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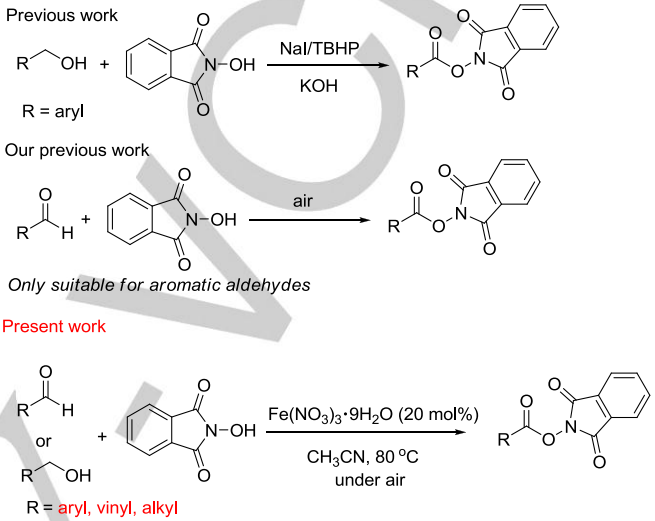
Iron Nitrate-Catalyzed Oxidative Esterification of Aldehydes and Alcohols with NHPI: Efficient Synthesis of *N*-hydroxyimide Esters

Xiaohe Xu,[a] Jian Sun,[a] Yuyan Lin,[a] Jingya Cheng,[a] Pingping Li,[a] Xiaoying Jiang,[a] Renren Bai[a] and Yuanyuan Xie*[a],[b]

Abstract: An $Fe(NO_3)_3{\cdot}9H_2O$-catalyzed cross-dehydrogenative coupling reaction between *N*-hydroxyphthalimide (NHPI) or *N*-hydroxysuccinimide (NHSI) and aldehydes or alcohols in the air has been described. This transformation provided an efficient approach to prepare *N*-hydroxyimide ester derivatives with wide substrate scope in moderate to excellent yields.

Introduction

Ester groups are important functional groups in organic chemistry and they can be found in natural products, medicinally relevant molecules, synthetic materials, and polymers.[1] Therefore, many methods have been developed for their efficient constructions.[2] *N*-Hydroxyimide ester, an important class of esters, is an active intermediate in preparing amides, esters, *O*-substituted hydroxylamine and in the formation of $Csp^2 - Csp^2$ bonds by cross-coupling.[3] Although *N*-hydroxyimide esters are frequently used intermediates, few approaches have been developed for their preparation. Traditionally, *N*-hydroxyamide esters were synthesized via direct coupling of carboxylic acids with *N*-hydroxyimides in the presence of *N*,*N*'-dicyclohexylcarbodiimide,[4] which suffers from several drawbacks such as poor atom efficiency and difficult purification process. The cross-dehydrogenative coupling approach is attractive because it is atom-economic without the requirement of the prefunctionalization of the substrates.[5] Recently, several groups have reported *N*-hydroxyamide ester formation from aldehydes *via* oxidative cross-dehydrogenative coupling either in the presence of a metal catalyst or under metal-free conditions.[6] These reported cross dehydrogenative coupling reactions between *N*-hydroxyphthalimide and aldehydes were normally applicable to aromatic aldehydes. The CDC reaction between NHPI and alkyl aldehydes, enaldehydes or alcohols still remains a major challenge. In 2013, Yu and co-workers developed a NaI catalyzed cross-coupling reaction of alcohols with NHPI in the presence of TBHP and KOH (Scheme 1).[7] Although the above-mentioned elegant methods appear to be general and efficient, they still suffer from some drawbacks, such as the stoichiometric oxidants and heavy-metal catalysts. Hence, milder, more environmentally benign synthetic methods with a wider scope of substrates are still required for the synthesis of *N*-hydroxyamide esters.



Scheme 1. Various approaches for synthesis of *N*-hydroxyamide esters

Iron is the second most abundant metal in the earth.[8] Compared with other metals, iron is relatively non-toxic, inexpensive and environmentally friendly. Thus a variety of organic transformations were carried out under iron-based catalyst systems.[9] Recently, iron (III) nitrate nonahydrate [$Fe(NO_3)_3{\cdot}9H_2O$] has been used as a nitration reagent, as well as a widely used catalyst to catalyze the oxidation of benzyl alcohols and oxidative coupling of NHPI with olefins.[10] Thermal decomposition of $Fe(NO_3)_3{\cdot}9H_2O$ is well-known to generate nitrogen dioxide (NO_2)[11] which is a radical species. In the presence of NO_2, NHPI can be oxidized to PINO, which may initiate radical oxidative reactions.[12] Herein, we disclosed an example of $Fe(NO_3)_3{\cdot}9H_2O$ catalyzed intermolecular C–O cross-coupling reaction of aldehydes or alcohols with NHPI.

Results and Discussion

In our initial study, propionaldehyde **1a** and NHPI **2a** were chosen as the model substrates to investigate the cross-dehydrogenative coupling reaction in CH_3CN at room temperature in the presence of $Fe(NO_3)_3{\cdot}9H_2O$ under the air for 1 h, and the desired *N*-hydroxyamide ester **3aa** was obtained in 13% yield (entry 1, Table 1). Intriguingly, elevated temperature gave the desired product in a much better yield (entry 2). When the temperature was increased up to 80 °C, the starting materials NHPI was totally converted into the desired product (entry 3). Other catalysts such as $Cu(NO_3)_2{\cdot}3H_2O$, $Mg(NO_3)_2{\cdot}6H_2O$ and $AgNO_3$ were also tested, but they were less active and gave **3aa** in 72%, 59% and 50% yields, respectively

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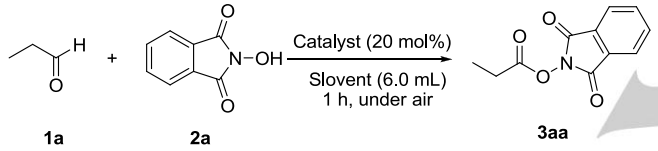
(entries 4-6). The reaction did not afford the coupling product when other iron salts such as $FeCl_3·6H_2O$ and $FeSO_4·7H_2O$ were used (entries 7-8), which indicated the importance of NO_3^- in the reaction. However, the reaction did not occur in the presence of $NaNO_2$ (entry 9). As reported, NO_2 can be generated from $NaNO_2$ under acidic conditions[13]. Considering this transformations, **3aa** was obtained in 55% when 3eq. CH_3COOH was added into the reaction mixture (entry 9). Additionally, the effect of different solvents including EtOAc, acetone, toluene and DMF were also investigated, and CH_3CN was showed to be the optimal solvent (entries 10-13). Decreasing the $Fe(NO_3)_3·9H_2O$ dosage reduced the yield. When the reaction time was prolonged to 4 h, the yield of **3aa** was increased to 76%, however, further prolonged the reaction time and the yield of **3aa** was unchanged (entry 14). Moreover, the reaction did not take place in the absence of $Fe(NO_3)_3·9H_2O$ (entry 15), when the reaction was performed under N_2 atmosphere, the yield of **3aa** was only 23% (entry 16).. The results proved that both catalysts and oxidants (air) are crucial for this transformation. Hence, the optimal conditions determined were **1a** (1.0 mmol) with **2a** (0.5 mmol) in the presence of $Fe(NO_3)_3·9H_2O$ (0.1 mmol) in CH_3CN at 80 °C under air to afford the desired product in 82% yield (entry 3).

Table 1. Optimization of the reaction conditions[a]



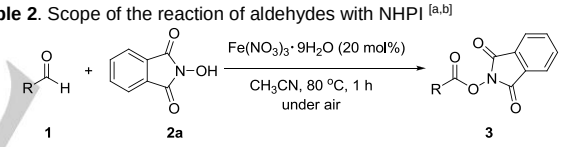
1a **2a** **3aa**

Entry	Catalyst	T. [°C]	Slovent	Yield [%][b]
1	$Fe(NO_3)_3·9H_2O$	r.t.	CH_3CN	13
2	$Fe(NO_3)_3·9H_2O$	60	CH_3CN	68
3	**$Fe(NO_3)_3·9H_2O$**	**80**	**CH_3CN**	**82**
4	$Cu(NO_3)_2·3H_2O$	80	CH_3CN	72
5	$Mg(NO_3)_2·6H_2O$	80	CH_3CN	59
6	$AgNO_3$	80	CH_3CN	50
7	$FeCl_3·6H_2O$	80	CH_3CN	n.d.
8	$FeSO_4·7H_2O$	80	CH_3CN	n.d.
9	$NaNO_2$	80	CH_3CN	trace [55][c]
10	$Fe(NO_3)_3·9H_2O$	77	EtOAc	46
11	$Fe(NO_3)_3·9H_2O$	56	acetone	35
12	$Fe(NO_3)_3·9H_2O$	80	toluene	13
13	$Fe(NO_3)_3·9H_2O$	80	DMF	30
14[d]	$Fe(NO_3)_3·9H_2O$	80	CH_3CN	65 [76][e]
15[f]	--	80	CH_3CN	n.d.
16[g]	$Fe(NO_3)_3·9H_2O$	80	CH_3CN	23

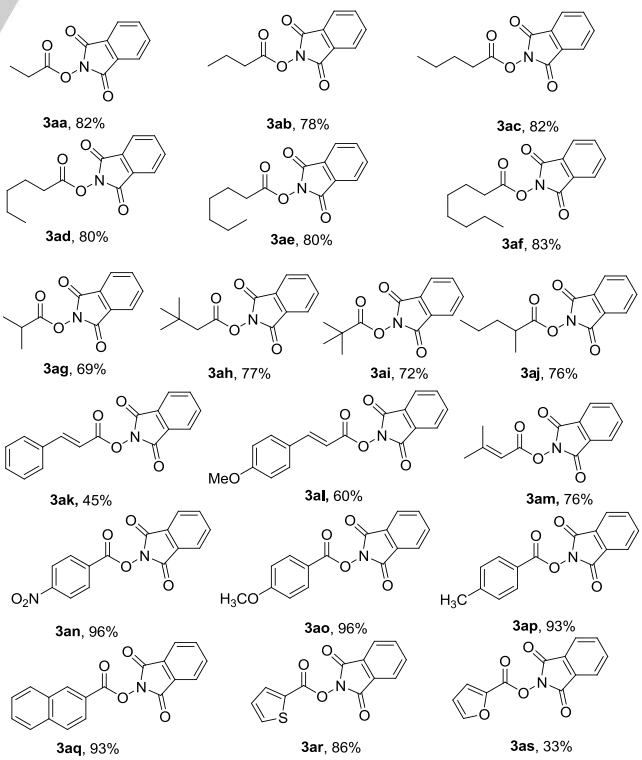
[a] Unless otherwise mentioned, the reactions were performed with **1a** (1.0 mmol), **2a** (0.5 mmol), catalyst (20 mol%.), solvent (6.0 mL) under air for 1 h. [b] Isolated yields based on **2a**. [c] CH_3COOH (3.0 equiv.) was added. [d] 10 mol% $Fe(NO_3)_3·9H_2O$ was used. [e] The reaction time was 4 h. [f] $Fe(NO_3)_3·9H_2O$ was not added. [g] The reaction was performed under N_2 atmosphere. n.d. = Not detected

With the optimized reaction conditions in hand, we then investigated the scope of aldehyde substrates. As shown in Table 2, a series of aldehydes were found to smoothly react with NHPI, giving the corresponding products in good to excellent yields. Aliphatic aldehydes like propionaldehyde, butyraldehyde and pentanaldehyde delivered the related products **3aa**, **3ab** and **3ac** in 82%, 78% and 82% yields, respectively. Increasing in linear chain showed little influence on the reaction efficiency, gaving products **3ad**-**3af** in 80-83%. Due to the steric effect, the reaction efficiency of branched aliphatic aldehydes was decreased. For example, when isobutyraldehyde was also used as the substrate, the cross-dehydrogenative coupling product **3ag** was obtained in only 69% yield (**3ag** *vs* **3aa**, **3ab**). Other branched aliphatic aldehydes such as 3,3-dimethylbutanal, pivalaldehyde and 2-methylpentanal displayed lower yields and products **3ah**, **3ai** and **3aj** were obtained in 77%, 72% and 76%, respectively. The enaldehydes were identified to be suitable substrates, providing esters **3ak**-**3am** in 45-76% yields. This work provides the first example of a direct transformation from an alkyl enaldehydes and NHPI to the corresponding *N*-hydroxyimide esters. The optimized conditions were also compatible with benzyl aldehydes and 2-naphthaldehyde, providing the desired products **3an**-**3aq** in 93-96% yields. Additionally, the heterocyclic aldehydes including thiophene-2-carbaldehyde and furfural were survived. For instance, 1,3-dioxoisoindolin-2-yl thiophene-2-carboxylate **3ar** was produced in 86% yield, and 1,3-dioxoisoindolin-2-yl furan-2-carboxylate **3as** was formed in 33% yield.

Table 2. Scope of the reaction of aldehydes with NHPI [a,b]



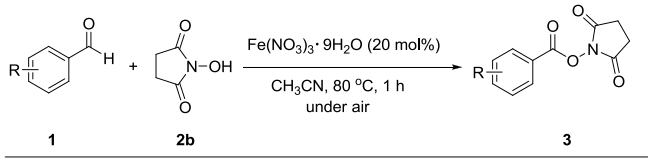
1 **2a** **3**

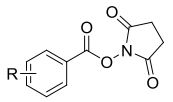


[a] All the reactions were performed with **1** (1 mmol), **2a** (0.5 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (0.1 mmol), CH_3CN (6.0 mL) at 80 °C under air for 1 h. [b] Isolated yields based on **2a**.

We then investigated the reaction of aldehydes with *N*-hydroxysuccinimide (**2b**, NHSI) under optimized reaction conditions. The results were displayed in Table 3. Aldehydes with electron-withdrawing groups performed well, giving the desired products **3ba**-**3bd** in 50-81% yields. Other substituents on the benzene ring of the aldehyde such as methoxyl, methyl, ester group were all well-tolerated and the corresponding products **3be**-**3bi** were obtained in good to excellent isolated yields. However, aliphatic aldehydes were turned out to be poor substrates for this transformation.

Table 3. Scope of the reaction of aldehydes with NHSI [a,b]





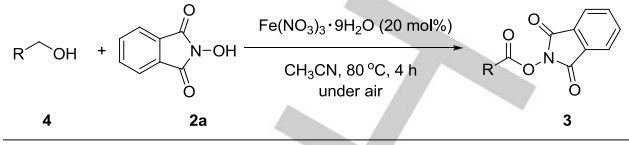
R = *p*-F, **3ba**, 71%; *p*-Cl, **3bb**, 61%; *p*-Br, **3bc**, 81%; *p*-NO_2, **3bd**, 50%; *p*-OCH_3, **3be**, 75%; *m*-OCH_3, **3bf**, 70%; *o*-OCH_3, **3bg**, 80%; *p*-CH_3, **3bh**, 91%; *p*-$COOCH_3$, **3bi**, 91%

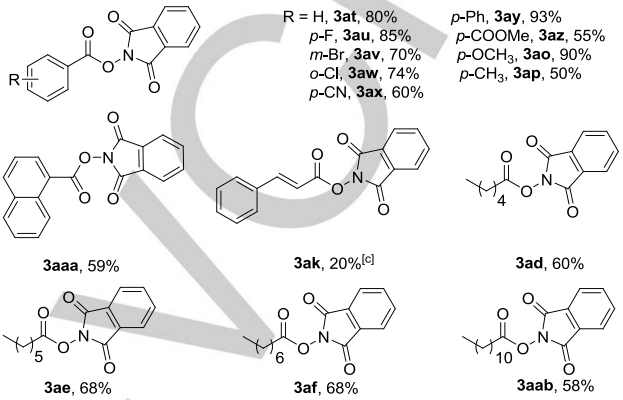
[a] All the reactions were performed with **1** (1 mmol), **2b** (0.5 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (0.1 mmol), CH_3CN (6.0 mL) at 80 °C under air for 1 h. [b] Isolated yields based on **2b**

Encouraged by the above mentioned discoveries, we further investigated the possibility of using alcohols as the coupling partner for NHPI since alcohols can be readily oxidized into aldehydes by NHPI and $Fe(NO_3)_3{\cdot}9H_2O$. As shown in Table 4, benzyl alcohols with various substituents, including electron-withdrawing groups such as F, Cl, Br, CN, Ph and COOMe, electron-donating groups such as OCH_3 and CH_3 could be converted to the desired products in moderate to excellent yields. Naphthalen-1-ylmethanol and 3-phenylprop-2-en-1-ol also furnished the desired products **3aaa** and **3ak** in 59% and 20% yields, respectively. Aliphatic alohols (**4d**-**4f**, **4b**) were investigated under the optimized conditions and resulted in the corresponding products **3ad**−**3af**, **3aab** albeit in moderate yields (58−68%)

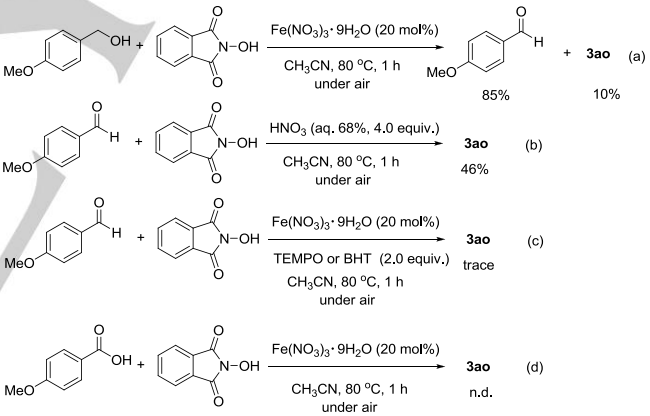
With the methods established, several control experiments were carried out to probe the reaction mechanism (Scheme 2). Under standard conditions, *p*-methoxybenzyl alcohol was oxidized to *p*-methoxybenzaldehyde in 85% yield in 1 h (Scheme 2, a). When the reaction was performed under HNO_3 condition, the desired product **3ao** was obtained in 46% yield (Scheme 2, b).The esterification of 4-methoxybenzaldehyde (**1o**) with NHPI (**2a**) was conducted in the presence of TEMPO under the standard reaction conditions and only trace product **3ao** was obtained (Scheme 2, c). The model reaction was also completely inhibited by another radical scavenger, 2,6-di-*tert*-butyl-4-methylphenol (BHT) (Scheme 2, c). In order to verify whether *p*-methoxybenzoic acid was formed in situ as an active intermediate, *p*-methoxybenzoic acid was applied to the cross-coupling reaction with NHPI (Scheme 2, d), and the *N*-hydroxyimide ester **3ao** was not found under standard conditions.

Table 4. Scope of the reaction of alcohols with NHPI [a,b]



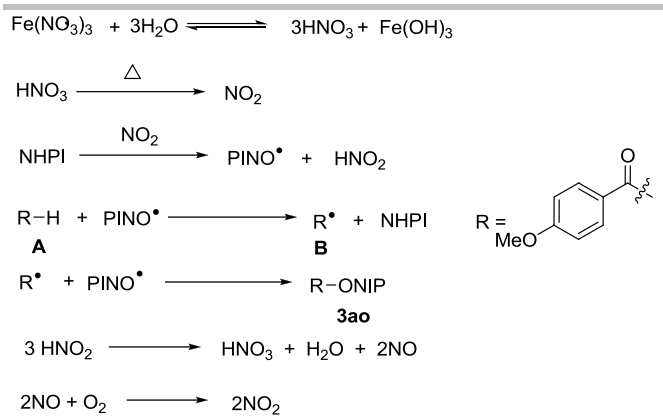


[a] All the reactions were performed with **1** (1 mmol), **2b** (0.5 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (0.1 mmol), CH_3CN (6.0 mL) at 80 °C under air for 4 h unless otherwise mentioned. [b] Isolated yields based on **2a**. [c] The reaction was performed for 24 h.



Scheme 2. Control experiments

Based on the results of above experiments and according to previous reports,[6,10-12] a plausible reaction pathway was illustrated in Scheme 3. Initially, ferric nitrate was hydrolyzed to HNO_3, and then thermal decomposition of HNO_3 generates NO_2. In the gas phase of the reaction mixture, the presence of NO_2 was confirmed by its characteristic reddish-brown color. In the presence of NO_2, NHPI could afford PINO radical, which induces the homolysis of the C–H bond of the aldehyde to afford the acyl radical **B** [14]. Finally, recombination of the acyl radical **B** and PINO radical forms the *N*-hydroxyimide ester **3ao**. On the other hand, HNO_2 decomposes into HNO_3 and NO. In the presence of O_2, NO was oxidized to NO_2 and NO_2 was recycled.



Scheme 3. Plausible mechanism

Conclusions

In conclusion, we have described an efficient method for the synthesis of *N*-hydroxyimide esters. The notable features of this reaction are a broad substrate scope, environmentally benign conditions. Using inexpensive, non-toxic catalyst and green oxidant (air) as the oxidant makes this protocol to be an attractive strategy in synthetic chemistry. This is also the first report that simultaneous employed aldehydes and alcohols (including alkyl aldehydes, enaldehydes and alcohols) as the coupling partners for NHPI to afford the corresponding esters. Further studies on the coupling of other compounds with *N*-hydroxyimides are under investigation.

Experimental Section

General procedure for synthesis of 3aa: Propionaldehyde **1a** (1.0 mmol), NHPI (0.5 mmol, 1.0 equiv.) and $Fe(NO_3)_3·9H_2O$ (0.1 mmol) were dissolved in CH_3CN (6.0 mL) and the mixture was stirred for 1 h at 80 °C under air until NHPI was completely consumption. Then, the solvent were removed under reduced pressure. The residue was washed with saturated $NaHCO_3$ and then extracted with EtOAc (2 × 20 mL). The combined organic layers was dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1) to afford the desired product **3aa** as a white solid; yield: 90 mg (82%); m.p.88–90 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.90-7.88 (m, 2 H), 7.81-7.79 (m, 2 H), 2.46 (q, *J* = 7.8 Hz, 2 H), 1.32 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (125MHz, $CDCl_3$) δ = 170.4, 162.0, 134.8, 128.9, 124.0, 24.5, 8.7 ppm. IR (KBr): *v* 3433, 3097, 2981, 2936, 1755, 1736, 1610, 1185, 1137, 1104, 1059, 1000, 876, 699, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{11}H_9NNaO_4$, m/z 242.0424, found 242.0423.

1,3-dioxoisoindolin-2-yl butyrate (3ab): Oil.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.90-7.87 (m, 2 H), 7.81-7.78 (m, 2 H), 2.66 (t, *J* = 7.2 Hz, 2 H), 1.86-1.80 (m, 2 H), 1.08 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.5, 162.0, 134.7, 128.9, 123.9, 32.8, 18.3, 13.4 ppm. IR (KBr): *v* 3521, 3068, 2969, 2937, 1788, 1743, 1609, 1467, 1367, 1186, 1127, 1062, 969, 877, 787, 696, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_4$, m/z 256.0580, found 254.0591.

1,3-dioxoisoindolin-2-yl pentanoate (3ac): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.87 (m, 2 H), 7.79-7.78 (m, 2 H), 2.67 (t, *J* = 7.8 Hz, 2 H), 1.80-1.75 (m, 2 H), 1.51-1.45 (m, 2 H), 0.97 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.6, 162.0, 134.7, 129.0, 123.9, 30.7, 26.7, 22.0, 13.6 ppm. IR (KBr): *v* 3521, 3045, 2960, 2932, 1788, 1743, 1670, 1467, 1364, 1186, 1128, 1064, 878, 696, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_{13}NNaO_4$, m/z 270.0737, found 270.0734.

1,3-dioxoisoindolin-2-yl hexanoate (3ad): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.91-7.90 (m, 2H), 7.81-7.80 (m, 2H), 2.68 (t, *J* = 7.2 Hz, 2 H), 1.84-1.79 (m, 2 H), 1.48-1.35 (m, 4 H), 0.95 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.7, 162.0, 134.7, 129.0, 123.9, 31.0, 24.4, 22.2, 13.8 ppm. IR (KBr): *v* 3059, 3031, 2957, 2931, 1789, 1744, 1467, 1363, 1186, 1311, 1066, 966, 878, 786, 697, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{14}H_{15}NNaO_4$, m/z 284.0893, found 284.0888.

1,3-dioxoisoindolin-2-yl heptanoate (3ae): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.87 (m, 2 H), 7.80-7.78 (m, 2 H), 2.67 (t, *J* =7.2 Hz, 2 H), 1.81-1.76 (m, 2 H), 1.47-1.43 (m, 2 H), 1.35-1.33 (m, 4 H), 0.91 (t, *J* =7.2 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.7, 162.0, 134.7, 128.9, 123.9, 31.3, 31.0, 28.5, 24.6, 22.4, 14.0 ppm. IR (KBr): *v* 3448, 3043, 3025, 2954, 2869, 1788, 1740, 1638, 1377, 1139, 1081, 877, 699, 521 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_{17}NNaO_4$, m/z 298.1050, found 298.1041.

1,3-dioxoisoindolin-2-yl octanoate (3af): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.90-7.86 (m, 2 H), 7.80-7.77 (m, 2 H), 2.67-2.65 m, 2 H), 1.801-1.76 (m, 2 H), 1.43 (s, 2 H), 1.33-1.30 (m, 6 H), 0.90 (t, *J* =3.6 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.7, 162.0, 134.7, 129.0, 123.9, 31.6, 31.0, 28.8, 24.7, 22.6, 14.1 ppm. IR (KBr): *v* 3065, 2958, 2928, 1789, 1745, 1467, 1364, 1185, 1072, 969, 878, 697, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{16}H_{19}NNaO_4$, m/z 312.1206, found 312.1202.

1,3-dioxoisoindolin-2-yl isobutyrate (3ag): white solid, m.p. 60-61 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.88 (m, 2 H), 7.79-7.78 (m, 2 H), 2.99-2.94 (m, 1 H),1.38 (d, *J* =7.2 Hz, 6 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 173.1, 162.0, 134.7, 129.0, 123.9, 31.8, 18.8 ppm. IR (KBr): *v* 3447, 3084, 2985, 2925, 1782, 1740, 1466, 1187, 1053, 967, 876, 694, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_4$, m/z 256.0580, found 256.0575.

1,3-dioxoisoindolin-2-yl 3,3-dimethylbutanoate (3ah): white solid, m.p. 92-93 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.87 (m, 2 H), 7.79-7.78 (m, 2 H), 2.53 (s, 2 H), 1.17 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 167.9, 162.1, 134.7, 129.0, 123.9, 44.6, 31.3, 29.5 ppm. IR (KBr): *v* 3428, 3072, 2963, 2926, 1782, 1736, 1468, 1370, 1286, 1182, 1090, 970, 878, 702, 519 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{14}H_{15}NNaO_4$, m/z 284.0893, found 284.0892.

1,3-dioxoisoindolin-2-yl pivalate (3ai): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.87 (m, 2 H), 7.79-7.77 (m, 2 H), 1.44 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 174.4, 162.1, 134.7, 129.1, 123.9, 38.4, 27.0 ppm. IR (KBr): *v* 3521, 3066, 3031, 2977, 2936, 1758, 1744, 1481, 1370, 1186, 1062, 1023, 965, 878, 696, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_{13}NNaO_4$, m/z 270.0737, found 270.0731.

1,3-dioxoisoindolin-2-yl 2-methylpentanoate (3aj): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.87 (m, 2 H), 7.79-7.78 (m, 2 H), 2.88-2.82 (m, 1 H), 1.86-1.80 (m, 1 H), 1.61-1.57 (m, 1 H), 1.53-1.48 (m, 2 H), 1.35 (d, *J* = 6.6 Hz, 3 H), 0.98 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 172.8, 162.1, 134.7, 129.0, 123.9, 36.9, 35.8, 20.1, 16.9, 13.9 ppm. IR (KBr): *v* 3431, 3097, 2958, 1768, 1743, 1635, 1377, 1141, 1102, 1045, 978, 877, 699, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{14}H_{15}NNaO_4$, m/z 284.0893, found 284.0885.

1,3-dioxoisoindolin-2-yl cinnamate (3ak): White solid, m.p. 143-145 °C. [6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.99 (d, *J* = 16.2 Hz, 1 H), 7.95-7.93 (m, 2 H), 7.84-7.82 (m, 2 H), 7.62 (d, *J* = 6.6 Hz, 2 H), 7.51-7.45 (m, 3 H), 6.69 (d, *J* = 16.2, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 162.1, 150.0, 134.8, 133.6, 131.6, 129.1, 129.0, 128.7, 124.0, 111.7 ppm. IR (KBr): *v* 3438, 3036, 2926, 1744, 1630, 1384, 1139, 980, 927, 876, 620,

532 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{17}H_{11}NNaO_4$, m/z 316.0580, found 316.0582.

1,3-dioxoisoindolin-2-yl-3-(4-methoxyphenyl)acrylate (3al): Brown solid, m.p. 135-137 °C. ^1H NMR (600 MHz, $CDCl_3$) δ = 7.95-7.93 (m, 3 H), 7.83-7.81 (m, 2 H), 7.57 (d, J = 9.0 Hz, 2 H), 6.97 (d, J = 9.0 Hz, 2 H), 6.54 (d, J = 16.2 Hz, 1 H), 3.89 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 163.3, 162.5, 162.2, 149.7, 134.7, 130.6, 129.1, 126.4, 124.0, 114.6, 108.8, 55.5 ppm. IR (KBr): v 3448, 3046, 2929, 1778, 1744, 1635, 1363, 1145, 987, 947, 878, 626, 528 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{18}H_{13}NNaO_5$, m/z 346.0690, found 346.0686.

1,3-dioxoisoindolin-2-yl 3-methylbut-2-enoate (3am): White solid, 118-119 °C. ^1H NMR (600 MHz, $CDCl_3$) δ = 7.90-7.88 (m, 2 H), 7.79-7.78 (m, 2 H), 6.00 (t, J = 1.2 Hz, 1 H), 2.23 (d, J = 1.2 Hz, 3 H), 2.05 (d, J = 0.6 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 165.5, 162.4, 161.8, 134.7, 129.1, 123.9, 110.0, 27.9, 21.1 ppm. IR (KBr): v 3521, 3034, 2960, 1786, 1743, 1614, 1467, 1367, 1186, 1135, 1065, 966, 878, 696, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_{11}NNaO_4$, m/z 268.0580, found 268.0586.

1,3-dioxoisoindolin-2-yl 4-nitrobenzoate (3an): White solid, m.p. 198-200 °C.[6b] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.39 (s, 4 H), 7.97-7.94 (m, 2 H), 7.87-7.84 (m, 2 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 161.7, 161.3, 151.6, 135.1, 131.9, 130.8, 128.8, 124.2, 124.0 ppm. IR (KBr): v 3434, 3050, 2926, 1769, 1741, 1608, 1239, 1136, 1053, 1010, 875, 710, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_8N_2NaO_4$, m/z 335.0275, found 335.0272.

1,3-dioxoisoindolin-2-yl 4-methoxybenzoate (3ao): White solid, m.p. 160-162 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.17 (d, J = 8.4 Hz, 2 H), 7.94-7.93 (m, 2 H), 7.83-7.82 (m, 2 H), 7.02 (d, J = 8.4 Hz, 2 H), 3.92 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 164.9, 162.4, 162.2, 134.7, 132.9, 129.0, 124.0, 117.3, 114.2, 55.6 ppm. IR (KBr): v 3441, 3024, 2986, 1738, 1605, 1248, 1117, 922, 875, 710, 521 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{16}H_{11}NNaO_5$, m/z 320.0529, found 320.0529.

1,3-dioxoisoindolin-2-yl 4-methylbenzoate (3ap): White solid, m.p. 164-165 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.10 (d, J = 7.8 Hz, 2 H), 7.95-7.93 (m, 2 H), 7.84-7.82 (m, 2 H), 7.35 (d, J = 8.4 Hz, 2 H), 2.48 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 162.8, 162.2, 146.1, 134.8, 130.7, 129.6, 129.1, 124.0, 122.4, 21.9. IR (KBr): v 3438, 3037, 2932, 1758, 1740, 1607, 1251, 1180, 1119, 1000, 875, 740, 701, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{16}H_{11}NNaO_4$, m/z 304.0580, found 304.0585.

1,3-dioxoisoindolin-2-yl 2-naphthoate (3aq): Yellow solid, m.p. 195-197 °C. ^1H NMR (600 MHz, $CDCl_3$) δ = 8.84 (s, 1 H), 8.16 (dd, J = 7.2, 1.2 Hz, 1 H), 8.02 (d, J = 8.4 Hz, 1 H), 7.99-7.94 (m, 4 H), 7.85-7.84 (m, 2H), 7.71-7.68 (m, 1 H), 7.64-7.61 (m, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 163.0, 162.2, 136.3, 134.8, 133.0, 132.3, 129.7, 129.4, 129.1, 128.8, 128.0, 127.2, 125.2, 124.1, 122.4 ppm. IR (KBr): v 3445, 3046, 2923, 1770, 1735, 1628, 1177, 1126, 1044, 875, 757, 697, 519 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{19}H_{11}NNaO_4$, m/z 320.0566, found 320.0580.

1,3-dioxoisoindolin-2-yl thiophene-2-carboxylate (3ar): White solid, m.p. 145-147 °C.[6b] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.09 (d, J = 4.2, 1 H), 7.94-7.92 (m, 2 H), 7.83-7.81 (m, 2H), 8.00 (d, J = 4.8 Hz, 1 H), 7.23 (t, J = 4.2 Hz, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 162.0, 158.3, 136.7, 135.7, 134.9, 128.9, 128.4, 127.0, 124.1 ppm. IR (KBr): v 3442, 3043, 3023, 2926, 1777, 1740, 1639, 1410, 1247, 1187, 1134, 992, 724, 695, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_7NNaO_4S$, m/z 295.9988, found 295.9995.

1,3-dioxoisoindolin-2-yl furan-2-carboxylate (3as): White solid, m.p. 148-150 °C.[6b] ^1H NMR (600 MHz, $CDCl_3$) δ = 7.95-7.93 (m, 2 H), 7.85-7.82 (m, 2 H), 7.78-7.77 (s, 1 H), 7.56 (d, J = 3.6 Hz, 1 H), 6.67-6.66 (m, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 161.8, 154.5, 148.8, 139.9, 134.9, 128.9, 124.1, 122.2, 112.7 ppm. IR (KBr): v 3444, 3038, 2955, 2924, 1742, 1646, 1466, 1286, 1155, 1081, 1038, 875, 766, 698, 532, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_7NNaO_5$, m/z 280.0216, found 280.0230.

1,3-dioxoisoindolin-2-yl benzoate (3at): White solid, m.p. 168-170 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.22 (d, J = 7.8 Hz, 2 H), 7.96-7.93 (m, 2 H), 7.85-7.83 (m, 2 H), 7.72 (t, J = 7.8 Hz, 1 H), 7.56 (t, J = 7.8 Hz, 2 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 162.8, 162.1, 134.9, 134.8, 130.7, 129.0, 128.9, 125.3, 124.0 ppm. IR (KBr): v 3438, 3040, 3018, 2917, 1773, 1734, 1640, 1237, 1140, 1021, 1007, 875, 697, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_9NNaO_4$, m/z 290.0424, found 290.0424.

1,3-dioxoisoindolin-2-yl 4-fluorobenzoate (3au): White solid, m.p. 189-192 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.25-8.22 (m, 2 H), 7.94-7.93 (m, 2 H), 7.83-7.82 (m, 2 H), 7.24-7.21 (m, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 166.9 ($^1J_{C\text{-}F}$ = 213.4 Hz), 162.0, 161.9, 134.9, 133.5 ($^3J_{C\text{-}F}$ = 8.1Hz), 129.0, 124.1, 121.6, ($^4J_{C\text{-}F}$ = 2.5 Hz), 116.3 ($^2J_{C\text{-}F}$ = 18.5 Hz) ppm. IR (KBr): v 3436, 3053, 3024, 2920, 1773, 1736, 1602, 1239, 1189, 1033, 1009, 875, 700, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_8FNNaO_4$, m/z 308.0330, found 308.0316.

1,3-dioxoisoindolin-2-yl-3-bromobenzoate (3av): White solid, m.p. 169-171 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.33 (t, J = 1.8 Hz, 1 H), 8.14-8.12 (m, 1 H), 7.95-7.92 (m, 2 H), 7.84-7.82 (m, 3 H), 7.43 (t, J = 8.4 Hz, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 161.8, 161.7, 137.9, 134.9, 133.4, 130.5, 129.2, 128.9, 127.2, 124.1, 122.9 ppm. IR (KBr): v 3448, 3094, 3037, 2925, 1774, 1736, 1644, 1185, 1136, 1024, 874, 851, 728, 517 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_8BrNNaO_4$, m/z 367.9529, found 367.9508.

1,3-dioxoisoindolin-2-yl-2-chlorobenzoate (3aw): White solid, m.p. 165-167 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.22 (dd, J = 6.6, 1.2 Hz, 1 H), 7.96-7.94 (m, 2 H), 7.85-7.83 (m, 2 H), 7.61-7.57 (m, 2 H), 7.46-7.44 (m, 1 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 161.9, 161.2, 134.9, 134.6, 132.5, 131.7, 129.0, 126.9, 124.8, 124.1, 123.6 ppm. IR (KBr): v 3419, 3068, 2958, 1777, 1751, 1638, 1588, 1229, 1185, 1137, 1075, 996, 875, 694, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{15}H_8ClNNaO_4$, m/z 324.0034, found 324.0050.

1,3-dioxoisoindolin-2-yl-4-cyanobenzoate (3ax): White solid, m.p. 218-220 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.33 (d, J = 8.4 Hz, 2 H), 7.98-7.96 (m, 2 H), 7.89-7.86 (m, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 161.7, 161.5, 135.0, 132.6, 131.1, 129.2, 128.8, 124.2, 118.3, 117.4 ppm. IR (KBr): v 3438, 3043, 3020, 2928, 2235, 1776, 1744, 1602, 1229, 1169, 1053, 1000, 876, 696, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{16}H_8N_2NaO_4$, m/z 315.0381, found 315.0388.

1,3-dioxoisoindolin-2-yl biphenyl-4-carboxylate (3ay): White solid, m.p. 208-210 °C. ^1H NMR (600 MHz, $CDCl_3$) δ = 8.29 (d, J = 8.4 Hz, 2 H), 7.98-7.95 (m, 2 H), 7.85-7.84 (m, 2 H), 7.79 (d, J = 8.4 Hz, 2 H), 7.69-7.68 (m, 2 H), 7.53 (t, J = 7.2 Hz, 2 H), 7.47-7.45 (m, 2 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 162.8, 162.1, 147.7, 139.5, 134.8, 131.2, 129.1, 128.7, 127.5, 127.4, 124.1, 123.9 ppm. IR (KBr): v 3418, 3069, 3021, 2923, 1775, 1744, 1607, 1376, 1254, 1187, 1141, 1035, 1001, 876, 740, 694, 518 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{21}H_{13}NNaO_4$, m/z 366.0737, found 366.0734.

1,3-dioxoisoindolin-2-yl methyl terephthalate (3az): White solid, m.p. 180-182 °C.[6c] ^1H NMR (600 MHz, $CDCl_3$) δ = 8.29 (d, J = 8.4 Hz, 2 H), 8.21 (d, J = 8.4 Hz, 2 H), 7.97-7.94 (m, 2 H), 7.86-7.84 (m, 2 H), 4.00 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 165.8, 162.2, 161.9, 135.6, 134.9, 130.6, 129.9, 129.0, 128.9, 124.1, 52.7 ppm. IR (KBr): v 3422, 3094, 2961, 1772, 1739, 1637, 1280, 1237, 1138, 1103, 1043, 1010, 875, 696, 519 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{17}H_{11}NNaO_6$, m/z 348.0468, found 348.0479.

1,3-dioxoisoindolin-2-yl-1-naphthoate (3aaa): White solid, m.p. 181-183 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.87 (d, J = 8.4 Hz, 1 H), 8.57 (dd, J = 6.6, 0.6 Hz, 1H), 8.19 (d, J = 7.8 Hz, 1H), 7.99-7.95 (m, 3 H), 7.85-7.84 (m, 2 H), 7.70-7.68 (m, 1 H), 7.63-7.60 (m, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 163.1, 162.3, 135.6, 134.8, 133.8, 131.9, 131.5, 129.1, 128.8, 128.7, 126.8, 125.4, 124.5, 124.1, 121.7 ppm. IR (KBr): v 3434, 3046, 2917, 1772, 1743, 1365, 1136, 1096, 978, 773, 695, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{20}H_{27}NNaO_4$, m/z 340.0580, found 340.0569.

1,3-dioxoisoindolin-2-yl dodecanoate (3aab): Oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.89-7.88 (m, 2 H), 7.80-7.78 (m, 2 H), 2.66 (t, J =7.2 Hz, 2 H), 1.81-1.76 (m, 2 H), 1.47-1.42 (m, 2 H), 1.34-1.27 (m, 14 H), 0.88 (t, J =7.2 Hz, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.7, 162.0, 134.7, 129.0, 123.9, 31.9, 31.0, 29.58, 29.57, 29.4, 29.3, 29.1, 28.8, 24.7, 22.7, 14.1 ppm. IR (KBr): v 3427, 3091, 3062, 2954, 2920, 1789, 1744, 1375, 1185, 1143, 962, 880, 696, 521 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{20}H_{27}NNaO_4$, m/z 368.1832, found 368.1815.

2,5-dioxopyrrolidin-1-yl 4-fluorobenzoate (3ba): White solid, m.p. 106-108 °C.[6b] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.20-8.17 (m, 2 H), 7.21 (t, J = 8.4 Hz, 2 H), 2.93 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.3, 166.9 ($^1J_{C\text{-}F}$ = 213.5 Hz), 161.0, 133.4 ($^3J_{C\text{-}F}$ = 8.1 Hz), 121.4 ($^4J_{C\text{-}F}$ = 2.5 Hz), 116.3 ($^2J_{C\text{-}F}$ = 18.5 Hz), 25.7 ppm. v 3430, 3074, 3023, 2964, 1778, 1730, 1598, 1400, 1085, 1056, 990, 846, 747, 530 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{11}H_8FNNaO_4$, m/z 260.0330, found 260.0320.

2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate (3bb): White solid, m.p. 205-207 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.09 (d, J = 8.4 Hz, 2 H), 7.56 (d, J = 8.4 Hz, 2 H), 2.93 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.2, 161.2, 141.7, 131.9, 129.4, 123.6, 25.7 ppm. IR (KBr): v 3442, 3080, 3070, 2967, 1770, 1744, 1567, 1211, 1070, 1060, 991, 848, 730, 520 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{11}H_8ClNNaO_4$, m/z 276.0034, found 276.0018.

2,5-dioxopyrrolidin-1-yl 4-bromobenzoate (3bc): White solid, m.p. 206-208 °C.[6f] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.01 (d, J = 8.4 Hz, 2 H), 7.69 (d, J = 8.4 Hz, 2 H), 2.93 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.1, 161.3, 132.4, 131.9, 130.5, 124.0, 25.7. IR (KBr): v 3432, 3084, 3069, 2974, 1773, 1727, 1588, 1406, 1211, 1080, 1066, 991, 848, 740, 531 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{11}H_8BrNNaO_4$, m/z 319.9529, found 319.9515.

2,5-dioxopyrrolidin-1-yl 4-nitrobenzoate (3bd): White solid, m.p. 200-203 °C.[6b] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.40 (d, J = 9.0 Hz, 2 H), 8.35 (d, J = 9.0 Hz, 2 H), 2.97 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 168.8, 160.3, 151.5, 131.8, 130.6, 124.0, 25.7 ppm. IR (KBr): v 3445, 3094, 3062, 2983, 1771, 1726, 1588, 1210, 1080, 1066, 991, 847, 740, 530 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{11}H_8NNaO_6$, m/z 287.0380, found 287.0392.

2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate (3be): White solid, m.p. 149-151 °C.[6b] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.11 (d, J = 9.0 Hz, 2 H), 7.00 (d, J = 9.0 Hz, 2 H), 3.91 (s, 3 H), 2.91 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.6, 164.9, 161.5, 132.9, 117.1, 114.2, 55.6, 25.7 ppm. IR (KBr): v 3445, 3081, 2974, 2932, 1766, 1736, 1602, 1514, 1428, 1217, 1183, 1074, 984, 848, 757, 595, 517 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_5$, m/z 272.0529, found 272.0532.

2,5-dioxopyrrolidin-1-yl 3-methoxybenzoate (3bf): White solid, m.p. 102-104 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.76 (dd, J = 7.2, 0.6 Hz, 1 H), 7.62 (t, J = 1.2 Hz, 1 H), 7.43 (t, J = 8.4 Hz, 1 H), 7.24-7.22 (m, 1 H), 3.87 (s, 3 H), 2.93 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.4, 161.8, 159.7, 130.0, 126.2, 123.0, 121.7, 114.6, 55.6, 25.7 ppm. IR (KBr): v 3445, 3094, 3049, 2963, 1770, 1744, 1570, 1426, 1201, 1089, 1076, 991, 858, 740, 696, 529 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_5$, m/z 272.0529, found 272.0516.

2,5-dioxopyrrolidin-1-yl-2-methoxybenzoate (3bg): White solid, m.p. 174-176 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.08 (dd, J = 6.0, 1.8 Hz, 1 H), 7.64-7.61 (m, 1 H), 7.06 (t, J = 7.8 Hz, 2 H), 3.95 (s, 3 H), 2.92 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.5, 160.7, 160.3, 136.0, 132.7, 120.3, 114.0, 112.2, 56.1, 25.7 ppm. IR (KBr): v 3434, 3074, 3029, 2983, 1768, 1740, 1470, 1231, 1100, 1076, 997, 898, 747, 700, 530 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_5$, m/z 272.0529, found 272.0539.

2,5-dioxopyrrolidin-1-yl 4-methylbenzoate (3bh): White solid, m.p. 173-175 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.04 (d, J = 8.4 Hz, 2 H), 7.33 (d, J = 8.4 Hz, 2 H), 2.91 (s, 4 H), 2.46 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ = 169.4, 161.9, 146.1, 130.6, 129.6, 122.3, 25.7, 21.9 ppm. IR (KBr): v 3444, 3094, 3039, 2983, 1771, 1743, 1577, 1436, 1200, 1089, 1046, 1000, 878, 749, 696, 529 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{12}H_{11}NNaO_4$, m/z 256.0580, found 250.0590.

2,5-dioxopyrrolidin-1-yl methyl terephthalate (3bi): White solid, m.p. 173-174 °C.[6c] ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.22 (d, J = 7.2 Hz, 2 H), 8.19 (d, J = 7.2 Hz, 2 H), 3.99 (s, 3 H), 2.94 (s, 4 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.1, 165.8, 161.2, 135.6, 130.6, 129.9, 128.9, 52.7, 25.7 ppm. IR (KBr): v 3435, 3008, 2951, 1732, 1710, 1288, 1235, 1207, 1110, 1067, 1001, 845, 720, 529 cm^{-1}. HRMS (ESI) calcd for $[M+Na]^+$ $C_{13}H_{11}NNaO_6$, m/z 300.0479, found 300.0490.

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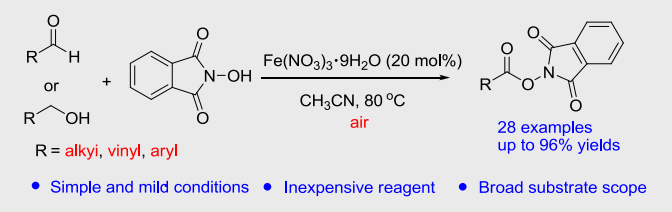

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Iron Nitrate-Catalyzed Oxidative Esterification of Aldehydes and Alcohols with NHPI: Efficient Synthesis of *N*-hydroxyimide Esters

An $Fe(NO_3)_3·9H_2O$-catalyzed cross-dehydrogenative coupling reaction between *N*-hydroxyphthalimide (NHPI) or *N*-hydroxysuccinimide (NHSI) and aldehydes or alcohols in air has been described.