37]. Some important information of the reactions is listed below: (i) the outcome of reaction is highly dependent on the electronic nature of the sunstrates; (ii) benzylic

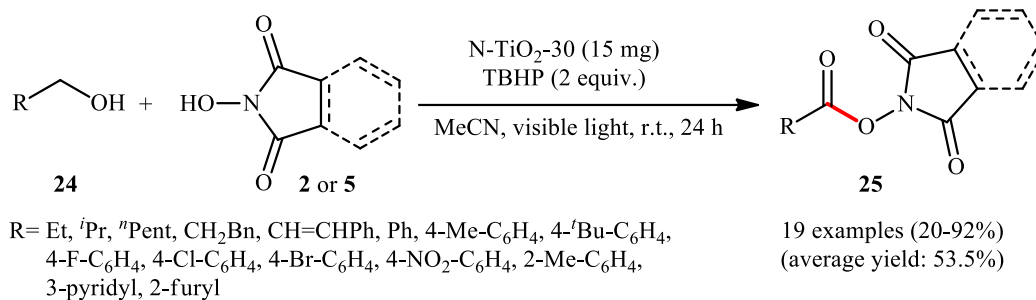
alcohols were high yielding compared to aliphatic and heterobenzylic alcohols; (iii) electron-rich amides compare to the electron-poor ones gave higher yield of the desired products; (iv) *N*-hydroxyphthalimide gave higher yields than *N*-hydroxysuccinimide; and (iv) the catalyst is reusable for at least four times with tangible decrease in its catalytic activity.



Scheme 12. Visible-light-mediated synthesis of *N*-hydroxyimides esters **23** developed by Rezaeifard



Scheme 13. Proposed mechanism for the formation of *N*-hydroxyimides esters **23**.



Scheme 14. N-TiO₂-catalyzed esterification of alcohols **24** and N-hydroxyimides **2** or **5**.

4. Conclusion

N-Hydroxyimide esters have been identified as extremely versatile building blocks in various organic transformations. Consequently, many efforts have been made to develop efficient synthetic strategies to access to this specific class of active esters. As illustrated, direct synthesis of these compounds through cross-dehydrogenative coupling reactions between aldehydes with N-hydroxyimides has attracted a lot of attentions in recent years as it avoids the use of pre-functionalized starting materials. Beside, easily accessible starting materials, broad substrate scope, high atom and step economy, as well as simplicity were other advantages of this synthetic process. This research area has still further possibilities for growth and we hope that this Mini-Review will be beneficial for stimulating researchers to further think and research on this topic.

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