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Silver-mediated oxidative 1,2-alkylesterification of styrenes with nitriles and acids *via* C(sp^3)–H functionalization†

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A new silver-mediated 1,2-alkylesterification of alkenes with nitriles and acids promoted by a catalytic amount of nickel catalyst for producing acyloxylated nitriles has been developed *via* a C(sp^3)–H functionalization process. By employing the NiI_2 and Ag_2CO_3 catalytic systems, the method features broad substrate scope with respect to carboxylic acids, including linear alkyl acids, cyclic acids, aryl acids and amino acids.

Nitriles have received increasing interest in recent years due to their wide applications in the synthesis of aldehydes, amides, amines, acids and other carboxy compounds.[1] Importantly, multifunctional ester-nitrile compounds bearing these functional groups are widely found in drugs and natural products (Fig. 1).[2] Thus, many methods have been developed to introduce cyano-containing moieties into the molecule. Traditionally, transition metal (Rh, Ru, Fe, Ni, *etc.*) catalyzed C–H activation of alkyl nitriles has been regarded as the most powerful tool to realize cyano-containing moiety insertion.[3–5] However, the classical α-functionalization of stable alkyl nitriles remains highly challenging for the following reasons: (1) the requirement of harsh reaction conditions; (2) slow reductive elimination of related metal–nitrile anion complexes; (3) the formation of insoluble oligomers with main group reagents.

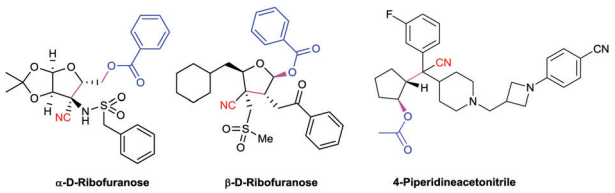
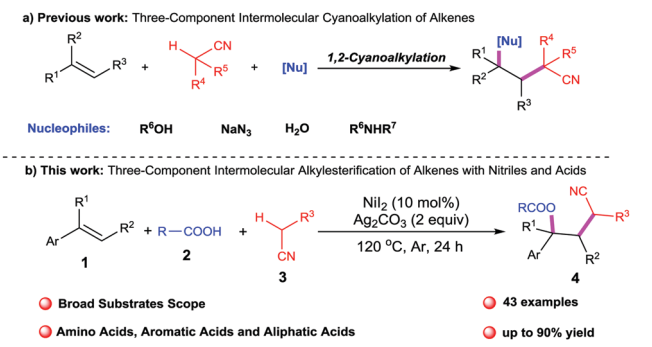


Fig. 1 Important examples of bioactive multifunctional ester-nitrile compounds.

1,2-Difunctionalization of alkenes has been becoming an ideal and highly step-economical method to convert highly functionalized compounds from simple and easily available starting materials.[6] In recent years, the free radical-initiated functionalization of less reactive C(sp^3)–H bonds of alkyl nitriles has been developed as an appealing strategy to prepare complex functional cyano-containing molecules.[7] For example, in 2015, Zhu and co-workers achieved copper-catalyzed carboetherification of α-substituted styrenes used DTBP as the initiator.[8] Sequentially, the Wang, Chen, Xing group and Zhu group also developed three-component oxyalkylation and carboazidation of alkenes with alkyl nitrile reactants, respectively (Scheme 1a).[9] However, most of these transformations suffer from limited scope of the substrates or the use of stoichiometric amounts of peroxides. Recently, our group has achieved three-component 1,2-carboamination of alkenes with alkyl nitriles and amines catalysed by a combination of Ag_2CO_3 and Fe reagents (Scheme 1a).[10] Inspired by the elegant studies on the alkyl nitrile-center radical-initiated α-C–H functionalization and combining our previous work in the development of multifunctional ester-carbon substitution synthesis, we envisioned that by employing



Scheme 1 Important examples for 1,2-cyanoalkylation of alkenes.

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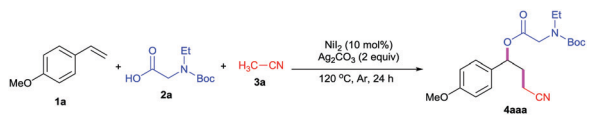
the strategy of difunctionalization of alkenes using an alkyl nitrile-center radical with styrenes and acids might initiate a new three-component alkylesterification reaction.[11] Herein, we reported a novel nickel-promoted, silver-mediated three-component oxidative 1,2-alkylesterifition of styrenes with nitriles and acids leading to acyoxylated nitriles (Scheme 1b). Importantly, not only carboxylic acids including linear alkyl acids, cyclic acids and aryl acids but also amino acids can work well under the NiI_2/Ag_2CO_3 catalyst system in this transformation.

Our initial attempt of this alkene difunctionalization process began with the reaction of 1-methoxy-4-vinylbenzene (**1a**), *N*-(*tert*-butoxycarbonyl)-*N*-ethylglycine (**2a**), and acetonitrile (**3a**) (Table 1). To our delight, 90% of the target 3-cyano-1-(4-methoxyphenyl)propyl *N*-(*tert*-butoxycarbonyl)-*N*-ethylglycinate (**4aaa**) can be successfully obtained with NiI_2 as the catalyst and Ag_2CO_3 as the additive at 120 °C for 24 h (entry 1). However, 60% yield of product **4aaa** can be isolated without a NiI_2 catalyst in the reaction (entry 2). Examination of the amount of NiI_2 catalyst indicated that 10 mol% of NiI_2 is the best amount for this transformation (entries 3 and 4). Changing NiI_2 to other Ni catalysts, such as $Ni(acac)_2$, $NiBr_2$, $Ni(OAc)_2$ and $Ni(PPh_3)_4$, all gave decreased yields (entries 5–8). However, only 35% yield of **4aaa** was isolated by the use of our previous conditions of 1,2-carboamination (entry 9).[10] In the absence of Ag_2CO_3, **4aaa** was not formed combined with the by-product 1-(4-methoxyphenyl)ethan-1-ol (**4aaa′**) in 49% yield (entry 10). When the amount of Ag_2CO_3 was screened, we found that 2 equiv. of Ag_2CO_3 was needed to give the best result (entries 11 and 12). Two other additives, including Ag_2O and AgOAc were also evaluated instead of Ag_2CO_3, and both of them resulted in lower yields (entries 13 and 14). Among the solvents and reaction temperatures tested, the reaction performed in MeCN at 120 °C gave the best yield (entries 15–18).

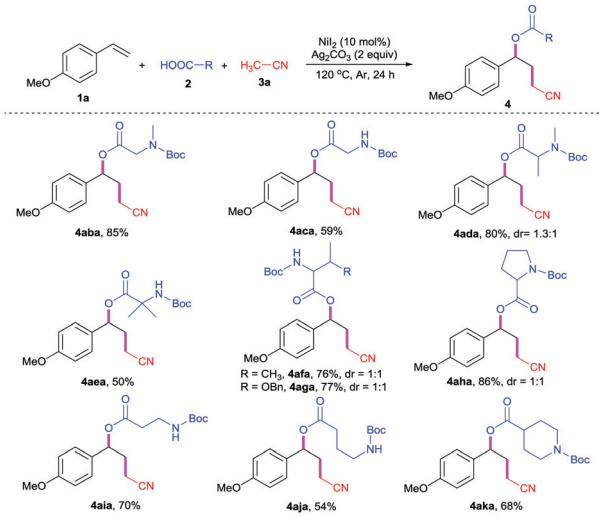
Table 1 Screening of optimal reaction conditions[a]



Entry	Variation from the standard conditions	Isolated yield (%)
1	None	90
2	Without NiI_2	60
3	NiI_2 (5 mol%)	74
4	NiI_2 (20 mol%)	82
5	$Ni(acac)_2$ instead of NiI_2	45
6	$NiBr_2$ instead of NiI_2	74
7	$Ni(OAc)_2$ instead of NiI_2	73
8	$Ni(PPh_3)_4$ instead of NiI_2	60
9	$Fe(OTf)_3$ or $FeCl_3$ (10 mol%), Ag_2CO_3	35
10	without Ag_2CO_3	0[b]
11	Ag_2CO_3 (1 equiv.)	59
12	Ag_2CO_3 (3 equiv.)	88
13	Ag_2O instead of Ag_2CO_3	65
14	AgOAc instead of Ag_2CO_3	58
15	**3a** (4 equiv.) in toluene	29
16	**3a** (4 equiv.) in DMF	48
17	at 140 °C	78
18	at 100 °C	62

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (2 equiv.), **3a** (2 mL), NiI_2 (10 mol%), Ag_2CO_3 (2 equiv.), argon, 120 °C, and 24 h. [b] By-product 1-(4-methoxyphenyl)ethan-1-ol (**4aaa′**) was isolated.

Table 2 Variation of amino acids (**2**)[a]

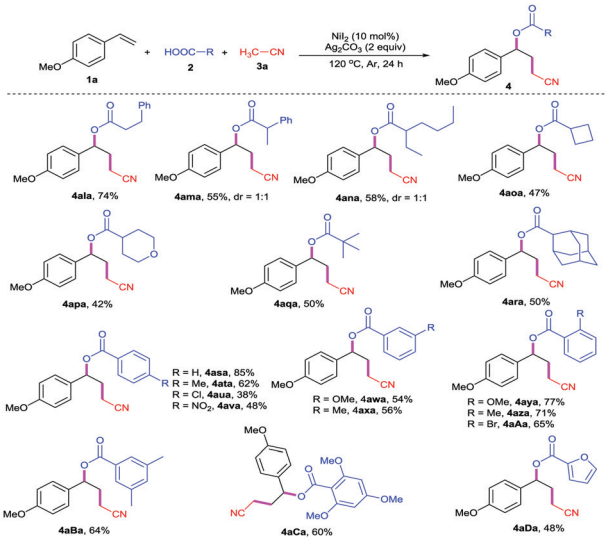


[a] Reaction conditions: **1a** (0.2 mmol), **2** (2 equiv.), **3a** (2 mL), NiI_2 (10 mol%), Ag_2CO_3 (2 equiv.), argon, 120 °C, and 24 h. The dr value was detected by ^1H NMR analysis.

With the optimized conditions in hand, we then explored the scope of this 1,2-alkylesterification reaction first with respect to amino acids (**2**) and the results are shown in Table 2. A variety of amino acids **2**, including α-amino acids **2b–h**, β-amino acid **2i** and γ-amino acids **2j–k**, were initially investigated in the presence of 1-methoxy-4-vinylbenzene (**1a**) and acetonitrile (**3a**). The results revealed that they exhibited good reactivities and afforded the corresponding 1,2-alkylesterification products **4aba–aha** in 50–86% yields. For example, substrates bearing an N–H free group **1c** or a hetero ring group **1h** were compatible with the reaction conditions and provided products **4aca** and **4aha** in 59% and 86% yields, respectively. β-Amino acid **2i** was also smoothly converted to the desired product **4aia** in 70% yield. Notably, γ-amino acids **2j** and **2k** were also suitable for this transformation and gave the desired products **4aja–aka.**

Subsequently, we examined a variety of alkyl- and aryl carboxylic acids **2** under the optimal conditions (Table 3). In general, various kinds of alkyl acids including linear acids and cyclic acids could smoothly react with 1-methoxy-4-vinylbenzene (**1a**) and acetonitrile (**3a**) to produce the target acyloxylated nitriles **4.** For instance, when linear acid 3-phenylpropanoic acid **1l** and cyclic acid 1-adamantyl carboxylic acid **1r** were subjected to the standard conditions, the C–C/C–O bonds could be directly formed to furnish complex nitriles **4ala** and **4ara** in 74% and 50% yields, respectively. Then the substrate scope of different types of aromatic acids was investigated. Moderate to good yields can be achieved in using aromatic acids including heterocyclic acids. The results showed that the electronic effect of the substituent on the aromatic ring of the carboxylic acids had a significant impact on the yields of the product. For example, substrates bearing a methyl group **2t** or nitro group **2v** were compatible with the reaction conditions and provided products **4ata** and **4ava** in 62% and 48% yields,

Table 3 Variation of carboxylic acids (**2**)[a]



[a] Reaction conditions: **1a** (0.2 mmol), **2** (2 equiv.), **3a** (2 mL), NiI_2 (10 mol%), Ag_2CO_3 (2 equiv.), argon, 120 °C, and 24 h. The dr value was detected by ^1H NMR analysis.

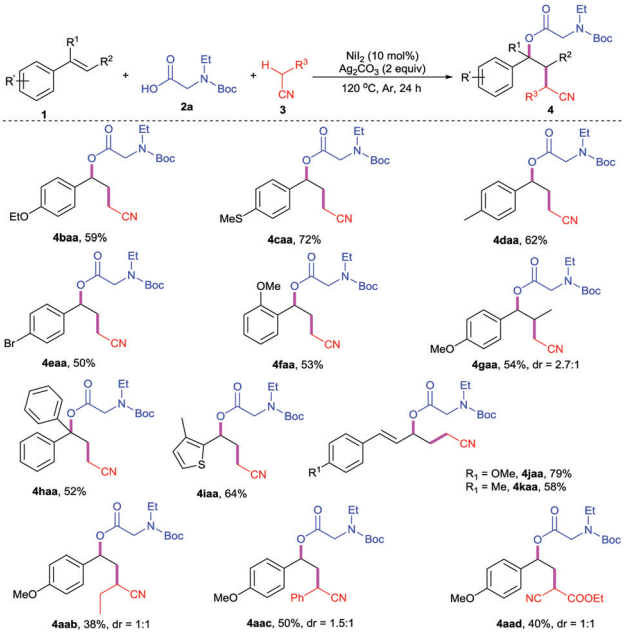
respectively. However, the steric hindrance had a little effect on the reaction. The *ortho*-substituted aromatic acid (**2z**) gave a higher yield than the *meta*- (**2x**) or *para*-substituted aryl acid (**2t**). Furthermore, heterocyclic acid **2D** also gave the desired product **4aDa** in moderate yield.

We turned our attention to examine the scope of various styrenes (**1**) and nitriles (**3**) for 1,2-alkylesterification (Table 4). The reaction can be performed with styrenes containing various functional groups including OEt, SMe, Me, Br and OMe. Satisfyingly, the SMe and Br substituents were well tolerated in this transformation; products **4caa** and **4eaa** could be isolated in 72% and 50% yields, respectively. Furthermore, 1,2-disubstituted alkenes **1g** and 1,1-disubsituted alkenes **1h** also proceeded smoothly and afforded the corresponding products **4gaa** and **4haa** in 54% and 52% yields, respectively. Alkene **1i** bearing a heterocycle reacted well and provided product **4iaa** in 64% yield. Furthermore, 1,3-dienes were also explored and the results revealed that (*E*)-1-(buta-1,3-dien-1-yl)-4-methoxybenzene **1j** and (*E*)-1-(buta-1,3-dien-1-yl)-4-methylbenzene **1k** were compatible with our protocol to give 1,4-difunctionalization products **4jaa** and **4kaa** in 58–79% yields. As expected, three other nitriles, including butyronitrile, 2-phenylacetonitrile and ethyl 2-cyanoacetate, were well-tolerated to deliver the products **4aab–aac** in 38–50% yields.

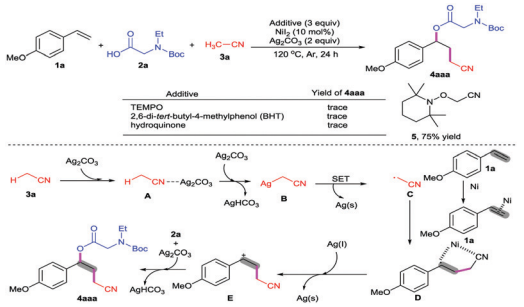
In order to gain insight into this 1,2-alkylesterification process, a series of control experiments were carried out (Scheme 2). When excessive radical inhibitor reagents, such as 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), 2,6-di-*tert*-butyl-4-methylphenol (BHT) and hydroquinone were used, only trace yield of product **4aaa** was detected by GC-MS analysis, which suggested that this reaction proceeds *via* a radical pathway.

On the basis of the control experiments and the literature,[12,13] a possible reaction mechanism is proposed (Scheme 2). At first,

Table 4 Variation of the styrenes (**1**) and nitriles (**3**)[a]



[a] Reaction conditions: **1** (0.2 mmol), **2a** (2 equiv.), **3** (2 mL), NiI_2 (10 mol%), Ag_2CO_3 (2 equiv.), argon, 120 °C, and 24 h. The dr value was detected by ^1H NMR analysis.



Scheme 2 Control experiments and possible mechanism.

the interaction of Ag_2CO_3 with CH_3CN formed the intermediate **A**, followed by reaction with Ag_2CO_3 to afford the $AgCH_2CN$ intermediate **B**. The alkyl radical intermediate **C** was then formed under heating *via* a single electron transfer (SET) process and the Ag^0 species [Ag(s)] was provided simultaneously. Subsequently, this radical intermediate **C** reacted with alkene **1a** to give the radical intermediate **D**. Intermediate **D** could convert to the cation intermediate **E** in the presence of Ag(I) species. Finally, oxidation and nucleophilic addition occurred to give the desired product **4aaa**. NiI_2 serves as a Lewis acid to coordinate alkene **1a** and stabilizes the radical intermediates, thus enhancing their reactivity.

In summary, we have described a nickel-promoted oxidative 1,2-alkylesterification of styrenes with nitriles and acids, in which a C–C bond and C–O bond were constructed simultaneously. Various acids including α-, β-, and γ-amino acids, aromatic acids and aliphatic acids were transformed to the corresponding acyloxylated nitrile derivatives in moderate to

good yields in the presence of NiI_2/Ag_2CO_3 catalyst systems under heating conditions. This convenient alkylesterification method may find utility in the synthesis of CH_2CN/COO-containing biologically active compounds.

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Conflicts of interest

The authors declare no conflict of interest.

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